TRITA-JOB PHD 1007

ISSN 1650-9501

PENETRABILITY DUE TO FILTRATION TENDENCY OF CEMENT BASED GROUTS

Daniel Eklund

Doctoral Thesis

Division of Soil and Rock Mechanics

Royal Institute of Technology

Stockholm, Sweden 2005

Summary

Grouting as a method of strengthening and sealing rock, soil and concrete is widely used. The possibilities of sealing structures are of great importance from both an economical and environmental point of view. The cost of grouting has in certain projects been as high as the cost for the blasting and excavation of the tunnel. To improve the technique of grouting with cement based material, it is necessary to focus on the properties of the used grout mixture. The ability of a grout to penetrate cavities, channels and porous material, the penetrability, depends on two things, the theology and the filtration tendency. Extensive laboratory tests on stable, low w/c-ratio, injection grouts show that the most significant limitation to their penetrability is the tendency of cement grains to agglomerate into an impermeable filter cake. The properties of a grout that may prevent passing obstructions in the flow path without the cement grains clogging and preventing further penetration is in this work called filtration tendency. An inert material mixture and a cement-based mixture are used for the investigations in this work. The inert material, which is crushed dolomite stone, does not react with the added water in the mixture. The used cement grouts are based upon three types of commercial available Portland cements and four Portland cements with modified grain size distribution curves.

Performed tests show that the grain size and grain size distribution is of great importance for the filtration tendency. According to performed experiments with inert and cement material, it seems to be advantageous for the penetrability to have a grain size distribution that contains neither too many fine or coarse grains. It is reasonable to believe that the grain size distribution should be relatively steep (narrow grain size range) between minimum and maximum grain size. The maximum grain size is of importance in terms of for example d_{95}. Too large maximum grain size will prevent penetration of the mixture through obstructions in the flow path. According to performed tests, the value of d_{95}, should be between 4-10 times smaller than the aperture to be penetrated by the cement based mixture. The small grain sizes are also of importance in order to achieve a low filtration tendency of the grout. This is because of the increased tendency for the small grains to flocculation into larger agglomerates, compared to larger grain sizes.

The filtration experiments with cement based grouts show that influences of parameters like surface chemistry (use of superplastisisers) and cement chemistry (hydration of cement grains) will strongly affect the filtration tendency of the mixture.

To visualize the phenomenon of filtration tendency it can be investigated on a larger scale than usually takes place. Filtration experiments in the scale of approximately 100:1 have been performed in order to see influences of grain concentration, grain shape and the penetrated slot aperture. It can be seen that used grain sizes (monodisperse and inert mixture) should be approximately at least 2-3 times smaller than the aperture to be penetrated by the mixture. Numerical experiments of filtration tendency have also been performed to investigate the possibilities to numerically simulate the influence of grain concentration and slot aperture. The numerical experiments are based on Eulerian flow modelling.

Preface

This doctoral thesis is a result of a project that started in the beginning of the year 2001. The thesis has been carried out in co-operation between the Division of Soil and Rock Mechanics at the Royal Institute of Technology (KTH) and Vattenfall Utveckling AB. The work has been generously financed by SveBeFo, Elforsk AB, SKB and Cementa AB.

The research presented in this work was initiated by Professor Jan Alemo, Vattenfall Utveckling. The supervisors for this work have been Professor Håkan Stille, KTH and Jan Alemo, to whom I want to express my appreciation and gratitude for their encouragement, advice and belief in me.

The reference group for the work consists of Lars Hammar, Elforsk, Tomas Franzén, SveBeFo, Magnus Eriksson, KTH, Gunnar Gustavsson, CTH, Ann Emmelin, SKB, Sten-Åke Pettersson, Atlas Copco AB, Staffan Hjertström, Cementa, Tommy Ellison, BESAB. Their participation is gratefully acknowledged.

Many people have been connected to this work during the past four years. Special thanks to:

- Professor Håkan Stille for all the help and support during these years.
- Professor Jan Alemo for all the help and support during these years.
- MSc Pär Hansson, Ericsson AB. Pär Hansson started this research work of filtration tendency of cementitious injection grouts, in the early 1990. He finished his employment at Vattenfall in the year of 2000 and handed over an excellent platform for further research within this field.
- The laboratory staff Peter Skärberg, Sven Isaksson and Kjell Fransson at Vattenfall Utveckling for the performance of laboratory testing.
- Urban Andersson at Vattenfall Utveckling for fruitful discussions regarding evaluation and design of experiments.
- Samir Saaidi at Vattenfall Utveckling for support with multiphase flow modelling.
- Anders Birgersson at Scancem Research AB in Slite for sieving dolomite and cement powder.
- Erik Viggh at Cementa for his support in the field of cement chemistry.
- The colleagues at Vattenfall Utveckling for a pleasant work environment.

Finally, I would like to thank my family for great support during this work.

TABLE OF CONTENTS

1 INTRODUCTION 1

1.1 Background 1
1.2 Objective 5
1.3 Hypothesis 6
1.4 Limitations 8
1.5 Disposition of the thesis 8

2 CHARACTERISTICS OF THE GROUTED STRUCTURE 11

2.1 General 11
2.2 Rock structure 11
2.3 Concrete structure 14
2.4 Conclusion and discussion 17

3 PENETRABILITY 19

3.1 General 19
3.2 Multiphase flow 20
3.2.1 General 20
3.2.2 Two-phase flow models 22
3.2.3 Basic governing equations 23
3.2.4 Closure laws 28
3.2.5 Rheology 30
3.3 Filtration tendency 31
3.3.1 Introduction 32
3.3.2 Theory of plug formation 34
3.3.3 Concentration and packing of grains 37
3.3.4 Crack aperture dependent rheology 41
3.3.5 Experiences of measuring with different devices for filtration tendency 45
3.3.6 Experiences of penetrability experiments 49
3.4 Tightness and durability 51
3.5 Conclusion and discussion 54

4 CEMENT BASED GROUT 57

4.1 General 57
4.2 Cement chemistry and early cement reactions 59
4.2.1 Hydration of cement 60
4.2.2 Development of structural strength and filtration tendency 62
4.3 Pore water 65
4.4 Additives 65
4.4.1 Superplastisisers 66
4.4.2 DLVO- theory 68
4.4.3 Accelerators 71
4.4.4 Retarders 71
4.4.5 Swelling agents 72
4.4.6 Stabilisation of grouts 72

4.5 Additive material 72
4.5.1 Silica 72
4.5.2 Slag 73
4.5.3 Fly ash 73
4.6 Conclusions 73

5 PERFORMED FILTRATION EXPERIMENTS 75

5.1 General 75
5.1.1 Characterisation of grain curves 75
5.1.2 Scanning electron microscopy (SEM) 77
5.1.3 Measuring device for filtration tendency 78
5.1.4 Rheological measurement system 79
5.1.5 Bleeding measurement 80
5.1.6 Evaluation of measuring results 80
5.1.7 Air sieving 84
5.2 Inert material 85
5.2.1 Physical experiments 85
5.2.2 Physical experiments with scaled-up plastic grains 89
5.2.3 Numerical experiments 94
5.3 Cement based material 96
5.3.1 Physical experiments 96
5.3.2 Evaluation of the test result 101

6 RESULTS OF EXPERIMENTS 103

6.1 General 103
6.2 Inert material 104
6.2.1 Influence of grain size and grain size distribution 106
6.2.2 Influence of grain concentration 111
6.2.3 Influence of mesh or slot geometry 115
6.2.4 Evaluation of measured values 122
6.2.5 Physical model 126
6.2.6 Numerical model 128
6.3 Comprehensive conclusions inert material 132
6.3.1 Influence of grain size and grain size distribution 132
6.3.2 Influence of grain concentration 133
6.3.3 Influence of mesh or slot geometry 134
6.3.4 Miscellaneous 134
6.4 Cement based material 135
6.4.1 UF 12, Influence of grain size, grain size distribution, grain concentration and slot aperture 138
6.4.2 UF 16, Influence of grain size, grain size distribution, grain concentration and slot aperture 141
6.4.3 IC 30, Influence of grain size, grain size distribution, grain concentration and slot aperture 144
6.4.4 Cem 2 and Cem 4, Influence of grain size, grain size distribution, grain concentration and slot or mesh aperture 146

6.4.5 UF 12 fine and UF 12 coarse, Influence of grain size, grain size distribution, grain concentration and slot aperture 147
6.4.6 Influence of superplastisisers 150
6.4.7 Evaluation of measured values. 153
6.5 Comprehensive conclusions cement based material 160
6.5.1 Influence of grain size, grain size distribution and slot aperture 160
6.5.2 Influence of grain concentration 162
6.5.3 Superplastisisers 163

7 ANALYSIS OF RESULTS 164

7.1 General 164
7.2 Requirements of filtration tendency 165
7.3 Main conclusions 166
7.4 Detailed conclusions 169
7.4.1 Grain size and grain size distribution in relation to the aperture 170
7.4.2 Grain concentration and bleeding 172
7.4.3 Grain concentration and numerical simulations 173
7.4.4 Rheology and superplastisisers 174
7.4.5 Cement chemistry 175

8 PROPOSAL TO CONTINUED RESEARCH 176

8.1 General 176
8.2 Development of test equipment for laboratory and field use 176
8.3 Filtration experiments 177
8.4 The physical model 177
8.5 The numerical model 178
8.6 Mixer design 179
8.7 Field tests 179

9 REFERENCES 180

APPENDIX

1 NUMERICAL MODEL PLOTS 1

2 FILTRATION EXPERIMENTS 6

3 CHARACTERISATION OF GRAIN CURVES (RRSB) 14

4 EVALUATION OF FLOWREGIME IN THE SLOT 15

5 AIR SIEVING OF INERT MATERIAL 17

6 SEDIMENTATION VELOCITY AND OIL VISCOSITY 19

7 DENSITY OF CEMENT AND INERT BASED MIXTURES 20

Nomenclature

Roman

a	grain surface distance	[µm]
a	normalised coefficient	[-]
Ar	*a*rchimedes number	[-]
b	slot aperture	[µm]
c	volumetric concentration	[%]
d	grain diameter	[µm]
$\hat{e}$	unit vector	[-]
D	strain rate tensor	[s^{-1}]
d′	parameter in the RRSB- distribution	[µm]
F	force between grains	[N]
g	gravitational constant	[ms^{-2}]
h	measuring gap	[µm]
I	penetration length	[m]
k	proportionality constant (Power-law model)	[-]
$k_{d\mu}$	factor for the viscosity dependency of the slot aperture	[m]
$k_{d\tau}$	factor for the yield values dependency of the slot aperture	[Pa*m]
KOV (x,y)	covariance of matrix x and y	[-]
L	length between obstacles	[m]
m	constant (Power-law model)	[-]
U	grain velocity	[ms^{-1}]
P	pressure	[Nm^{-2}]
P	transformation matrix	[-]
Q^2	sensivity of disturbances in the model	[-]
R^2	deviation according to the least square method	[-]
Re	Reynolds number	[-]
T	external force on the arch	[N]
T	scores	[-]
t	time	[s]
V	volume	[m^3]
v	velocity	[ms^{-1}]
V_p	volume of particle p	[m^3]
W/ C	Water/ Cement ratio	[-]
W/ S	Water/ Solid ratio	[-]
$\bar{x}$	mean average of observations	[-]
x	input variable	[-]
y	output variable	[-]

Greek

$\dot{\gamma}$	shear rate	[1/s]
σ	standard deviation	[-]
ε	residual in the PC-space	[-]
ϕ	porosity	[%]
τ	shear stress	[Pa]
μ	shear viscosity	[Pas]
τ_0	yield value	[Pa]
ρ_c	compact density	[kg/m^3]
ρ_s	density of suspension	[kg/m^3]
ρ_w	density of water	[kg/m^3]
δ_{xy}	coefficient of correlation	[-]
σ_x	standard deviation of matrix x	[-]
σ_y	standard deviation of matrix y	[-]
τ_∞	yield value at indefinite slot aperture	[Pa]
μ_∞	viscosity at indefinite slot aperture	[Pas]
γ	arch angel of plug formation	[rad.]
β	slope angel of plug formation	[rad.]
β	interphase drag constant	[$kgm^{-3}s^{-1}$]
α	slope of a regular arch formation	[rad]
α	volume fraction	[-]
$\hat{\rho}$	bulk density	[kgm^{-3}]
ρ_s	grain density	[kgm^{-3}]
$\overline{\overline{\sigma}}$	stress tensor	[Nm^{-2}]
$\overline{\overline{\tau}}$	viscous stress tensor	[Nm^{-2}]
ν	dynamic viscosity	[m^2s^{-1}]

Subscripts

f	fluid-phase
l	liquid-phase
q	arbitrary phase
hyd	hydraulic
s	solid-phase
crit	critical
req	required
rel	relative
min	minimum
PC	principal component

1 Introduction

1.1 Background

Grouting as a method of strengthening and sealing rock and concrete is widely used. A historical review of the grouting technology on an international level has been made by A.C Houlsby, 1990. Generally, holes are drilled into the structure of rock/concrete mass in order to make the cracks or leached channels accessible to pump a grout mixture into the same. Knowledge about the grouting technique and grouting material is to a high extent founded on empirical relations and improvisation. Possibilities of sealing structures are of great importance from both an economic and environmental point of view. For example, the requirements of closeness on a rock structure affect both the functions as achieving a dry tunnel and the influence on the surrounding environment (Palmqvist K, 1983). Problems with settlement of the surrounding ground surface around a tunnel, caused by insufficient sealing of the tunnel, can be caused by an insufficient grouting operation.

Grouting is, in this work the method where liquid material flow into cracks in rock or concrete structures. The force that creates the flow is commonly created by overpressure from pumping. The term grouting is usually synonymous with the whole technique and execution of the grouting operation. The used pressure in the grouting operation is the pressure applied to the liquid. The pressure applied to the grout has probably no influence on the properties of the liquid, unless the liquid contains air bubbles. The air bubbles can give rise to the air being compressed in different ways, which create differences in the behaviour of the liquid (Crowe, 1998). The differential pressure that pushes the flow is the important factor. The differential pressure is the difference of pressure between the ground water pressure and the used pump pressure (adjusted by the loss of pressure in pipes). This pressure, in this work, is called the grouting pressure. If the grouting pressure is divided by the length of the flow direction of the liquid that is grouted, the pressure gradient is found. The pressure gradient is an important parameter in order to predict the flow rate and penetrated length for a given liquid in a given geometry.

Penetrability is a summarised term for the grout´s ability to penetrate crack apertures, cracks and channels. The limiting factor can be rheology (flow properties like viscosity and yield value) or plug formation when the grains stick together (filtration tendency).

Also the method chosen for drilling and cleaning of the drill hole is probably of great importance for the penetrability. Concerning the drilling method (hammer drill) it is probable that the design of the drill head and drilling rod influence the shape of the crossing between drill hole and crack. The texture of the drill cuttings is also a possible critical parameter to avoid initialisation of plug formation at the crack entrance. Description of grouting equipment and grouting procedures can be found in Pettersson (1999).

which makes the composition of the grout mixture difficult. Work has been done by Eriksson M, (2002) to develop a model for prediction of grout spread and sealing effect based on the penetrability of the grout.

Even lack of accurate testing methods is a consequence of the lack of fundamental understanding of the performance of the grouting material. One of the major difficulties when accurately measuring the properties of the grout mixture is its changing properties with time.

The crack system and crack apertures are often relatively unknown (Fransson, 2001). Above all these uncertainties it is difficult to predict the grouting result and attempts to do this are seldom performed with a more theoretical approach.

Figure 1.1, Description of the ingredients required for a good grouting result.

Several requirements have to be fulfilled for a good grouting result, see Figure 1.1. The requirements can roughly be divided into two groups, first the requirements of the grouting operation (short run) and second the long term requirements (long run). The short run requirements are for example to avoid hydraulic cracking, rewind pressure and to avoid washout of the grout in the borehole. It is necessary to control the setting time of the grout and the design of the packers. In the long run one has to secure the sealing effect when the construction has been taken into use. To achieve a resistant grouting result one has to know the grouts bleeding during setting, hardening time and even its solubility in the surrounding water environment (chemical resistence). Of course, the penetrability of small cracks is also of certain interest to achieve a resistant grouting result. More about performance and requirements of grouting can be read in Nonveiller E (1989) and Dalmalm (2004).

To fulfil the requirements of more water tight tunnels with requirements of limited ingress of water in urban areas of 1-4 l/ min/ 100 m tunnel, cracks wihh an aperture of approximately 50 µm have to be sealed against ingress of water. Experiences from several tunnelling projects show that one can reduce the inflow of water to approximately 10^{-7} m/s with the first pregrouting operation. It is then only possible to reduce the permeability to approximately $0{,}3*10^{-7}$ m/s with normally existing regrouting technique (Stille H, 2001). The developments after 2001 indicate that 10 times better results can be achieved (Emmelin et al 2004). The conclusion regarding water sealing of tunnels with cement based grouting is probably that one has to via pregrouting seal the whole tunnel in order to fulfil the requirements. The cost of grouting have, in certain projects, been as high as the cost for the blasting and excavation of the tunnel. To improve the technique of pregrouting with cement based material it is necessary to focus on the properties of the used grout mixture.

Other types of structures, which are subject for grouting, are hydropower structures. Hydropower structures are often subjected to internal damage that can impair both structural integrity and water tightness. One of the most common types of damage are cracks due to thermal movement shortly after pouring and porous areas due to leaching, the latter often caused by the former or by pervious concrete. Injection grouting is often an economically advantageous method to repair this kind of damage. Grouting is, in this case, a method for rejuvenation of the concrete structure (VAST, 1991).

The grouting material can be based on solutions of e.g. alkali-silicate-hydrate, epoxy or polyurethane or it can consist of cement and other mineral binders. This classification of the chemical grouts is rough and their only common denominator is that they are all solutions. On the contrary the cementitious ones are suspensions of grains in water. Each category of grouts has its advantages and disadvantages. The main advantages of cementitious injection grouts are their low cost, compatibility with the environment and predictable durability. Concrete is always pervious to water to some extent and if impervious layers of e.g. epoxy are introduced into for example a hydropower structure, water enrichments can occur and give rise to spalling due to frost action in cold climates. This is not likely to happen if a cement grout is used since the hardened grout has approximately the same permeability as the original concrete. Other benefits are environmental friendliness and that they can be handled without special safety equipment for the workers. The disadvantages, compared to the solution grouts, are the limited ability to penetrate fine cracks due to its content of solid grains.

Classification of grouts has also been made in the handbook *Preliminary Glossary of Terms Relating to Grouting (1980).* This handbook is published by the Geo Institute of America (Committee on Grouting, 1980). Four broad categories of grouts are classified:

- Cementitious
- Chemical solution
- Resinous
- Miscellaneous

The cementitious ones are those that use hydraulic cement as a primary binding component. Chemical solutions are defined as those compounds that have a basically waterlike appearance prior to injection. The resinous category is mainly solvent based and normally supplied in two or more components that have to be mixed properly in order to harden and cure. The fourth category (Miscellaneous) includes the ones that do not fit into the other categories, like for example bitumen and clays.

Grouts can also be classified due to other properties like its engineering and rheological properties. The engineering classification is based on grout characteristics and its engineering performance. It includes the flow, penetrability and strength characteristics of grouts, but also the strength and permeability of the grouted mass as related to its interaction with the grout. Based on their initial viscosity, rheologically, particulate and chemical grouts are classed as granular Bingham and non-granular Newtonian grouts respectively.

Initial viscosity is determined from the shear stress- shear rate relationship of the grout using a rheometer. The flow curve for a Newtonian grout is a straight line that passes through the origin, for a Bingham grout the line no longer passes through the origin but makes an intercept with the shear stress axis, which is commonly referred to as the yield value.

To obtain a durable and high strength hardened cementitious grout it is necessary that the grout is stable in terms of bleeding and sedimentation. Sedimentation and bleeding can cause incomplete filling of the crack volume, which creates paths for leaching through a grouted crack, see Figure 1.2. Furthermore, the w/c-ratio has to be kept as low as possible to avoid a hardened cement paste with an extensive pore system. A hardened cement paste with an originally low w/c-ratio has few capillary pores which connect to each other. Few connected pores make the paste more insensitive to leaching of binder material in the paste (Hansson P, 1994, Alemo J 1988).

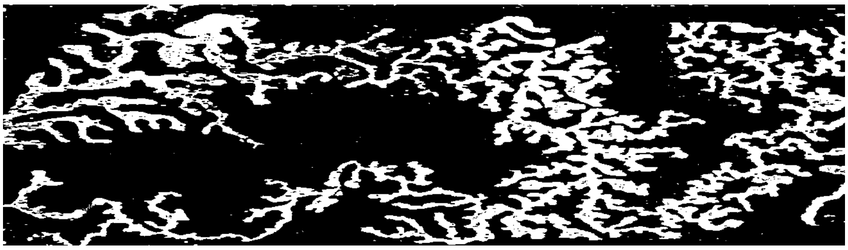


Figure 1.2, Illustration of a crack that is partly filled with grout, the plane of the crack is in the plane of the paper. The white colour indicates areas not filled with grout, black colour is areas filled with grout. Hansson P. (1994).

Regarding grouting in hard rock and concrete structures, it is said that it is possible to grout a crack when its aperture exceeds three times the maximum grain size of the cement (A.M Crawford, 1984). A similar rule of a thumb for soil grouting is that the soil can be grouted if the quotient of the soil's grain size at 15 percent passing to the cement's grain size at 85 percent passing (D_{15}/d_{85}) is more than 20 to 25 (Mitchell J, 1970).

A summary of different authors views of a groutable crack aperture can be found in Brantberger et al, 1998. Bergman (1970) stated that a crack could be penetrated if the crack aperture was 3 times bigger than d_{95} of grains in the dry cement powder. None of these established rules-of-thumb concerning the filtration effect is applicable to predict penetration ability for grouts with low w/c-ratio. These kind rules of thumb are probably more valid for older types of grout mixes with high W/C ratio and no superplasticizer agent added. These older types of grouting cements generally also contained a coarser grain size distribution then that is used today. Laboratory work at Vattenfall Utveckling AB has shown that a quotient between groutable crack aperture and maximum grain size can be as high as about 10, for more modern types of micro cements with a grain diameter <30 µm. (Alemo J, Hansson P 1997).

The penetration characteristics of particulate Bingham grouts are very different from those of Newtonian chemical grouts. Commercial laboratory tests at Vattenfall Utveckling on stable, low w/c-ratio, Bingham grouts show that the most significant limitation to their penetrability is the tendency of cement grains to agglomerate into an impermeable filter cake (plug formation). Grout refusal due to inappropriate rheology, can often be avoided by using superplastisiser (Hansson P, Eklund D, 2001). In Newtonian grouts the penetrability is mainly dependent of the initial viscosity and gel time.

1.2 Objective

The main target with this project is to improve the knowledge about how to compose grout mixtures to fulfil the requirements of penetrability. The detailed objectives of this thesis can be summarised in:

- Map and explain the mechanisms of the fresh mixed grout that govern the plug formation.
- Recommendations of how a grout should be composed to avoid plug formation.

1.3 Hypothesis

Plug formation occurs when the grains in the grout sticks together and create a plug. Plug formation can occur from a constriction in the flow path or at the entrance of a crack aperture. The property of a grout to penetrate cracks and porous material, the penetrability, depends on two things, the rheology and the plug formation. When grouting fine cracks one has to take into account the fact that cement grouts contain solid grains.

The properties of a grout that may prevent passing obstructions in the flow path is in this thesis called filtration tendency, see Figure 1.3. Low filtration tendency is according to this thesis, both a question of total passed amount of mixture and the quality of the passed mixture (concentration of grains in the mixture after filtration).

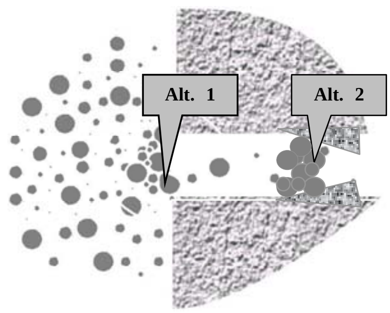


Figure 1.3, The figure show the influence of plug formation. Arches and agglomerates are formed (plug formation) at the entrances of cracks (Alt. 1) and at changes of crack aperture (Alt. 2), which obstruct further penetration of the grout. Hansson (1994).

One of the hypotheses of this work is that the filtration tendency of a grout is not solely governed by the maximum grain size of the cement, or by some other single point of the grain size distribution curve.

The maximum grain size of the cement (d_{100}), d_{95} or d_{85}, is not expected to be as influential on the penetrability as it was considered earlier. The appearance of the entire grain size distribution curve contributes to the behaviour of the grout as well as pore water chemistry, physiochemical aspects and practical issues such as mixing efficiency. Filtration tendency becomes the property of the fresh mixed grout that dominates the penetrability of the grout when the aperture is in the range of 0.3 mm or less, according to tests by (Hansson P, 1996).

The basic idea, in this work, of evaluating the filtration tendency of different grouts is to measure the minimum aperture a certain grout may pass. The aperture that the grout passes is a property of the grout mixture.

The chemical composition is dependent on for example, superplastisiser used and type of cement. The chemical composition of the cement has in this work been kept constant, with the study being confined to the use of Portland clinker. Variation of the grain distribution (grain sizes) will affect the speed of reaction and the flocculation effect in the grout mixture.

The influence of grain concentration, grain size, grain size distribution and chemical properties, on the minimum aperture a certain grout may pass has by Eriksson (2003) been summarised in parameters like b_{crit}, and b_{min} (Eriksson 2003), see Figure 1.4.

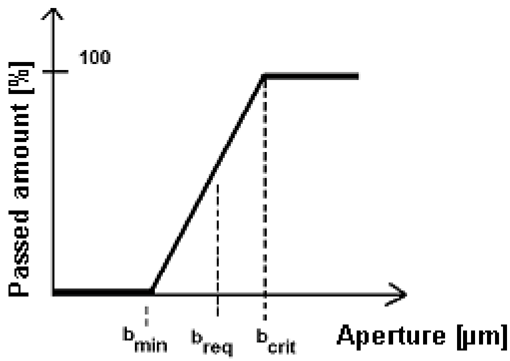


Figure 1.4, Simplified model of the passed amount as a function of used aperture (Eriksson 2003).

The aperture size below which plug formation occurs is denoted b_{crit}. The aperture size below which no mixture at all passes is denoted b_{min}. The parameter b_{req} which will further be described in this work, is dependent on the performance of the grouting operation and should therefore not be seen as a pure property of the grout mixture. b_{req} represents the slot aperture when a sufficient amount of mixture can pass. To fulfil the requirements of b_{req} a sufficient amount of mixture shall with a sufficient quality pass the actual slot aperture.

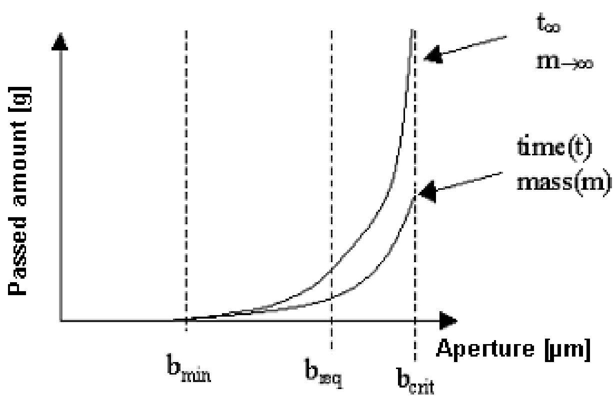


Figure 1.5, More detailed model of the passed amount as a function of used aperture.

When using a larger aperture than b_{crit}, the passed amount will approaches infinity if the available amount of mixture and time is infinite, see Figure 1.5. In practice, during penetration experiments, will b_{crit} will represent a certain amount of passed mixture during a certain time.

Practically b_{min} is relatively hard to define from the measurements when this aperture represents the aperture when no mixture is passing. Practically there will almost always be some mixture that is passing, containing more or less cement grains (different W/C ratio).

b_{req} has to be connected to an actual case to be evaluated (requirements of the results of the grouting operation).

1.4 Limitations

The design and even the results of the experiments have been influenced by the limited available amount of material (dry powder in the used mixtures). It might be possible that a larger passed amount of mixture has caused plug formation even for the filtration experiments were no plug formation has occurred (low filtration tendency). The design of the apparatus for filtration experiments can practically not be designed for use of an infinite passed mixture volume through the filter. The limited amount of used material is in these experiments due to problems in producing larger amount of powder (inert and cement) in certain grain fraction intervals.

The filtration tendency has just been studied at the entrance of for example a slot aperture, not in a constriction further in along a simulated slot plane. It might be reasonable to believe that the initialisation of the plug formation further in along a slot plane, can be influenced by other properties along the slot plane then in the case of plug formation at the entrance of the slot. The plug formation at the slot aperture is mainly a two dimensional phenomenon (slot length is much longer than slot aperture and the extension of the slot plane is small compared to slot length).

Plug formation along the slot plane with a larger extension will probably even be affected by a third dimension, the extension of the slot plane. Surface roughness and electrostatic surface charge along the slot plane can probably affect the plug formation.

1.5 Disposition of the thesis

The first tests carried out deal with an inert material in order to study the filtration tendency. Inert material is used in order to eliminate the time dependent and chemical changes in ordinary cement based grout mixture. The inert material, which is crushed dolomite stone, does not react with the added water in the suspension. The first tests also include scaled-up filtration experiments with plastic grains (2-4mm) and numerical simulation of plug formation in the scaled-up model.

The second tests deal with cementitious grouts based on Portland cement. This work also mainly includes mixtures with a limited maximum bleeding of approximately 5%.

The grouted mediums that are considered are cracks in hard rock and concrete structures. In order to get an overview of the structure of this thesis, a short description of each chapters´s content is given below.

Chapter 2 contains a description of the medium that is supposed to be grouted. The mediums, which are described, are fractured rock structures and fractured concrete structures. Short descriptions of different sealing methods are given in this chapter.

Chapter 3 contains a fundamental description of the mechanisms and parameters, which obstruct grout penetration of crack volumes. Attempts to develop theoretical models for filtration tendency have been made.

Chapter 4 introduces cementitious grouts. Used materials like cement and superplastisisers are presented. Experiences from former penetrability experiments with cement based mixtures are discussed.

Chapter 5. Test equipment and test methods are described in detail. A concept to quantify filtration tendency has been developed. Description of used numerical and physical models.

Chapter 6. The results of the penetration experiments with inert and cement based material are presented.

Chapter 7 analyses the test results presented in chapter 6. Feedback of the results is given. Some general guidelines for the composition of a cement based grout mixture with low filtration tendency are given.

Chapter 8 deals with a proposal to further research based on the conclusions and analyse made in chapter 6 and 7. Five different fields for further research are identified.

2 Characteristics of the grouted structure

2.1 General

The geometries, which in this work, are supposed to be penetrated are cracks or porous material. Much work has done in the field of characterisation of cracks in rock and concrete structures. The purpose of characterisation of the cracks is mainly to describe the properties of interest for the grouting process, such as crack apertures, roughness of the crack plane and water conditions of the crack. Different models of cracks in rock structures have been tested in order to better predict the grouting process (Jansson 1998). This section of the work will very briefly illustrate some parts of the knowledge about the medium that is supposed to be grouted.

The aims of grouting structures are often to fill voids, cracks and pores and sometimes strengthen the structure. The sealing of the structure prevents further attack on the structure. The structure consists of rock, concrete or soil. This work deals with rock and concrete grouting, as the technique is almost the same for both. Cracks can be stiff or flexible. The movement in cracks can depend on different causes, daily temperature changes or movement due to seasonal variation. Cracks can be either dry or wet. The choice of grouting agent will be made, depending on the requirements and purpose of the grouting, construction material, type of cracks, aperture, movements and degree of moisture in the cracks.

2.2 Rock structure

Little is known about the way in which cracks are formed (geological background) in a rock (Fransson 2001). Hard rock can be described as a system of cracks and zones of weakness in a solid rock mass. The cracks and zones of weakness can be due to tectonic, thermal, lithostatic and high water pressure. Their appearance mainly depends on the stress configuration in the rock, both at present and during petrogenesis, and on the mechanical and physical properties of the rock mass. There are different types of cracks, cracks with flow of water and tight cracks. Cracks can be very conductive due to crack aperture. From an engineers point of view it is of importance to characterize the aperture, frequency and direction of the cracks, in order to succeed with the grouting operation.

A rock mass structure contains discontinuities, which may be described individually or as an entire system. Flow of water in cracks is usually a problem when constructing tunnels or other underground facilities. Models for describing the crack or crack systems has been made by several researchers, see for example (Fransson 1999). There are mainly two approaches, the continuums and the discrete modelling. The continuum models describe a homogenous and porous material, where the pores are fully connected. The continuum approach is limited due to the scale of the problem.

The volume of rock needed for a continuum approach is often relatively large, about 100-1000 m^3, even if the frequency of cracks is high (~10 st/m) (Rehbinder et al 1995).

Discrete crack modelling attempts to include every important conductive crack in a volume. Fractures in a discrete crack model are defined by a number of characteristics (Dershowitz and Doe 1997). The characteristics are location, shape, orientation, size, intensity, transmissivity and storativity. Usually there is a problem to define the actual geometry of the crack on the basis of performed measurements as for example a water loss test. The basic idea of crack modelling is to via results (conductivity or transmissivity) from water loss test, translate the hydraulic crack aperture into a geometric crack aperture. The possibility to evaluate the actual geometric crack aperture (b) is fundamental to using the crack aperture as a measure of the grout mixtures filtration tendency.

In order to predict the behaviour of mass from a grouting point of view it is of interest to look closer to the discrete modelling of cracks. Channel network is one type of discrete modelling of cracks and has been used by several authors for different application. Hässler (1991), Gylling (1997) and Eriksson (2003) used this model to predict the spread of grout.

To obtain a suitable model, Hakami (1995) listed a number of important crack properties, which affects the flow behaviour in the crack. The crack should be seen as a three dimensional geometry (crack volume), which has some specific properties. The important properties of the crack are aperture, roughness, contact area, matedness, spatial correlation, tortuosity, channelling and stiffness.

The three dimensional behaviour of the water flow in cracks makes it interesting to not only analyse the crack aperture, but even the roughness of the walls and asperity regions (contact areas), i.e. where the two opposite faces of the crack walls are in contact to each other. Pure measurement of the water flow through the crack, is not sufficient to predict the penetrability of a particulate mixture (grout). It is of interest to estimate the distribution and sizes of the crack apertures along a crack plane, in order to predict the penetrability of the crack. The variable crack aperture is a widely studied problem. (Tsang & Tsang, 1989; Hakami, 1995; Nordqvist et al, 1992; Larsson, 1997 among others).

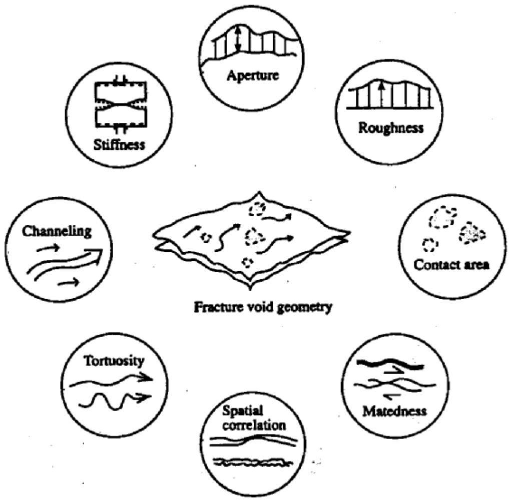


Figure 2.1, Characterization of crack properties that controls the flow in a crack, Hakami (1995).

A proposal to relationship between the hydraulic aperture and the physical aperture was made by Zimmerman et al (1991) and Cheng et al (2000). The relationship stated that an increasing difference between the hydraulic aperture (b_{hyd}) and the physical aperture (b) was found as the standard deviation (σ) of b increases. The relation is described by the eq 2.1.

eq 2.1

$$(b_{hyd}/b)^3 = 1 - e^{-0.56 \cdot b/\sigma}$$

The characterisation of a cracks in a hard rock structure can be done in several ways. Experiments (Gustavsson, 2004) show that the crack planes are hardly horizontally parallel to each other, the apertures will vary and fragments of rock pieces (alteration products from rock and ground water) can be found in the cracks. Different crack apertures and fillings along the crack plane are connected to each other via a network of fine cracks that cross between the planes. The boreholes into the rock structure will cross a number of these crack planes. A number cracks and voids will be accessible to grout.

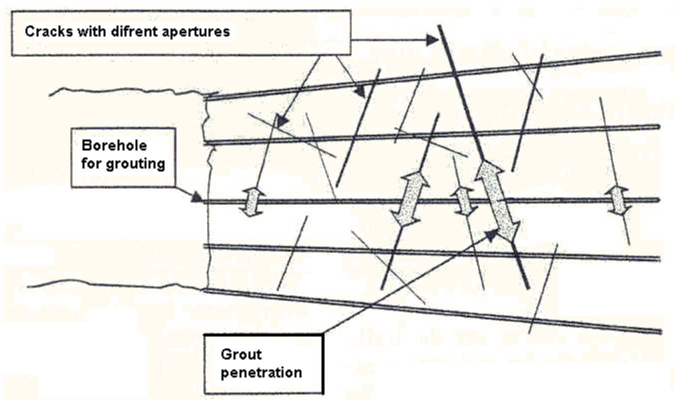


Figure 2.2, Description of grout spread in the rock structure (Gustavsson, 2004).

2.3 Concrete structure

Concrete structures are often subjected to internal damage that can impair both structural integrity and water tightness. The most common types of damage are cracks due to thermal movement shortly after pouring and porous areas due to leaching, the latter often caused by the former or by pervious concrete. Injection grouting is often an economically advantageous method to repair this kind of damage. Grouting is in this case, a method for rejuvenation of the concrete structure. The cause and extension of the damage and its influence on the structure must be made. The crack system and crack apertures are often relatively unknown. Above all these uncertainties is the fact that it is difficult to evaluate the grouting result and attempts are seldom performed.

Crack patterns, crack apertures, depth and orientation must be surveyed as well as the location and size of the voids. The moisture state, cleanness and possible movement of the cracks are also of significance. The moisture state can vary from dry to flowing water. The movements can be frequent (traffic load), daily or yearly. The examination can be visual, include core drilling and use of non-destructive methods or water loss tests.

The requirements of the structure and the repaired structure as load bearing capacity must be clear before starting the grouting operation. Grouts for force transmitting filling of cracks are products, which are able to bond to the concrete surface and transmit forces across them. Grouts for ductile fillings of cracks are products, which are able to accommodate subsequent movements. When the grout has filled the cracks, pre-stressed reinforcement can be mounted in order to increase the strength of the structure. It is of great importance to fill all cracks, because if movements occur in the structure it can cause loss of pre-stress in the reinforcement bars.

It is normal with cracks in reinforced concrete structures. In some cases measures have to be taken in order to reduce their effects on the structure, see Table 2.1.

Table 2.1, Characterisation of cracks in concrete structures. (Alemo, 2003)

<table>
<tr><th>Type of cracking</th><th>Sub-division</th><th>Time of appearance</th><th>Phase</th></tr>
<tr><td rowspan="3">Plastic settlement</td><td>Over reinforcement</td><td rowspan="4">Ten minutes to three hours</td><td rowspan="7">Very early age</td></tr>
<tr><td>Arching</td></tr>
<tr><td>Change of depth</td></tr>
<tr><td>Formwork settlement</td><td></td></tr>
<tr><td rowspan="3">Plastic shrinkage</td><td>Random</td><td rowspan="3">Half an hour to six hours</td></tr>
<tr><td>Over reinforcement</td></tr>
<tr><td>Parallel</td></tr>
<tr><td>Self desiccation</td><td>W/C < 0.45</td><td>During hardening</td><td rowspan="5">Early age during the hardening of the concrete</td></tr>
<tr><td rowspan="2">Crazing</td><td>After surface treatment</td><td rowspan="2">One to seven days, sometimes much later</td></tr>
<tr><td>Against formwork</td></tr>
<tr><td rowspan="2">Thermal cracking</td><td>Surface cracking</td><td rowspan="2">One day to some weeks</td></tr>
<tr><td>Through cracking</td></tr>
<tr><td rowspan="3">Drying shrinkage</td><td>One-side drying</td><td rowspan="3">One to several months</td><td rowspan="6">After part of the structure has been completed</td></tr>
<tr><td>External restraint</td></tr>
<tr><td>Differential final shrinkage</td></tr>
<tr><td rowspan="2">Thermal cracking</td><td>Surface cracking</td><td rowspan="2">During cooling to long-term ambient temperature</td></tr>
<tr><td>Through cracking</td></tr>
<tr><td>Pre-stressed concrete</td><td>Cracks at anchorage</td><td>After pre-stressing</td></tr>
<tr><td rowspan="5">Loading cracks</td><td>Micro cracks</td><td rowspan="5">At loading</td><td rowspan="15">During service life</td></tr>
<tr><td>Tensile cracks</td></tr>
<tr><td>Flexural cracks</td></tr>
<tr><td>Shear cracks</td></tr>
<tr><td>Torsional cracks</td></tr>
<tr><td>Long-term loading cracks</td><td></td><td>After completion of creep</td></tr>
<tr><td>Imposed deformation</td><td colspan="2" rowspan="2"></td></tr>
<tr><td>Ground settlement</td></tr>
<tr><td rowspan="2">Corrosion in reinforcement</td><td>Chloride initiated</td><td>More than one year</td></tr>
<tr><td>Carbonation initiated</td><td>More than five years</td></tr>
<tr><td>Sulphate attack</td><td></td><td>More than five years</td></tr>
<tr><td>Alkali-silica reaction</td><td></td><td>More than five years</td></tr>
<tr><td>Alkali-carbonate reaction</td><td></td><td>More than five years</td></tr>
<tr><td>Freezing and thawing</td><td></td><td></td></tr>
<tr><td>Fire</td><td></td><td></td></tr>
</table>

According to Table 2.1 there exist a vast number of reasons for a concrete to crack and each type of crack shall be treated in different way depending on their origin. Two main methods exist to take care of sealing of cracks in concrete structures (Injection and surface sealing). Injections is an internal treatment to fill most of the cracks and voids and thus seal the cracks. The crack apertures of damaged concrete structures can vary between small ones of approximately 50 µm up to apertures of several millimetres. The ones that are necessary and possible to seal will of course vary depending on the actual case. In the case of making a structure waterproof it is generally necessary to seal cracks down to 100 µm and even smaller. The requirements of grouting in concrete structures will also be linked to the configuration of the crack plane. A crack plane that crosses the whole structure (for example a dam wall) has to be penetrated to a larger extent than a crack plane that does not cross to whole structure. In cases of structural repair (mounting of pre-stressed reinforcements in the grouted structure) it can generally be enough to fill the apertures in the region of 0,5-1.0 mm. In the case of filing these larger cracks with grout, the purpose is to be able to transmit the force from the reinforcements through the crack plane, without deformation of the structure. Grouting is the predominant method of repairing concrete cracks. The method of grouting concrete structures will also vary according to the type of crack, generally holes are drilled into the structure (like pre-grouting of rock structures) but even surface sealing methods can be used.

Surface sealing is the other alternative method. The penetration of the sealing products (as for example cement mixtures) is driven into the cracks by the force of gravity. Surface sealing which can be subdivided into two groups one with membranes applied either as liquids or preformed (bonded or unbonded) sheets and another one in which a suitable dimensioned groove is made and filled with an appropriate sealant. At surface sealing of cracks it is important to make the sealing on the most humid side of the structure. A sealing on the wet side will better withstand possible water pressure and there is less risk for an increase of the humidity behind the sealing, which can lead to frost damage. There are examples where sealing on the wrong side have decreased the durability (VAST, 1991).

2.4 Conclusion and discussion

Based on literature studies the following important factors, regarding the properties of the grouted medium (rock and concrete), can be stated with respect to the penetrability of cracks.

- The size of the crack aperture is of critical importance for the penetrability of the grout into the crack.

- The geometric crack aperture is practically hard to measure in the structure. Estimation of the hydraulic aperture can be made by methods like water loss test. Translation of the hydraulic aperture into a geometrical aperture is essential in order to evaluate the grout mixtures filtration tendency in terms of the geometrical crack aperture (b).

- The aperture varies widely along a crack plane, with the result that the main flow of fluids takes place along a few channels and that a large proportion of the surface is tight, these are referred to as contact areas.

- It is of great importance to investigate the cause of cracks in a concrete structure, in order to repair the cracks in a proper way. This includes, of course, selection of material, equipment and method of the grouting operation.

- The distribution and location of minor and larger crack apertures and voids have to be located in order to propose a plan for the grouting operation.

- In order to get a representative mean value of the hydraulic conductivity of the rock mass, a large rock mass must be tested. The hydraulic conductivity can be interpreted to a hydraulic aperture if the number of cracks is known.

3 Penetrability

3.1 General

The penetrability of the fresh mixed grout can probably be seen as the single most important property in order to achieve a good grouting result. A good grouting result is usually synonymous to a durable filling of the grouted geometries. Durability of the hardened grout requires that the W/C ratio should be kept as low as possible (Hansson, 1998). The lowest possible W/C ratio will vary depending on the properties of the mixture and the purpose of the grouting performance (for example grain size, grain size distribution of the dry cement powder, superplastisiser and crack aperture). Commonly, with the use of modern micro cements, W/C ratio will vary between 0.7-1.5 and still maintain a durable grouting performance. Performed laboratory experiments (Alemo, 1988) show that an increase of the W/C ratio from 0.3 to 0.5 may cause leaching in the paste to increase with 90 %. The penetrability of this type of highly concentrated mixture can not be described solely by a rheological model because the grains are more or less in constant contact with each other. In a grout with low W/C ratio the packing ratio of grains can be as high as in the dry cement powder. That can be illustrated through a grout mixture with a W/C ratio of 0.8, which has almost the same packing ratio as a dry cement powder. The packing ratio, in the mixture, is defined as the ratio between the volume of solid grains and the volume of surrounding fluid in the sample. In a dry powder is the packing ratio the quotient between volume of grains and the surrounding volume of air in a specified volume of sample.

The packing ratio is generally expressed as the volumetric concentration (c) of grains in the mixture. The porosity (ϕ) in the mixture is equal to one minus the volumetric concentration (ϕ=1-c).

There exist models for predication of the penetrability of grout mixtures with low grain concentrations (high W/C ratio). Most of these types of models are based upon a rheological approach, where the mixture is seen as a homogeneous fluid.

An equilibrium equation can be written (Hässler, 1991) in which the pushing force from the pump pressure and the shear force from the yield value of the grout is in balance. In a given geometry the penetration length can then be calculated for the grout. Eriksson 2002, developed Hässlers models further by introducing a restriction in the penetrability due to grain size of the grout.

To achieve a good penetrability of the grout it is probably necessary to both optimize the rheology (flow properties) and the filtration tendency of the fresly mixed grout.

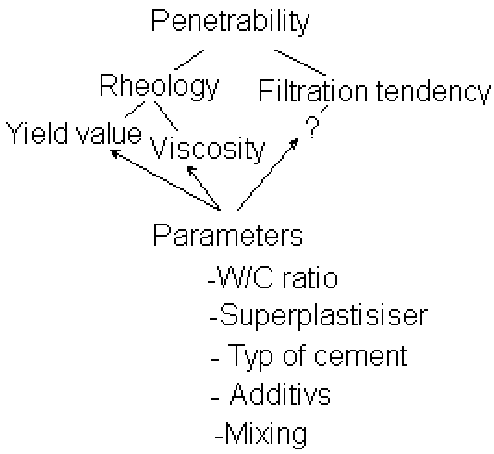


Figure 3.1, Parameters that influence the penetrability of the fresly mixed grout. The parameters (?) of the freshly mixed grout that govern the filtration tendency of the grout is today not fully understood.

Exactly which properties influence the penetrability, and the magnitude of their influence, are still not entirely understood, see Figure 3.1. The lack of knowledge about filtration tendency of cement based grouts is the background to this project. A method of assessing the penetrability of a grout by measuring the filtration tendency has been developed in this project. The testing is performed on freshly mixed grout and properties such as cement quality, admixtures and mixing efficiency that affect the penetrability are considered.

3.2 Multiphase flow

3.2.1 General

The basic idea of using the theory and tools of multiphase flow in order to predict plug formation in a grouting process, aims to explain in a theoretical manner the important mechanisms. The theory and mathematics of multiphase flow of dense mixtures is generally difficult and rapidly becomes complex. Therefore numerical methods have to be utilized in order to solve the equations that govern such flows. In general the equations that govern the flow of dense mixtures are a system of partial differential equations. CFD (Computational Fluid Dynamics) calculations have proven to be a successful approach to model multiphase flow. The method of CFD usually solves the equations by using a numerical scheme called the finite volume method.

This section of the work will highlight the multiphase flow approach as a complementary aid to the physical experiments in the work of understanding and explaining the mechanisms of plug formation. This chapter is a summary of the work performed in a master thesis (Saaidi 2004) performed within the field of multiphase flow.

Multiphase flow covers a wide range of applications from the flow of mud to the flow field in gas turbine engines. In the past few years there have been significant advances in the science and technology of multiphase flows.

The field is evolving from one where empiricism played a major role to one in which analysis and modelling can now be used to complement design and control. However, the technology is far from mature and many challenges still remain (Crowe et al, 1998).

Multiphase flows are important in a vast number of industrial applications. In general the flow situations that arise in real applications are very complex. Multiphase flow is the most common flow of fluids in nature. Some examples of multiphase flows are the flow of blood, the drifts of clouds in the atmosphere, boiling liquids, transport of grains in rivers etc. (Hestroni, 1982)

Multiphase flows can be divided into a number of subcategories depending on the components in the flow. The flow of grains in liquid is a subcategory of multiphase flows. A phase refers to the liquid, solid or vapour state of matter. The best example of a single-phase flow is the flow of air, which is a mixture of gases. These types of flows are treated as single component flows with a viscosity and thermal conductivity representative of the mixture of gases as a whole.

The multicomponent nature of air will be important at high temperatures where dissociation can occur, or at very low temperatures where some species may condense out. Examples of four different categories of multiphase flows are given in Table 3.1.

Table 3.1, Categories and examples of multiphase flows. (Crow, 1998)

Gas-liquid flows	Bubbly flows Gas-droplets flows
Gas-solid flows	Gas-grain flows Pneumatic transport
Liquid-solid flows	Slurry flows Sediment transport
Three-phase flows	Bubbles in a slurry flow Grains in gaseous flow

Liquid-solid flows are flows where liquid carries solid particles. This type of flow is of considerable interest in the study of the filtration tendency of cementitious grouts. The liquid-solid flow is often called slurry flow or dispersed flow.

Single phase flow of fluids has occupied the attention of scientists and engineers for many years. The governing equation (Navier-Stokes equations) for the motion and thermal properties of single-phase fluids are well accepted.

The major difficulty in single-phase flows is the modelling and quantification of turbulence and its influence on mass, momentum and energy transfer. Contrary to single-phase flows the field of multiphase flows are much more primitive in that correct formulations of the governing equations are used.

Multiphase flows can be modelled in many different ways, see Table 3.1. In some two-phase flow applications it might be sufficient to model each phase with well established single-phase flow equations with a moving boundary between the phases.

However depending on the applications different types of methods are more or less suitable to use. However the method of modelling each phase with a moving boundary between them is not useful when the phases are well mixed, as in the case of a cementitious injection grout. In a dense and well mixed mixture the number of grains are large thus averaging procedures are necessary to make the equations solvable. The most common and important averaging procedures are space averaging, time averaging and ensemble averaging.

The developments of numerical modelling are in many cases driven by the improved capacity of computers. With modern computer technology it is possible to solve the partial differential equations describing multidimensional, time dependent two-phase flow problems. Development of the theoretical background is needs to be done in order to perform better numerical simulations and to understand the physics behind the flows. In addition empirical data from carefully conducted experiments are needed in order to close the set of equations that govern the flow. Hence successful numerical modelling is strongly dependent on accurate experimental data (Enwald et al 1996).

3.2.2 Two-phase flow models

There are numerous ways to model two-phase flow problems depending on the nature of the problem. One way is to use a numerical mesh finer than the smallest length scales and time steps smaller than the fastest fluctuations of the flow. This method is usually called Direct Numerical Modelling (DNS). DNS is the subject for an extensive amount of research but is today not feasible to be used in problems of interest regarding penetrability of grouts, due to lack of computer capacity. In the techniques described below the local instantaneous equations for the continuous phases are averaged in a suitable way that allows a coarser mesh and a longer time step to be used in the calculations.

Another way to model multiphase flows is the Lagrangian modelling technique. However this method keeps track of each individual solid particle (grain), i.e., it solves Newtons's second law of motion for each grain. Thus it generates an unrealistic amount of equations if the volume concentration of grains is high, which unfortunately is the case for cement based grouts. The Lagrangian technique would demand an unrealistic amount of computer power.

Due to the extensive amount of computer power needed to use the Lagrangian technique it is obviously not a technique that is feasible to use for dense mixtures with many grains. As an example it can be mentioned that if the CFD model would calculate the position and interaction of each grain (ϕ= 50 μm) in a mixture with a volume concentration of 1%, the number of grains in one cm^3 would approximately be one million. In the case of mixtures used for grouting the volume concentration of grains seldom goes under 15 % (W/C ratio~1.5).

A third way of modelling multiphase flows is the Eulerian modelling technique. In Eulerian modelling both phases are modelled as two intersecting continua, therefore it is not possible to model the interaction between specific grains. Moreover it is not possible to explicitly account for the size of grains, meaning that the grains can be specified to have a larger diameter than the actual crack aperture and still penetrate through the crack. This is possible since the grain phase is interpreted as a continuous phase. However the size of the grains has an indirect effect on the penetrability of the grain phase, because of the direct effect it has on to the shear viscosity for the grain phase. The main focus of the present study is the Eulerian two-dimensional modelling technique.

The Eulerian models can be divided into two different sub categories:

- *Diffusion models*

  Diffusion models are formulated by considering the mixture as a whole, they can be represented by one continuity and one momentum equation in each coordinate direction. Additionally there should be one energy equation and one diffusion equation to account for the effect of concentration gradients.

- *Two-fluid models*

  A two-fluid model consists of two continuity equations and two momentum equations in each coordinate direction. Moreover there are two energy equations. To close the set of equations different closure laws are needed. The Eulerian model is a two-fluid model.

In the present work the Eulerian two-phase model is used.

3.2.3 Basic governing equations

To be able to translate Navier-Stokes equations for the fluid and Newton´s equation of a single grain directly into continuum equations representing momentum balances for the fluid and solid phase, researchers mathematically define local mean values. The point variables are averaged over regions which are large with respect to the solid grain diameter but small with respect to the characteristic dimension of the complete system. There are two types of equations that govern the flow of the mixture, the continuity equation for mass balance and the Navier Stokes equation for momentum transfer.

For a single-phase fluid model, a single set of conservation equations for momentum and continuity are solved. However, in a multiphase flow model additional sets of conservation equations must be introduced, in this process the original set must also be modified. This modification involves the introduction of volume fractions for the different phases, moreover on extra term has to be added to account for the momentum exchange between the phases. The equations presented in this chapter can be found in any fundamental multiphase flow textbook.

Continuity equation, mass balance

In general form the continuity equation can be written as:

eq 3.1

$$\frac{\partial}{\partial t}(\alpha_i \rho_i) + \nabla \cdot (\alpha_i \rho_i \vec{v}_i) = 0$$

The continuum assumption is the foundation for all the basic laws in fluid mechanics. The continuum assumption states that macroscopic quantities such as; density, velocity and pressure varies continuously from point to point throughout a fluid.

The continuity equations for the fluid and solid phases can be expressed as:
Fluid phase:

eq 3.2

$$\frac{\partial}{\partial t}(\alpha_f \rho_f) + \Delta \cdot \left(\alpha_f \rho_f \vec{v}_f \right) = 0$$

Solid phase:

eq 3.3

$$\frac{\partial}{\partial t}(\alpha_s \rho_s) + \Delta \cdot \left(\alpha_s \rho_s \vec{v}_s \right) = 0$$

Where α is the void fraction of grains. By definition the sum of the void fractions (α_{fluid} and α_{solid}) must be unity.

$$\alpha_f + \alpha_s = 1$$

The volume of phase q is defined as:

eq 3.4

$$V_q = \int_V \alpha_q dV$$

The effective density (or bulk density) of phase q is:

eq 3.5

$$\hat{\rho} = \alpha_q \rho_q$$

The bulk density is the density of the q:th phase in a given material region with respect to the total volume in that specific region.

The physical principle underlying the continuity equation is the conservation of mass. In words the equation may be stated as "the time rate of change of the mass of a material region is zero". A material region is defined to be a volume where the velocity of the surface enclosing the volume is equal to the fluid velocity (Panton, 1996).

To obtain a better physical understanding of the terms in the continuity equation it can be evaluated at a fixed point, i.e. a fixed point P in space, at the center of a fixed differential volume. From this point of view the continuity equation is a balance between the rate of accumulation of mass and the net outflow of mass. The first term on the left hand side (eq 3.2 and eq 3.3) describes the rate of accumulation of mass per unit volume at P. The second term describes the net flow of mass out of P per unit volume.

Navier-Stokes equation, momentum transfer

Navier-Stokes equation for a single phase fluid can be written as:

eq 3.6

$$\rho\left(\frac{\partial \vec{v}}{\partial t} + \vec{v}\cdot\nabla\vec{v}\right) = -\nabla p + \mu\cdot\nabla^2\vec{v} + \vec{F}$$

Where $\frac{\partial \vec{v}}{\partial t}$ is the material derivative, which has the physical interpretation of the time derivative, following a fluid particle[1]. eq 3.6 can be interpreted from the same point of view as the continuity equation, i.e., we evaluate the equation at a fixed point, P, in space at the center of a differential volume.

The first term on the left hand side in eq 3.6 describes the rate of momentum increase at the fixed point P, sometimes referred to as the local rate of change. The second term on the left and side describes the net rate of momentum that is carried into P by the fluid flow, commonly referred to as the convective change.

[1] In this context a fluid particle and a material region is exactly the same thing.

The first term on the right hand side denotes the net pressure force at P. The second term on the right hand side represents the net viscous force at P and the third term describes the body forces at P (gravity is an example of a body force).

In words the equation states that the mass per unit volume (ρ) times the acceleration of a fluid particle (the left hand side) is equal to the net force acting on the particle, which corresponds to Newton's second law of motion.

Extended Navier-Stokes equation

Focusing on two-phase flows, which are the flow of interest regarding the filtration tendency problem, the conservation of momentum for the fluid-phase is given by an extended Navier-Stokes equation. The effective (bulk) density replaces the density in the single-phase Navier-Stokes equation. Moreover one extra term (β) is added to account for the interphase momentum transfer.

The momentum balance equation for the fluid-phase is given by:

eq 3.7

$$\frac{\partial}{\partial t}\left(\alpha_f \rho_f \vec{v}_f\right) + \nabla \cdot \left(\alpha_f \rho_f \vec{v}_f \vec{v}_f\right) = \nabla \cdot \overline{\overline{\tau}}_f + \alpha_f \rho_f \vec{g} - \alpha_f \nabla P - \beta\left(\vec{v}_f - \vec{v}_s\right)$$

Here P is the fluid phase pressure, g is the gravity acceleration, β is the interphase momentum transfer coefficient, τ_f is the fluid phase shear stress tensor which is assumed to be Newtonian.

The momentum balance equation for the solid-phase is even more modified to account for the particle-particle (grain-grain) interactions. Thus comparing it to the single-phase Navier-Stokes equation there are two additional terms added. As in the case of the fluid phase momentum equation, the single-phase density is replaced by the bulk density. The momentum transfer term (β) is also included but with the opposite sign compared to the fluid phase equation. Additionally, a solid-phase stress tensor, sometimes referred to as the solid-phase pressure (P_s) is included in the equation. This term should account for the grain-grain interactions.

eq 3.8

$$\frac{\partial}{\partial t}\left(\alpha_s \rho_s \vec{v}_s\right) + \nabla \cdot \left(\alpha_s \rho_s \vec{v}_s \vec{v}_s\right) = \nabla \cdot \overline{\overline{\tau}}_s + \alpha_s \rho_s \vec{g} - \alpha_s \nabla P - \nabla P^* - \beta\left(\vec{v}_f - \vec{v}_s\right)$$

Here P_s is the solids phase stress tensor with a kinetic, collisional and frictional contribution. The two first contributions are derived from kinetic theory (Saaidi 2004) and the latter one is given by empirical or semi-empirical relations. Hence the solid phase stress tensor reads:

eq 3.9

$$\overline{\overline{P}}_s = \overline{\overline{P}}_{s,kinetic} + \overline{\overline{P}}_{s,collision} + \overline{\overline{P}}_{s,friction}$$

Since the main interest in the present work is to investigate the clogging of grains in a narrow slot it is believed that the frictional contribution will be the most dominant contribution. Hence it is of great importance to have a high-quality frictional model (Saaidi, 2004).

The interphase momentum transfer coefficient (β) is an empirical parameter that describes the momentum transfer between the fluid and grain phases. The drag coefficient is typically obtained from pressure drop measurements in fixed, fluidized or settling beds. As seen from the equation, the term containing the interphase transfer coefficient is directly proportional to the velocity difference between the phases. As a consequence this term will become increasingly important as the velocity difference between the phases increases, which is a logical argument from a physical point of view. Thus the momentum exchange between the phases will increase as the velocity difference increase. In a grout this term will probably be of significant importance if the phases start to separate (Saaidi, 2004).

The fluid phase shear stress tensor (τ_f) describes the shear stresses acting in the fluid because of the viscous forces. In a Newtonian fluid these stresses are proportional to the rate of strain within the fluid.

The solid´s pressure ($\overline{\overline{P}}_{s,kinetic}$ and $\overline{\overline{P}}_{s,collision}$) is more difficult to interpret than the fluid phase pressure. Both the kinetic and collision part is derived from the kinetic theory of granular flow, which is an extension of the kinetic theory of dense gases. The collisional part denotes the momentum transferred by direct collisions and the kinetic part physically represents the momentum transferred through the system by grains moving across imaginary shear layers in the flow. Figure 3.2 gives a schematic description of the kinetic, collision and frictional part of the solid phase stresses. At high solid volume fractions, sustained contacts between grains occur. The resulting frictional stresses ($P_{s,friction}$) must be accounted for in the description of the solid-phase stress. Hence the frictional stress is added to the stresses predicted by the kinetic theory for $\alpha_s \geq \alpha_{s,\min}$.

eq 3.10

$$\overline{\overline{P}}_s = \overbrace{\overline{\overline{P}}_{s,\,kinetic\ theory}}^{\overline{\overline{P}}_{s,\,kinetic}+\overline{\overline{P}}_{s,\,collision}} + \overbrace{\overline{\overline{P}}_{s,\ friction}}^{if\ \alpha_s \geq \alpha_{s,\min}}$$

where $\alpha_{s,\ \min}$ is the smallest value for which the frictional stresses start to become important. When the solid´s volume fractions become high the frictional stress part will dominate the solid phase stresses. Thus to obtain correct numerical results it is crucial to have a good model for the frictional stress. Since most of the frictional stress models are semi-empirical a correct model for the frictional stress could be obtained through carefully conducted experiments.

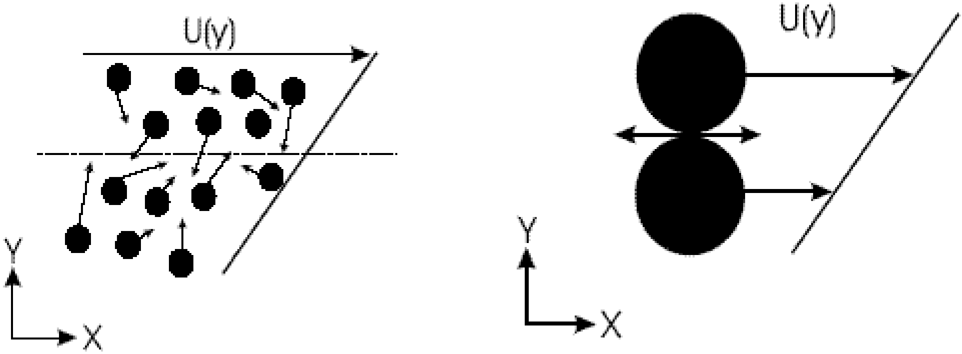


Figure 3.2, Kinetic effect (left hand side), random grain motions across a plane. The mean fluid velocity gradient gives rise to momentum transfer across the plane. Collisional effect, (right hand side) frictional interparticulate forces may become very large when the solids concentration is high

The three different the particle pressures described above (eq 3.10) contribute to the solid phase stress tensor. Hence they will have an impact on the outcome of the numerical simulations for the filtration tendency problem. Thus it is crucial to have accurate models for these physical parameters. As mentioned above kinetic theory gives the values for the normal forces due to grain-grain interactions, whereas for the frictional part we have to rely on empirical relations. An extensive review on kinetic theory can be found in (Wachem, 2000).

3.2.4 Closure laws

For the Eulerian modelling approach the local instantaneous equations are averaged. The averaging procedure introduces a closure problem, i.e., the system gets more unknown variables than equations. In order to solve the closure problem a set of additional expressions is needed. These expressions are commonly referred to as closure laws. There exist three different types of closure laws:

- *Topological laws*
  The topological closure laws describe the spatial distribution of the phase specific quantities.

- *Constitutive laws*
  The constitutive closure laws describe the physical properties of the phases.

- *Transfer laws*
  The transfer closure laws describe the different interactions between the phases.

Since most of the closure laws are empirical, experiments are vital for the development and verification of the closure laws. The key to accurate numerical modelling is to a large extent dependent on the formulation of the closure laws that are used. A considerable amount of different closure laws have been used in two-phase flow modelling, but still it can be difficult to find suitable closure laws for a specific case (Enwald et al. 1996).

The different types of closure laws are sometimes grouped together and simply referred to as constitutive laws. The laws do not describe the transport of mass, momentum or energy across the interface between the phases. For the solid phase there are two different ways to model such physical properties as dynamic viscosity, bulk viscosity and the solids pressure.

One way, sometimes referred to as the traditional way, is when the empirical models are constructed based on the grain properties and the local grain concentration. The other way is to apply the so-called kinetic theory of granular flow that many researchers believe have a wider range of applicability. The empirical models are simpler and hence easier to implement in a computer code. Figure 3.3 schematically shows how a two-fluid model is constructed.

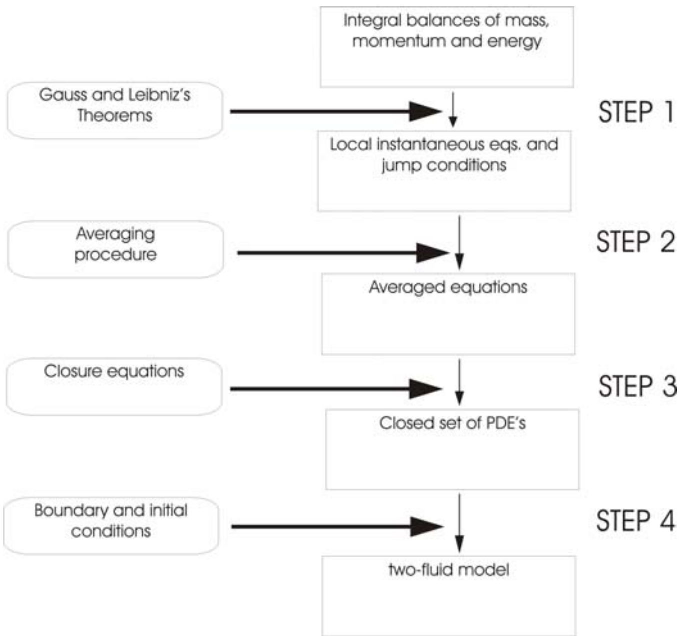


Figure 3.3, General outline for the formulation of a two-fluid model.

An even more detailed description on how eq 3.9 is derived and simplified can be found in Wachem (2000). eq 3.9 do not say anything about the mechanism of plug formation (the filtration tendency of the mixture), however it is fundamental in order to understand the numerical modelling of flow behaviour of dense mixtures like grout. It is possible to predict the risk of plug formation by the help of changes in void fractions (continuity equation) in different places in an injected geometry.

High concentration of grains (void fraction of fluid is low) can indicate an increased risk of plug formation at this specific place.

By the help of Eulerian modelling of flow, differences can be seen between the volume fractions of fluid and solid phase. Modelling of flow of densely packed mixtures is today just possible with the type of continuum model like the Eulerian.

3.2.5 Rheology

Rheology is a way of describing fluid properties without paying any attention to whether it is a homogenous liquid or a mixture of grains in a liquid. Several rheological models are available to describe the relationship between shear rate and shear stress in a fluid in movement (Eklund, 2000). The simplest rheological model is the Newton model where the fluid is described only with a linear relationship between shear rate and shear stress. The derivative of the curve is the viscosity of the fluid. A two-parameter model that is often used for cement grouts is the Bingham model where the viscosity is completed with a yield stress.

The yield stress is a shear strength that has to be exceeded before the fluid begins to flow (the ketchup effect). The Bingham model can be refined with a parameter describing a non-linear viscosity relationship, which gives us the Herschel-Bulkley model (Mork,, 1994).

The Herschel-Bulkley model also covers the two other models. The Newton model applies for very thin grouts with high water content and/or high dosage of superplasticisers where the yield value often can be neglected. For slightly thicker mixes the Bingham model can be used.

The Herschel-Bulkley model can describe grouts with high grain concentrations since the shear thinning behaviour begins to be more pronounced with higher grain concentration (Schwarz, 1997).

The parameter in the Bingham and the Herschel-Bulkley models that determines the potential penetration of a grout is probably the yield value. At a certain penetration length the yield value will balance the grouting pressure at which the flow will stop. The viscosity probably determines the rate of penetration. The validity of the yield value as a determinant of penetrability is probably limited to quite wide apertures without sharp obstructions in the flow path. Down to an aperture of about 0.5 mm one can probably regard the yield value as a valid penetrability determinant (Q.D Nguyen, 1986). For an aperture below 0,5 mm, the effect of the grouts filtration tendency has to be taken into consideration in order to predict the penetrability. Non Newtonian mixtures have been subjects for several different industrial applications. Many authors have also discussed the importance of the right grout rheology like Håkansson, (1993), Hässler, (1991) and Eriksson, (2002). Some special difficulties concerning rheology measurements of cementitious grout have to be made.

Håkansson (1993) stated some important parameters for good grout rheology:

- The rheology of a grout mixture is strongly influenced by chemically dependent factors during hydration of the cement.
- Cementitious grout has a thixotropic behaviour when measuring in short cycle times.
- The shape and concentration of the suspended grains is of importance.
- The fluid property of the medium that surrounds the grains is essential to prevent flocculation of grains.

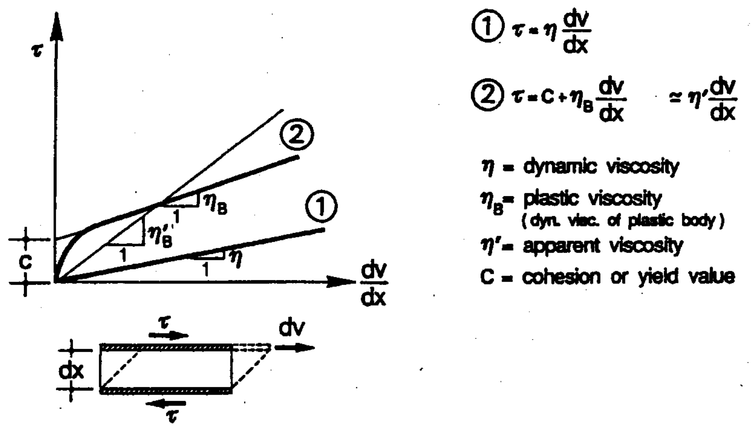


Figure 3.4, Rheological behaviour. 1) Newtonian fluid, 2) Bingham fluid. (De Paoli et al 1992).

3.3 Filtration tendency

When dealing with stable grouts there are commonly two approaches, the old- and the new one. The old one says that you should first grout with a thin and unstable mixture. The unstable grout will then be dewatered and create a stable plug in the crack. The new one recommends a stable mixture from the beginning.

The approach of dewatering the grout to create a sealing plug is not acceptable, because one cannot control where the plug is formed. If the sealing effect is dependent on a tight plug and the rest of the crack volume is badly filled, small movements in the structure can cause seepage through the plug. This old approach is in some sense a paradox because thin grouts are more difficult to dewater than stable ones (Hansson, 1995). The meaning of dewatering is in this case the same as plug formation (plug formation will occur more seldom if the W/C ratio is high compared to a mixture with low W/C ratio), this has also been shown by (Eriksson, 2002).

When grouting fine cracks or slots one has to take into account the fact that cement grouts contain solid grains. The cement grains can clog slots of roughly three to ten times the grains maximum size, even though the grains may be initially well dispersed by an effective mixer.

Many parameters influence the filtration tendency and since their influence is not yet fully investigated, the filtration tendency is, in this study measured on the freshly mixed grout.

3.3.1 Introduction

Within other industrial applications of filtration, two types of filtration phenomenon are mentioned, *cake filtration* and *deep bed filtration* (Svarovsky, 1985). The principle of cake filtration is that the solid material in the mixture is filtrated from the liquid by a relatively thin and coarse sieve cloth. The filtrated material will after a while create a thicker and thicker layer upon the sieve clot. Then the filter cake will, after some time of flow of mixture through the filter, create the filter itself, see Figure 3.5. Cake filtration can be compared with a grout mixture which is filtered (plug formation) within or at the entrances of a crack aperture.

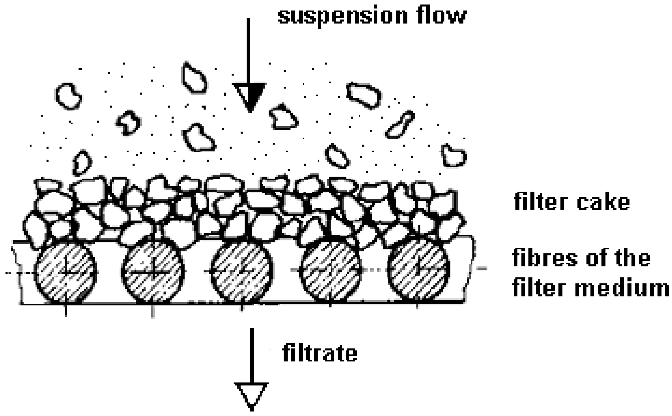


Figure 3.5, Cake filtration of cement based grout (Svarovsky, 1985).

Deep bed filtration is a gradual filtration process in a porous medium. Deep bed filtration can be seen as cake filtration in many tiny places, see Figure 3.6. Deep bed filtration can be compared to a grout mixture which is gradually filtered along a crack or in a porous material like soil. (Svarovsky, 1985).

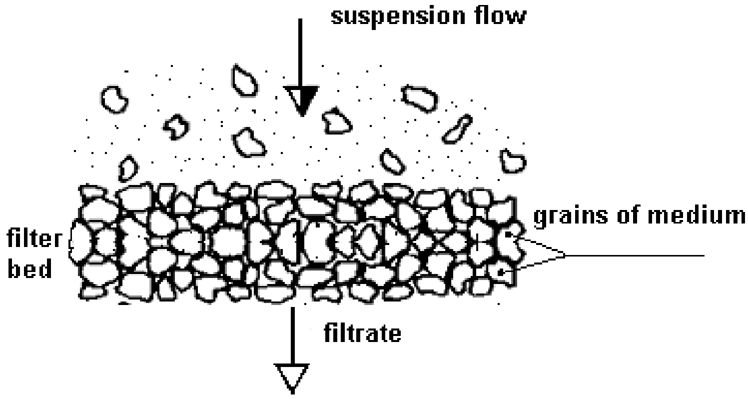


Figure 3.6, Deep Bed filtration of cement based grout (Svarovsky, 1985).

The behaviour for cake filtration and deep bed filtration has been the basic idea behind the rule of a thumb regarding soil grouting. The soil can be grouted if the quotient of the soil's grain size at 15 percent passing to the cement's grain size at 85 percent passing (D_{15}/d_{85}) is more than 20 to 25 (Mitchell, 1970). As mentioned earlier, this is not applicable in order to understand the mechanism of plug formation and what properties in the grout mixture govern the plug formation.

The scope of the experiences of penetrability of cement based grouts, is in this work focused on to laboratory work. In this work, the grouts that are of interest to investigate are those with low W/C ratio and stable in terms of bleeding and sedimentation. Different types of penetrability tests can be performed. Example of penetrability tests are tests like the filter pump (prEN 14497) and pressure chamber (VU:SC 48), sand column tests and injection in concrete crack specimen.

The filtration tendency for a grout is, as mentioned above, dependent on the mixture's overall properties and not for example on one single property like the maximum grain size or the specific surface of the grains. Rough rules can however be stated for the relation between the filtration tendency and some parameters of the grout. The author has made a preliminary evaluation based upon commercial test at Vattenfall Utveckling. The influence of different parameters can be preliminary presented according to Table 3.2.

Table 3.2, Parameters which influence the filtration tendency (Hansson, 1995).

Parameter	**Influence on filtration tendency (FT)**
Increased W/C ratio	Lower FT
Added superplasticizer	Lower FT
Added stabilisation agent	Higher FT
Added swelling agent	Higher FT
Added accelerator agent	Higher FT
Narrow grain size distribution	Lower FT
Effective mixer	Lower FT

Lower filtration tendency means that a larger amount of mixture will pass the filter in the test equipment. Higher filtration tendency will obviously signify a smaller passed amount of mixture. Of course, some exceptional cases do not fit in to this description. Generally according to performed experiments at Vattenfall Utveckling (Hansson, 1995) a fine-grained cement is better (better penetrability) than the coarser ones. Depending on, for example, the used mixing equipment, cement batch and surrounding air temperature, this relation can be the opposite.

3.3.2 Theory of plug formation

A theoretical approach can be used to look upon the phenomenon of plug formation. It is desirable to predict plug formation from the basis of the ingredients in the mixture like grain size distribution, shape of grains and concentration of grains. A theory has been developed to describe this behaviour by looking at cylindrical grains that create a plug (arch) between two obstacles, see Figure 3.7 (Martinet, 1998). To simplify the model the arch is assumed regular with the positive slope angle of γ. The friction between the grains ($P_1 \ldots P_N$) is negligible in the model, because a thin film of fluid is supposed to separate the grains. The cohesive forces between the grains are also considered to be zero.

The only force that acts between the grains is the force F, due to the external force T. The obstacle C_1 is cylindrical. The arch is assumed to be stable if the condition $\beta_1 \geq \gamma$ is fulfilled (stable if $\beta_1 - \beta_0 \geq 0$ is true, because $\beta_1 - \beta_0 = \gamma$), see Figure 3.7.

The length between the obstacles can be assumed L and the diameter of the grain is d_p. A simple equation can be set for the formation of a stable arch.

eq 3.11

$$\sum_{i=1}^{N-1} \sin(\beta_1 + \gamma(i-1)) = \frac{L}{d_p})$$

$$\sum_{i=1}^{N-1} \cos(\beta_1 + \gamma(i-1)) = 0$$

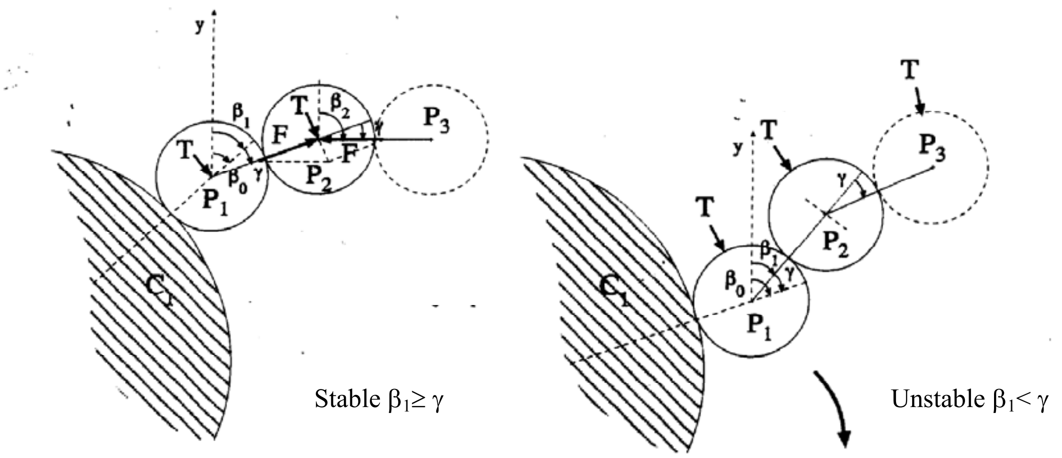


Figure 3.7, Regular arches in contact with cylindrical obstacle (Martinet, 1998).

A more flattened arch is associated to increasing forces F between the grains. Smaller forces F mean that the arch is more sensitive to movements of individual grains within the arch. Small movements of grains can then cause collapse of the arch (plug). The relation of the F/T can be written as:

eq 3.12

$$\frac{F}{T} = \left(2 * \sin\left(\frac{\gamma}{2}\right)\right)^{-1}$$

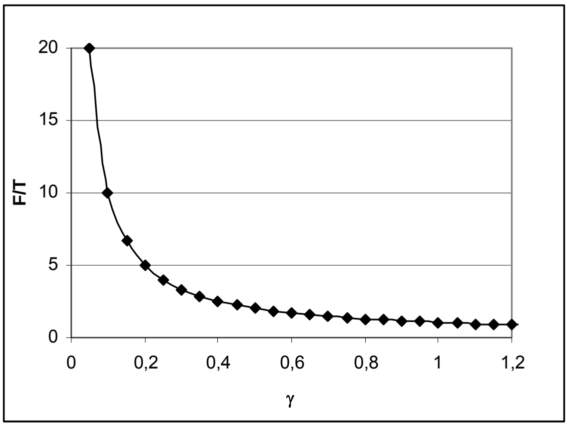


Figure 3.8, Relation between interparticular force F and external force T as a function of the friction angle γ (Martinet, 1998).

The relation F/T can be seen as an indication of the stability of the formed arch between the cylindrical obstacles (Figure 3.7). For small values of the angle γ the grains will be placed along a line, the ratio F/T will become infinite, see eq 3.12 and Figure 3.8

A similar way of describing arch formation based upon Martinet (1998) is to look at the relation between the number of grains (n) with diameter d and the distance L between two obstacles.

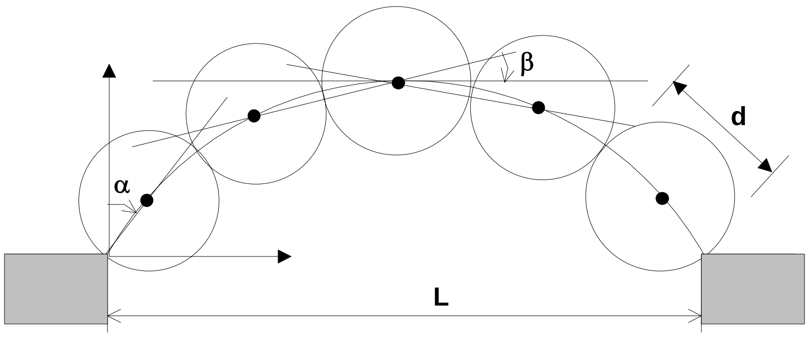


Figure 3.9, Regular arch of cylindrical grains between two obstacles.

The relation L/d can be written by the equations:

$$\frac{L}{d} = 1 + \sin\alpha + 2\sum_{i=2,4,6..}^{n} \sin(\alpha + (i-2)\beta)$$ **eq 3.13**

$$\frac{L}{d} = \sin\alpha + 2\sum_{i=3,5,7...}^{n} \sin(\alpha + (i-1)\beta)$$ **eq 3.14**

$$\alpha = \frac{\pi}{2} - n\beta \quad ; \quad 0 \prec \alpha \prec \frac{\pi}{2}$$ **eq 3.15**

Equation 1.13 is valid for an even numbers of grains and 1.14 is valid for an odd number. The solution of equation 1.13 and 1.14 describes the number of grains that is necessary for a stable arch.

The solution can be plotted as:

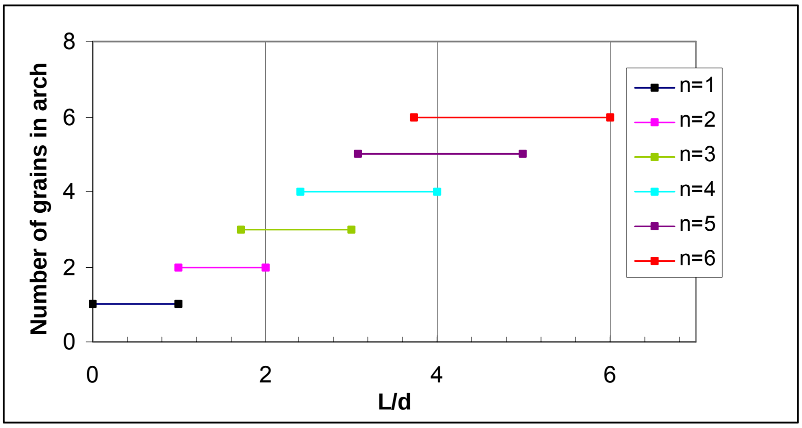


Figure 3.10, Number of grains in a stable arch between two obstacles spaced with the distance L.

The result from the theoretical approaches presented in Figure 3.8 to Figure 3.10 is of limited value for the understanding of the plug formation. The approach is to a wide extent a large simplification of a complex system including irregular grain shapes, electrostatic forces and chemical reactivity. A more detailed approach is needed to increase the knowledge of plug formation on a theoretical level.

3.3.3 Concentration and packing of grains

A granulate, such as cement is commonly described by a number of geometric parameters which are of importance for its properties. Packing of grains can often be regarded as a property that is of importance for the behaviour of the mixture as briefly discussed in 3.1. Packing of grains regards the surface distance between adjacent grain surfaces. Packing ratio is therefore of certain interest when looking at flow of mixtures in a thin slot. Different packing ratios are found for different grain size distribution and grain shapes. A number of packing theories can be found in literature (Cumberland, 1985). The packing ratio is also of importance for the mixtures susceptibility of flocculation and bleeding (Hansson, 1996). Commonly in grout mixtures, the grain concentration is used as a measure of the packing ratio in the mixture. A common way of describing the concentration for grout mixtures is the W/C ratio. The W/C ratio is correlated to the volumetric concentration according to:

$$c = \frac{1}{1+\frac{\rho_C \cdot W/C}{\rho_W}})$$ **eq 3.16**

där :

$c =$ Volumetric concentration

$\rho_C =$ Compact density

$\rho_W =$ Density, water

$W/C =$ Water Cement ratio

A typical value for grain concentration in cement based grout mixtures is W/C 0,7, which corresponds to a volumetric concentration of 33 %. This is a highly concentrated mixture. Most industrially used mixtures have as a maximum grain concentration of approximately 5 % (pulp slurry, mineral slurry etc) (Lohmander, 2000). The susceptibility of the mixture to flocculate is also dependent of the surface distances (free distance) between the grains. The surface distance is both influenced by the characteristic diameter of the grain and volumetric concentration of grains.

According to the FCC- model (Face Centred Cube) for packing of mono sized spheres, the surface distance between adjacent spheres is given by (Graton, 1935):

eq 3.17

$$a = d \cdot \left(\sqrt[3]{\frac{\pi}{3 \cdot \sqrt{2} \cdot c}} - 1 \right)$$

$a =$ Particle surface distance, μm

$d =$ Particle diameter, μm

$c =$ Volumetric concentration, [-]

This model gives the maximum volumetric concentration of 74 % (when a=0), which corresponds to a W/C ratio of 0,11.

A way of illustrating the difference in surface distances with changed grain diameter is to plot the surface distance versus grain diameter. The surface distance (a) is defined as the shortest distance between nearby surfaces of spherical grains. The distance (a) also assumes that all grains are in the same size (monodisperse mixture).

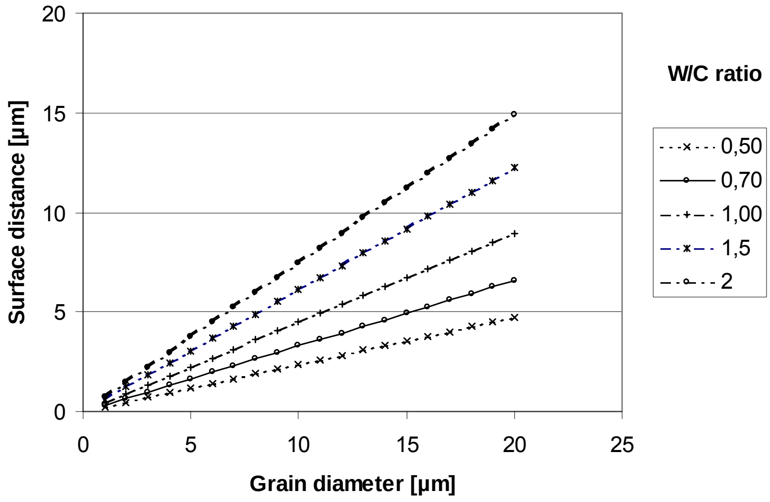


Figure 3.11, Correlation between surface distance and grain diameter.

To illustrate the correlation between packing of grains one can simplify the investigation to just look at packing of two-dimensional circles instead of three-dimensional grains. A perfect packing of circles occur if they are inscribed in hexagon head and the difference between the circle area and the hexagon head area is the porosity. Maximum packing ratio will then in this case be approximately 91 %.

The packing ratio of grains is affected by the space into which the grains are packed. If the grains for example are packed into a crack with an aperture of the same size as the grain diameter, the packing ratio will be much lower than if the aperture had been remarkably bigger than the grain diameter, see Figure 3.12.

Extra water often needs to be added in the crack aperture to maintain the flowability of the grains in the small aperture (due to decreased packing ratio). This phenomenon of decreased packing ratio of grains (increased porosity), is probably an important reason why grouts with a high W/C ratio have a better penetrability than a grout with lower W/C ratio.

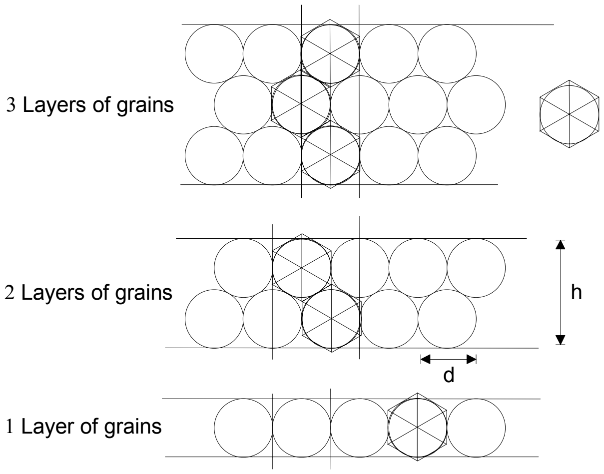


Figure 3.12, Packing of grains (circles) into different spaces.

The ability of the grains to reorganise in a densely packed system, as in the case with 2 or 3 layers of grains, is limited, see Figure 3.12. The limited ability to reorganise in these cases is mainly due to interlocking between the grains. Interlocking means that a single grain cannot move independently of the surrounding grains. Consequently, all the surrounding grains in the crack have to be reorganised when a single grain is moved. Plug flow is a common way of describing this flow of densely packed systems of grains (Håkansson, 1993). The plug flow is the basis for the Bingham behaviour of flow of grout.

Figure 3.12 illustrates the relationship between crack aperture/ grain diameter and the increase of the porosity of the packed grains, see Figure 3.13.

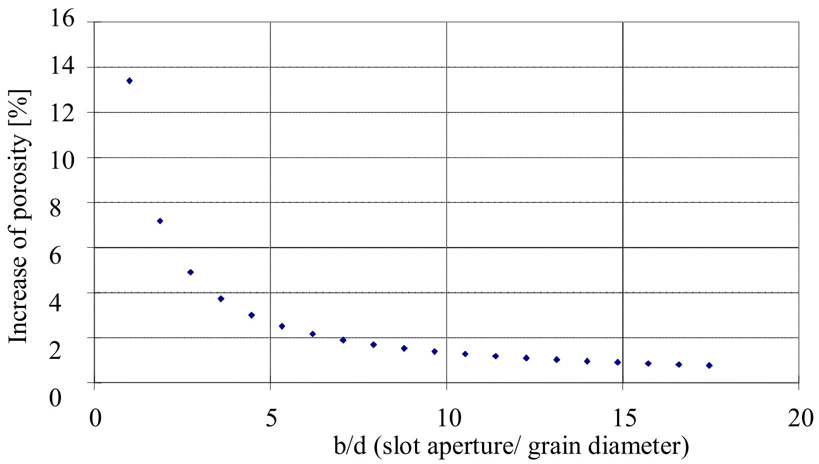


Figure 3.13, Packing of grains (circles) versus increase of porosity (increase from infinite slot aperture). The Porosity ϕ is 1-c (c is the volumetric concentration of grains in the mixture). The increase of porosity [%] is consequently calculated as one minus the porosity.

One should observe that Figure 3.13 is not a continuous function and that it is just a way of illustrating a phenomenon. This phenomenon is of importance when measuring the rheology of the grout in a plate-plate rheometer (Figure 3.15). Increased porosity makes that the water content in the mixture in some cases is not sufficient when the measuring gap gets close to the grain sizes. More water has to be added to compensate for this increased porosity in order to get representative measuring values. If more water is not added, the measuring plate will get into mechanical contact to the single grains and cause an increase of the friction between the measuring plate and the mixture. The increase of friction is probably due to an under pressure that is created in the measuring gap. The increased friction will cause the measuring values (yield and viscosity) to be out of the measuring range for the rheometer.

The phenomenon of decreased packing ratio may cause it to be difficult for the grains to maintain the flowability when they come to a constriction (same aperture as the grain size). More water is then needed to separate the grains and to avoid plug formation. This phenomenon is of importance if the original porosity in the mixture is low (high volumetric concentration of grains).

3.3.4 Crack aperture dependent rheology

When bleeding-stable grouts with low W/C-ratios are used, the packing ratio of grains is, in such a case, about as dense as for the dry cement. Flow of a stable grout has therefore probably to be looked upon as the transport and deformation of a body of continuously interacting grains. The grains have consequently small possibilities to rearrange within the paste, which are supposed to govern the filtration tendency. Some results of performed experiments support this theory (Hansson, 2000).

Measurement of the needed energy to deform an elastoplastic element through a constriction may be quantified with an oscillated measuring system in a rheometer. Some initial experiments have been made to investigate if it is possible to perform this measurement in an oscillatory rheometer (Eklund, 2000). Due to the very narrow elastic interval of the fresh grout mixture, it is difficult to measure the deformability of the mixture.

The penetration length (I) should then be characterised by the relationship between the used pressure gradient over the constriction (ΔP), yield value (τ_0) of the mixture and the aperture (b) of the slot, according to eq 3.18**.** The criteria for the grout mixture to pass the constriction (b) would then be when I>L.

eq 3.18

$$I = \frac{\Delta P \cdot b}{2 \cdot \tau_0}$$

The yield value (τ_0) should then be characterised by oscillatory measurement in a rheometer. The used rheological measurement system can, for example, be the plate-plate measuring system. A too high value of the τ_0 (within the elastic region) will mean that the element of mixture will not pass the constriction in the slot, see Figure 3.14.

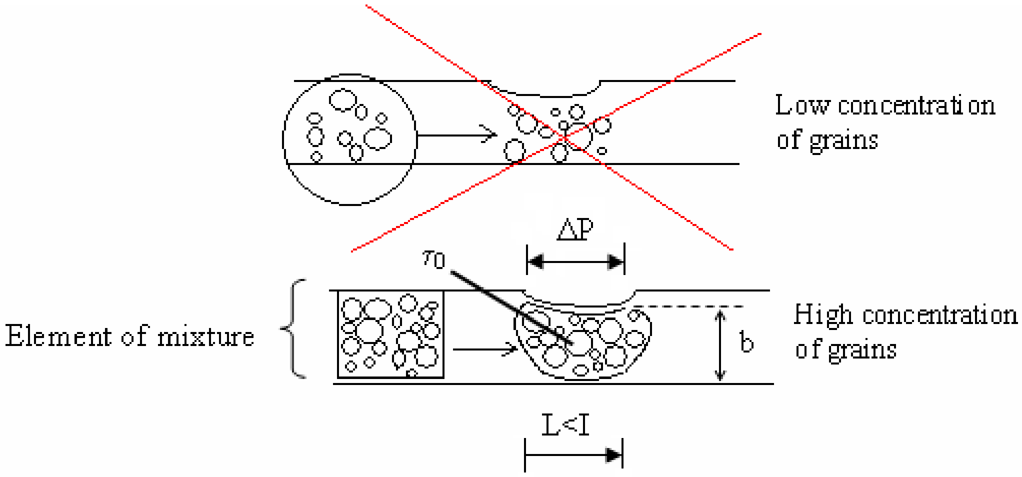


Figure 3.14, Deformation of stable (highly concentrated) mixture.

The models available today for the prediction of flow of mixture, like the Bingham- or Herschel- Bulkley model, do not take into account that the flow resistance increases when the crack aperture is close to the grains diameter. The Bingham model is valid

for flow of mixture in cracks with an aperture of approximately 5 to 10 times the maximum grain size (Mezger, 2000). An effort has been made (Hansson, 1997) to modify the Bingham model to even be valid for prediction of flow in cracks, with an aperture in the same region as the maximum grain diameter.

The Bingham model was written as:

eq 3.19

$$\tau = \tau_{0\infty} + \mu_{p\infty} \cdot \dot{\gamma}$$

$\tau = Yield\ value$

$\tau_{0\infty} = Yield\ value$ when "indefinite" slot width

$\mu_{p\infty}$ = Plastic viscousity when "indefinite" slot width

$\dot{\gamma} = Shear\ rate$

The modified Bingham model aim is to introduce size dependent factors into the model. The size dependency aim is to take into account that the yield value approaches infinity when the slot aperture approaches a critical value. The fact that the yield value approaches infinity when the measuring gap approaches the maximum grain diameter introduced the idea of an inverse function as the modified Bingham model.

The measuring device that was used to measure the yield value of the mixture was a rotating plate-plate rheometer, see Figure 3.15.

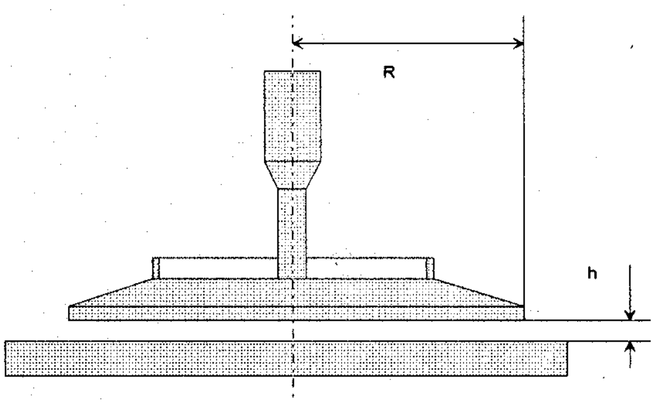


Figure 3.15, The plate-plate measuring system, h is the measuring gap (slot width) and R is the radius of the probe.

The suggestion of a modified Bingham model was written:

eq 3.20

$$\tau = \frac{k_{d\tau}}{h - h_{cr}} + \tau_{0\infty} + \left(1 + \frac{k_{d\mu}}{h - h_{cr}}\right) \cdot \mu_{p\infty} \cdot \dot{\gamma}$$

Där:

$h =$ Slot width [m]

$h_{cr} =$ Critical slot width (asyptote value) [m]

$k_{d\tau} =$ Factor for the yield values dependency of the slot width [Pa · m]

$k_{d\mu} =$ Factor the viscousitys dependency of the slot width [m]

Since the model contains parameters like $k_{d\tau}$ and $k_{d\mu}$ which has no physical anchorage, one should mainly see the model as a curve fitting function. Performed measurements at Vattenfall Utveckling show that the critical slot aperture h_{cr} widely varies for the same mixture. Measurement of τ is done by a plate-plate system (Figure 3.15), and evaluated by the Bingham model.

The measured value of τ is put into eq 3.20, to determine the value of $k_{d\tau}$, $k_{d\mu}$, h_{cr}, τ_0 and μ. The five parameters are determined by adjusting the measured value of τ to the calculated value of τ according to eq 3.20. The adjustment is done by the least square method. The critical slot aperture h_{cr} should respond to the minimum possible groutable crack aperture. Performed measurement indicates that the minimum possible groutable crack aperture is probably significantly larger than h_{cr}.

One can plot the result from the model in a three dimensional graph that shows the slot aperture where the yield value starts to approach infinity (h_{cr}), see Figure 3.16.

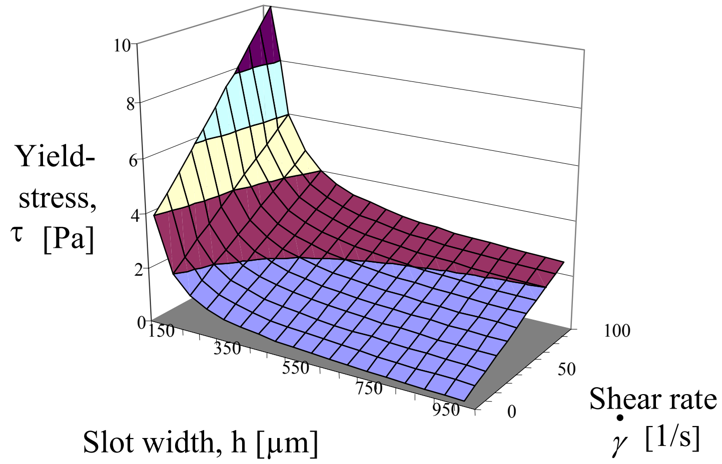


Figure 3.16, Presentation of a three dimensional plot from the model (Hansson 1997).

Performed correlations between filtration measurements (filter pump) and the calculated parameter h_{cr}, from the model, show no great correlation (Eklund, 2000b). The reasons for the bad correlation can be different.

Firstly, the measurement set up (plate-plate system) can cause problems with un-reproducible measurements of yield values. A problem that can occur during this kind of measurement is bleeding of the mixture. Bleeding in the measurement causes a thin layer of water to be created on top of the mixture's volume between the two plates. The water film causes a non representable and irreproducible value of τ to be measured.

Secondly, the parameter that governs plug formation was not reproducible in the rheology measurement. The model can maybe serve as an evaluation tool for characterisation of a mixture with good or bad filtration tendency. The work presented here has so far mainly been focused on finding key parameters that have the greatest influence on the formation of plugs.

The work with this model has in this thesis not been further developed because of the above mentioned reasons (practical problems with measuring the yield value and no significant correlation between measured value and result from other measurement methods like the filter pump).

3.3.5 Experiences of measuring with different devices for filtration tendency

There are some different measuring devices that are of importance for control of the penetrability of the freshly mixed grout. Some of the devices are made for measurements on site and others are designed for laboratory measurements. In this work only the measurement devices suited for laboratory are used.

Vattenfall Utveckling has developed test equipment (prEN 14497) suitable for measuring filtration tendency both in laboratory and on site. An outline of the equipment can be found in Figure 3.17.

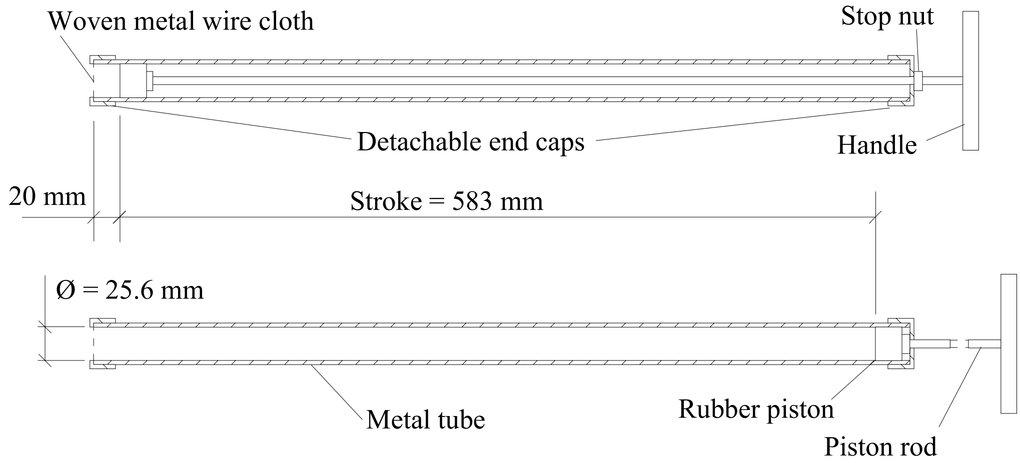


Figure 3.17, Measuring device (filter pump, prEN 14497) for filtration tendency.

The grout is drawn into the device by pulling the handle to full stroke for five seconds. The device is kept immersed in the grout for ten seconds. The grout is then pressed out into a measuring vessel. The amount of grout that was able to pass the sieve is measured and used as a relative measure of filtration tendency. This measurement gives a fair assessment of the grouts penetrability. This is a far more informative measurement than the Marsh-cone viscosity, which does not say anything about the penetrability.

It is of great importance that one sucks (pushes) up the grout in the pump with a representable pressure through the filter. The pressure over the filter in the pump can never exceed the atmospheric pressure (0,1 MPa). This is important of two reasons, first to avoid cavitation or release of air in the mixture, second to avoid pressing a plug through the filter. Although the absolute grouting pressure is much higher than 0,1 MPa, this pressure gradient is probably in the region at which the dewatering process starts. It is probably unlikely to believe that a plug formation would be pressed through a constriction in a real grouting case (Hansson, 1995).

Using this test equipment (filter pump), Vattenfall Utveckling has discovered that the commercially available products vary with in wide limits with respect to filtration tendency. Figure 3.18 shows six examples where each example represents one composition of grout subjected to identical mixing effort based on different batches of cement. The tested cement mixtures in Figure 3.18 are done with W/C ratios between 0,7-2, containing grouting cements (based on Portland clinker) and with the use of the same type of superplastisisers. Each mixture composition number contains the same composition of cement and superplastisiser. Six different combinations of mixtures were made (Hansson, 1995b).

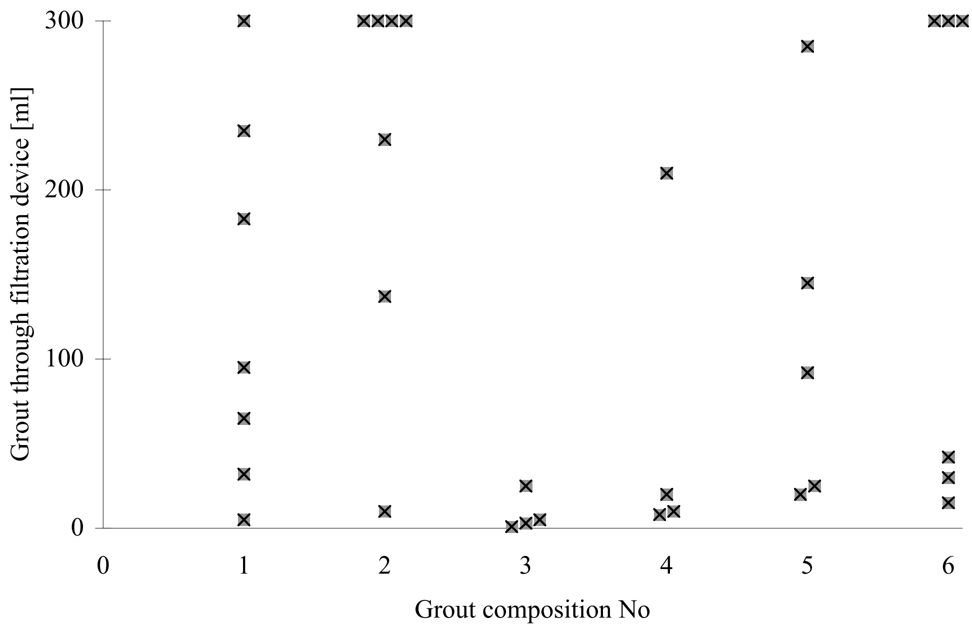


Figure 3.18. **Natural variations in filtration tendency, due to batchwise variations in cement and mixture properties (Hansson 1995b).**

Figure 3.18 illustrates the differences in the passed mount of mixture through the filter (net mesh) in the filter pump. If the tested mixture is close to the b_{crit} value, the passed amount of mixture can vary within wide limits (as for example in mix no 1,2, 5 and 6). Other mixtures like 3 and 4 show a significant lower spread then the rest of the 6 mixtures. Mixture 3 and 4 are probably not in the region of the mixtures b_{crit} value. The variations have been tried to be reduced by controlling, for example, used cement batches and the performance of the testing procedures.

The filter pump has been compared with other devices like the NES apparatus for measurements of filtration tendency (Brantberger & Nelsson, 1998). In Eriksson (1999) it was stated that no difference could be noticed between the filter pump and the NES apparatus. The main advantage of using the NES apparatus (compared to, for example the filter pump or sand column test) are the easy operation of filtration pressure and adjustment of the crack aperture. The NES apparatus is a reliable tool with consistent results (Brantberger & Nelson, 1998).

The sand column test is another method and is primarily a test method for grouts suitable for porous medium. Dalmalm, (2001) found that there was a difference in the passed amount of mixture between the sand column test and the other two methods (filter pump and NES apparatus). The sand column test is a method where sand with a specific grain distribution is packed into a plexiglas tub. The sand is injected and the front of the grout can be followed through the transparent tub.

The penetration property of the grout can be studied under similar conditions as in a real operation of grouting in a porous medium. The length that the grout front has penetrated through the sand column in the tub is measured. Both filtration tendency and rheology of the grout will affect the result of the test. Feder (1993) has also investigated penetrability with a sand column test apparatus.

Comparative tests have also been made by Hansson (1995) in order to compare results from the filtration measuring device with the sand column measuring device. The filtration measuring device was according to VU:SC 48, but with the use of a larger amount of mixture (4000 ml instead of 600 ml). The sand column test was performed according to the SS-EN-standard for cement based injection grouts.

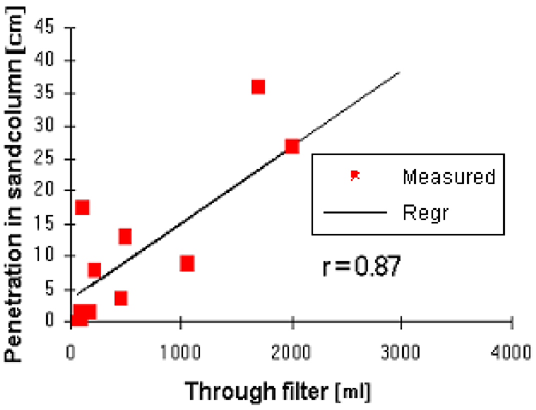


Figure 3.19. Relation between the sand column test and filtration test in a 125 µm filter and packed sand with grain sizes between 0,63-1,25 mm in the colon (Hansson 1995).

Although the performed tests in Figure 3.19 are based on relatively few measuring points it can be seen that some correlation between the two measuring devices exists. The factor of correlation is 0,87 (least square method).

A penetration test through an aperture with a specific length is another way of testing the penetrability of a grout. A slot with an approximate length of one meter is made of, for example, two plane steel plates, which are fixed together. The mixture's flow behaviour through the slot can then be studied. This test method is not used in this work, mainly because this work has focused on plug formation at the entrance of the slot. The second reason is that this type of experiment is practically hard to perform and is more time consuming than other penetrability tests like the prEN 14497 or VU:SC 48 (filter pump or pressure chamber).

Although, some initial testing has been performed by Hansson (1995), see Figure 3.20. The test is performed in order to compare the plate penetration device and the filtration device similar to VU-SC 48.

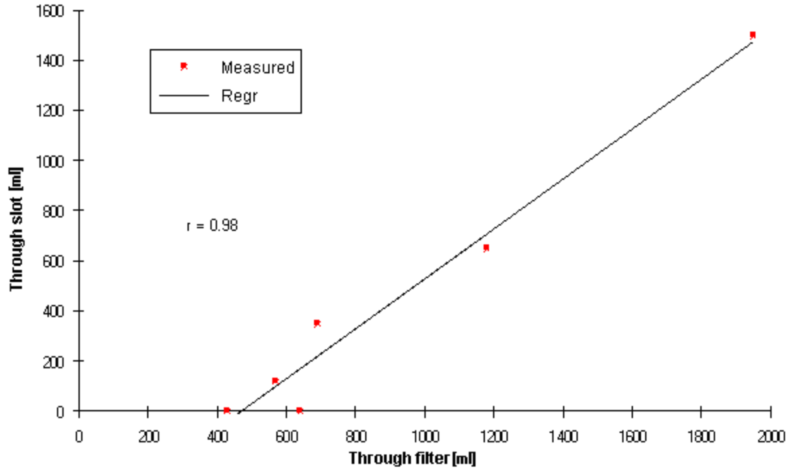


Figure 3.20. Relationship between the plate penetration test and filtration test in a 125 μm filter and a slot aperture of 125 μm (Hansson 1995).

The plate penetration tests is performed with two plates with a width of approximately 300 mm and a length of approximately1000 mm. The used volume of mixture is at least 2000 ml. The filtration tests is performed with a equipment according to VU:SC 48, but with a mixture volume of at least 2000 ml (Hansson 1995). Although the performed test in Figure 3.20 are based on relatively few measuring points it can be seen that some correlation between the two measuring devices exist. The factor of correlation is 0,98 (least square method). Generally a slightly larger amount passes through the filter compared to the slot geometry

The device that has been used in this and many other research work for measuring filtration tendency is a pressure chamber that contains a volume of grout mixture. The mixture is pressed through a filter with a specific aperture. The filter cake can then be saved and different filter apertures can be tested (Eriksson 2002). Eriksson (2002) presented an apparatus in order to determine the penetrability of a cement based grout. The method is to inject the grout through a filter of a specific aperture.

The filter is placed in a tube that makes it possible to collect the sample (filter cake) for analysis. The measured volume, filter cake length and density are the results of a measurement. Different filter apertures can be used in order to determine the penetrability of the grout. For a detailed description of the filtration equipment (VU:SC 48) used in this work, see 5.1.3.

Because no major differences could be identified and a relatively good correlation between the different measuring devices could be found (pressure chamber, plate penetration and sand column), the pressure chamber according to VU-SC 48 has been chosen to be used in this work.

The filter pump is mainly the same method as the pressure chamber when using the sieve cloth as the filter geometry. The NES- apparatus has not been tested in this work, but according to available literature (Brantberger & Nelsson, 1998) there is no major difference between this method and the ones tested in this work.

3.3.6 Experiences of penetrability experiments

Penetrability tests with cement based injection grouts are made in different works, e.g. by Hansson (1995), Eriksson (2003). Two phenomenon of filtration can occur when using cement based grouts, chemical and mechanical filtration. The chemical filtration causes the smaller grains to be filtered due to physiochemical properties. The physiochemical properties that are concluded to be of importance are the ionic strength of the pore water and the zeta potential around the grain surface. Lower ionic strength (resulting ionic strength of K^+, Ca^{2+}, SO^{4-} and OH^-) and high zeta potential decreases the influence of chemical filtration, Schwartz (1997). The mechanical filtration phenomenon is the blocking of grains due to the aperture being too small. Both of these two filtration criteria have to be fulfilled in order to achieve a good penetrability of the grout. This thesis will mainly deal with mechanical filtration (plug formation).

Performed tests by Fjällberg (2003) indicate that the flow properties of a grout with a W/C ratio of approximately 0,8 or higher is to recommend in order to penetrate cracks with apertures smaller than 100 µm. Low temperature and low W/C ratio in the grout, makes the grout stiffer and the demand of superplastisiser is increased in order to maintain good flow properties. The flow properties were measured in a mini slump cone and a March cone. The flow property is as earlier mentioned in this work, probably one important mixture property to achieve a good penetrability.

In the case of flow properties of more densely packed mixtures, this is the case with mixtures containing a low W/C ratio. Fjällberg (2003) found that the steric superplastisisers are probably is more effective than the electrostatic ones in order to avoid flocculation of grains in the mixture. The difference decreases when the W/C ratio gets larger. Minor flocculation of grains will influence the flow properties in a positive way (decrease of yield- and viscosity values).

The required flow properties can be achieved with both slow and more rapid hardening cement types. A longer setting time is to be preferred if the time for the hydration process is not crucial for the specific application. For example if the purpose of the grouting is to seal a structure against an ongoing inflow of water, it is of great importance to use a grout with a rapid setting time. The negative influence of a rapid setting time is an uncontrolled hardening process, which can obstruct the penetrability of the grout mixture. Cements with rapid setting time are also more sensitive to changes in temperatures than a grout with a slower setting time. Dosage of additives like accelerators and superplastisisers are even more sensitive when using grouts with a rapid setting time.

From the filtration experiments carried out by Eriksson (1999) it can be seen that a change of the mixture's density occurred when the flow of mixture passes a constriction. The increase in density is due to plug formation of grains at the entrance of the constriction. A decrease of mixture density could be seen after the constriction, the mixture has obviously been filtered.

Feder (1993) reported that a number of parameters are of great importance in order to avoid plug formation. These parameters are:

- W/C ratio of the fresh mixture.
- Joint width.
- The amount of different additives in the grout mixture.

Eriksson 2002 concluded that cement based grouts are not easily described and characterised because of their changing properties with time and because mixtures containing the same type of cement can have differences in penetrability. Work done by Eriksson 2002 uses the aperture b_{min} and b_{crit} to evaluate the filtration properties of the grout. b_{min} indicates the minimum aperture below which no grout can pass, and b_{crit} is the minimum aperture where no plug formation will occur, as earlier discussed in 1.3.

In Eriksson et al (1999) it is concluded that the mixing method is of great importance for the penetrability of the freshly mixed grout. The mixing time is also of important for the penetrability. Laboratory tests showed that the filtration tendency decreases differently depending on the composition of the grout. In Hansson (1996) and others works, it is found that the filtration tendency and bleeding properties of the grout can be in opposition to each other. In order to achieve stipulated requirements of a grouting operation, a priority between bleed and filtration tendency has to often be made.

Axelsson and Turesson (1996) also investigated the influence of mixing efficiency on the filtration tendency. Micro fine cements require a longer mixing time and more effective mixers then coarser ones in order to get the same penetrability of the mixture. A mixer of colloidal type is recommended to use and the rotor in the mixer (colloidal type) is found vital to the result of the mixtures penetrability. An even better penetrability was found when using an ultra-turrax or jet stream mixer compared to the colloidal type mixer.

Hjertström and Pettersson (2004) concluded that the rotational speed (rpm) used in the colloidal type of mixer is of importance for the penetrability of the grout mixture. The rotational speed should be increased to approximately 1750 rpm when using fine grained grouting cement in order to improve the dispersion of the mixture.

Penetrability experiments have been performed in a sand column apparatus by Arenzana (1989). The experiments concluded that micro fine cements were able to penetrate approximately 0,50 m into the column. The sand has d_{10} of 0,15 mm (10 % of the grains were smaller than 0,15mm). These results correspond to the results from penetrability tests at Vattenfall Utveckling. This test concluded a penetration length of the micro cement of 0,25-0,7 m, in sand with grain sizes between 0,125-0,25 mm. Both these experiments use grouts with W/C ratios of approximately 2,0. Reducing the W/C ratio will strongly reduce the penetration length of the grout mixture into the sand column. These test results are primarily of interest in the case of permeability injection in porous mediums as, for example, embankment dams or soil stabilisation. Penetrability of cement based grouts is anyway not a problem isolated to injection in hard rock or concrete structures.

Performed tests at Vattenfall Utveckling (Hansson, 1998) concluded that improved penetrability of a cement based grout can be achieved by using fairly instable grout mixtures and delay its setting time. The instable grouts that were used have W/C ratios of about 1,2-1,5 and with a bleed of between 5-10%. In order to delay the setting time it is recommended to overdose the amount of superplastisiser.

Comparison of filtration tendency has been made between standard Portland cement (d_{95}=120 μm) and grouting cement (d_{95}= 30 μm) (Bernstone, 2001). The filtration tests were performed with the filter pump (prEN 14497), and with a net mesh of 125 μm. The passed volume of the mixture based on grouting cement was 300 ml (maximal passed volume) and the passed volume of standard cement mixture was just 5 ml. Used W/C ratio was 0,8 and the mixture was mixed in a commercially available colloidal type of mixer. Samples were taken from the fresh mixture in the mixer.

3.4 Tightness and durability

A good penetrability is not the single governing factor for a successful grouting operation. The grout which is placed in the cracks or cavities also has to be resistant against leakage of water and other destructive processes. The first basic demand is water tightness and the second is durability. The two requirements are in agreement with to each other in the sense that if the grout has a bad water tightness, it will also mean a bad durability of the grout.

The tightness of the hardened grout depends on:

- W/C ratio
- The specific surface of the cement grains
- Degree of hydration

The W/C ratio and the specific surface area decide how close the grains can be to each other. Lower W/C ratio and lower specific surface area makes the grains get closer and a more durable grout is created, see Figure 3.21.

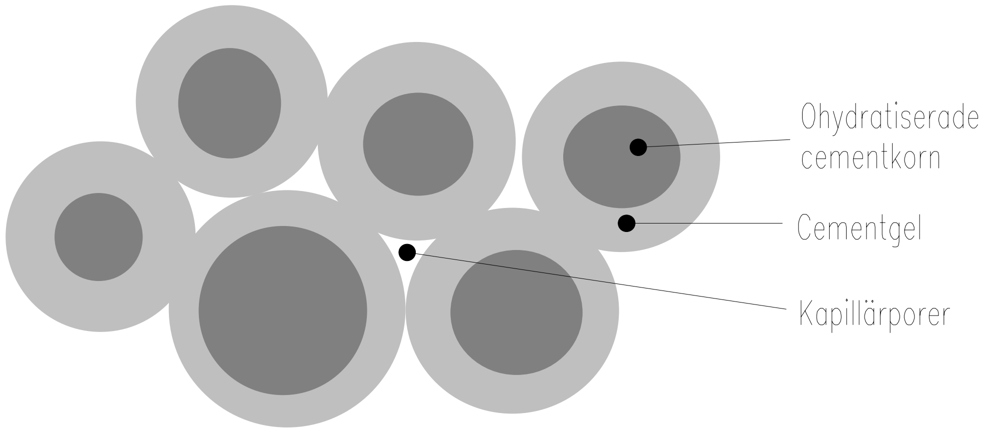


Figure 3.21, Outline of cement grains undergoing hydration (Hansson 1994).

Basically it is the capillary pores in Figure 3.21 that decides the tightness of the grout. Normally there are no connected capillary pores when the W/C ratio is below 0,7. If the W/C ratio is below ~0,39, the capillary pores are completely filled with cement gel. A higher degree of hydration of the cement means that the capillary pores are to a higher extent filled with cement gel and a more water tight hardened grout is achieved. A grout that has cured during one month is in general tighter then one that just has hardened for a week (Hansson 1994).

The grouts ability to seal a crack is not just a matter of the tightness of the grout itself, the grout has also to fill out the injected crack volume. In a case where the grout experiences a major bleeding, there is a chance that the water in the grout creates a layer on top of the mixture which then creates a possible path for leaching to occur through the hardened grout, see Figure 3.22.

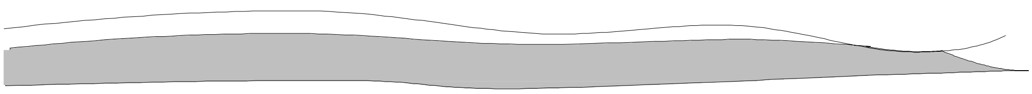


Figure 3.22, Incomplete filling of wide crack due to bleeding. The plane of the crack is perpendicular to the plane of the paper (Hansson 1994).

This mentioned reason indicates that it can be important to contain the bleeding of the grout mixture to a reasonably low level. This is a common acceptable theory which however has not been scientifically investigated.

Grouts with a reasonably low bleeding are often called stable grouts. Stable grouts are generally achieved by:

- Keeping to a low W/C ratio.
- Using an effective mixer.
- Using fine grained cement.
- Adding stabilizing agents.

The cases where the coarser grains or agglomerates of smaller grains fall to the bottom are called sedimentation. The sedimentation causes an increase in the mixture's W/C ratio near the bottom, with the consequence of a higher W/C ratio at the upper part. Sedimentation can be a sign of insufficient dispersion rate, wrong dosage of additive or maybe some contamination in the fresh grout mixture. Knowledge of the governing factors about sedimentation and bleeding is, today, a subject for further research.

The degradation processes of grouts are normally due to:

- Leaching of chalk
- Sulphate attack

Leaching is commonly due to the flow of water through the grout or beside it and the grouts most soluble component, the calcium hydroxide, is dissolved into the water. This type of leaching is recognized when the leached water comes in contact with the air, the calcium hydroxide reacts with the carbondioxid in the air and creates a precipitation of chalk on to the structure. These precipitations of chalk can have a positive side effect, the leaching paths self heal and further leaching can be stopped.

3.5 Conclusion and discussion

The penetrability of the freshly mixed grout can be divided into two parts, one concerning rheology (flow properties) and the other dealing with filtration tendency (plug formation). Both of these two parts have to be optimized in order to get a good penetrability of the grout. Understanding the mechanisms and the properties of the freshly mixed grout, which govern the plug formation, has to be developed. The mixtures filtration tendency means formation of an arch of grains at the entrance of the slot aperture or at a constriction along the slot.

According to performed tests of arch formation of grains (Martinet 1998) it is necessary to have a certain relationship between the internal force (F) between the grains and the external force (T) from the surrounding grains in order to create a stable arch.

Multiphase flow is maybe a way of dealing with plug formation of dense grain mixtures. Multiphase flow can be subdivided into a number of different applications depending on the nature of the flow. In order to investigate the penetrability of grout mixtures it may be suitable to study the two-phase flow between the liquid and the solid phase (water and cement grains). Two-phase flow can be analysed both numerical and by the help of physical experiments.

The development of numerical models is occurring rapidly and is in many cases driven by the improved computer capacity. Today’s two-phase flow models concerning dense grain mixtures probably have to be developed in order to achieve a better understanding of the mechanisms and the physics behind penetrability. At first most of the attention should be devoted to the different empirical models for the frictional viscosity between the grains. Most probably it is these forces that dominate the flow when the plug formation is initialised within a crack. Since the relationships describing the frictional mechanisms in a dense grain-fluid flow are based on semi-empirical relationships it is very important to get these relationships right (eq 3.10) (Saaidi 2004). In the literature several approaches on how to model the frictional stress can be found. Most of these approaches originate from geological research groups (Wachem 2000).

Today’s models can be used in order to obtain a qualitative measure between different flow configurations regarding for example grain concentration and slot aperture (filter geometry). Existing numerical models for this kind of dense mixture flows are continuum models, which means that the grain phase and the fluid phase are considered as two interpenetrating continua. The numerical models can serve as a good complement to physical experiments in order to understand the mechanisms and the grout properties, which govern the penetrability.

Different rheological models are available to predict the flow behaviour of the mixture. The most commonly used model is the Bingham model. The Bingham model describes the mixtures yield and viscosity value.

The yield value is, according to many authors (Eriksson 2002, Hässler 1991 etc) the governing rheological parameter for the penetrability of the fresh mixed grout. The viscosity mainly governs the flow rate of mixture during the grouting process (pumping) (Håkansson 1993, Schwarz 1997).

This approach, i.e. that the viscosity is of limited value for the penetration length (I), assumes that an infinite time is available for the pumping of grout, which of course is not the case. The practical limited available time means that the viscosity is of importance for the penetration length. This is also true because of the time dependent properties of the grout. The viscosity and yield value of the mixture will increase with time after mixing during its flow in the grouted medium. Increased viscosity and yield value will after a while probably prevent further penetration.

The available of today's models for the prediction of flow of mixture, like the Bingham- or Herschel- Bulkley model, do not take into account that the flow resistance increases when the crack aperture is close to the grains diameter. An effort has been made (Hansson, 1997) to modify the Bingham model to even be valid for prediction of flow in cracks, with an aperture in the same region of size as the maximum grain diameter. The aim for the model including size dependency is to take into account the fact that the yield value approaches infinity when the slot aperture approaches a critical value. The results of these tests points out that it seems to be difficult (with the used equipment Eklund, 2000b) to measure the increased yield value in thin slot apertures.

The packing of grains is of great importance when the slot aperture approaches the maximum grain size. The packing ratio determines the grains ability to redistribute and the resulting W/C ratio in the given geometry. Low initial packing ratio (in the fresh mixture) means that the grains can redistribute (better avoiding plug formation) but the injected geometry will be filled with a mixture with poor durability.

Different measuring devices have been tested in order to identify if there is any significant difference in terms of measuring the grouts penetrability. Sand column, filter pump, pressure chamber and plate penetration are the methods compared in this work. No major difference has been found. A literature review indicates that even the NES apparatus did not show any significant difference in the measuring results from the tested devices.

There is a significant variation in the freshly mixed grout's filtration tendency. According to performed filtration tests (Hansson, 1995), with different grout compositions the filtration tendency can (measured by the filter pump) vary within wide limits. This is probably due to batchwise variations in cement and mixture properties and also to the stochastic process of filtration.

A good penetrability is not the single governing factor for a successful grouting operation, the grout which is placed in the cracks or cavities also has to be resistant against leakage of water and other destructive processes. The first basic demand is water tightness and the second is durability. The two requirements are in agreement with each other in the sense that if the grout has a bad water tightness, it will also mean a bad durability of the grout.

Eriksson 2002 concluded that cement based grouts are not easily described and characterised, because of the properties change with time and because mixtures containing the same type of cement can have a difference in penetrability. Eriksson 2002 uses the aperture b_{min} and b_{crit} to evaluate the filtration properties of the grout. b_{min} indicates the minimum aperture below which no grout can pass, and b_{crit} is the minimum aperture where no plug formation will occur.

In this laboratory and others works (Hansson 1996), it is found that the filtration tendency and bleeding properties of the grout are often in opposition to each other. In order to achieve stipulated requirements of a grouting operation a priority between bleed and filtration tendency have to often be made.

Axelsson and Turesson (1996) investigated the influence of mixing efficiency onto the filtration tendency. Micro fine cements require a longer mixing time and more effective mixers then coarser ones in order to get the same penetrability of the mixture. Hjertström and Pettersson (2004) concluded that the rotational speed (rpm) used in the colloidal type of mixer should be increased in order to achieve a better penetrability of fine grained grout mixture.

4 Cement based grout

4.1 General

There is still after more than 100 years of use, some uncertainties about the mechanism of hydration of Portland cement. The main reason for this is that it takes place at a solid –liquid interface and produces solids of variable composition which are either amorphous or, if crystalline, adopt more than one morphology. This makes the reaction difficult to study experimentally. Today's more sophisticated measuring equipment have made it possible to better understand the reactions (Widman 1993).

Cement is composed of a number of minerals, which when in contact with water start to react (hydratisation) and create a hard solid mass. Hardening of grout has nothing to do with drying, as for example when clay is drying and turns into a more solid mass. As an example it can be mentioned that grout hardens even under water. Cement contains grains of different size and shape, which for example is because of the used milling technology. As mentioned, cement contains grains not just of a single size, but a distribution of sizes. The grain curve in a grouting cement can be described by a maximum grain size and a distribution of grain sizes smaller then the maximum size. The grain curve is produced by air sieving of the cement. The principle of an air sieve is that smaller grains are carried a longer distance by the air stream than larger ones.

Cementitious injection grouts are a mixture of cement, water and additives in a predetermined proportion, in its simplest form just containing cement and water. The cement is not soluble in water, it remains in its solid phase. The cement grains are kept vagued in the mixture by natural motion in the water phase of the mixture. The receipt of a grout is predetermined in a balance between water-cement ratio and dosage of different additives to obtain certain properties.

In the past a grouting operation was often performed with ordinary cements intended for concrete manufacturing. The ordinary cement is not that suitable for grouting of fine cracks and pores when the cement grains are relatively large compared to the crack apertures. For a couple of years it has been common with cements intended for grouting, mainly due to the fine grounding of the grout cement. To describe fineness of the cement two measures are used, maximal grain size and specific surface area. The term maximum grain size is a bit misleading, when in most cases it is meant the sieve width where 95 % of the amount of material passes. Specific area is the total surface area of a certain weight of grains.

Binders for concrete manufacturing can be divided into hydraulic, latent hydraulic and pozzolanic. Hydraulic binders are materials with the consistency of a powder, which when mixed with water, will create a hardening product (Portland cement and aluminate cement). Latent hydraulic binders are materials that are hydraulic when mixed with water and in addition of an activator (Betonghandboken 1994).

Examples of a latent hydraulic binder is blast furnace slag that in combination with Portland cement will be activated by the $Ca(OH)_2$ and alkali and create a hardened product. Pozzolanic binders react with $Ca(OH)_2$ in the pore water and create a hard CSH-gel (calcium silicate hydrate) Examples of this binder are a large amount of natural pozzolanes and synthetic ones like silica fume. For grouting purpose, the hydraulic binders are like Portland cement and aluminate cement, the most common ones (Wieker, 1989). Some commercially available cement of today also contain fine-grained blast furnace slag, fly ash and silica fume mixed with Portland cement.

Apart from the fineness of the cement grains there can also be a difference in the chemical composition of the cement. Cement for grouting purpose can mainly be divided into two groups, Portland cement and slag cement. Portland cement mainly consists of limestone and clay, which are burned together to make the cement clinker. The clinker is then milled together with different additives, for example gypsum and material to facilitate the milling process in order to produce the final cement.

Slag cement usually contains Portland cement, but 45-80 % of the content is fine grained and specially treated slag from iron manufacturing. The slag cement has both advantages and disadvantages compared to Portland cement. The slag cement is, for example, more resistant to leaching because of the lower or no content of free calcium hydroxide. The slag cement gives very little or no contribution to the self-healing process of concrete structures and to protect the reinforcement or bolts from corrosion.

Variations in chemical composition within these two groups (Portland and slag cement) of cement are due to the variation of the raw material. The raw material varies depending on the geographical place where it is taken. The chemical composition is often adjusted to different requirements of the cement, for example sulphur resistance and low alkaline cement. Apart from these two cements, Portland and slag cement, aluminate cement can also be used for grouting. It's very rapid hardening process characterizes this cement. The disadvantage with the cement is that it is hard to control and predict the hardening process, which can create great difficulties during the mixing process (Betonghandboken 1994).

4.2 Cement chemistry and early cement reactions

This study just includes use of Portland cement. Portland cement contains Portland clinker, gypsum and other inorganic substances, as for example limestone filler material. Important properties for the penetrability of the fresh grout mixture are the amount and type (the solubility) of added gypsum and the liquid’s content of ions. Cement chemistry and early cement reactions, which affects for example the penetrability and hardening of the grouting cement, are principally the same as in regular construction cement (Portland cement). The major difference between grouting and regular cement is that the grinding of the grouting cement is finer (Fjällberg, 2003). For the composition of a Portland clinker, see Table 4.1.

Table 4.1, **Composition of the clinker mineral in a Portland cement.**

Clinker mineral	C_3S	C_2S	C_3A	C_4AF
Share [%]	53	23	1,4	14

Generally most literature about cement chemistry, mainly focuses on concrete manufacturing, the different clinker mineral can be designated different tasks during its hydration process. The presented clinker properties are even valid in terms of grout manufacturing (Taylor 1990) (Betonghandboken 1994).

C_3S, Alit, The most important component to build-up the strength of the grout during the first 28 hours.

C_2S, Belit, The long-term strength is decided by the relationships between Alit and Belit.

C_3A, Aluminat, This component reacts very fast if gypsum is not added as a retarder. False setting can be an undesirable consequence of a mixture containing too much C_3A. An amount of more than 7% (C_3A) in grouting cement will normally create false setting in the fresh mixture.

C_4AF, Ferrit, This component generally reacts very fast in the fresh grout mixture.

Other key components for the behaviour of the fresh and hardened grout is as previous mentioned (Taylor 1990) (Betonghandboken 1994):

Calcium sulphate (gypsum), Gypsum is added in the manufacturing process as more hard to dissolve dihydrate ($CaSO_4 \cdot 2H_2O$) or the easier soluble anhydrite ($CaSO_4$). Milling of the dihydrate makes that some of it converts to easy soluble half hydrate ($CaSO_4 \cdot 0{,}5H_2O$). The gypsum has as its main task to retard the C_3A reaction.

Alkalisulphate, Sodium and potassium are called alkali. The alkali is bound to the clinkers sulphate as alkali sulphate. Alkali sulphate is an easily soluble sulphate, which affects the early rheology of the grout mixture. The alkali amount is expressed as the equivalent amount of $(Na_2O)_{eq}=0{,}66K_2O+Na_2O$. Alkali in the cement can also cause problems with alkali-silica reaction (creation of swelling products) but this will hardly be a problem in case of grouting mixtures.

CaO, free chalk, The chalk which has not been bound during the manufacturing, are left as free CaO. The amount of free chalk after the manufacturing process should not be higher than 1 % of the cement weight. If the free chalk reacts with water after the grout has hardened, cracks can be detected due to the fact that the free chalk creates swelling products in the grout.

4.2.1 Hydration of cement

When the cement gets in contact with the water, mainly two reactions occur (Fjällberg 1998);

1. When dissolving easy soluble compounds such as alkali sulphates, the water gets saturated of K^+, Na^+, Ca^{2+}, SO_4^{2-} and OH^- -ions (the pore water solution). The pH in the water is quickly increased up to about 13. Crystals of $Ca(OH)_2$ and ettringite are produced.

2. Different types of reactions will continue in parallel. The surface of the grains will be covered with reaction products. The tightness and composition of the surface layer will decide how fast the water can penetrate into unreacted cement and then continue to produce reaction products. The volume between the cement grains decreases and fills up with reaction products (cement gel).

Basically three types of chemical reactions can be studied during hydration of cement(Taylor H.F.W 1990) (Betonghandboken 1994):

1. **Aluminate reaction**, Reactions between C_3A and water immediately create CAH (calcium aluminate hydrates), this reaction makes the grout to harden immediately, which is why this reaction has to be stopped. Gypsum is added in order to stop this reaction. A short time after the addition of water, the grains will be covered by ettringite, the reaction speed will then decrease. The C_3A is consequently of importance for the setting time of the grout mixture.

   When the concentration of sulphates decreases, the production of ettringite will become a production of monosulphate. Grouting cement with a low amount of C_3A will react more slowly during a lower generation of heat than cement containing a larger amount. Low amounts of C_3A in the cement will therefore contribute to a durable hardened paste (sulphate resistance for example). (Fjällberg, 2003).

2. **Silicate reaction**, Reactions that involve C_3S and C_2S. The reactions means that a compact cementgel is created. The cement gel consists of CSH (calcium silicate hydrate) and chrystalls of $Ca(OH)_2$. The silicate reaction governs the final strength of the grout mixture.

3. **Ferrit reaction**s, The ferrite reacts with the gypsum and creates neadle shaped crystals.

4.2.2 Development of structural strength and filtration tendency

Dissolving of components and chemical reactions start immediately after the cement has been exposed to water. The free lime and sulphate dissolve and surface skins of hydrated minerals form on the other constituents. Reactions then proceed very slowly for the next 2-5 hours (the setting period, when the mixture change from a viscous to a solid material) before accelerating when the surface skins break. Hydration products grow away from the surfaces into the spaces between grains and finally result in the development of strength in the cement paste, see Figure 4.1.

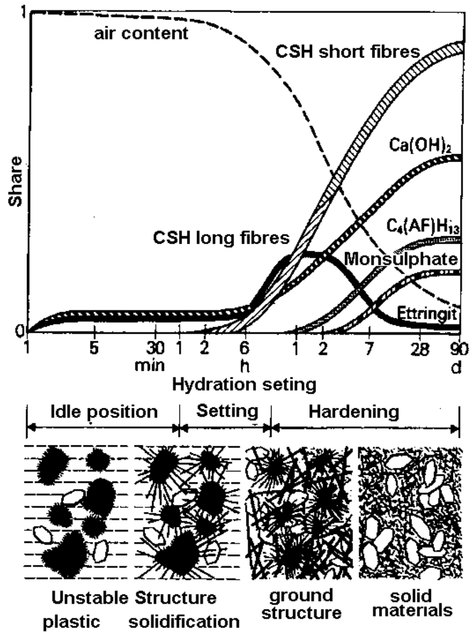


Figure 4.1. Cement reactions as a function of time (Betonghandboken 1994).

Development of the strength of the grout can be divided into two phases:

Gel phase: The cement grains are weakly bounded to each other, some shear strength about a few kPa can be achieved. With in a period of approximately 10 hours after mixing depending on temperature and composition, the grout is thixotrop. The thixotropic nature means that the grout can retain its fluidity even if it is disturbed by, e.g. some kind of vibration.

Hardened phase: This is the period when the real hardening process starts. The grout has no longer a thixotropic behaviour. The grout is no longer affected by for example blast-induced vibrations that can destroy or delay the hardening process (hydration). The hydration process is going on during the whole lifetime of the grout.

The growth of the strength during the gel phase is strongly dependent on the additives that are added in the fresh mixture.

Superplastisisers extend the bonding time and decrease the gel strength but do not decrease the final strength of the hardened grout.

Some accelerators strongly increase the rate of gel strength during the gel phase, but do not affect the bonding time to the same extent. Some accelerators will decrease the final strength of the grout. Final strength is not a good term for the expression of the strength of a grout, though the hardening process will, as mentioned earlier, continue almost for ever as long as the solid grouted mass exists.

Approximately one hour after mixing the chemical reaction that mainly governs the filtration tendency has occurred. The reaction between the tricalciumaluminate and gypsum is among the first reaction that starts. This reaction creates ettringite and slows the rapid hydration of the aluminate phase. The ettringite covers the cement grains and creates what usually is called false setting (Bradley, 1986). The governing factor for the overall fresh properties (rheology and filtration tendency) of cement based grout is probably the gypsum reaction. Gypsum is added to the cement powder in the manufacturing process. When the sulphate is consumed, hydration can accelerate and a secondary transformation of ettringite to monosulphate occurs. Monosulphate is a needle like crystal, which contributes to an increase of viscosity and to the setting of the paste. Sulphate quantity must therefore be carefully controlled to leave little excess aluminate (C_3A) to hydrate directly. Too low sulphate content makes precipitation of mono sulphate crystals in the mixture and a too high content makes too much ettringite. None of these cases are good for the fresh properties like rheology and filtration tendency of the grout, see Figure 4.2.

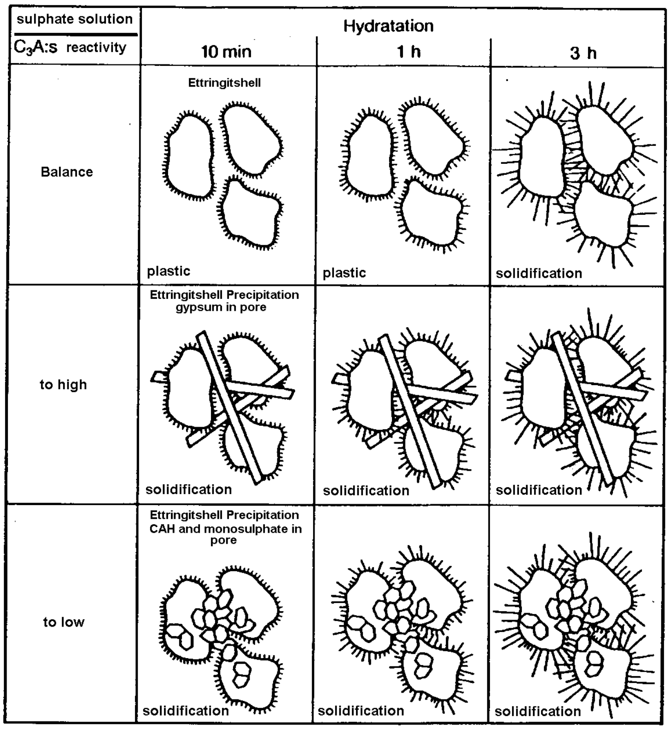


Figure 4.2, Schematic presentation of the gypsum reactions as a function of the amount of C_3A, Tricalciumaluminte (Betonghandboken 1994).

Even other reactions occur simultaneously and the common denominator is that the produced crystals all have a large specific surface area. The large specific surface area increases their tendency to flocculate. The flocculation of crystals makes a gel between the cement grains, which deteriorates the rheology of the grout. The fineness also affects the reaction speed of the grout.

Cements with smaller grains reacts faster than coarser ones, due to the fact that more calcium silica hydroxide and ettringite are created (the total surface area to cover is larger when the grains are small). The final setting of the paste is caused principally by the growth and interlocking of the calcium silica hydroxide (C-S-H) gel.

Commercially grouting cement without addition of gypsum and low sulphate content is available today. This type of cement has the advantage of a hardening time that can be controlled by the addition of retarders like citric acid and lignosulphates. The first setting is then false and the final setting of the grout which creates the final strength, starts later.

The composition of typical Swedish cement for grouting purpose consists of a number of clinker minerals, see Table 4.2. Dry cement powder consists of a number of ingredients. The terminology of cement ingredients can mainly be divided into three groups:

Cement= Milled clinker and gypsum
Clinker= Silicate
Silicate= Alite and Belite

Table 4.2, Approximate amount of mineral content of the used Portland cement in the tests. (for example Injekteringscement 30) (Betonghandboken 1994)

Alite	Belite	Ferrit	Aluminate	Gypsum
[%]	[%]	[%]	[%]	[%]
53	23	14	1	5

One of the governing factors for a given type of cement when dealing with early cement reactions is the W/C ratio in the mixture. The setting time (change from a viscous to a solid material) is largely governed by the distance between the grains in the mixture. The chemical reactivity in the mixture is often measured as a rising of the temperature. A rising temperature is an indication that the silicate (mainly alite) reactions have started in the mixture. A high W/C ratio is associated with a longer time before this reaction starts (setting time is delayed).

4.3 Pore water

It is usually mentioned that water is suitable for mixing with cement to grout, should be drinkable. The most important factor is that it should not contain any organic substances. Organic substances can destroy the hardening process of the grout. Sulphates in the mixture water can also contribute to a retarding effect of the grout. A bad smell from the water can often be a sign of that it contains sulphates or organic substances.

The properties of the pore water are of importance for the mixture's rheology. The interaction of forces between the grains in the mixture is dependent on the electrical properties of the pore water. The ion content in pore water will change with time during the hydration process of the grout. The chemical composition of the grout will also strongly affect the ion content in the pore water. The composition of the pore water, at a specific time, with a specific grout, is therefore difficult to influence, but there are some possibilities. If one discovers that a specific type of ion contributes with bad properties, it is possible to neutralize the ion by a complex binder. There are some ideas that a lower ionic strength is associated to a better penetrability of the grout. (Yang et al 1997). However, a low ionic strength is probably also associated with a lower reactivity of the cement. The pH value can be measured in order to follow the development of the ionic content in the pore water. Normally the pH value of the pore water is about 12,5, 10 minutes after mixing water and cement. The pH value will then increase up to about 13.5 after about one hour after mixing, due to precipitation of OH^- ions.

The ion strength in the water has a small effect on the LvdW forces, while the electrostatic forces are strongly affected. The release of alkali sulphates in the grout mixture during the first twenty-four hours makes the pore water saturated with ions as K^+, Ca^{2+}, SO^{4-} and OH^- (Wieker 1989). Alkali consists of Natrium (Na) and Potassium (K). The amount of alkali affects the amount of water that can be kept in the mixture. High amounts of alkali mean that a larger amount of water can be kept in the mixture. Cements with a high amount of alkali requires a large amount of mixing water.

4.4 Additives

A number of different additives can be used to change the properties of the grout. Dosage and combination of additives always have to be tested in order to optimise the performance of the grout mixture.

4.4.1 Superplastisisers

The most widely used additive is a superplastisiser. The purpose of using additives in cement-based grouts is mainly to improve the rheological properties (viscosity, yield value) of the grout in order to enhance the penetrability and flow characteristics of the grouting material.

The addition of superplastisisers makes it possible to reduce the required amount of water with a maintained rheology (flowability). The addition of superplastisisers makes it possible to use fine-grained cements (micro cements) without using an increased amount of water in the mixture (constant W/C ratio). There are numerous commercially available types of superplastisisers on the market today. Basically are all these superplastisisers are based on different types of polymers with different origin, length and subdivisions at which an active substance is mounted. The active substance is mainly based on a melamine, naphthalene, lignosulfonate or a group carboxylic group (copolymers) (Ramachandran et al 1998).

A cementitious grout in it simplest form consists as mentioned earlier of suspended grains in water. In the mixture the grains are attract each other by London-van der Whaal forces (LvdW), which are attracting and electrostatic forces that can be both attractive or repulsive. The chemistry of the water, the ion strength, has a small influence on the LvdW-forces but the electrostatic forces are highly affected (Bradley, Howarth, 1986). The LvdW forces are always present and cannot be eliminated or manipulated. The repulsive forces can be manipulated and that is what happens when a superplastisiser is added to the grout mixture. The cement grain consists of several minerals with different surface charges, even different polarity, which makes the grout mixture a highly flocculated system. The active substance in the superplastisiser is adhered onto the surface of the cement grains and affects its surface property. Melamine, naphthalene and lignosulfonate affect the cement grains surface charge and create a repellent force between the grains (electrostatic stabilisation). The active substance in the superplastisisers based on copolymers, is also adhered to the grain surface, but the grains are separated by steric stabilisation. Steric stabilisation is based upon that long polymer chains are adhered to the grain surface, the grains will then repel each other when the polymer chains come into contact with each other, see Figure 4.3.

The flocculation is probably not completely broken when the superplastisiser is added to the mixture (Vovk, 1989). The effect of the superplastisisers is influenced by the cement properties like the grain sizes, grain distribution and amount of C_3A, alkali and sulphate. According to the different effects in different types of cements, it is of great importance to optimize the dosage of a specific superplastisisers to a specific grout mixture. The most common types of superplastisisers on the market are based on melamine, naphthalene or different co-polymers.

Table 4.3, **Some common superplastisisers.**

Short name	More complete name	Example of product
Melamin	Sulfonerat melaminformaldehydharts Sulfonsyremodifierat melaminformaldehydharts Natriumpolymelaminsulfonat	Peramin (F, FP, FS, FXP), Cementa (V33, 92M), Rheobuild 475-S, Rescon HP- SF, Melcrete
Naftalen	Sulfonerat naftalenformaldehydharts Natriumpolynaftalensulfonat Kalciumpolynaftalensulfonat	Mighty 150, Rescon HP L 15
Co-polymer		Cemflux, Glenium 51

As can be seen from Table 4.3, the composition of the superplastisisers can vary within different categories. A clear difference can be seen within the group of naphthalene, there are two clearly different formulas, and considerably different effect can be identified. The formula with sodium is the most common. Generally the product seems to be neutralised to Na- or Ca salts (Gu Ping 1994). Besides differences of the formulas of the active molecule there are differences in additives and modifications of the superplastisiser. Some are modified to maintain a longer (retarder) or shorter (accelerator) time until setting, i.e. the time that passes from addition of superplastisiser until the superplastisiser has lost its effect. An example of a retarder is phosphate and an accelerator is potassium or calcium. It should be mentioned that all superplastisisers have a more or less retarding effect upon the setting time of the fresh grout mixture. This is because they all are surface-active substances (adhere to the cement grains surface).

The chemicals in superplastisisers are not unique for the construction industry, similar products has been used in other industrial mixture to improve the storage durability of for example coal powder, grinding agents etc. The chemical engineers calling it to stabilise the mixture, which means to prevent flocculation by modifying the surface chemistry of the grains in the mixture. The traditional method of stabilisation of mixture is the electrostatic stabilisation. Another type of superplastisiser that relatively recent has made its appearance at the market is the steric superplastisisers. They are based on copolymers (Shaw 1980). The differences between the superplastisisers are described by Figure 4.3

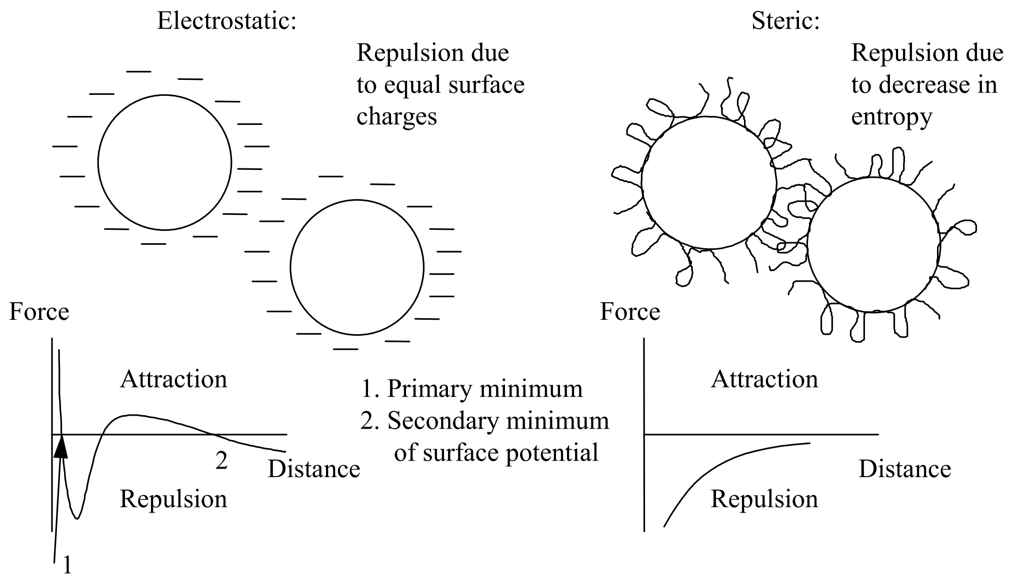


Figure 4.3, Principals for electrostatic and steric stabilisation of mixtures.

The force that trys to bring the grain together in a mixture and cause flocculation and agglomeration is the earlier mentioned London-van der Waahls force, however, the different surface charge of the grain's surfaces can contribute to a deteriorated dispersion. It is this attraction forces that the stabilisers are supposed to neutralize. Measurements of the repulsive forces between the grains in the mixture have not been performed in this work.

4.4.2 DLVO- theory

The different modes of action give different distance-force relationships according to the DLVO-theory (Bradley 1986 and Shaw 1980). The electrostatic stabilisers suffer from two equilibriums along the curve, the primary and the secondary minimum of energy. The secondary minimum causes loose flocculation that is noticed by a thixotropic behaviour of the mixture. The primary minimum gives a rather stable bond between grains, which can form hard cakes on the bottom of a container. The grains have to be submitted to certain forces exceeding the electrostatic repulsion forces to come within the minima. For the secondary minimum, it can be sufficient with the natural Brownian movement of the grains, or the mixture is so highly concentrated that the grains are already in reach of each other. In order to be trapped in the primary minimum the grains have to be affected by greater forces, e.g. gravity or mechanical forces by filtration (Shaw 1980 and Bradley et al 1986). The steric stabilisers should not exhibit corresponding equilibrium along the force-distance curve. The intergrain force should be repelling regardless of the distance. This indicates that a grout with a steric stabiliser, i.e. a copolymer SP, should allow a more severe filtration stress without the grains flocculating.

In case regarding electrostatic stabilisation of the grout (melamine etc.) the polymer part of the molecule is absorbed at the grain surface, because the polymer part is hydrophobic and therefore searches towards the grain surface. The anion sulphonate group creates the negative surface charge.

The left part of the force-distance curve is created by deriving the sum of the surface potentials by the LvdW forces and the electrostatic forces, according to the DLVO theory (Shaw 1980). The electrostatic stabilisers increase the magnitude of the surface potentials and give it the same sign for all surfaces in the mixture. At the same time the shear plane is moved a longer distance from the grain surface, which makes that the ξ-potential (zeta) measurement sometimes give an unrepresentative result for the mixture's other properties. The only point where it is possible to measure the potential is just in the shear plane. The fact that you do not know how the additives are absorbed on to the grain's surface makes the effect of the superplastisisers more difficult to explain. This is because you cannot be sure of the position of the shear plane relative the grain surface. The right curve in Figure 4.3 is made in the same way as the left curve, but the repulsive force/ potential is represented by a steric stabilisation. To achieve a sufficient repulsion it is necessary that the polymer molecule is heavy enough.

The steric stabilisators work by using the fact that they are composed of different homopolymers (Hansson 1996). In its simplest case with two polymers, one that is hydrophobic and the other is hydrophilic. This means that the hydrophilic parts is absorbed on to the grains surface while the hydrophobic parts are extended in the water and create a hairy texture at the grains surfaces.

When the hairy surfaces of the grains get close to each other, the hairy texture starts to interact and shear its space, the entropy decreases and a repulsive force is created. The repulsive force can also be created by a compression of the hairy texture.

The most common type of design of the copolymers and their manner of absorption is that they are linked together in each others ends and the "fur" consists of loops and tails.

There is also another alternative, the copolymers are composed of a hydrophobic backbone polymer which is extended into the liquid as tails, see figure 3.2 (Bradley, Howarth 1986).

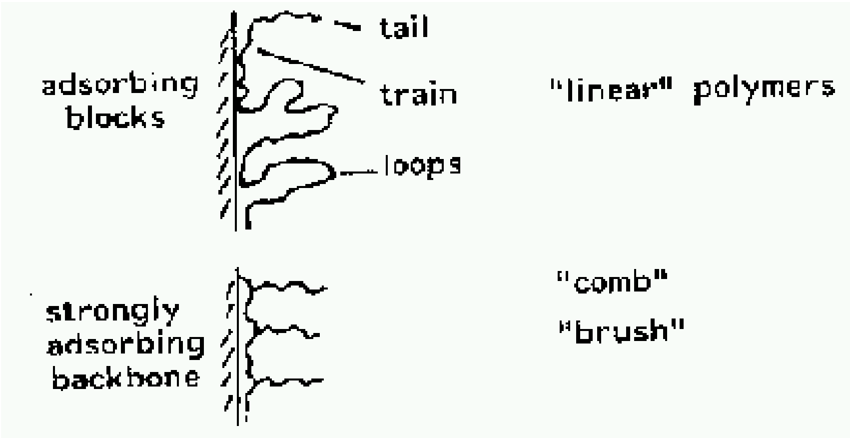


Figure 4.4, Two different types of absorption to a grain surface (Shaw 1980).

The length of the copolymers in the steric superplastisisers is supposed to have influence on the dispersion effect of the fresh grout mixture. A longer polymer chain is found to have a greater dispersion effect than a shorter one (Kinoshita 1999 and Yamada 2000).

Electrostatic stabilisation does not create a continuous repulsion, there exists two minimum of potential, where equilibrium of forces are created along the axis of distance, see Figure 4.3. The primary minimum gives rise to a strong bonding between the grains, they coagulate or agglomerate.

This phenomenon has been experienced if one has ever mixed a grout with a high dosage of superplastisiser and a moderate to high w/c ratio (0,5-1) and felt the consistency of the material next to the bottom of the bowl. The material has often created a hard cake on to the bottom. The cakes consist of coarser cement grains that are pressed together by gravity so the distance between the grains are within the primary minimum. The secondary minimum gives rise to a weaker bounding between the grains or agglomerates.

In a study (Bradley, Howarth, 1986) the difference between melamine and copolymer superplastisisers have been investigated in grout mixtures. The study showed no significant difference between the two categories, which had been expected due to the different force-distance relationships illustrated in Figure 4.3. The grouts were evaluated by letting them pass through a sieve with the mesh width of 100 µm, their ability to create a plug on to the filter was investigated. Analysis of the copolymers chemical content revealed that they contained anionic groups, combined with a low molecule weight, the main mode of action can be electrostatic.

Other types of additives like retarders and air entraining agents which also can improve the properties of the grout, also contain an ionic, cat ion or surface-active groups. These types of additives should also be able to act like superplastisisers.

4.4.3 Accelerators

Accelerators are used in order to make the hardening (hydration) process faster. Accelerators can generally be divided into two groups, depending on their effect. One group is the ones that affect the setting time and the other group affects the final strength. Practically most of the common used superplastisisers affect both setting time and final strength, which is why it is hard to make a distinct classification. The accelerators are commonly based on inorganic salts, some examples of accelerators are:

- Calcium chloride
- Sodium hydroxide
- Potassium carbonate
- Sodium aluminates

A common type of material that is often mentioned as an accelerator is alkali silicate, this material is actually not a true accelerator. The alkali silicate reacts with substances in the cement and creates a gel that makes the grout stiffer, the hydration of the cement grains is not affected. The dosage of an accelerator in the grout has to be done carefully, wrong dosage can result in retardation or that the grout gets a too rapid hydration. Accelerators can in some cases affect the durability of the grout. Use of chlorides in accelerators can cause problems with reinforcement corrosion.

In grout mixtures it can be desirable to delay the setting time. There are some works (Justnes and Nygaard 1995) where calcium nitrate has been used as an accelerator in grout mixtures (delay setting time). They concluded that the dosage of the accelerator is crucial to its effect on a specific cement type. Dosage and combination of accelerator/ cement type is generally of great importance in order to not deteriorate the properties of the fresh grout mixture.

4.4.4 Retarders

Retarders have an opposite influence on the grout properties compared to accelerators. Use of retarders can in grouting purpose aim to extend the time before the beginning of the hydration process. Dosage of retarders also has to be done with precision. Examples of retarders are:

- Sugar
- Phosphates
- Citric acid
- Lignosulphonates

4.4.5 Swelling agents

Swelling agents are used in grouts to prevent the decrease of volume that always happens when the cement grains reacts with water (hydration). The purpose of the swelling agent is to fill the cracks completely and create a durable result of the grouting operation. This type of agent is usually quite unusual in common grouting operations.

4.4.6 Stabilisation of grouts

Stabilisation agents are used to prevent bleeding of the grout. Bentonite, which is a sort of clay (montmoriolite), is a common type of stabilisation agent. Bentonite decreases the strength and penetrability of the grout. The material is often delivered in a dry powder form. The amount of Bentonite is often related to the used amount of water, normally one part of Bentonite is mixed with fifteen parts of water.

4.5 Additive material

Additive materials are added on purpose to fill up the cement, lower the generation of heat or to make the hardened cement paste more massive or solid. The additive material that are described here are also called pozzolanes, which are a group of materials that can create a solid paste in reaction with water, without the addition of Portland cement or chalk to make it hydrate. A common factor for these materials is that they all consume calcium hydroxide during their hydration, the risk of leaching is then consequently decreased. Many of the additive materials contain the same oxides like those in Portland cement. Common types of additives are silica fume, slag and fly ash. The grout's ability to self-healing actually decreases when the amount of additive material increases. The corrosion protection also decreases when using a larger amount of additive material in the grout mixture. Additive materials are used in some special applications of grouting cements. Some tests indicate that the penetrability can be increased in fine grained grouting cements when using, for example, fine grained blast furnace slag in the cement (Hiroshi Uchikawa, 1994).

4.5.1 Silica

Silica, or dust of silica is a bi-product from the manufacturing of alloy material for steel. Silica is an amorphous type of silicon dioxide (SiO_2) and is very fine grained, the maximum grain size is approximately 0.1-1 μm and a specific surface of ~20 000 m^2/kg. The size of the grains can be compared to the grain sizes in cigar smoke. Silica is often added in dosages of 2-8 % in order to increase the solidness or decrease the bleeding of the grout. The use of silica in grouting purpose is relatively unusual. In the alkaline environment in the grout the silica will rapidly create a gel, which increases the viscosity and yield value in the mixture. Silica is usually delivered as slurry or as a dry powder.

Another type of silica is the colloidal silica. This type of silica has a grain size of less than 0,1 µm and with a low viscosity of its mixture. Colloidal silica can be used in grouting of fine cracks and with the addition of e.g. NaCl or $CaCl_2$, the mixture starts to gel (Axelsson, 2002).

4.5.2 Slag

Slag has been described in previous chapters as a type of cement, slag can also be added separately. Usually about 45% of the cement is replaced with slag, but in rare cases up to 80 % can be replaced. The slag that is commonly used is specially treated blast furnace slag from steel manufacturing.

4.5.3 Fly ash

Fly ash is for example produced when the smoke from the chimneys at a coal fired power plan is purred. The quality of the fly ash is commonly very varied depending on the quality of the used fuel (coal) in the power plant. Fly ash consists of spherical and amorphous grains in the size of between 1-150 µm. This material is as earlier mentioned, a pozzolanic material that consumes $Ca(OH)_2$ and creates CSH gel like that in cement. Fly ash is not a common additive material for grout mixtures.

4.6 Conclusions

- The chemical reactions of interest for the filtration tendency occur during the first one-hour after mixing. The gypsum reactions are one of the dominant early cement reactions that influence the filtration tendency of the fresh mixed grout. Other early cement reactions of interest are the aluminates and silicate reactions, which affect the setting time of the mixture. W/C ratio is one important parameter on the setting time. Additives like retarders or accelerators can, of course also, control the setting time.

- Surface chemistry property is of great importance to prevent flocculation of grains in the mixture. Flocculation of grains is important to avoid in order to get a mixture with a low filtration tendency. A common way to avoid flocculation is to use superplastisiser. Superplastisisers are the most widely used additive in grout mixtures. There are mainly two types of superplastisisers, the electrostatic and the steric ones. Experiences from (Fjällberg 2003) show the steric ones are probably the most effective in order to get a grout mixture with good penetrability (low yield and viscosity value).

- The ion content of the mixing water is of importance for the mixture's rheology and probably even the filtration tendency. The ion strength in the water will strongly affect the electrostatic forces. The ion content in pore water will change with time during the hydration process of the grout. The composition of the pore water at a specific time with a specific grout is therefore difficult to influence. There are some ideas that a lower ionic strength is associated with a better penetrability of the grout. (Yang, et al, 1997). Ions in the mixture (pore water) can be neutralized by the addition of some kind of complex binder, depending on the ion type.

5 Performed filtration experiments

5.1 General

In this chapter, the mechanisms, which govern the penetrability properties, are studied. Relevant parameters that describe the function of the grout will be identified and methods of measuring them will be developed. In order to achieve this it is impoetant to describe and characterise the different components in the grout, like binder agent, additives and added material. The interactions between these are essential to outline.

The problem of filtration tendency is, however, very complex. The research work has therefore followed a trial and error philosophy in the sense that new and complementary tests have been designed and carried out based on the results from previous tests. The overall objective has been to investigate the influence on the filtration tendency from different parameters and also to some extent the complex interaction between them. This more deductive approach has implied that the search for understanding has been encouraged to the sacrifice of a more rigorous testing program covering all possible combination of parameters.

Tests were first carried out with inert material to be able to look upon filtration tendency without any influence from the cement chemistry. Tests on cement based grout were then carried out, both on standard portland and on specially prepared cement material.

5.1.1 Characterisation of grain curves

The grain size distribution of a dry cement powder is of great importance for the penetrability of the grout mixture. There are different ways to indirectly or directly describe the distribution of grain sizes. A common way of describing the grain content is to present the dry powders d_{95} value, which corresponds to the sieve aperture where 95 % of the material passes. This value does not say that much of the distribution of grains. Another common way to characterize the grain content in the dry powder is to present the blaine fineness. This value characterizes the amount of surface area available for reaction, rather than the specific size of a specific grain. None of these two values are anyhow that illustrative for the overall grain size distribution.

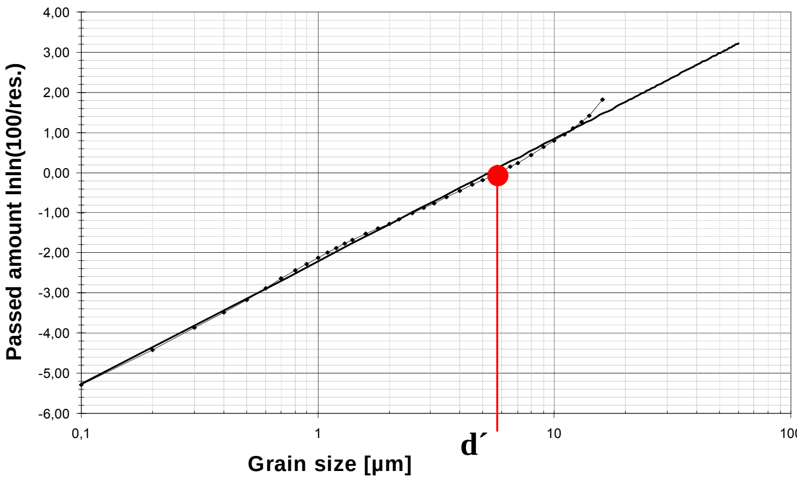
In order to achieve a better characterisation of the grain curve an alternative method RRSB has been tested. This way of describing the distribution of grains differs from the traditional way in terms of plotting the grain sizes against the amount of it. The RRSB distribution (Taylor, 1990) is more influenced by the amount of small sized grains. d´ is evaluated as the grain size which corresponds to the intersection between the straight line (adjusted line) and the cumulative residue value of zero.

The output parameters from the distribution are d´ and n, see figure 3.6. d´ is a grain size in µm which corresponds to an amount of grains, normally about 50-60% of the total amount of grain sizes in grouting cements. n is a measure that describes the spread of the grain sizes and is presented as a slope of the line of best fit, between the min. and max. grain size.

The RRSB- distribution is a commonly used method to describe the grain content in powders with a wide range of grain sizes and special consideration has to be taken to the small grain sizes.

A passed amount of 50-60% corresponds to d_{50}-d_{60} if the grain size distribution is considered as being normally distributed, with the standard deviation of σ and mean value µ. The plots in figures 3.6 and 3.7 illustrate the difference between RRSB-distribution and the ordinary way of plotting grain size distribution.

The reason for using this type of distribution model is to make the output parameters (d´ and n) more dependent on the small grain sizes rather than e.g. the parameter d_{95}. The parameter d´ and n will be dependent on an equation of the type $y=k\cdot x+m$, compared to d_{95} which is solely determined by a single point in the grain size



distribution.

Figure 5.1, RRSB-distribution plot of a grain curve.

The cumulative residue as (lnln 100/res.) is plotted against log grain size.

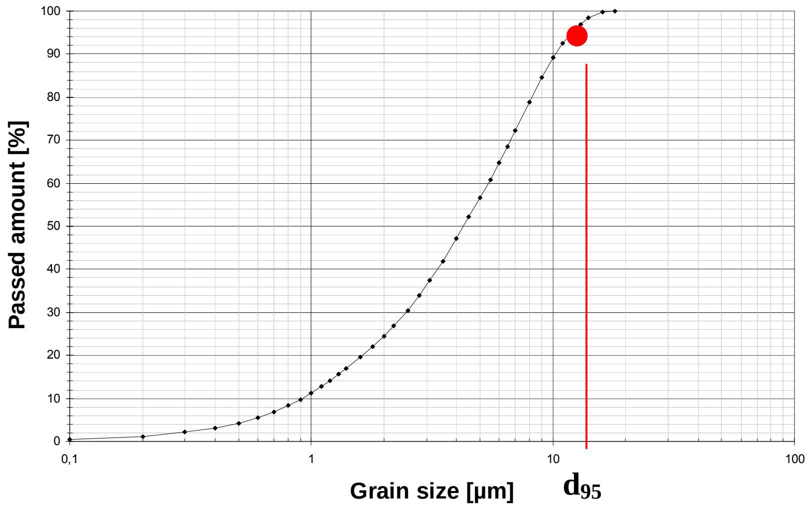


Figure 5.2, Ordinary-distribution plot of a grain curve.

5.1.2 Scanning electron microscopy (SEM)

Analysis of material has been made in a scanning electron microscopy (SEM) in order to investigate the shape and agglomeration of grains in the dry powder. The used magnification is between 1000 and 4000. Some investigations have been made to visually analyse filter cakes with SEM-technology. The idea was to see if any certain type of grain initialises the plug and if some significant strata of grains could be seen. Small pieces of the filter cake were cut out and put into the vacuum chamber in the microscopy. Different adjustments of the microscopy were tested, in order to get a detailed picture of the grain sizes and the distribution of the grain sizes.

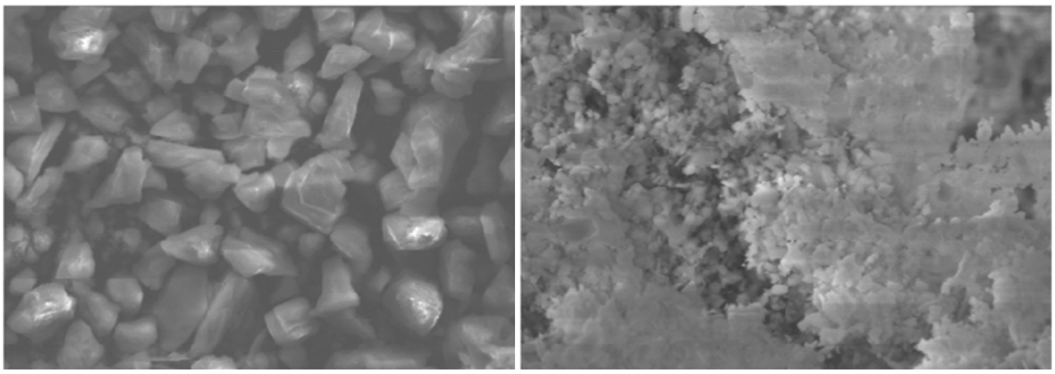


Figure 5.3, Pictures taken in 1000x magnification. To the left, coarse grains of dry dolomite powder and to the right fine grains.

5.1.3 Measuring device for filtration tendency

Filtration tendency is quantified in this work by a measuring device that presses the mixture through a net or slot with a specific mesh aperture or aperture. The idea of using this type of testing equipment was suggested by Pär Hansson, Vattenfall Utveckling, in the beginning of the 90´th. The equipment is further developed by Eriksson, 2002a. The reason why the filter pump was not used is that the pump needs a relative large amount of mixture for each test. The available amount of the sieved material (dolomite powder) is limited, due to it being very expensive and time consuming to produce these fraction intervals in the air separator. In order to compare the filtration results between the dolomite powder and the cement-based material, the same test equipment has been used in both test cases. The equipment used in this work is according to the standard VU:SC 48, see Figure 5.4.

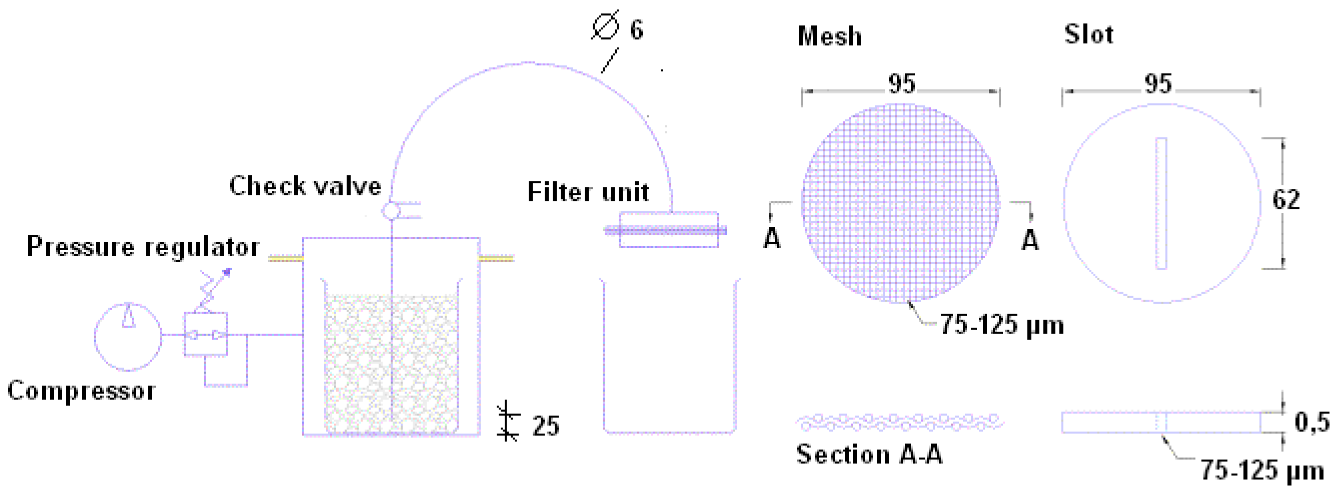


Figure 5.4, Test equipment according to VU:SC 48.

A pressure gradient is applied over the filter. The amount of mixture that passes the filter is a measure of filtration tendency. When a large amount passes the filter, then the mixture is said to have low filtration tendency. A small amount indicates that a filter cake builds up at the filter, which after a while makes it impossible for the mixture to pass through the filter. To evaluate different mixture's filtration tendency a measuring procedure has been set up.

The results from the filtration experiments with this test equipment may depend on the amount (volume) of mixture that passes the filter.

The available original amount of mixture is therefore of importance. Performed filtration tests with different available amount of mixture will therefore probably generate different passed amount through the filter (different quantification of filtration tendency).

Two types of filter geometry have been tested. The first geometry, a mesh of thin woven steel wires makes a filter with quadratic mesh design. The second geometry consists of, a thin steel plate in which a thin slot has been cut by a laser beam, see Figure 5.4.

The filter area in the mesh geometry has in some experiments been reduced by a 5*65 mm opening in a steel plate, which was placed upon the mesh. The filter area is reduced in order to see the influence of the filtration area relative to the passed volume.

Filter of a single slot is associated with the case where the grain can just build an arch from two sides of the slot. The initialisation of the plug is of great interest for predicting the moment when no more mixture can pass the filter.

The filter area is of importance for the investigation of how many grains pass per filter area. The filter area is defined as the area of the sieve cloth that does not consist of steel wires (mesh geometry). In the case of a slot geometry the filter area is defined as the area of the slot (aperture with a specified length), see Figure 5.4. From a statistical point of view a certain amount (number) of grains probably has to pass the mesh or slot in order to create a plug. Many passed grains per area unit increase the probability that some of the grains get stuck and a plug is initialised. The principle of how a plug can be initialised may be different dependent on the design of the filter geometry. A filter with a mesh design means that the grain can build an arch (plug) from four sides around a mesh opening in the filter, compared to slot geometry where an arch can be created from just two sides.

To reduce the necessary amount of mixture the filtration experiments has been focused on the slot geometry.

5.1.4 Rheological measurement system

Viscosity and yield value has been measured in a plate-plate rheometer (Paar Physica UDS 200). The measuring loop consists of shear rates from 0 to 50 s^{-1} and back to 0 again. The duration of the loop is approximately 5 minutes. The used measuring gap is 100 μm. Rheology measurements are performed in order to investigate if there are some correlations between the passed amount of mixture in the filter device (filtration tendency) and viscosity and yield value. Rheology measurements also serve as a check of the mixing quality of each mixture. Varying mixing quality means also varying viscosity and yield value. It is certainly important to have the mixing procedure as equal as possible between different mixtures.

5.1.5 Bleeding measurement

Bleeding tests of mixtures have been performed in order to investigate the influence of grain size distribution and concentration of grains. The bleed tests have been performed according to the principles of SS-EN 445, but the amount of available inert mixture volume is just 110 cm^3 (used volume according to SS-EN 445 should be approximately 390 cm^3). The limited volume of mixture means that the measured bleeding (%) is probably estimated to a lower value than if the measurements had been performed with a larger volume. The used measure vessel has the same diameter but a lower height, approximately 56 mm in the inert bleeding tests (ordinary height is 200 mm).

The bleeding measurements, regarding the cement based mixtures, are performed according SS-EN 445. Because of the relative small passed amounts of mixture in some cases, the measurement of the bleeding has not been directly measured on the passed amount of mixture. The W/C ratio in the passed amount of mixture has been calculated via the measured density of the passed mixture (see eq 5.5), the bleeding has then been evaluated corresponding to a mixture with the same W/C ratio and recipe as used in the experiment.

Low bleeding is an important parameter in order to reduce the probability of achieving an inhomogeneous mixture with varying properties. Varying properties in the mixture can influence the measurements of filtration and rheological measurements in a negative way.

5.1.6 Evaluation of measuring results

The work of finding essential parameters from the grain size and grain size distribution, that influence the filtration tendency is probably much a work of finding representable parameters to characterize the grain size and grain size distribution. Attempts have been made to correlate certain parameters of the grain size and grain size distribution like for example d_{95} and d' to the passed amount of mixture through the filter device, see eq 5.1. Even quotes of used parameters have been used for correlation. One of the correlation methods that have been used is written as:

eq 5.1

$$\delta_{xy} = \frac{Kov(X,Y)}{\sigma_x \times \sigma_y}$$

δ_{xy} = Coefficientof Correlation, $-1 \le \delta_{xy} \le 1$

$Kov(X,Y)$ = Covarianse of matrix X and Y

$\sigma_{x,y}$ = Standard deviation for matrix x and y

The parameter δ_{xy} is a measure of how well two series of data correlate to each other. Good correlation is associated with a δ_{xy} near 1.0. The presented correlation values in the diagrams in chapter 6.2.4 are the R^2 value, which is the squared value of δ.

If the relationships between two measured series are strongly divergent from a linear relationship, the coefficient of correlation δ_{xy} is not that good estimation of the relation. δ_{xy} correlation can identify linear relations and relationships close to linear.

Another way of predicting or analysing the results from the filtration experiments is to use statistical tools like multivariate data analysis (MVDA). There are a vast number of methods for MVDA, that are often used in order to get an overview of a large amount of data as, for example, classification of data and regression modelling (Johnson, 1998). The multivariate software used in this work is Simca©, which uses projection methods (PM) for the MVDA.

The projection method is also rather easy to understand when the solution can be interpreted geometrically. Projection methods can be described as projection of collected data onto a new coordinate system. PM is often used in order to reduce the variables in a problem, which will often facilitate the evaluation of test results. As an example, it can be mentioned that temperature and pressure are often well correlated to each other in a process. By the help of PM, one of the variables can be eliminated (two variables can be handled as a single variable) in the evaluation of the result. The purpose of using multivariate analyse in this work is to find the parameters and limits which significantly influence the amount and quality of the passed mixture through the used filter (filtration tendency). The performance of PM includes in this work both PCA- and PLS-analysis. More in depth information about PM methods can be found in Eriksson (1999).

5.1.6.1 PCA-analyses (Principal Component Analyses)

Data that can be analysed with projection method, typically consist of N observations in K variables. Each observation can be interpreted as a point in a K-dimensional space. To be able to compare different types of variables and get unit independent solutions, data is scaled and centred. One common projection method is the PCA (Principal Component Analysis). In PCA the first step is to determine the Principal Component (PC) that bests describes the variation of data, using e.g. a Least Square (LS) criteria.

Additional PCs can be added to better describe the variation in the data (Figure 5.5). Each new PC has to improve the approximation of data as much as possible but also has to be orthogonal to all earlier PCs. There are several ways to calculate the new PC-space. In this work commercial software Simca P 8.0 from Umetrics has been used and the algorithm used in the programme is described in the software manual (Umetrics 2000).

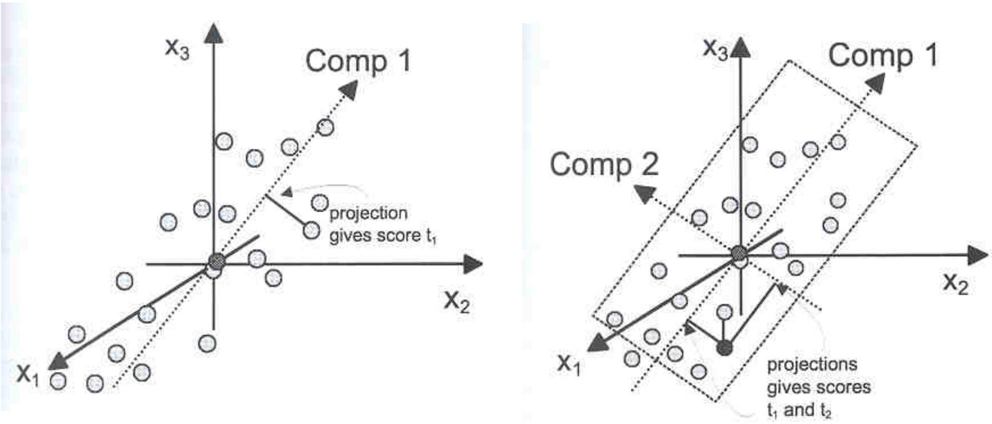


Figure 5.5, The first Principal Component (PC) is the component that bests describes the variation of data. The second PC is the component orthogonal to PC 1 that bests describes the variation of data. The projections of the observations to the PCs are called scores. (L. Eriksson, 1999).

Geometrically this can be seen as an incomplete coordinate transformation from X-space to the new PC-space:

eq 5.2

$$\hat{e}_{PC} = P\hat{e}_X$$

where $\hat{e}_{PC}$ is the unit vectors of the PC space, $\hat{e}_X$ the unit vectors of the X-space and P is the transformation matrix. In PCA the transformation matrix P is called **loadings**, hence, P describes the load/effect of the individual variables on the model.

The transformation can be regarded as incomplete since only the most important components are calculated of the new PC-space and therefore the observations are projected to the new space, i.e. the new point in the PC-space (the projection) is not an exact representation of the observation, it's the best approximation of the observation. The projections of the observations to the new PC space are called **scores** (T).

In PCA loading and score plots are an important complement to each other. The loading plots tell how important different variables are and the score plots reveal trends and groups in the data. Mathematically the observations (x_i) are modelled as:

eq 5.3

$$x_i = \bar{x}_i + TP^{-1} + \varepsilon$$

where $\bar{x}_i$ is the mean average of the observations and ε is the residuals that are a combination of noise and weak interaction from the variables, that the PC-space cannot explain.

5.1.6.2 PLS-analyses (Partial Least Square Analyses)

In PLS a regression analysis is carried out between two groups of variables with corresponding data sets. In a process the most common one data set is determined by Factors, input variables of a process, i.e. X-space and one data set of resulting Responses, output variables of the same process, i.e. the Y-space.

The method results in a linear model that describes how the Factors (x_i) affect the Responses (y_i). Normally, coefficients (a_i) are given in a normalised form so that the importance of different variables is clearly shown.

eq 5.4

$$y_i = a_0 + a_1 x_1 + a_2 x_2 + a_3 x_3 + ... = \sum a_{ij} x_i$$

The difference between the PLS and the algebraic (linear) LS regression is that the regression is done between the scores of each individual measurement in X and Y-space, instead of the original values of the measurement in X and Y-space. This can be seen as a form of noise elimination, i.e. a variable with low influence, e.g. due to noise, in the X-space will have a low impact on variables in the Y-space. This also allows for handling of missing data for some variables of a measurement.

The analysis is itself linear so in order to be able to handle non-linear variations, new variables have to be added, e.g. x_i^2, $x_{i\ t\text{-}\Delta t}$ or exp(x_i). In this way non-linear processes and time series can be studied by this method.

To assess a model, the goodness of fit, R^2, is calculated. A high R^2 means that the observations are well predicted by the model. However, increasing the number of parameters in the model will increase the agreement so R^2 will increase towards unity as the degrees of freedom are reduced.

Hence, R^2 is not a sufficient assessment parameter of the model. Therefore, the goodness of prediction, Q^2, has to be evaluated. A high Q^2 tells that the model is independent of the value of single observations. As the model parameters increase Q^2 will first increase as the fit of the model is improved, however, as the model starts to be ‘over predicted’, i.e. starts to explain noise for individual observation the value will decrease. So the best model is obtained by maximizing Q^2, see Figure 5.6.

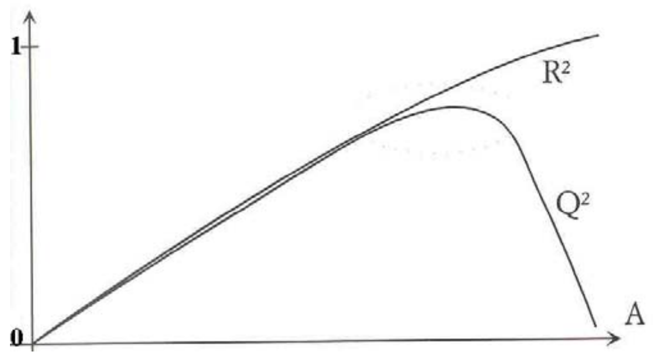


Figure 5.6, The principal variation of R^2 and Q^2 with an increased number of model parameters.

5.1.7 Air sieving

Air sieving was used to manufacture both the inert and cement based material. A vast number of difficulties were detected when the sieved material was manufactured, which are here described. The principal for the manufacturing procedure was almost the same for the inert and the cement based material. General description of the air sieving technique of dry powders is found in Appendix F. General explanation of the used sieving method and some technical data of the used apparatus is described.

The air stream separator that was used is Alpine 100 MZR. The problem with incomplete splitting of grains can both be explained by the sieving technology and the combination of sieved material and the material in the sieving device. Air stream separation is associated with having to calibrate the device so that one would theoretically get a powder with narrower grain size distribution then actually received. The overlapping of grain limits is associated with the difficulties in splitting the powder into narrow and adjacent grain fractions.

Electrostatic charge of the small grains in the powder makes the small grains adhere to larger ones (flocculation). The electrostatic charging of grains is mainly due to electron transport from the metal in the sieving device to the dolomite powder. The flocculation phenomenon makes it difficult to separate the small grains from the larger grains.

A relatively small amount of the sieved inert materials (mix 1, 2 and 3) was produced in the apparatus. This was due to that each bath of sieved material was very time consuming to produce. The lack of inert material makes that each mixture in the filtration tests have to the rather small (~110 cm^3 of mixture). Originally the amount of mixture was set to be based upon about 1000 g (600 cm^3 of mixture volume).

The purpose of sieving the original material was to create a number of dry powder mixtures, containing different grain sizes and grain size distributions. The basic three dry powder mixtures were then enough to create these six dry powder mixtures, see Figure 5.7. The d_{95} value of the dry powder mixtures will vary between the finest and the coarsest of approximately 5 µm (mix 1) up to 12 µm (mix 3). The six different dry powder mixtures were regarded to be divergent enough (grain sizes and grain distributions) in order to analyse the influence of grain size and grain size distribution on the filtration phenomenon.

Sieving the raw material (dolomite powder) produces the powder mixtures 1, 2 and 3. The basic idea of sieving the material was to divide the raw material into four different groups, depending on their minimum and maximum grain size. The grain size limits were set to 0-2, 2-5, 5-10 and 10-30 µm. It is desirable to get as sharp fraction interval as possible when investigating the influence of the grain size distribution. Due to some problems with splitting the grains into different fraction intervals, the results can be seen in figure 3.1 (mix 1, 2 and 3). 95 % of the grain sizes in mix 1 are between 0-5 µm, mix 2 is a middle fraction containing 40 % of grain sizes between 5-10 µm and mix 3 is a coarser fraction with 80 % of the grains between 10-30 µm.

Cement based material were also air sieved into a number of powder mixtures. The powder mixtures were named Cem 2 (d_{95}= 10 µm), Cem 4 (d_{95}= 9 µm), UF 12 Fine (d_{95}= 8 µm) and UF 12 Coarse (d_{95}= 16 µm). Cem 2 has a steeper grain size distribution compared to Cem 4 (Cem 4 is consequently containing a larger amount of small grains compared to Cem 2). The steepness of UF 12 Fine and UF 12 Coarse are almost the same. Larger amount of the cement based material could be manufactured (within reasonably amount of time for the sieving operation) compared to the amount of inert material. This was probably mainly because of the properties (surface charge, hardness and shape of grains etc.) of the dry cement powder compared to the dolomite powder.

5.2 Inert material

The inert experiments can be divided into two categories, one that deals with the physical experiments of inert material (dolomite powder and scaled up experiments with plastic grains). The other part of experiments deals with numerical simulations.

5.2.1 Physical experiments

The geometrical properties (maximum and minimum grain size, grain size distribution, grain shape and W/C ratio) which influence the penetrability are investigated by the use of inert grains. The reason for using inert grains is to avoid influences from chemical reactions and time dependent properties of cement. The inert material that is used is crushed dolomite stone, in the fraction of 0- 30 µm.

The trading name of the powder is Myanit. Dolomite is a mineral of mainly Calcium, Magnesium and Carbonate with the formula $CaMg(CO_3)_2$. It has a grain density of 2850 kg/m^3 and a pH value of approximately 9,5- 10,5.

The influence from the grain size distribution is investigated by mixing three different fraction intervals of the powder, into a number of new mixes with different grain size distributions, see Figure 5.7. The original fraction intervals are Mix 1, 2 and 3. The fraction mixes are then mixed with different amounts of water before the penetrability analysis.

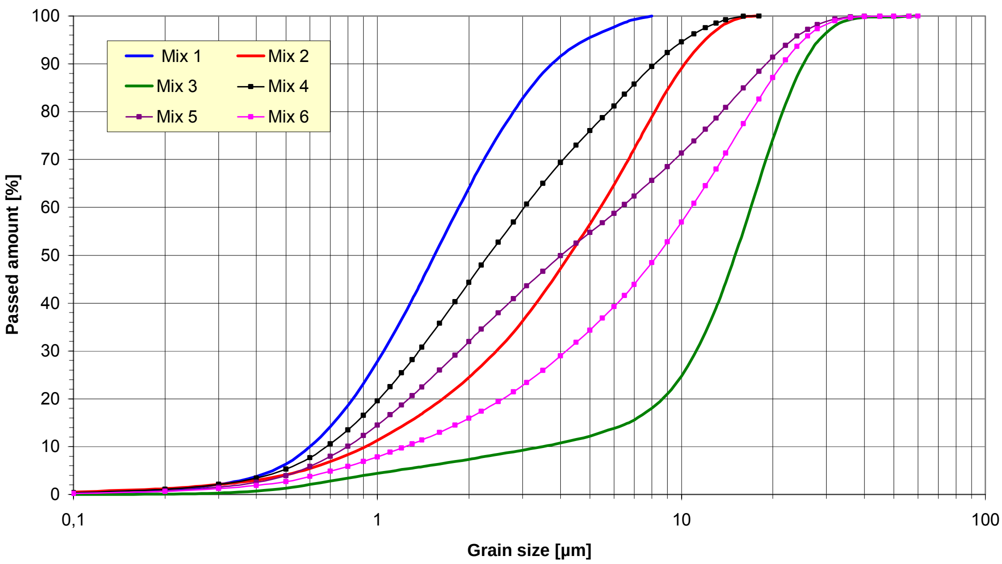


Figure 5.7, Grain size distribution curves for different mixtures.

Table 5.1, Characteristic data for used mixtures.

Mixture	d_{95}	d`
	[µm]	[µm]
1	5	2,0
2	12	5,2
3	28	10,6
4	10	10,0
5	24	3,7
6	26	7,0

According to the relatively small volume of inert mixture (~110 cm^3) in the filtration experiments with inert mixtures compared to the volume of cement-based mixtures (~600 cm^3) in the filtration experiments with cement mixtures, just a rough measuring scale has been defined to quantify the difference in filtration tendency between the mixtures of inert material.

The small amount of inert material means that the result (passed amount of mixture) will be more uncertain than if a larger volume of mixture had been used (like in the cement based experiments). b_{Stop}, $b_{Filtration}$ and b_{All} refer to the interval of filter aperture that cause or do not cause plug formation at the filter. b_{stop} indicates that a very small and filtrated amount of mixture (almost pure water) has passed the filter. $b_{Filtration}$ means that an obvious filtration of grains has occurred. b_{All} indicates that the mixture has low filtration tendency and no significant filtration has occurred.

The results (passed amount of mixture) through these filter intervals (based on inert filtration experiments) should therefore not be directly compared to the results from the ones noteded with b_{min} and b_{crit} (from the cement based mixtures), see chapter 3, because of the difference in the used amount of mixture. b_{crit} has been interpreted to the minimum of b_{All}. b_{min} has been interpreted to the maximum of $b_{Stop.}$

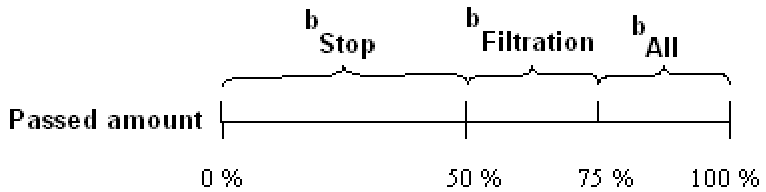


Figure 5.8, Rough measuring scale for filtration tendency of the inert material.

It has been observed that some minor material was stuck in the equipment. It has been estimated that this could go up to about 25% of used material. The used limit of b_{crit} corresponds therefore to a passed amount of 75% of used material.

To quantify the filtration properties one has to also look at the concentration of grains in the passed mixture. A large passed amount (large weight) of mixture is not necessarily equal with a large amount of passed grains. Grains could be stuck in the filter and the passing mixture is then mainly consistent with that of water. The quality of the mixture that passed the filter has to be investigated by comparing the W/C ratio of the mixture before and after the passage through the filter. Almost the same W/C ratio before and after filtration is an indication of a good quality of the passed mixture.

The ratio between the amount of water and the amount of grains could be determined by the following relationship (Hansson 1994);

eq 5.5

$$W/S = \frac{\rho_w \cdot (\rho_s - \rho_c)}{\rho_c \cdot (\rho_w - \rho_s)}$$

ρ_w = Density, water

ρ_s = Density, suspension

ρ_c = Compact density, dolomite powder

W/S = Water/ Solid ratio

W/S-ratio (Water/ Solid) is used in this work for the mixtures content of dolomite powder and W/C-ratio is used for mixtures of cement and water. eq 5.5 is valid for both these cases.

In this work, approximately 110 cm^3 of mixture is used in each test (depending on the desired volumetric concentration of grains in the mixture). Each mixture consists of 100 g dolomite powder and a different amount of water, depending on the desired volumetric concentration of grains. The powder was first mixed in a paddle mixer during one minute then in a blender (Ultra Turrax) at approximately 20 000 rpm during two minutes. The ready mixed mixture was then placed in the pressure chamber. The mixture was put under pressure (30 kPa) and continuously stirred while pressing the mixture through the filter. The maximum pressure time was set to one minute. The passed mixture through the filter was collected and weighted in a collecting chamber. Some mixture will always adhere to walls in the collecting chamber, hose and the filter.

The amount of mixture that will adhere to the equipment is mainly dependent on the W/S ratio in the mixture. A low W/S ratio will increase the amount of mixture that adheres to the equipment compared to a mixture with a higher W/S ratio.

Mainly three types of parameters have been varied in the experiments, variation of grain concentration (W/S-ratio), slot and mesh aperture and variation of used grain curve. One parameter in each test has been varied, while the others are constant. Six different grain size distributions have been used according to Figure 5.7 (mixture one to six). The influence of grain concentration on the filtration tendency is done by varying the W/S-ratio between 0,6 to 1,4. The influence of crack aperture (filter geometry) on the filtration tendency has been studied by using different types of filter geometrie's and filter apertures, see Table 5.2

Table 5.2, Description of the used parameters and their variation. Note that all of the parameters have not been tested in all combinations.

Parameter			
W/S-ratio	**Slot aperture**	**Net mesh**	**Mixture no.**
	[μm]	**[μm]**	
0,6	45	36	1
0,7	75	45	2
0,8	125	75	3
1,4			4
			5
			6

5.2.2 Physical experiments with scaled-up plastic grains

To visualize the phenomenon of plug formation of grains an investigation has been carried out in a larger scale than usually takes place in reality. A number of hypothesises can be stated in order to analyse the filtration tendency in the physical model.

- Due to difference in shape of grains in the mixture, it can be proposed that irregular (larger angularity) grain shapes are more inclined to agglomerate at the crack entrance than grains with a regular shape.

- The probability of agglomeration at the crack entrance increases as the grain concentration is increased.

- The probability for the grains to agglomerate at the crack entrance increases as the crack aperture is narrowed.

- The clogging procedure is a statistical phenomenon**.**

To investigate the clogging phenomena, a plexiglas model was built. Figure 5.9 below illustrates the physical model. One basic problem with physical modelling is to recreate a model that is similar, preferably identical, to the real scale situation referring to the underlying physics. One of the physical quantities that have to be taken into account is the sedimentation velocity. It is important that the sedimentation velocity of the grains in the scaled-up model is of approximately the same magnitude as the grains in the real scale application.

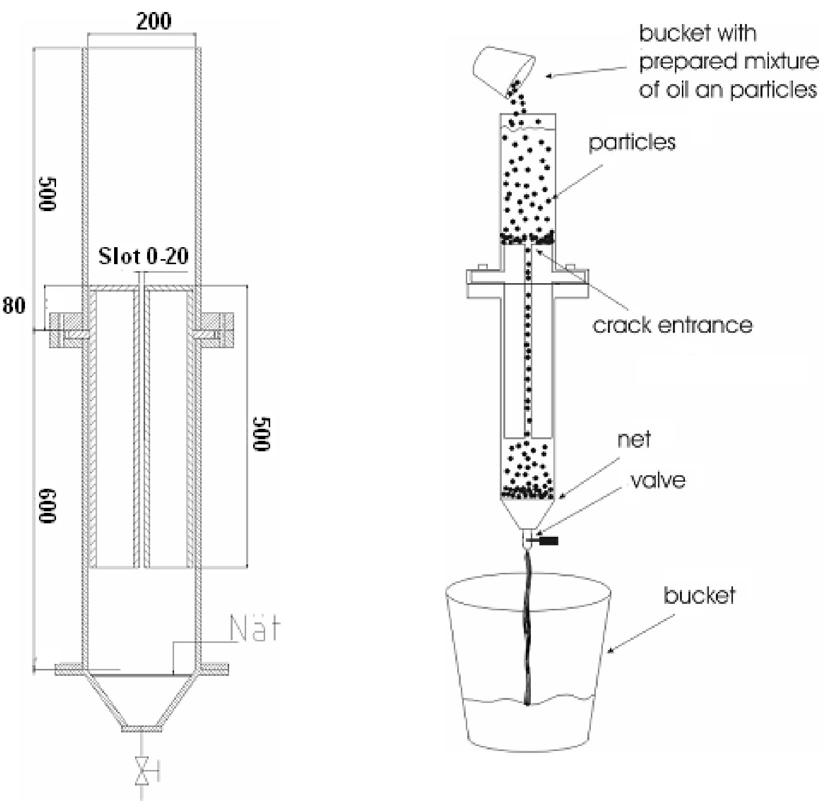


Figure 5.9, Outline of the physical model (Saaidi, 2004).

The upper part of the model was made twenty times bigger than the crack geometry in the spanwise direction. This was done to minimize the effect of the upper container walls.

In order to compare the experiment with the two-dimensional numerical simulations the ideal case would be to have a "two-dimensional" experiment, meaning that it would be desirable to design the experiment in a way that would make the front and back wall effects negligible. Nothing could be done to completely eliminate the front and back wall effects. However it is believed that they have a limited effect on the flow-field within the crack.

From a fluid-mechanical point of view it is possible to scale-up the flow field without changing the underlying physics of the phenomena by using a fluid with a larger viscosity than the fluid in the real application. Cementitious grouting materials are mixtures of grains in water, hence it was found suitable to scale up the geometry and the grains 100 times by using oil with a viscosity 100 times larger than the viscosity of water $(\nu_{oil} = 100 \cdot \nu_{H_2O})$. The main idea was to keep the Reynolds number and the sedimentation velocity constant.

The Reynolds number is defined as:

eq 5.6

$$\mathrm{Re} = \frac{U \cdot d}{\nu}$$

where d is the diameter of the sphere (grain), U is the velocity of the grains and ν is the viscosity of the oil. At first glance it may seem possible to perform the scale up by just varying the diameter of the grain and the viscosity of the oil to keep the Reynolds number constant. However, there is another important dimensionless number called the Archimedes number that has to be taken into account.

The Archimedes number is defined as:

eq 5.7

$$Ar = \frac{gd^3}{\nu^2}\left(\frac{\rho_d}{\rho_c} - 1\right)$$

Where d is the diameter of the sphere, g is the gravity force, ν is the viscosity of the oil and ρ_d and ρ_c are the density of the grain and the oil respectively. eq 5.7 illustrates that it is not possible to just perform the scale-up based solely upon the Reynolds number. Archimedes number which is taking the difference in densities between the grains and the fluid into account in order to calculate the resulting sedimentation velocity of the grains in the oil (Saaidi 2004). It should be noted that the physical experiments described in this section were performed with inert plastic grains, hence no adhesive forces and chemical reactions are present as in the case of a real grout. The scaled-up experiment is therefore based upon that both the Archimedes number and Reynolds number should be kept constant.

The physical tests were performed in a plexiglas model, see Figure 5.9.

To obtain the desired viscosity of the oil, three different 20 liter containers of oil were heated to 60°C. Thereafter, the oil in the different containers were carefully mixed in a big plastic vessel. The viscosity of the oil were measured by the usage of a capillary viscosimeter that was normalized for comparison with water, see Figure 5.10.

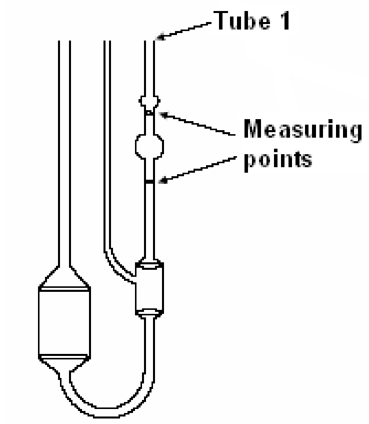


Figure 5.10, Capillary tube for measurement of the oil viscosity (Saaidi, 2004).

By measuring the necessary time (at a certain temperature of the oil) it took for the oil to flow through Tube 1, from the upper measuring point to the lower measuring point, it was possible to calculate the oil's viscosity according to eq 5.8.

eq 5.8

$$\nu_{oil} = t_m \cdot \nu_{H_2O}$$

where t_m is the measured time in seconds.

By the use of eq 5.8 and the measurements in the capillary viscosimeter it was possible to calculate the desired viscosity. The achieved viscosity of the oil in this study was found to be 98.4 times larger than the viscosity of H_2O, which is very close to the desired value of 100 times the viscosity of water. The density of the oil was measured to be 850 kg/m^3.

Inert plastic grains with a density in the proximity of the oil density were used to simulate the grains in a grout. The plastic grains were quite irregularly shaped, and to characterize the dimensions of the grains a representative sample of each type were passed through a sieve. Two different kinds of grains were used (black and white grains). All of the grains where found to have a characteristic dimension between 2 and 4 mm. The dimensions specified in Figure 5.11 have been measured by hand. Two types of grains are used in the experiment, one with a round shape and another with more sharpened edges. The white grains had a more spherical shape than the black ones. The white grains also had a slightly wider grain size distribution.

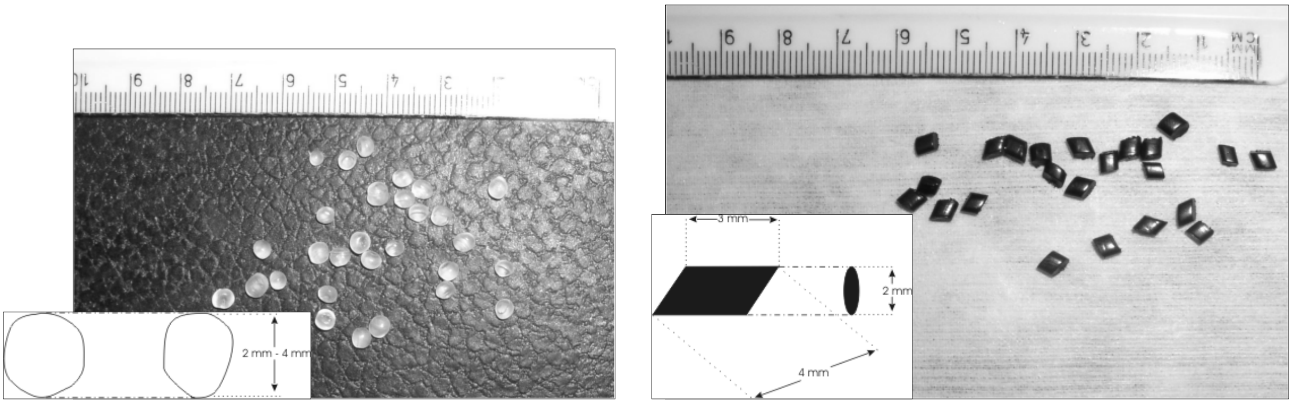


Figure 5.11, The two types of grains used in the experiments, the left picture shows the round shaped grains, the right picture shows the sharp edge grains (Saaidi, 2004).

Table 5.3, Grain characteristics.

Grain type	Density [kg/ m^3]	Size [mm]
Black	933	≈ 3
White	917	≈ 2-4

Due to the scaling of the experiment a grain in the model corresponds to a 100 times larger grain than in the real grout mixture. Hence a grain of 2-4 mm in the model corresponds to 20-40 μm in the real grout mixture. The scaling of the experiment presume as mentioned earlier, a certain viscosity and density of the used oil and grains. For further description of the physics behind the scaling of experiments, see appendix F.

Each test in the plexiglas model was made up of a series of events:

1. The plexiglas model was filled with oil.
2. The bucket was filled with the amount of grains and oil so as to get the desired bulk concentration.
3. The valve was opened to get the desired fluid flow velocity within the model.
4. The grain mixture was carefully poured into the top of the model. If not, the result would have been undesirable disturbances of the flow field close to the crack, because of the high viscosity of the oil.
5. A new grain mixture was prepared in the bucket and the same procedure as outlined above was repeated.

As a consequence of the limited access to oil there was a clear limitation as to how many new grain mixtures that could be poured through the model. To change the crack aperture the model had to be emptied of oil and demounted. Thereafter the plexiglas blocks, had to be adjusted by hand.

5.2.3 Numerical experiments

The simulations in this work were done with the commercial CFD code Fluent 6.1 from Fluent©.
The objectives of the numerical simulations can be summarised in the following way:

- Perform a two-dimensional Eulerian multiphase flow calculation of a dispersed two-phase flow through a narrow slot.

- Implement a new frictional model to better account for the frictional stresses between the grains when the local grain concentration becomes high.

- Compare the results with the physical experiments with inert particles (plastic grains).

The numerical calculation has been conducted on two different geometries (slot aperture 5 and 10 mm). The geometries are shown in appendix A. Moreover four different volumetric grain concentrations have been used in the simulations; 5, 10, 20 and 30%. No comparative studies are done between the available models instead the models that are believed to be most suited for the particular application is used. The results of the simulations are compared to the physical simulations described above. The mesh is refined at the locations in the model where the largest gradients are expected to be present, see Figure 5.12.

For the 5 mm crack mesh there is a gradual increase of the spacing between the grid nodes in the upward direction. This gradual increase makes the interpolation error between the fine mesh and the coarse mesh regions smaller. However, there is an error at the interface between these regions, however it is not believed to have a very big impact on the flow field in the vicinity of the crack entrance.

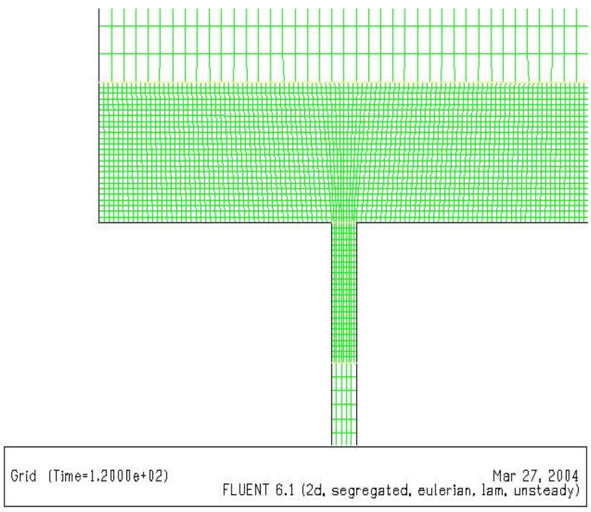


Figure 5.12, Used mesh sizes at different places in the model.

All the simulations were carried out in a two-dimensional space in which front and back wall effects were neglected. For the fluid-phase all walls are treated as no-slip velocity boundary conditions. Moreover the velocity for both phases is specified at the top of the model, this is sometimes referred to as a Dirichlet boundary condition. At the bottom of the model a Neumann boundary condition is applied, which means that the velocity gradients of both phases are zero across the boundary. For the grain phase a free-slip velocity boundary condition is employed for the horizontal walls and a no-slip boundary condition is applied for the vertical walls. Figure 5.13 below illustrates the applied boundary conditions for the grain phase.

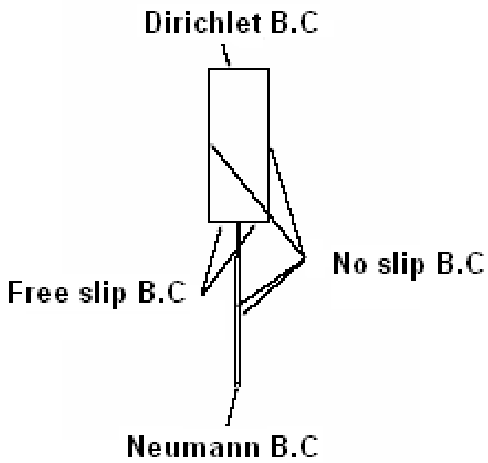


Figure 5.13, The grain phase boundary conditions.

It would have been more correct to use a partial slip boundary condition at the wall for the grain phase. This type of boundary condition is however not an option in Fluent[2]. It is however necessary to use the no slip boundary condition for the grain phase. If the free slip boundary condition would have been used all of the grains would slide unhindered through the model since there would have been no force to counteract the vertical shear forces of the grain phase.

[2] Such boundary conditions have to be programmed by the user, due to time limitations this was not done.

5.3 Cement based material

5.3.1 Physical experiments

The amount of used mixture in the mixing process is approximately 2000 cm^3. Filtration tests with cement-based material are performed with approximately 600 cm^3 of the mixture. This volume of mixture is chosen due because too small amount will create an undesirable development of a high temperature in the mixture. A high initial temperature in the mixture will speed up the hydration process in the mixture. The temperature of the mixing water is set to approximately +8°C. A rapid hydration process of the cement grains will increase the risk for flocculation of the grains in the mixture. The flocculation of grains affects the filtration tendency in a negative way (higher filtration tendency). The temperature of the mixture at testing is assumed to be approximately 20°C according to spot sample measurements.

It is also of importance to mix the ingredients in a specific order i.e. water, cement and finally superplastisisers. The same pressure and mixing method has been used for the cement-based mixtures as in the case of the dolomite based mixtures. The time dependency of the cement reactions in the mixture means that it is of great importance to perform the penetrability tests at a certain time after mixing. According to the literature (Betonghandboken 1994) it is suitable to start the penetrability tests approximately 3-5 minutes after the mixing procedure is finished. This moment is chosen because the chemical reactions during this period have been stabilized (the dormant period).

The time dependent properties of the grout mixtures have also been tested at some specific moments after the mixing is finished. Filtration tests have been performed at 10, 20, 40 and 60 minutes after the mixing procedure was terminated.

As mentioned earlier, a different amount of material will be used in different tests. Variations in the W/C ratio (see Table 5.7) will generate a different amount (weight) of mixture, see Table 5.4.

Table 5.4, The left table illustrates the initial weight of the mixtures (mixture in the filtration test equipment). The right table illustrates the available weight of mixture through the filter (not all the amount of mixture is available in the filtration test).

	UF 12	UF 16	IC 30
W/C ratiot	[g]	[g]	[g]
0,7	989	1008	988
0,85	951	915	896
1,0	902	868	866
1,25	701	704	-
1,50	674	677	-
1,75	652	656	-

UF 12	UF 16	IC 30
[g]	[g]	[g]
813	827	809
722	744	724
741	704	702
551	550	-
526	525	-
509	510	-

As can be calculated from Table 5.4 and appendix G (density measurements), at least approximately 110 cm^3 can be directly cut off (not available) from the initial 600 cm^3 of mixture volume. The amount of mixture that gets stuck in the filtration equipment (container, hose, tube) will vary depending on the W/C ratio used in the mixture. A lower W/C ratio will according to performed tests, means that a slightly larger amount of the mixture will get stuck in the equipment. The tests with cement were carried out with a larger container than for inert material which reduced the available amount to pass the filter.

The available amount of the cements of the sieved cements Cem 2 and Cem 4 is 774g when the W/C ratio is 0,7. The used amount of UF 12, Fine and Coarse is in the same range as the original UF 12 cement (951-701 g), when using a W/C ratio between 0,85 to 1,25.

Different cement-based grain curves have been used in the filtration experiments. Figure 5.14 show the grain size distribution of the commercial available cements Ultrafint cement 12 (UF 12), Ultrafint cement 16 (UF 16) and Injekterings cement 30 (IC 30).

As can be seen in the figure below the grain size distributions are, for UF 12 and UF 16, quite similar, except the coarser grains are larger than by approximately 6 µm. The IC 30 cement has a major difference of its grain curve compared to the two other.

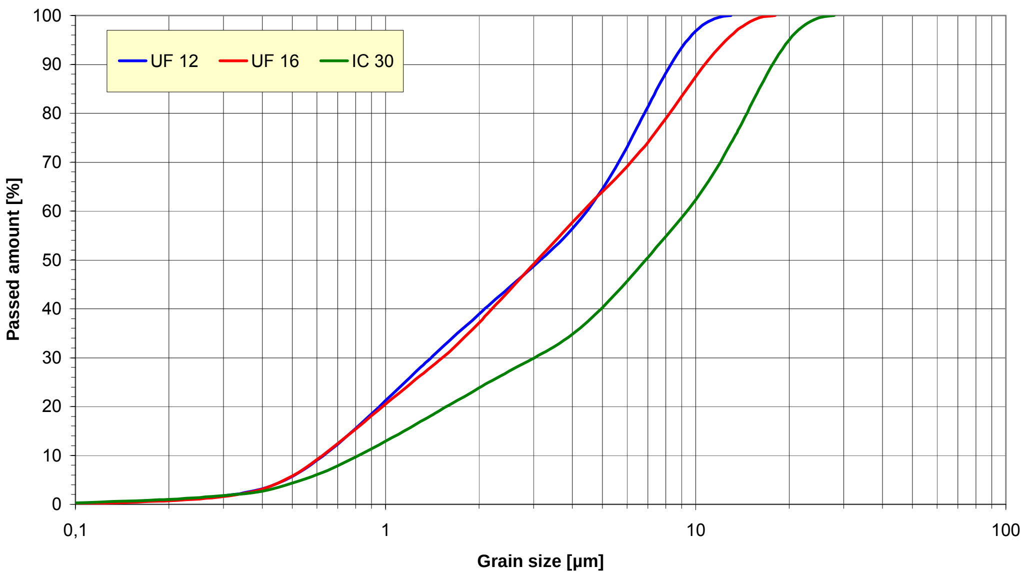


Figure 5.14, Grain size distribution curves of cement-based material (Uf 12, Uf 16 and IC 30).

Attempts have also been made to create new grain curves of cement-based material. The cement-based grain curves were fitted to the inert material's grain curve that showed the best filtration tendency (the largest passed amount). For the result of the sieved cement-based-material, see Figure 5.15.

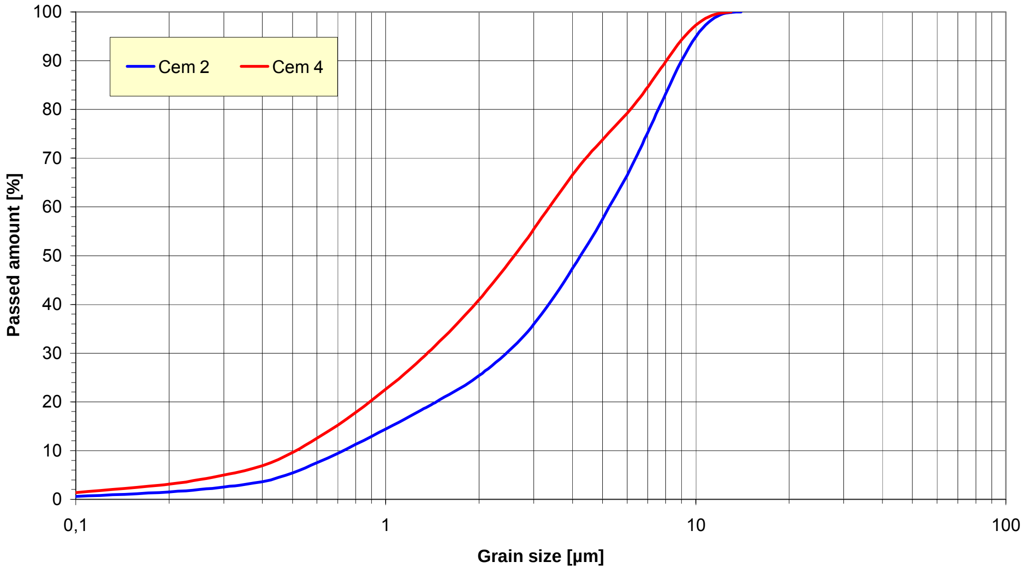


Figure 5.15, Grain size distribution curves of cement-based material (Cem 2 and Cem 4).

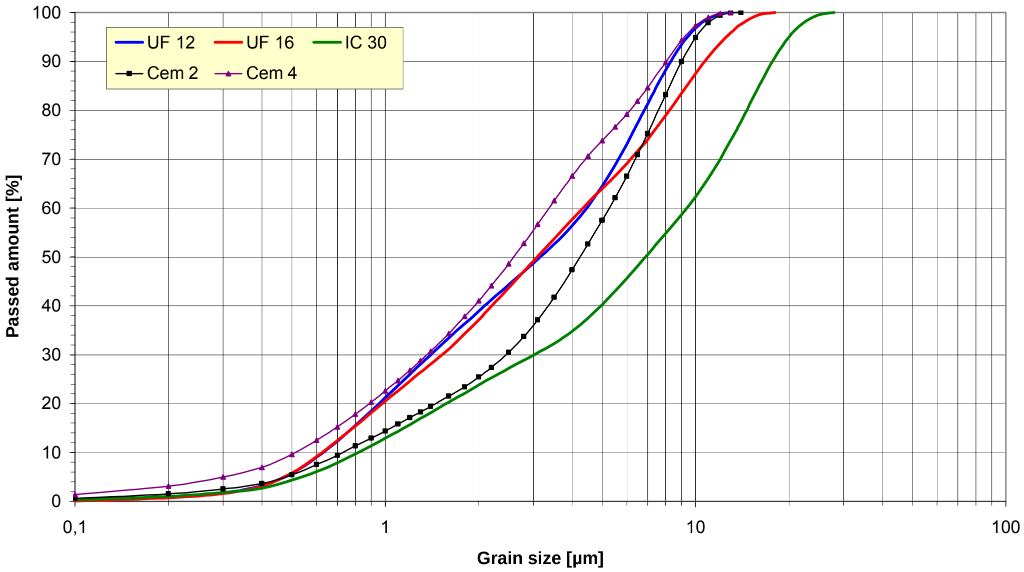


Figure 5.16, Grain size distribution curves of cement based material (Uf 12, Uf 16, IC 30 and Cem 2, Cem 4).

Table 5.5, Characterisation of grain size distribution, in Figure 5.16.

Cement	d_{95}
	[μm]
UF 12	12
UF 16	16
IC 30	30
Cem 2	10
Cem 4	9

As can be seen from Figure 5.16 there are not any major differences between the grain curves. The simulated cement based grain curves Cem 2 and Cem 4 should be compared to the inert grain curves 2 and 4.

Finally two cements, based upon UF 12, have been tested which have been sieved according two criteria. The first criterion was to remove a major part of the fine grains, i.e. those smaller than 3 μm from the original UF 12 cement. The second criterion was to remove the grains larger than 9 μm from the UF 12 cement. Consequently, the idea was to analyse the influence of the fine and coarser grains on the filtration tendency of the cement based mixture.

Unfortunately the apparatus for the analysis of the grain size distributions in the cement powder were changed in the end of the sieving work. This causes a difference that can be detected when comparing the grain size distribution curves from the old apparatus (Cilas 850 GR) and the newer one (Malvern Masterziser 2000).

This primarily concerns the sieved cements of UF 12, Fine and Coarse (Figure 5.17), which have been analysed by the new apparatus (Malvern Masterziser 2000), see Figure 5.16 compared to Figure 5.17 regarding the grain size distribution for UF 12.

The different results from the analyze of the sieved powder indicate the difficulties involved in such process. Before this has been more rigorously investigated cautiousness with conclusions regarding the grain size distribution's influence on to the results should be considered.

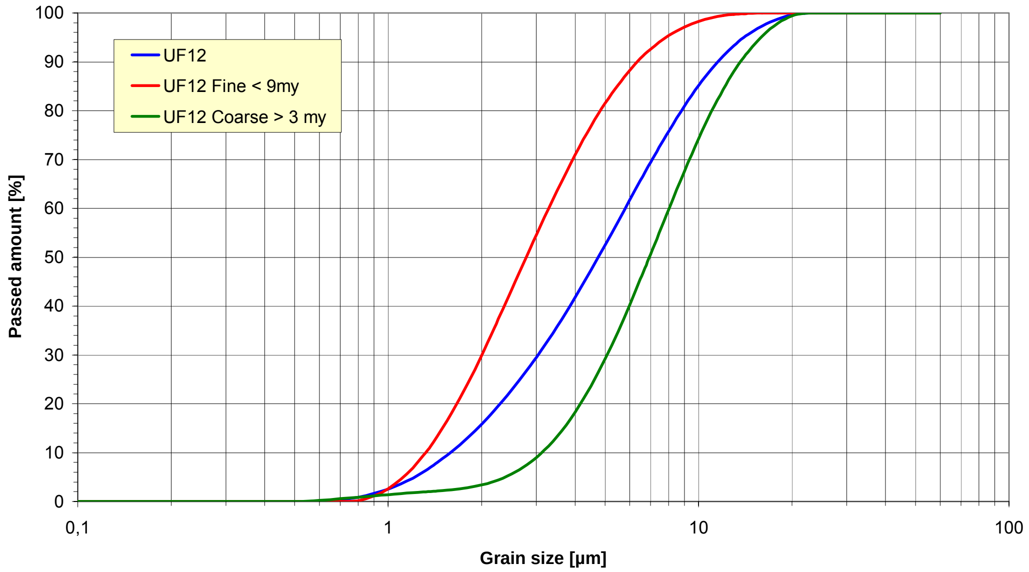


Figure 5.17, Grain size distribution curves of cement based material (UF 12 Fine and UF 12 Coarse).

Table 5.6, Characterisation of grain size distribution in Figure 5.17.

Cement	**d_{95}**
	[µm]
UF 12	12
UF 12, fine	8
UF 12, coarse	16

The presentation of the results from the filtration and the bleeding tests, will mainly be made for each used cement (UF 12, UF 16 and IC 30).

Three different superplastisisers have been used in the experiments. The superplastisisers Cemflux, Melcrete and L 15 are mainly two different types of superplastisisers. Cemflux is a co-polymer based additive and the other two are based on Melamine and Naphthalene.

The co-polymer based one is a steric stabilisator (steric superplastisiser) and the other two are electrostatic stabilisators (electrostatic superplastisiser).

The filtration tendency has been tested for the slot aperture 75, 100 and 125 µm. The differences in filtration tendency have been tested using different slot aperture, W/C ratio, superplastisisers and used cements (grain curves). The parameters have been varied in different combinations according to Table 5.7.

Table 5.7, Description of the used parameters and its variation. Note that all of the parameters not have been tested in all combinations.

Parameter				
W/C-ratio	**Slot aperture**	**Net mesh**	**Grain curves no.**	**Superplastisisers**
	[µm]	**[µm]**		**[]**
0,7	75	75	UF 12	Cemflux
0,85	100		UF 16	Melcrete
1,0	125		IC 30	L 15
1,25			Cem 2	
1,5			Cem 4	
1,75			UF 12, Fine	
			UF 12, Coarse	

Each tested combination has also been tested twice due to the uncertainties in the repetition of the experiments. If all these experiments in Table 5.7 were going to be investigated it would generate a relatively large amount of experiments (a quantity of (6*4*7*3=504), 504*2=1008 experiments).

Some of the experiments have therefore been excluded, mainly because of bad rheology or major bleeding.

5.3.2 Evaluation of the test result

The filtration tendency of the tested mixture has been evaluated in three steps. Firstly the penetration length has been calculated for every test. The penetration length has been defined as the length the passed volume of the mixture fill up given the cross area that corresponds to the filter area (opening in the filter).

Secondly the filtration tendency is expressed with the parameters b_{crit}, b_{req} and b_{min}, see Figure 5.18.

b_{crit} is measured as the smallest slot aperture where nearly all of the available amount of mixture passes the filter, this corresponds to a mixture volume of more than 75% of the available amount of 490 cm^3. This corresponds to a penetration length of 79, 60 and 48 m for a slot aperture of 75, 100 and 125 µm.

b_{min} is characterized as the slot aperture where no mixture is passes the specific slot. The evaluation of b_{req} has in this work been defined as the penetration length of 5m. This corresponds to roughly about 10% passed amount of mixture.

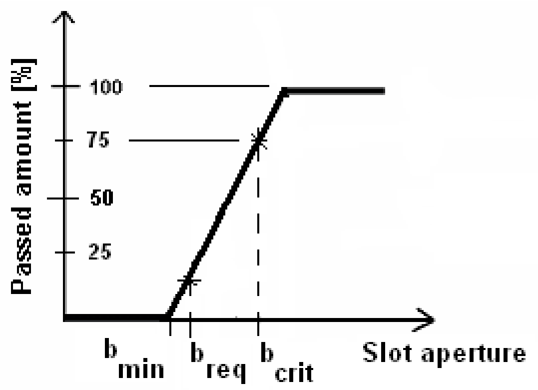


Figure 5.18, Definition of b_{crit}, b_{req} and b_{min},

To quantify low filtration tendency one also has to look at the concentration of grains in the passed mixture. A large passed amount (big weight) of mixture is not necessarily equal with a big amount of passed grains. Grains could be stuck in the filter and the passing mixture is then mainly water. Check of change in grain concentration has to be made.

The demand of the grain concentration of the passed mixture is in this work expressed in terms of the bleeding of the passed mixture. The upper limit of the bleeding is in this work set to 5 %. A bleeding of more than 5 % in the passed mixture is expected to generate problems with the durability of the hardened grout mixture in the crack volume. The quality of passed mixture that fulfils the requirement of penetrability length has therefore been evaluated according to the above mentioned criteria of bleeding.

As the final step the evaluated b_{crit} and b_{req} for different W/C ratios are compared to the requirement of quality in order to define the limits of what can be acceptable (defined by the slot aperture, W/C ratio and passed amount of mixture.

6 Results of experiments

6.1 General

The performance and design of the filtration experiments have been continuously developed during this work. In the beginning of the work, there were no suitable measuring methods to evaluate the tested mixtures filtration tendency. Because of the limited amount of mixture (sieved inert and cement powder), the available equipment (prEN 14497, Eriksson 2004, NES-apparatus etc.) was not possible to use in this work. The limited amount of mixture was basically (as earlier mentioned in chapter 5) due to a very time consuming (consequently even very expensive) performance of the manufacturing process (air sieving) of the powder. A partly new equipment and method for evaluating the mixtures filtration tendency had to be developed.

The new equipment (VU:SC 48) was developed. In the beginning of the work, the filter geometry consisted both of a mesh and slot geometry. It was found during the performance of the filtration experiments that the passed amount of mixture probably would have to be relatively large compared to the filter area (relatively large amount passed volume/ filter area) in order to evaluate the mixture's filtration tendency (plug formation) in a proper way. The filtration tendency can probably be seen as a stochastic process, where a relatively large amount of mixture should be available (used initial amount of mixture) to pass the used aperture. The probability of plug formation during the filtration experiment will probably increase as an increased amount of mixture passes the filter. Agglomerates or larger grains in the mixture may initialize the plug at the entrance of the aperture.

Theoretically an infinite amount of mixture should be available in order to evaluate the passed amount of mixture. Practically a limited amount of mixture is available for each filtration experiment. The limited amount of mixture meant that the filter area had to be reduced (compared to existing testing equipments) in order to generate a higher passed amount of mixture through the specific filter area (volume/ filter area). This was even the reason why the filter geometry in the later experiments in this work was focused on the slot geometry instead of the mesh geometry.

In the inert mixtures an initial volume of approximately 110 cm^3 has been used. Filter areas vary between 0,05 cm^2 (slot aperture) to 13,9 cm^2 (mesh aperture). The cement based powder was generally easier to manufacture (air sieving), this caused that a larger volume of mixture could be used in these filtration experiments. Initially the volume of cement based mixture used was approximately 600 cm^3. The filter geometry has been focused on the slot aperture with filter areas between 0,05-0,08 cm^2.

The passed weight of mixture through the filter is mainly expressed as a relative percentage of the initial available weight of mixture. The passed amount of mixture through a mesh filter should not be compared to a passed amount through a slot filter, even though the slot/ mesh aperture is the same. This is because of differences in design and size of filter area between the mesh and slot geometry.

Due to the influence of the stochastic plug formation it is likely to believe that the variations in passed amounts will increase when the used filter area decreases. Plug formation at the mesh geometry can probably be seen as an averaging process in relation to plug formation at the slot geometry. Filtration through the mesh geometry can be seen as filtration through many slot geometries.

However, the relative amount of mixture passed is used in some of the evaluations of filtration tests with inert and cement based material. This is mainly because it is an easy way to evaluate the results. The basic evaluation of the passed amount of mixture should regard the used filter area, filter geometry and available amount of mixture to a larger extent. Evaluation of the mixture's penetrability length is a way of taking these parameters into account, which has been done for the filtration experiments performed with cement based mixtures.

The presented results are divided into two major parts, one which deals with inert material and the second one that deals with cement based material.

The first part will present the results from the investigation of three main headlines. The headlines are penetrability experiments with inert material, scaled up experiments with plastic grains and numerical simulations of filtration tendency. The penetrability experiments will present parameters that in different ways affect the filtration tendency of a mixture. The parameters are grain size, grain size distribution, grain concentration and slot or mesh geometry. Each headline is finished with a summary and discussion of the presented results.

The second part deals with the filtration tendency when using cement based material that is reactive and changes its properties with time. Effects of using different W/C ratio, grain size, grain size distribution and superplastisiser are analysed in combination with slot or mesh geometries. The filter geometry in this second part mainly focuses on the slot geometry.

6.2 Inert material

The first section 6.2.1 in the first headline is dealing with the influence of different grain sizes and grain size distributions on to the filtration tendency. In this section, all mixtures have a W/S ratio of 0,7 and a mesh aperture of 45 μm. Measurements of filtration tendency, rheology and bleeding are preformed for six mixtures with different grain sizes and grain size distributions, see Figure 5.7.

The second section 6.2.2 in the first headline deals with the influence of different W/S-ratio on the filtration tendency. Measurements are preformed with a constant mesh aperture of 45 µm. Measurements of filtration tendency, rheology and bleeding are performed for six mixtures with different grain sizes and grain size distributions, see Figure 5.7.

The third section 6.2.3 in the first headline deals with the influence of different filter geometries on the filtration tendency. Different types of filter geometries were simulated by using a mesh or slot geometry, the size of the aperture was simulated by different slot and mesh apertures. Measurements of filtration tendency, rheology and bleeding are performed for six mixtures with different grain sizes and grain size distributions, see Figure 5.7.

At the end of each section 6.2.1, 6.2.2 and 6.2.3 there is a short summary of the significant results from the tests. The section regarding inert material is concluded with a section containing the comprehensive conclusions from all the preformed tests with inert material.

Finally, correlation tests are performed to detect and quantify connections between measured values of the mixture like for example $d_{95,}$ d´ and measured filtration tendency (passed amount through the filter). The standard deviation of each set (results) of experiment is presented.

The second headline deals with the scaled up filtration experiments using plastic grains (2-4 mm) and slot apertures of 5 to 10 mm. The fluid in the mixture is oil.

The third headline deals with numerical simulations of filtration tendency. In the numerical model attempts have been made to predict the risk of plug formation via concentration gradients and flow velocities of grains.

The amount of passed mixture [%] concerns the passed weight of mixture. The used initial weight of mixture will vary depending on the used W/S ratio, 100 g of the dolomite powder is always used in each mixture. As an example it can be mentioned that a mixture with a W/C ratio of 0,7 will consequently consist of 100 g dolomite powder and 70 g water.

6.2.1 Influence of grain size and grain size distribution

Influence of different grain sizes and grain size distributions on to the filtration tendency has been investigated for six mixtures with different grain size grain size distributions. The filtration geometry was kept constant using a mesh with a quadratic aperture of 45 μm. Reproducible measuring values were obtained with this geometry and a distinction between different passed amounts (weights) of mixtures through this mesh aperture was seen. The W/S ratio was set to 0.7, which corresponds to a volumetric concentration of grains of approximately 33 %. Mix 1 is not tested because of its bad rheological properties at the tested W/S ratio. A high content of small grains caused a too high yield- and viscosity value of mix 1.

Table 6.1, Result of filtration measurements. Mesh aperture 45 μm and mixtures with W/S 0,7. No. corresponds to number of performed measurements and Dev. is the standard deviation of the measured values . Cov. regards the coefficient of variance of the measured values.

	Passed Amount			
Mixture	Mean	No.	Dev.	Cov.
	[%]			
2	81	8	5,7	0,07
3	11	3	3,1	0,28
4	52	4	7,7	0,15
5	18	4	6,2	0,34
6	8	3	1,4	0,18

As can be seen from Table 6.1 the coefficient of variance decreases as the passed amount (based upon the passed weight) increases. It should be noted that there are relatively few measuring values for each mixture. The mixtures 3, 5 and 6, which contain the largest grains (expressed as d_{95}, see Table 5.1) are the ones that show the smallest passed amount.

To check the passed amount (weight) of grains through the filter the difference in density before and after filtration of the mixtures has been measured. The initial W/S ratio of 0,7 (before filtration) corresponds to a volumetric grain concentration c of approximately 33 %. The results in Table 6.2 illustrate the difference between different mixtures content of grains after filtration compared to before filtration.

Table 6.2, Result of density measurements. Initial W/ S ratio is 0,7. V is total passed mixture volume, c is volumetric concentration of grains and V_p is passed volume of grains.

	Passed Amount	ρ	V	W/S	c	V_p
Mixture	[%]	[kg/m^3]	[ml]		[%]	[ml]
2	81	1570	72,2	0,79	31	22,4
3	11	1005	18,6	130	0,3	0,06
4	52	1600	51,9	0,73	33	17,1
5	18	1193	10,8	3,01	10	1,1
6	8	1053	12,9	11,8	3	0,6

Calculated W/S ratio (in Table 6.2) is in accordance with eq 5.5. Mixture number 3, 5 and 6 showed barely any passed volume of grains through the filter. The passed amount of mixture no. 3, 5 and 6 presented in Table 6.1, mainly consists of water, see Table 6.2.

Table 6.3, Result of rheology measurements. W/S ratio 0,7. No. corresponds to number of performed measurements and Dev. is the standard deviation of the measures. Cov. regards the coefficient of variance of the measured values.

	Yield value				Viscosity			
Mixture	Mean	No.	Dev.	Cov.	Mean	No.	Dev.	Cov.
	[Pa]				[Pas]			
2	1,95	8	0,46	0,24	0,087	8	0,011	0,13
3	1,3	3	0,52	0,4	0,002	3	0,002	1
4	10,35	4	2,8	0,27	0,255	4	0,071	0,28
5	2,19	4	0,95	0,43	0,139	4	0,011	0,08
6	0,68	3	0,17	0,25	0,029	3	0,006	0,21

A rather large difference in the yield and viscosity values can be seen between the different mixtures in Table 6.3. As can be seen from Table 6.3 the yield and viscosity values of mixture 2, 4 and 5 are relatively high compared to the other mixtures 3 and 6. A high yield and viscosity value would according to the Bingham model generate a shorter penetration length (smaller passed weight of mixture through the filter) compared to a mixture with a lower yield and viscosity value.

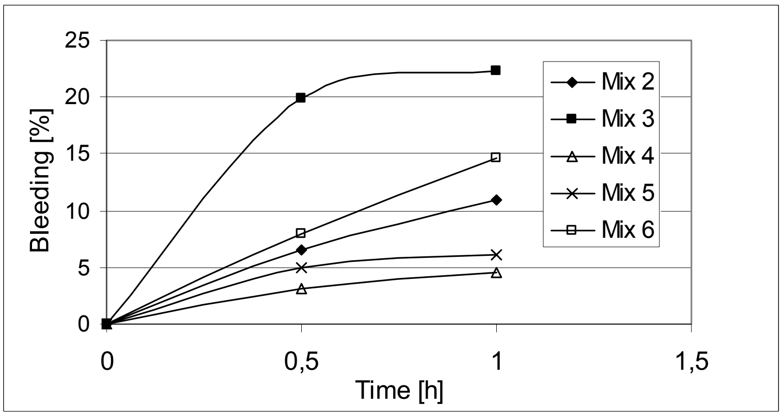


Figure 6.1, Result of bleeding measurements. W/ S ratio is 0,7.

A great difference can be seen when comparing the results from the bleeding tests of different mixtures. The bleeding tests are performed according to chapter 5. Mixtures containing coarser grains show a larger bleeding than mixtures containing smaller grains.

Experimental work has also been done to simulate the grain size distribution of the grouting cements (IC 30 and UF 12) with dolomite powder, see Figure 6.2.

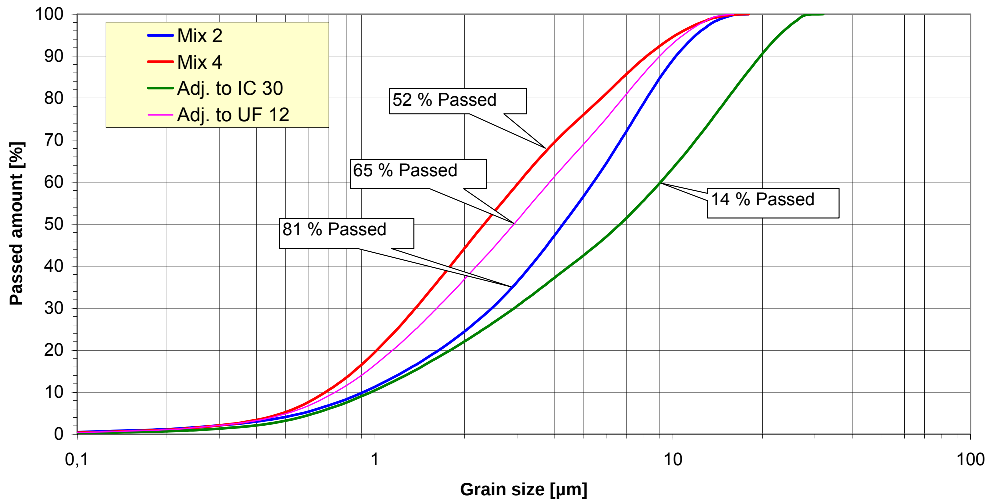


Figure 6.2, Result of filtration measurements. The mesh aperture was 45 µm and the mixtures had a W/S of 0,7.

As can be seen from Figure 6.2 and Table 6.4, a grain size distribution that consists of coarser grains (increased d_{95}) will generate a decreased passed amount of mixture. A steeper grain size distribution with the same value of d_{95} will increase the passed amount of mixture.

The correspondence between the simulated grain curve and grain curve of IC 30 is within a difference of ± 10 %. The simulation of UF 12 differs approximately by 10 to 20 % in the grain sizes range between 1-5 µm. The simulated grain curve is contains too small amount of grains between 1-5 µm. Three measurements each are performed with the grain curve adjusted to IC 30 and UF 12.

Table 6.4, Result of filtration measurements. Mesh aperture is 45 µm. W/S ratio is 0,7. No. corresponds to number of performed measurements and Dev. is the standard deviation of the measurements. Cov. regards the coefficient of variance of the measured values .

	Passed Amount			
Mixture	Mean	No.	Dev.	Cov.
	[%]			
Adj. to UF 12	65	3	6.3	0,09
Adj. to IC 30	14	3	5,5	0,39

Table 6.5, Result of density measurements. Initial W/ S ratio is 0,7. V is the total passed mixture volume, c is the volumetric concentration of grains and V_p is the passed volume of grains.

	ρ	**V**	**W/S**	**c**	**V_p**
Mixture	(kg/m^3)	[ml]		[%]	[ml]
Adj. to UF 12	1600	70,8	0,79	31	21,8
Adj. to IC 30	1067	22,3	9,39	4	0,8

The quality (concentration of grains after filtration) of the passed mixture of the coarser mixture (Adj. to IC 30) show that the passed mixture consists of a low concentration of grains compared to the mixture containing smaller grains (Adj. to UF 12).

Table 6.6, Result of rheology measurements. W/S ratio 0,7. No. corresponds to number of performed measurements and Dev. is the standard deviation of the measures.

	Yield value			**Viscosity**		
Mixture	Mean	No.	Dev.	Mean	No.	Dev.
	[Pa]			[Pas]		
Adj. to UF 12	5,9	3	0,41	0,17	3	0,008
Adj. to IC 30	1,3	3	0,55	0,04	3	0,010

A difference in rheology can be seen between the two mixtures in Table 6.2. Although the mixture based upon Adj. to UF 12, has a yield value which is more than four times greater than that of the other mixture, the passed amount (weight) of mixture is larger for the fine grained mixture (Adj. to UF 12) than the coarser one (Adj. to IC 30).

6.2.1.1 Summary and discussion (grain size and grain size distribution)

A relatively small change in the mixtures grain size and grain size distribution, affects the amount passed through the filter. The conclusions can be summarised as follows:

- Based upon the performed tests it can be seen that the grain size that corresponds to d_{95}, is of importance for the filtration tendency of the mixture. The coarser mixtures like 3, 5, 6 and Adj. to IC 30 with a d_{95} which are larger than 24 µm, show a higher filtration tendency compared to the fine grained mixtures of 2, 4 and Adj. to UF 12, with a d_{95} of less than 12µm. The coarser grains in the mixture can be shown to increase the filtration tendency of the mixture.

- The grain size of d_{95} should according to the tests be smaller than a certain value, approximately not less than 3-4 times smaller than the mesh aperture in order to have a low filtration tendency. This seems to be fairly valid for mixtures like 2, 4 and Adj. to UF 12. The relation between the grain size of d_{95} and the used mesh aperture can be seen to be 45µm/ 12µm for mixture 2 and Adj. to UF 12 respectively 45µm/10µm for mixture 4.

- When the mixtures contain grains with the same d_{95} value, the slope of the grain curve (between d_{95} and the small grains) will be of importance. A relatively steep grain size distribution seems to be desired, in order to achieve a low filtration tendency of the mixture.

- As earlier mentioned in chapter 1, low filtration tendency is both a question of the quantity of passed amount (weight) and the quality of the passed amount. Bad quality of the passed mixture is achieved when a large difference in W/S ratio can be seen before and after filtration of the mixture. Table 6.2 indicates that a significant filtration of grains occurs in certain mixtures like no. 3, 5 and 6. The W/S ratios for these passed amounts of mixture vary between 3 and 130. Mixtures like no. 2 and 4 indicate a more or less unaffected W/S ratio in the passed amount. The initial used W/S ratio is 0,7 in the mixtures.

- Some uncertainties are associated to all of the above statements because of the small and limited amounts (volumes) that are used in the tests. Some results could therefore be different than presented in this work.

6.2.2 Influence of grain concentration

The influence of varied grain concentration is investigated by varying the W/S ratio of the mixtures. The mesh aperture is set to 45 μm.

Table 6.7, Result of filtration measurements. Mesh aperture is 45 μm. No. corresponds to number of performed measurements and Dev. is the standard deviation of the measured quantities.

W/ S ratio	0,6			0,7			0,8			1,4		
Mixture	Passed	No.	Dev.		No.	Dev.		No.	Dev.		No.	Dev.
	Mean	No.	Dev.	Mean	No.	Dev.	Mean	No.	Dev.	Mean	No.	Dev.
	[%]			[%]			[%]			[%]		
1	-			-			63	1		-		
2	60	3	5,2	81	8	5,7	86	1		99	2	
3	11	3	2,9	11	3	3,1	8	1		-		
4	29	3	6,1	52	4	7,7	81	1		97	2	
5	-			18	4	6,2	14	1		-		
6	-			8	3	1,4	16	1		-		

As can be seen in Table 6.7 some of the mixtures are more influenced (larger or smaller passed amounts) than others, by varying the W/S ratio. The passed amounts of the mixtures with coarser grain sizes (larger d_{95}) like no. 3, 5 and 6 are not that influenced by varying the W/S ratio compared to mixtures like no. 2 and 4. The passed amounts of the mixtures with coarser grains (mixture 3, 5 and 6) mainly consisted of water, see Table 6.2.

A steeper grain size distribution seems to cause a larger passed amount at low W/S ratios. Mixture 2 has a steeper grain size distribution than mixture 4, significantly larger passed amounts of mixture are achieved at W/S ratios of for example 0,6 and 0,7.

Table 6.8, Result of rheology measurements. Mixture 2. No. corresponds to number of performed measurements and Dev. is the standard deviation of the measures.

W/ S ratio	Yield value			Viscosity		
	Mean	No.	Dev.	Mean	No.	Dev.
	[Pa]			[Pas]		
0,5	15,3	1	-	0,425	1	-
0,6	6,68	3	3,06	0,187	3	0,048
0,7	1,95	8	0,46	0,087	8	0,011
0,8	1,59	1	-	0,062	1	-
1,4	0,23	1	-	0,010	1	-

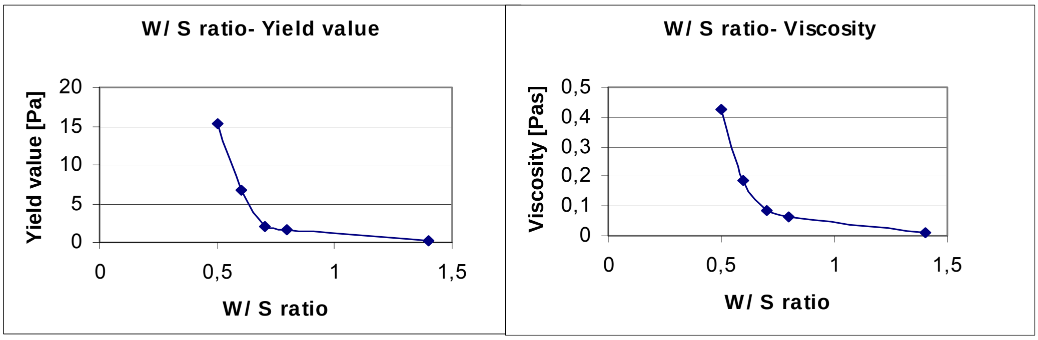


Figure 6.3, Influence of W/ S ratio on to the yield- and viscosity value for mix 2.

Table 6.8 and Figure 6.3 illustrates that the yield- and viscosity values rapidly increase when the W/S ratio approaches approximately 0,6.

Pictures have been taken by a SEM- microscopy to check if it is possible to identify any difference in concentration of grain sizes in the cross section of the filter cake. The magnification is 400x. As can be seen from Figure 6.5 no major difference can be identified. Although some amounts of larger grains or agglomerates are although identified in the part near the filter. This may be due to larger grains or agglomerates which initialise the plug (creation of an arch as described in chapter 3). This type of filtration is usually noted as cake filtration, Figure 6.4.

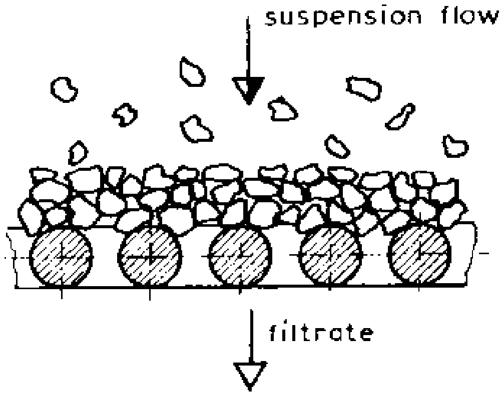
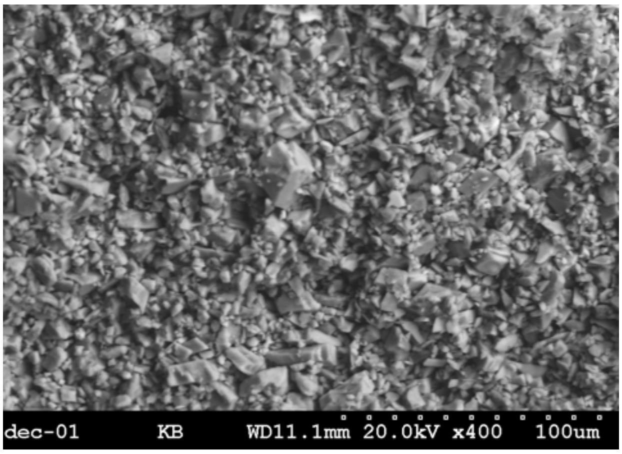


Figure 6.4, Cake filtration with a mesh filter.



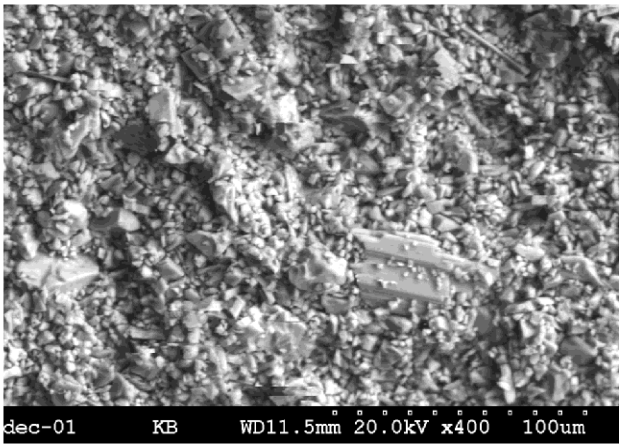


Figure 6.5, SEM- picture, cross section of a filter cake from mixture 4 (mesh filter, W/C ratio 0,7). Sample taken in the upper part of the cake (left), sample from the part near the mesh filter (right).

The SEM- picture in Figure 6.5 has only been taken for the tested mixture 4. The reason for studying the mixture 4 was that a plug formation occurred despite the fact that no filtration could be observed in the passed mixture (see Table 6.2). One explanation may be that the plug formation in this case might be initialised by some few oversized grains.

Variation of the grain concentration in the mixture resulted in the following variation in bleeding of the mixtures, see Figure 6.6.

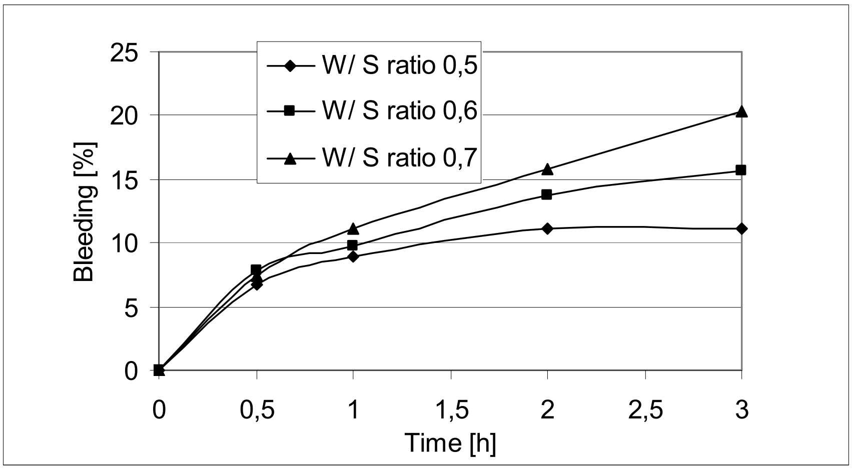


Figure 6.6, Result of bleeding measurements. Mixture 2.

Measurement of the bleeding has been made during a longer time (3 h) than for the case of using different grain size distributions (1 h, in Figure 6.1). The longer time of measurement is needed in order to analyse the influence of time on to the bleeding of a certain mixture (mixture 2) in a more detailed way.

6.2.2.1 Summary and discussion (grain concentration)

According to this work the mixture is influenced in different ways due to changes in W/S ratio. The magnitude of the influence of a change in W/S ratio, is to a large extent dependent on the grain size distribution in the mixture.

- If the value of d_{95} is small enough compared to the used mesh aperture (d_{95} is not less than 3-4 times smaller than the mesh aperture), the passed amount will generally increase if the used W/S ratio increases. As an example, this can be seen from the lowered filtration tendency of mixture 2 and 4 when the W/S ratio is increased. The passed amounts of the coarser mixtures 3, 5 and 6 are not that influenced by an increased W/S ratio.

- The influence of a steep grain size distribution is most significant at low W/S ratios. As an example, it can be noticed that mixture 2 has a steeper grain size distribution compared to mixture 4. Mixture 2 shows a larger passed amount at lower W/S ratios compared to mixture 4.

- At a low W/S ratio the grains are to a larger extent in contact with each other compared to the case of a higher W/S ratio. If the grains are in contact with each other the possibilities for the grains to rearrange is limited. A steep grain size distribution will probably make it easier to rearrange the grains than in a wider grain size distribution. The ability for the grains to more easily rearrange within the mixture is probably of importance to avoid plug formation at the entrance of the aperture.

- The rheology is also to a large extent dependent on the W/S ratio. The experiments show that the viscosity and yield value approaches infinity when the W/S ratio goes below 0,5 to 0,6, see Figure 6.3. There is a difference in bleeding when changing the W/S ratio. A variation from 0,5 to 0,7 in W/S ratio causes a bleeding which is two times higher for mix 2 with a W/S of 0,7 compared to a W/S ratio of 0,5, Figure 6.6. The difference had probably been even larger if the measurement had been continued for a longer time after mixing (more than three hours).

6.2.3 Influence of mesh or slot geometry

The influence of mesh or slot geometry has been investigated by using different mesh and slot apertures in the filtration experiments. A relative passed amount of mixture between 75-100% can, in these presented experiments, be regarded as all of the available amount of mixture has passed the filter aperture. Plug formation could have occurred if a larger passed amount was used, consequently the relative passed amount could have been different.

Table 6.9, Result of filtration measurements. Different mesh apertures. No. corresponds to number of performed measurements and Dev. is the standard deviation of the measures. W/S ratio is 0,7.

Mesh aperture	36 μm			45 μm			75 μm		
	Passed			Passed			Passed		
	Mean	No.	Dev.	Mean	No.	Dev.	Mean	No.	Dev.
Mixture	[%]			[%]			[%]		
2	79	1	-	81	4	6,3	83	3	1,0
3	-	0	-	11	3	3,1	34	3	15,3
4	69	1	-	52	3	7,7	68	3	2,6
5	5	1	-	18	3	4,4	72	3	9,2
6	11	1	-	8	3	1,4	51	3	20,8

Table 6.9 indicates that the mesh aperture influences the passed amount of mixture in different ways. Some mixtures are strongly influenced by an increased mesh aperture (3, 5 and 6), while others like 2 and 4 are not that influenced. As can be seen from Table 6.9 (mixture 2, 36 μm aperture, mixture 4, 36 μm and mixture 5, 75 μm) the ratio between the aperture (36μm and 75 μm) and the used grain size of d_{95} (12, 10 and 24μm) has to be at least approximately 3 in order to pass nearly all (75-100%) of the available amount of mixture.

The mesh area has, in the experiments presented in Table 6.10 been reduced by a 5*65 mm opening in a steel plate, which was placed above the mesh. The relative passed amount of mixture was generally lowered when the mesh area was reduced, compared the mesh area in Table 6.9 and Table 6.10.

Table 6.10, Result of filtration measurements. Different meshes with reduced areas. No. corresponds to number of performed measurements and Dev. is the standard deviation of the measures. W/S ratio is 0,7.

Mesh/ slot	**45 µm**			**75 µm**		
aperture	**Passed**			**Passed**		
	Mean	No.	Dev.	Mean	No.	Dev.
Mixture	[%]			[%]		
2	72	2	-	81	2	-
3	0	2	-	15	2	-
4	32	2	-	51	2	-
5	17	2	-	56	2	-
6	11	2	-	42	2	-

The mesh has also been replaced by a slot area with different apertures in the results presented in Table 6.11.

Table 6.11, Result of filtration measurements. Different slot apertures. No. corresponds to number of performed measurements and Dev. is the standard deviation of the measures. W/S ratio is 0,7.

Slot aperture	**45 µm**			**75 µm**			**125 µm**		
	Passed			**Passed**			**Passed**		
	Mean	No.	Dev.	Mean	No.	Dev.	Mean	No.	Dev.
Mixture	[%]			[%]			[%]		
2	3	1	-	8	1	-	33	1	-
3	0	1	-	0	1	-	17	1	-
4	0	1	-	22	1	-	62	1	-
5	0	1	-	5	1	-	44	1	-
6	0	1	-	0	1	-	27	1	-

As can be seen in Table 6.11 the relative passed amount of mixture is significantly lower when using the slot aperture compared to the filtration experiments when using the mesh aperture. According to Table 6.11 the slot aperture has to be at least approximately 125 µm, in order to pass any major amounts of mixture. Comparative measurements have been made with the adjusted mixtures UF 12 and IC 30 with the slot aperture 125 µm and W/ S ratio 0,7, see Table 6.12.

It should be noted that the total filter areas in Table 6.9 to Table 6.11 are different which is why a direct comparison between the relative passed amounts not can be carried out.

Table 6.12, Result of filtration measurements. Slot aperture 125 µm. No. corresponds to number of performed measurements and Dev. is the standard deviation of the measures. W/S ratio 0,7.

Slot aperture	125 µm		
	Passed		
	Mean	No.	Dev.
Mixture	[%]		
Adj. to UF 12	51	1	-
Adj. to IC 30	12	1	-

The different passed amounts of mixture through the used filters can be analysed together with the area of the used filters, according to Table 6.13 and Table 6.14.

Table 6.13, Illustration of the passed amount through different filter areas. The initial mixture volume is approximately 110 cm^3. W/S ratio is 0,7.

	Slot aperture		Mesh/ slot aperture		Mesh aperture		
	75 µm	125 µm	45 µm	75 µm	36 µm	45 µm	75 µm
Filter area	0,05 cm^2	0,08 cm^2	1,04 cm^2	1,56 cm^2	8,3 cm^2	10,6 cm^2	13,9 cm^2
	Passed	Passed	Passed	Passed	Passed	Passed	Passed
Mixture	[%]	[%]	[%]	[%]	[%]	[%]	[%]
2	8	33	72	81	79	81	83
3	0	17	0	15	-	11	34
4	22	62	32	51	69	52	68
5	5	44	17	56	5	18	72
6	0	27	11	42	11	8	51

As can be seen from Table 6.13 an increased filter area is not always followed by an increased amount of passed mixture. Not surprisingly, the geometry of the filter aperture also influences the passed amount of mixture.

The geometry of the filter area seems therefore to be an important factor to analyse regarding filtration tendency. In order to be able to compare different passed amounts of mixtures from experiments performed with different filter geometries it can be suitable to analyse the penetration length (L) that corresponds to the passed amount (volume) of mixture. The penetration length considers the used filter area and the passed volume. Penetration length is consequently calculated as if the extension of the filter area creates the volume that is supposed to be filled, see eq 6.1.

The filter area is designated an area (A) and varies between 0,05- 13,9 cm^2, passed volume of mixture is (V). The penetration length (L) is calculated as :

eq 6.1

$$L = \frac{V}{A}$$

As can be seen in Table 6.14 the quotient (passed volume/ filter area) is larger in the case of using a slot aperture compared to the case using a mesh aperture. The result can be expressed as a larger amount of grains per filter area that pass the slot aperture than pass the mesh aperture. The quotient (passed volume/ filter area) is consequently equal to the presented penetrability length in eq 6.1.

The relative passed amounts (weights) in Table 6.13 is in Table 6.14 presented as the penetrated lengths. The passed volume (V) used in eq 6.1 has been calculated by the use of measured densities of the mixture after their passage through the used filter, see appendix G. Some of the penetrability lengths in Table 6.14 are not presented, because of the density of the mixtures density was not measured (-).

Table 6.14, Illustration of the quotient (penetration length) between passed volume [cm^3] of mixture and filter area [µm]. The initial mixture volume is approximately 110 cm^3. W/S ratio is 0,7. Relative passed amounts see Table 6.13.

Penetration length							
	Slot aperture		Mesh/ slot aperture		Mesh aperture		
	75 µm	125 µm	45 µm	75 µm	36 µm	45 µm	75 µm
Filter area	0,05 cm^2	0,08 cm^2	1,04 cm^2	1,56 cm^2	8,3 cm^2	10,6 cm^2	13,9 cm^2
	Passed volume/ filter area	Passed volume/ filter area	Passed volume/ filter area	Passed volume/ filter area	Passed volume/ filter area	Passed volume/ filter area	Passed volume/ filter area
Mixture	[m]	[m]	[m]	[m]	[m]	[m]	[m]
2	1,94	4,68	0,78	0,56*	-	0,09*	0,07*
3	0,00	2,79	0	0,15	-	-	-
4	4,79	8,38	0,33	0,35	-	0,05	0,05
5	1,10	6,07	0,23	0,41	-	-	0,06
6	0,00	3,86	0,17	0,36	-	-	-

Penetration lengths marked with an asterisk* are not applicable because no plug formation occurred. As earlier mentioned, experiments that have resulted in a passed amount between 75-100% can not be included in the analysis. If a larger amount of mixture had been available (for the penetration lengths marked with*), the penetration length could have been different.

As can be seen from Table 6.14, the penetration length is significantly larger in the case using the slot aperture compared to the case using a mesh aperture with regard to mixture 4 and 5. Plug formation has occurred both for mixture 4 and 5, regarding both mesh and slot geometry.

The tests indicate that more mixture passes per unit filter area for the slot compared with the mesh. An explanation may be that the geometry of the opening is of importance but it may also depend on the equipment.

The used filter geometries (mesh and slot) can be compared to the different used d_{95} values of each mixture, see Table 6.15 and Table 6.16.

Table 6.15, Relationship (quotient) between the used mesh apertures and the grain size (d_{95}). Quotients that are shaded are the ones that show a relative passed amount between 75-100% (b_{All}).

		mesh aperture		
Mixture	**d_{95} [µm]**	**36 µm**	**45 µm**	**75 µm**
2	12	≥ 3,0	3,8	6,3
3	28	2,3	1,6	2,7
4	10	≥ 3,6	4,5	7,5
5	24	1,5	1,9	≥ 3,1
6	26	1,4	1,7	2,9

This result indicates that the grain size expressed as d_{95} compared to the mesh aperture, is of importance for the filtration tendency of the tested mixtures. According to Table 6.15 the quotient should be approximately at least 3 in order to pass a larger amount of mixture through the used mesh aperture.

Table 6.16, Relation (quotient) between the used slot apertures and the grain size (d_{95}). Quotients that are shaded are the ones that show a relative passed amount between 75-100% (b_{All}).

		slot aperture	
Mixture	**d_{95} [µm]**	**75 µm**	**125 µm**
2	12	6,3	10,4
3	28	2,7	4,5
4	10	7,5	12,5*
5	24	3,1	5,2
6	26	2,9	4,8

None of the tested slot apertures or mixtures generated a passed amount of mixture between 75-100%. According to Table 6.16 the quotient should be larger than approximately 12,5* in order to pass a larger amount of mixture through the used slot aperture.

It must be kept in mind that Table 6.15 and Table 6.16 can not be directly compared with each other, because of the different filter areas and the same used available amount of mixture (110 cm^3). All that can be said of the result in Table 6.16 and Table 6.15 is that a larger amount of grains per filter area have passed the slot aperture when compared to the mesh aperture.

The larger passed amount of grains per filter area in the slot aperture cause that a larger quotient is required to be used in order to pass the same amount (75-100%) of mixture through the slot as in the mesh aperture. This is probably due to the stochastic process of plug formation. The probability of a plug formation occurring during the filtration experiment will probably increase as an increased amount of mixture passes the filter. Agglomerates or larger grains in the mixture may initialize the plug at the entrance of the aperture.

6.2.3.1 Summary and discussion (mesh or slot geometry)

The performed tests indicate that the choive between a mesh and slot aperture is a critical parameter for the passed amount of mixture through the used filter.

- What is noticeable when looking at the filtration tests in Table 6.13 is that mixture 2 generally shows a passed amount of approximately 75-100%, except for the case of using slot geometry. The relative passed amount of mixture 2 could consequently been different if a larger passed amount of mixture had been available. Mixture 4 show a larger passed amount when using the slot geometry compared to mixture 2 (test with a slot geometry and mixture 4 is just performed as a single test).

- According to Table 6.9 and Table 6.11, it seems that a difference can be seen between the relative passed amount of mixture through the mesh and the slot filter area. One explanation maybe due to the different filter areas.

- A direct comparison when using a slot aperture in the filtration tests, show that the slot has to be approximately 125 µm before a relative passed amount (> 50 %) of mixture shall pass the filter. When using a mesh aperture, the slot has to be approximately 45 µm before a relative passed amount (> 50 %) of mixture shall pass the filter.

- In order to be able to compare the results (passed amounts) from the mesh and slot apertures, the penetrability length (passed volume of mixture/ filter area) has been calculated. The calculation of the penetrability length regarding the slot and mesh aperture of 75 µm, resulted in 20-100 times larger penetration lengths for the slot aperture (as regards mixture 5 and 4). The result can also be expressed as nearly 20-100 times more mixture volume/ filter area passes the slot aperture compared to the mesh aperture.

- The filtration tendency can probably be seen as a stochastic process, where a relatively large amount of mixture should be available (used initial amount of mixture) to pass the used aperture. The probability of plug formation during the filtration experiment will probably increase as an increased amount of mixture passes the filter, see Figure 6.7. Agglomerates or larger grains in the mixture can initialize the plug at the entrance of the aperture. The stochastic behaviour of the plug formation can probably not easily be predicted.

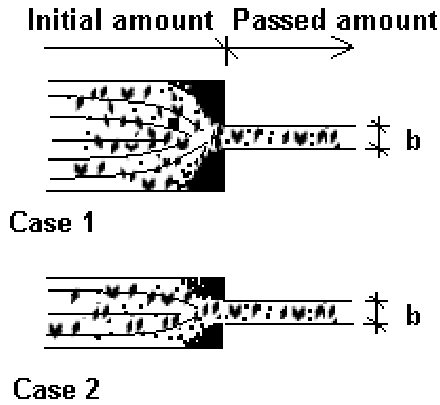


Figure 6.7, The initial amount of mixture that is supposed to pass the slot aperture of b, is probably of importance for the plug formation. Case 1 and Case 2 describes a larger and a smaller initial amount of mixture.

- The densities of the passed amount of mixture seem to be slightly higher in the case when using a mesh aperture (Case 2) compared to that of a slot geometry (Case 1), see appendix F.

- According to the performed tests, as regards mixture 4 and 5 in Table 6.14, the slot generates a larger penetration length compared to the mesh geometry. This can be caused by differences in geometrical design and build up of grains at the entrance of the slot and mesh aperture. In the case of using the mesh, a plug of grains can be built-up in four directions around the aperture compared to the case of using a slot aperture the build-up of grains can only be done in two directions, see Figure 6.8. The abutment for plug formation against the wires (mesh) can also be better compared to the edges of the slot aperture. Better abutment can create a more stable plug formation.

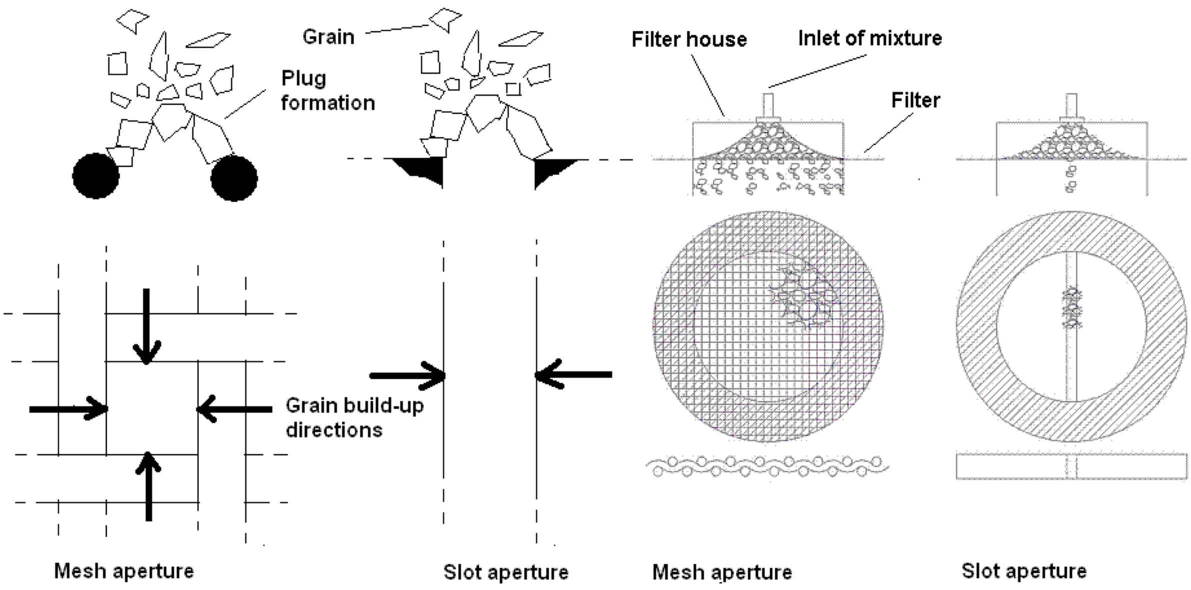


Figure 6.8, Build-up of grains around a mesh and slot aperture.

6.2.4 Evaluation of measured values

A number of statistical correlations have been made for the purpose of analysing correlations between filtration tendency and grain sizes, grain size distributions, grain concentrations and filter geometries. It can be suspected from the results presented in this chapter that the passed amount of mixture through the aperture is a question of a combination of parameters like grain size, grain size distribution, grain concentration in the mixture and used filter aperture. In this chapter, the performed correlations do not take into considerations to combine the influences of different parameters. Just one parameter is studied against the passed amount of mixture in each correlation.

The performed correlations will make it probable that a combined influence of different parameters onto the passed amount is present, because of the generally bad achieved correlations in Figure 6.9 to Figure 6.11. However, some tendencies can be stated for the correlations between different parameters.

The Figure 6.9 to Figure 6.11 illustrates the correlations that were found to be of further interest in this work. The different dots of d_{95} and d´ in Figure 6.9 to Figure 6.11 represent the five mixtures of 2, 3, 4, 5 and 6.

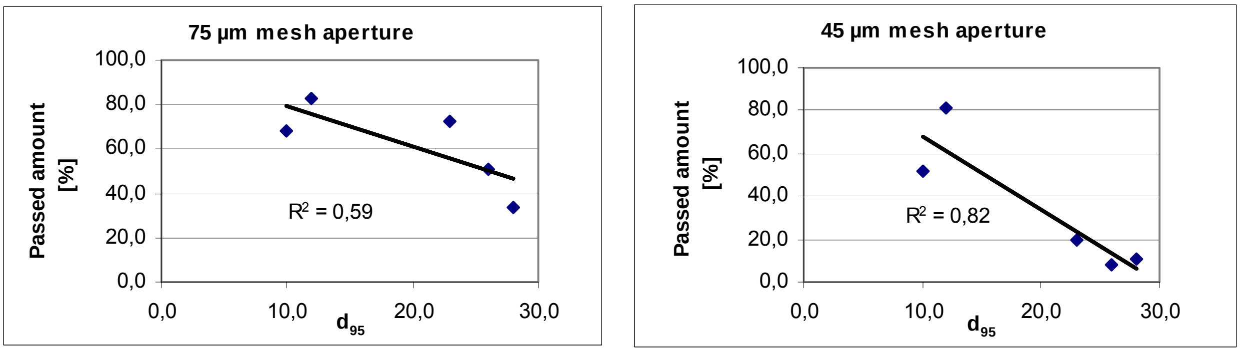


Figure 6.9, Relation between passed amount of mixture and d_{95} (mixture 2-6). W/S ratio 0,7.

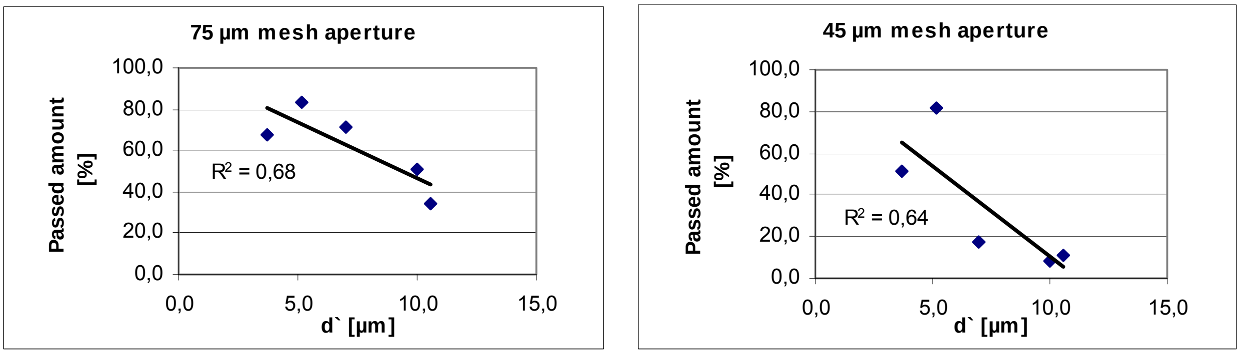


Figure 6.10, Relationship between passed amount of mixture and d` of grain size distribution in the mixture (mixture 2-6). W/S ratio 0,7.

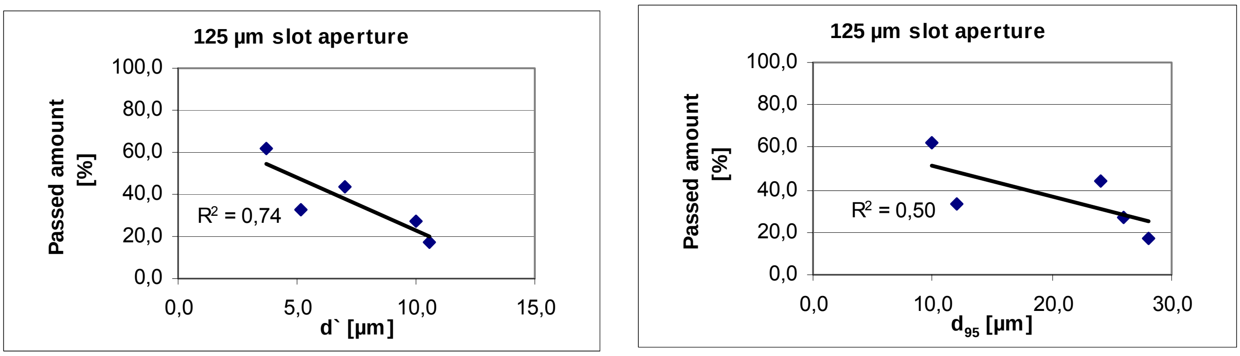
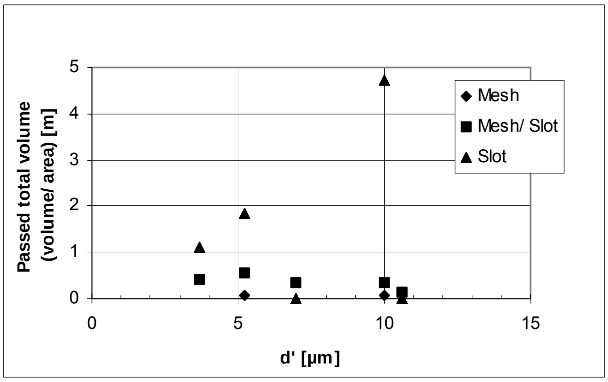


Figure 6.11, Relationship between passed amount of suspension and d` of the grain size distribution in the mixture (mixture 2-6). W/S ratio 0,7.

As been mentioned earlier in the text about Table 6.11, the passed amount of mixture through the slot aperture of 75 µm was so small that correlation analysis was useless to perform.

The passed amount of mixture through a mesh filter compared to a slot filter is as mentioned earlier in this work of importance to study. As mentioned earlier, three types of filters were used, mesh, mesh with a delimited steel plate and slot geometry. A difference in the relative passed amount of mixture was seen between the two mesh filters. A difference in the relative passed amount of mixture was also observed between the mesh filters and the slot filter. The results presented in Figure 6.12 to Figure 6.13 are the passed **total volume/ filter area** and the **grain volume/ filter area**.



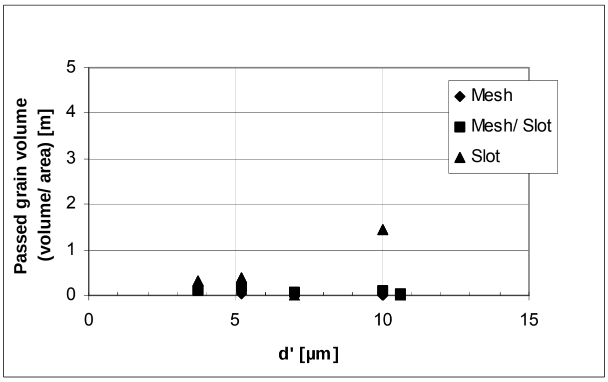
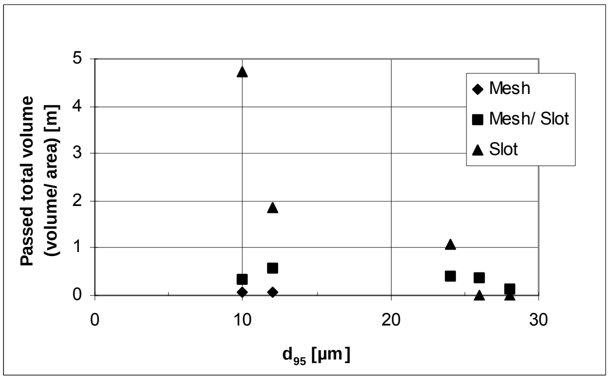


Figure 6.12, Left figure, relation between total passed volume per filter unit area and d´. Right figure, relation between passed volume of grains per filter area and d´. W/S ratio 0,7 and filter aperture 75 µm.



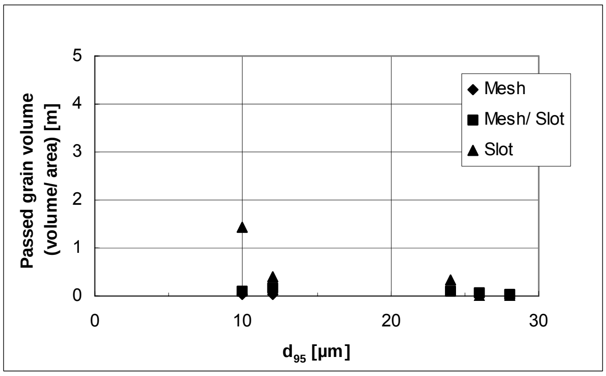


Figure 6.13, Left figure, relationship between total passed volume per filter unit area and d_{95}. Right figure, relationship between passed volume of grains per filter area and d_{95} W/S ratio 0,7 and filter aperture 75 µm.

Figure 6.12 to Figure 6.13 shows that the passed amount of grain volume is lower than the total passed amount of mixture, which is quite natural. Some of the passed volume of mixture consists of pure water. As can be seen in the left figures in Table 6.11 and Table 6.12, a larger passed amount of total mixture volume is obtained with the slot aperture compared to the mesh aperture.

When looking at the right figures in Table 6.11 and Table 6.12 it can be seen that there also seems to be a larger passed amount of grain volume passing the slot aperture compared to the mesh aperture.

The stochastic plug formation could probably be seen as an increased coefficient of variance (Cov) of the passed amount of mixture through the filter (mesh), see Table 6.1. The coefficient of variance is increasing, when the relative passed amount is decreasing, see Figure 6.14

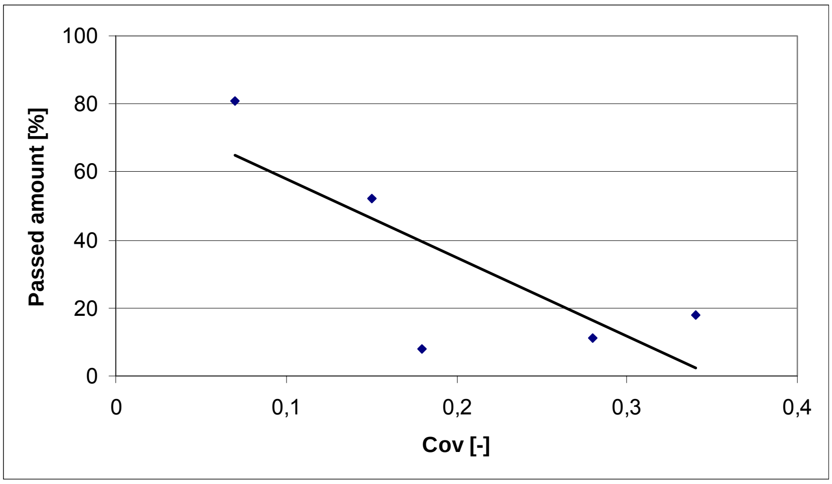


Figure 6.14, Passed amount of mixture as a function of the coefficient of variance.

6.2.4.1 Summary and discussion (evaluation of measured values)

Generally, few of the performed correlations tests show any high values of correlation, it is probably of interest to achieve correlation values in the region of 0,8 to 0,9, this is in order to determine the influence of a single parameter on the filtration tendency of the mixture. The bad correlation of the single parameters is probably due to the fact a combination of the investigated parameters that will influence the passed amount of mixture through the used filter.

- According to preformed correlation tests (Figure 6.9 and Figure 6.10) it can be identified that a decreased d_{95} and d´ are followed by an increased passed amount.

- As can be seen from Figure 6.9 and Figure 6.10, the passed amount of mixture is more influenced by changes in the grain size and grain size distribution when using a small mesh aperture compared to a wider mesh aperture. The slope of the line of the best fit is steeper in the case of smaller mesh apertures.

- According to Figure 6.12 and Figure 6.13, there is a difference between the three different filter types in terms of passed mixture volume per filter area. If a larger volume of mixture had been available, it is probable to believe that the difference had been a smaller compared to the performed tests. This assumption is based upon the assumption that a larger filter area will need a larger amount than a smaller filter area, in order to show the same relative passed amount of mixture.

- Larger filter area and smaller amount of mixture to be passed through the filter will decrease the influence of stochastic plug formation.

6.2.5 Physical model

The results of the filtration experiments in the physical model show that different grain concentration, slot aperture and grain shape in some cases generates a plug formation and sometimes not. The geometry and grains are scaled-up 100 times by using an oil with 100 times larger viscosity than the viscosity of water. The basic idea of the scale-up is to make the phenomena of penetrability visible.

To achieve a constant sedimentation velocity it is suitable to use the Reynolds and Archimedes number to describe the relationship between the grain sedimentation velocity and the liquid viscosity. The resulting sedimentation velocity in the scaled-up experiment is supposed to be the same as in the case of grains in a real grout suspension.

The boxes that are empty (no text) are test cases that were not performed. In the experiment the volume concentrations of grains varied between 5-20%. This can be compared to a W/S ratio of between 6,1- 1,3. The first row and column of the table represents the grain volume fraction and the crack aperture respectively.

If a plug formation occurs, see Figure 6.15, at some part of the crack, it is indicated by a "Yes" at the relevant position in the Table 6.17. On the other hand, if the grains pass through the crack without a plug forming it is denoted by a "No" in the table. The superscript indicates which kind of grains that were used in the experiment, where "w" and "b" stands for white and black grains respectively. The subscript (1 and 2) indicates in which try the plug formation occurred.

Table 6.17, Results from the filtration experiments in the physical model.

Volumetric concentration of grains [%]

Slot aperture [mm]	20%	15%	10%	5%
10 mm	No^w			
9 mm				
8 mm	No^b	No^b		
7 mm	Yes_1^b ; Yes_2^b	Yes_2^w ; Yes_2^w	Yes_1^w	No^w
6 mm				Yes_1^w
5 mm			Yes_1^w	Yes_2^w

If the plug formation appeared in the first test, the second test is not performed. If no plug formation occurred in the first test, the second test is performed. The experiments related to the slot aperture of 7mm and the volumetric concentration of 20 and 15% are performed twice.

However it should be kept in mind that the outcome of one test with a given set of initial conditions can in one test give a plug formation and in the next test the same experiment with "the same" set of initial conditions[3] can render a result that is different. Hence, it would have been desirable to do each experiment with the same initial conditions many times. On the other hand, when the parameters reach their critical ranges (as for example when the grain size is close to the slot aperture or the volumetric concentration of grains approaches its maximum for the certain grain size distribution) the outcome of each test can be highly random.

The initialisation of the plug at the entrance of the slot can be seen to grow in the longitudinal direction of the slot. The initialisation starts at several different points at the same time, see Figure 6.15.

Exactly at which location/locations the initialisation starts, seem to be random. The small bridges then merge to form larger bridges, while the filtration continuous. After a while the small bridges have grown together and no more grains are able to pass the slot.

[3] It is of course not possible to have exactly the same initial conditions in two independent tries. "The same" initial conditions in this context refers to the same particle concentration in the mixture that is poured into the model.

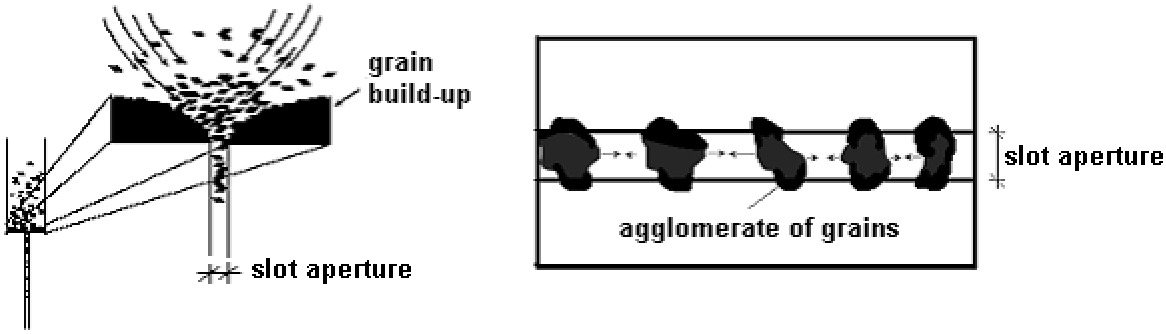


Figure 6.15, Initialisation of the plug at the entrance of the slot. The left figure shows the entire physical model and the built-up of grains around the slot entrance. The right figure shows the initialisation of plug formation at the slot entrance.

No difference in plug formation could be seen due to the performed tests with different grain shapes (rounded and more angular grains).

The conclusions of the performed experiments can be summarized in:

- The critical slot apertures seem to be 7 – 8 mm, which is 2 – 3 times the maximal grain size. The critical slot aperture is a bit smaller (6-7 mm) when using a lower grain concentration (higher W/S ratio) of approximately 5 %.

- The plug formation procedure is probably influenced by a stochastic phenomenon. The time for the plug to occur varies in the different tries. The position where the plug initialization occurs also varies in the different tries.

- No differences of the passed amount of mixture were observed through the slot due to the different shape of the grains.

6.2.6 Numerical model

Three different initial volumetric concentrations (10, 20 and 30%) were used in the numerical model. Time dependent simulations from 0-120 s have been performed. The different colors in Figure 6.16- Figure 6.18 indicate different grain concentrations at different places in the numerical model. The simulated slot aperture is 10 mm. The red color corresponds to a volumetric grain concentration of approximately 50%. A volumetric grain concentration of 50 % in a real grout mixture means that the grains are in more or less constant contact with each other.It can by mentioned that a monodisper mixture (all grains of the same size) and with spherical grain shapes, can, as a maximum, achieve a volumetric grain concentration of approximately 74% (according to the FCC-model, see chapter 3, eq 1.18). The grains are then arranged in a way that no more grain volume per mixture volume can be achieved at a certain geometry. The maximum grain concentration in the numerical model was set to 63%.

Plug formation can probably occur at lower volumetric concentrations than 74 %, probably even lower than 50 %. The critical volumetric concentration for plug formation (near a constriction) is probably dependent on the; flow velocity of the grains near the constriction, grain size, grain size distribution and grain shape in the mixture. It should be kept in mind that the performed calculations (Figure 6.16 to Figure 6.21) are Eulerian simulations and the purpose is to illustrate the possibilities of predicting the risk of plug formation via concentration and velocity criteria in a continuum model.

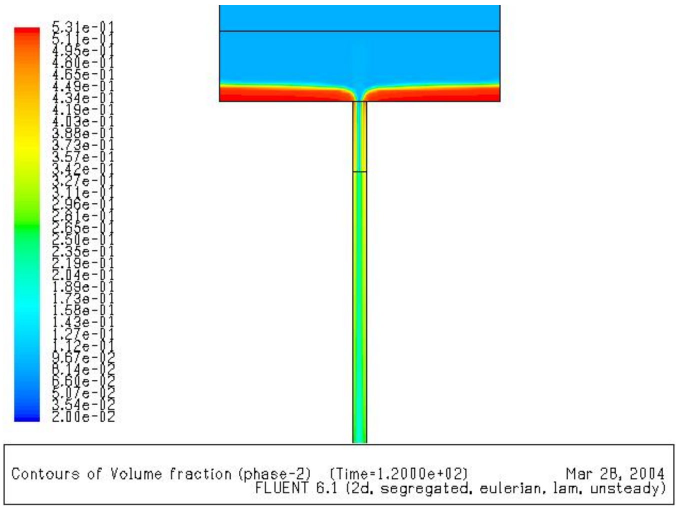


Figure 6.16, Results, c=10 %, 10 mm, t=120 s.

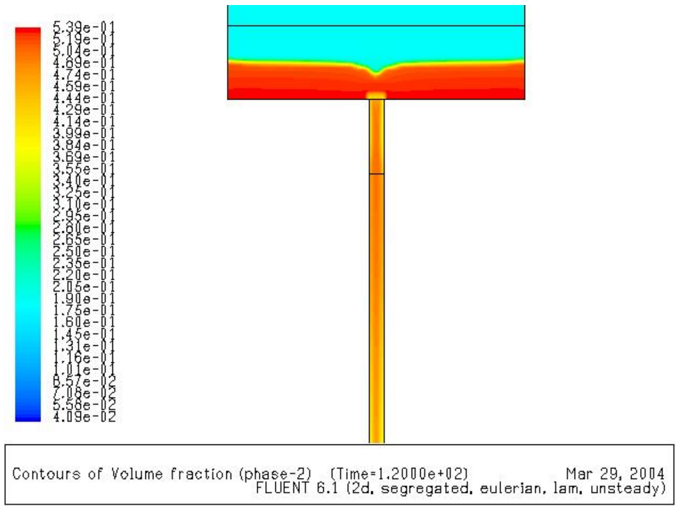


Figure 6.17, Results, c=20 %, 10 mm, t=120 s

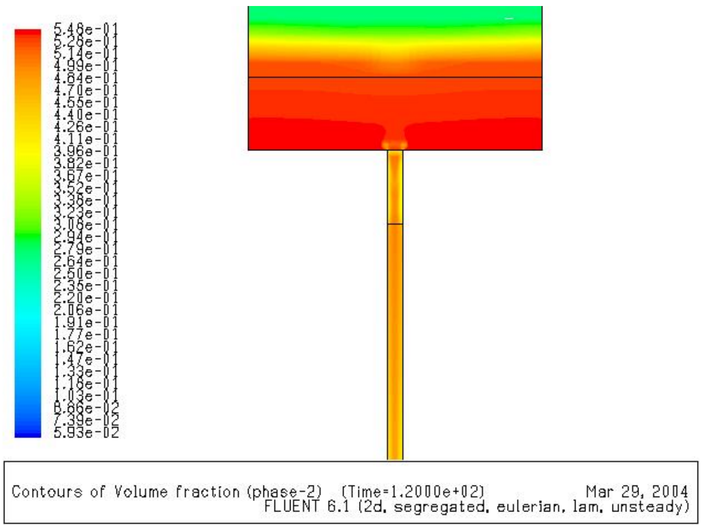


Figure 6.18, Results, c=30 %, 10 mm, t=120 s.

Figure 6.16 to Figure 6.18 illustrate that a higher initial grain concentration makes the grain concentration near the slot entrance increase, also see appendix1.

Figure 6.16 to Figure 6.21 show the concentration and velocity vectors of the grains at the lower part of the model near the slot entrance.

The different colors and length of the arrows in Figure 6.19 indicate the velocity of the grains in the mixture, red color corresponds to a velocity of approximately 0,04 m/s.

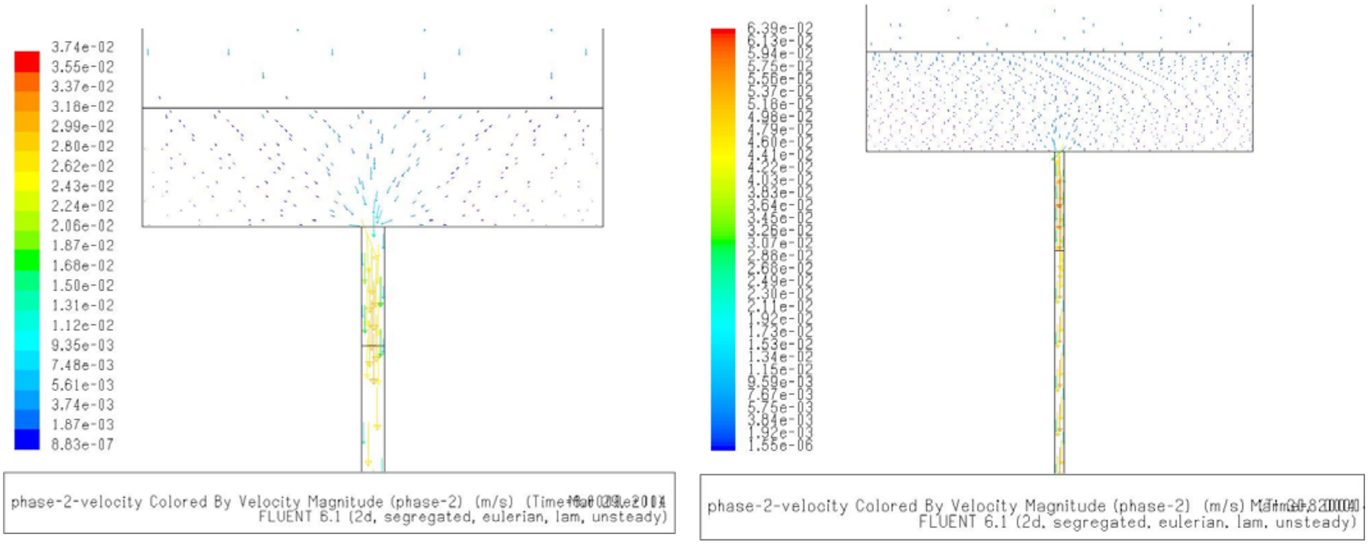


Figure 6.19, To the left, c=30 %, slot aperture 10 mm, t=120 s. The right picture represents a 5 mm wide slot and the same initial grain concentration and time of simulation (t=120 s).

As seen in Figure 6.19, the velocity of the grains is much higher in the simulation with the 10 mm wide slot entrance case than in the 5 mm wide slot entrance case. The different colors in Figure 6.20 indicate different grain concentrations at different places in the numerical model. Once again the red color corresponds to a volumetric grain concentration of approximately 50%.

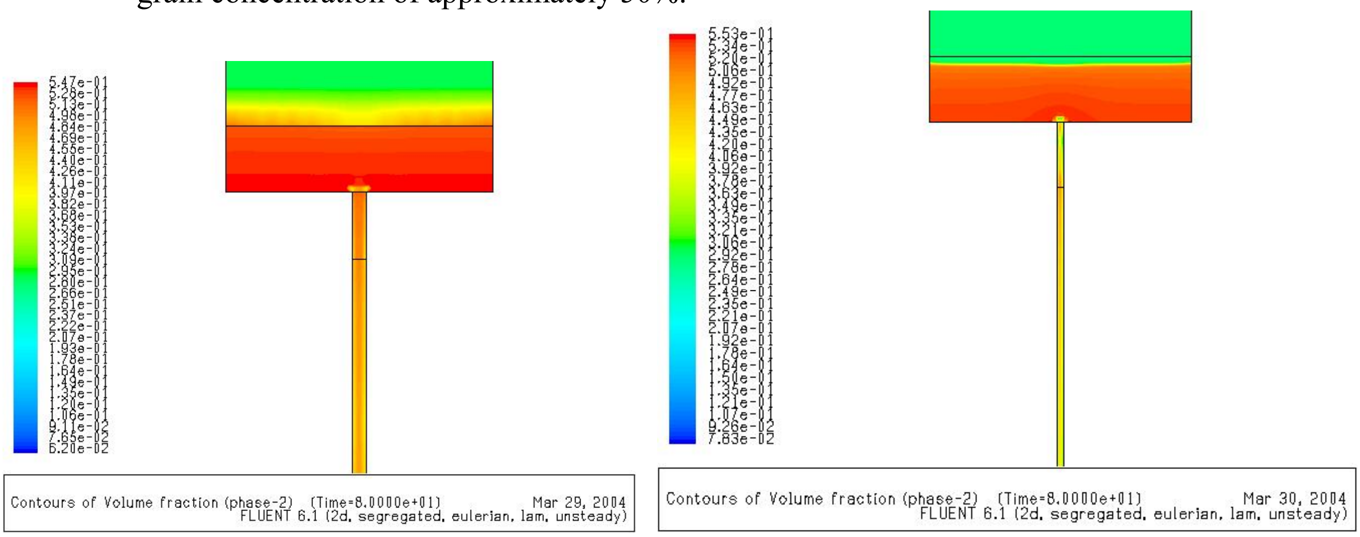


Figure 6.20, To the left, c=30 %, slot aperture 10 mm, t=120 s. The right picture represents a 5 mm wide slot and the same initial grain concentration and time of simulation (t=120 s).

As seen in Figure 6.19 and Figure 6.20, the grain concentration near the slot entrance is almost the same in both cases (slot aperture 10 and 5 mm).

The difference between the two tests is that the velocity of the grains in the 10 mm slot entrance is higher than in the 5 mm case, see Figure 6.19.

A higher grain velocity means that it is likely to believe that there is a minor risk for plug formation than in the case with a lower grain velocity near the slot entrance. If the mass flow of grains near the slot entrance is lower than the inlet grain mass flow, an accumulation of grains occurs near the slot entrance (grain build-up, see Figure 6.15).

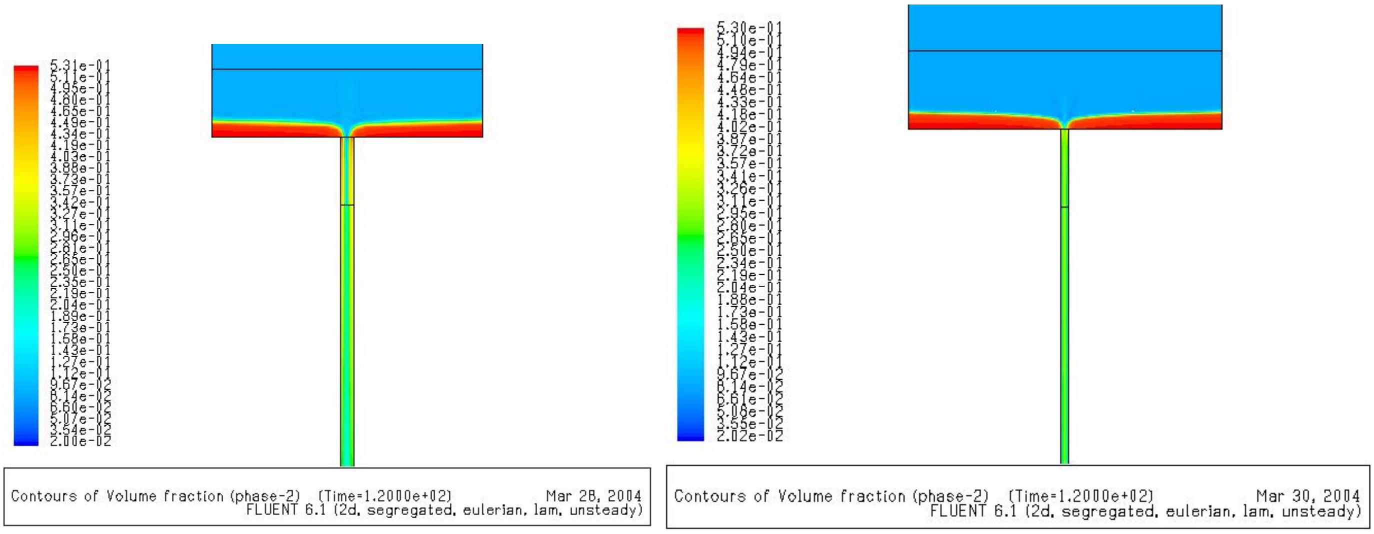


Figure 6.21, To the left, c=10 %, slot aperture 10 mm, t=120 s. The right picture represents a 5 mm wide slot and the same initial grain concentration and time of simulation (t=120 s).

Figure 6.21 illustrates that when the grain concentration is lowered to c=10% instead of 30 %, it makes the grain concentration near the slot entrance lower. Moreover it can be seen that there is no major difference between the two slot apertures (10 and 5 mm) when the grain concentration (c) is 10%. The conclusions of the performed numerical simulations can be summarized as:

- No real similarities between the numerical simulations and physical experiments were found (filtration and scaled-up experiments). Hence, further development of the numerical models is necessary. A higher initial concentration will, however, cause a higher concentration (red color) near the slot entrance. Smaller slot aperture will also cause higher concentration near the slot entrance.

- It is believed according to the filtration and scaled-up experiments that plug formation occur when the surface distance between the grains gets small (high volumetric grain concentration, which is indicated by the red color in the above figures). When the surface distance gets small, the friction between the grains will increase. It can be concluded that the frictional model used in the numerical simulation is of great importance for the plug formation. The frictional model has to be developed in order to simulate the plug formation.

- The approaches of multiphase flow simulations can be a fruitful way of dealing with the phenomena of plug formation. High concentration of grains and low flow velocities near the entrance of the slot can be an indication of a plug starting to form. When time dependent simulations of plug formation are performed one of the major problems is to set a limiting value for when the grains have formed a plug. In a frictional model representative of the real application the frictional forces between the grains should become very large. The velocity of the grain phase will thus become successively smaller until a limiting value is reached where problems due to numerical accuracy start to become an issue. Therefore, it is of importance to set a limiting value on the velocity, i.e., when the velocity through the crack is so small that a plug can be considered to have formed.

6.3 Comprehensive conclusions inert material

Low filtration tendency of the mixture is not equal to a large passed amount through the used filter (mesh or slot). The content in the passed amount of mixture has to be analysed. It is desirable to have the same grain concentration in the passed mixture amount as in the initial amount. The analysis of the relative passed amounts can only consider the mixtures where plug formation has occurred during the filtration test (comparison to other mixtures or filter geometries).

6.3.1 Influence of grain size and grain size distribution

The tests show that the grain size and grain size distribution are of great importance for the filtration tendency.

- Based upon the performed tests it can be stated that the grain size that corresponds to d_{95}, is of importance for the filtration tendency of the mixture. The grain size of d_{95} should be smaller than a certain value, approximately not less than 3-4 times smaller than the used aperture. This confirms the old rule of a thumb that says that the aperture has to be 3-4 times larger than the d_{95} value of the grains.

- According to performed experiments with inert material, it seems to be advantageous for the penetrability to have a grain size distribution not containing too many fine grains but not too many coarse grains either. The shape of the grain size curve should, according to performed tests, be relatively steep (narrow grain size range) between minimum and maximum grain size.

- For a monodisperse (one grain size) mixture the relationship between slot aperture and grain size has been found to be 2-3 in order to pass a major amount of the available mixture through the used slot.

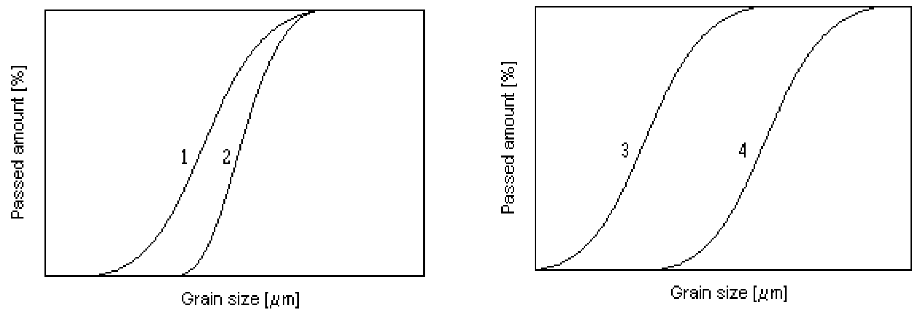


Figure 6.22, The left figure illustrates steepness of the grain size distribution, the right figure shows the variations in the mixtures content of finer and coarser grain sizes. When the mixtures contain grains with approximately the same value of d_{95} (left figure), the slope of the grain curve (between d_{95} and the smallest grains) will be of importance. A relatively steep grain size distribution is desired, in order to achieve a low filtration tendency of the mixture.

6.3.2 Influence of grain concentration

A critical parameter to achieve a low filtration tendency of the mixture is the grain concentration.

- If the value of d_{95} is small enough compared to the used slot aperture (d_{95} is not less than 3-4 times smaller than the aperture), the passed amount will increase with an increased W/S ratio.

- The influence of a steep grain size distribution is most significant at low W/S ratios. At a low W/S ratio the grains are to a larger extent in constant contact to each other then at a higher W/S ratio. A steeper grain size distribution probably makes it easier for the grains to rearrange within the mixture and because of that avoid plug formation.

- The grain concentration affects the filtration tendency in the scaled-up experiments. According to the physical simulations it can be concluded that a grain concentration of 20 % (W/S ~1,3) will make trouble with plug formation in a 7-8 mm wide slot aperture. Decreased grain concentration to a W/S ratio of 6 will decrease the possible slot aperture to be passed to approximately 6-7 mm.

- The rheology is to a high extent dependent on the W/S ratio. The experiments show that the viscosity and yield value increase rapidly when the W/ S ratio goes below 0,5 to 0,6.

- The bleeding of the mixture is to a high extent influenced by the used grain concentration. Mixtures with a major amount of fine grains can be used with higher W/S ratios then mixtures with coarse grain size distributions and still show the same bleeding value.

6.3.3 Influence of mesh or slot geometry

In this work, three types of filtration geometries have been used. The performed tests show there is a difference between the slot geometry compared to the mesh geometry as regards the relative amounts they allow to pass.

- According to performed tests, there is a difference in the relative passed amount of mixture between the filter geometries of mesh, mesh/ slot and slot design. The experiments show that the same filter aperture (slot and mesh) generate different relative passed amount of mixtures.

- A smaller relative filter area will consequently cause that a larger number of grains (when using the same initial volume and for the same passed volume of mixture) to pass the specific slot or mesh area of the filter.

- If a larger amount of grains passes the aperture, the probability of plug formation at some place along the slot or mesh aperture will probably increase compared to a case of a larger filter area. Agglomerates or larger grains in the mixture can initialize the plug at the entrance of the aperture. Probably, this stochastic behaviour of the plug formation can not easily be predicted.

- Calculation of the penetrability length that corresponds to the relative passed amount of a mixture is a way of comparing measurements of filtration tendency based upon different filter geometries. Presented results indicate that plug formation more easily can occur at a mesh geometry compared to a slot geometry. This result only regards mixture 4 and 5, at a W/S ratio of 0,7 and based upon the use of an aperture of 75 μm. The comparison of penetration length was not possible to perform for mixture 2, 3 and 6, because there was no occurrence of plug formation when using the mesh aperture of 75 μm (mixture 2) and no passed amount of mixture through the slot aperture of 75 μm (mixture 3 and 6).

6.3.4 Miscellaneous

A number of correlations tests have been made with the purpose of analysing connections between filtration tendency and grain size, grain size distribution, grain concentration and filter aperture. The performed correlation tests do not taken into consideration the combined influences of different parameters. Just one parameter was studied against the passed amount of mixture in each correlation test. The performed correlation tests show that it is probable that a combined influence of different parameters on the passed amount is present, because generally bad correlation values were achieved.

- Regarding the quantity compared to the quality of the passed mixture it can be said that a larger passed amount is generally associated with a good quality of the mixture (same grain concentration before and after passage through the filter). A small passed amount of mixture is consequently more often associated with a larger difference in grain concentration before and after filtration (due to plug formation at the entrance of the aperture).

- No real similarities between the numerical simulations and physical experiments were found. Further development of the numerical flow model is necessary. Physical experiments with inert material can be used in order to better understand and visualize the mechanisms of the plug formation.

- The time scale for the plug to occur in the scaled up model varies in the different tests. The position where the plug initialization occurs varies in the different tests. The influence of a stochastic behaviour on the total passed amount becomes more dominant when the slot aperture becomes closer to the maximum grain (expressed as for example as d_{95}).

6.4 Cement based material

Due to the complex interactions between physical properties (grain size, grain size distribution, grain concentration and the used slot aperture) and chemical influences because of hydration of the cement grains and use of additives (superplastisiser) in the fresh mixture, the results of the filtration experiments are presented due to used type of cement. The used type of cement is characterized by the grain sizes and grain size distributions. The three types of used commercial cements are Ultrafint cement 12 (UF 12, d_{95}=12µm), Ultrafint cement 16 (UF 16 d_{95}=16µm) and Injekterings cement 30 (IC 30, d_{95}=30µm). Three sets of experiments are performed. Every set of experiments includes the same variable parameters (one type of cement, three to six different grain concentration, three used superplastisisers and three used slot apertures). At the end of this chapter sieved cement is used (Cem 2, Cem 4, UF 12 fine and UF 12 coarse), these cements are produced according to the grain sizes and grain size distributions that were found to show the lowest filtration tendency of the inert mixtures. Measurements of filtration tendency, rheology and bleeding are preformed for all the tested mixtures. Only slot apertures have been tested here.

The analysis of the passed volume of mixture through the used filter area has been illustrated by the penetration length, (see eq 6.1, section 6.2.3). The calculated penetration length is the ratio between the passed volume and filter area. Used slot apertures are 75, 100 or 125 µm and with a width of 62 mm, see Figure 5.4. The filtration tendency expressed as the penetrated length is a way of reducing the influence of the used relative passed amounts (passed amount through the aperture related to the initial available amount of mixture). b_{req}. represents the slot aperture (75, 100 or 125 µm) that fulfills the requirements of penetration length.

The demand of penetrated length is in this work set to 5 m (which corresponds to a relative passed volume between 5-8%). The maximum penetration length corresponds to approximately 100-60 m, depending on the used slot aperture (75, 100 or 125 μm). b_{crit} is defined as the slot aperture where 75% of the available passed volume of 490 cm^3 passes the slot (which consequently corresponds to a penetration length of 79-47m).

The demand of a grout's resistance to leaching means that the W/C ratio should be kept as low as possible, in order to obtain both a tight hardened cement paste and to avoid extensive bleeding of the paste. Either parts aims to avoid leaching because leach paths are created due to a porous paste or after bleeding of the paste. The passed amount of mixture shall therefore, in this work, show a bleeding of less then 5%.

The requirements of the passed amount of mixture in order to fulfill the requirements of $b_{req.}$, can as earlier mentioned be described by a penetration length of more then 5m and with a bleeding of less than 5 %.

The first section 6.4.1 deals with the influence of the UF 12 cement. Six different W/C ratios between 0,7 and 1,75 are used. The used slot apertures are 75, 100 and 125 μm.

The second section 6.4.2 deals with the influence of the UF 16 cement. Six different W/C ratios between 0,7 and 1,75 are used. The used slot apertures are 75, 100 and 125 μm.

The third section 6.4.3 deals with the influence of the IC 30 cement. Three different W/C ratios between 0,7 and 1,0 are used. The used slot apertures are 75, 100 and 125 μm.

The fourth section 6.4.4 deals with the influence of the sieved cements Cem 2 and Cem 4. Used W/C ratio is 0,7. Used mesh aperture is 75 μm.

The fifth section 6.4.5 deals with the influence of the sieved cements UF 12 fine and UF 12, coarse. Three different W/C ratios between 0,85 and 1,25 are used. Used slot apertures are 100 and 125 μm.

At the end of these five sections a statistical analysis is performed on the presented results in section 6.4.1 to 6.4.5. Finally, the significant results from the tests in section 6.4.1 to 6.4.5 are presented at the end of this chapter.

The relative amount of passed mixture concerns the passed weight of mixture (relative to the initial amount). The used initial weight of mixture will vary in the cement based filtration experiments depending on the used W/C ratio.

Figure 6.23 shows the passed amounts of all the mixtures containing UF 12, UF 16 and IC 30 cement.

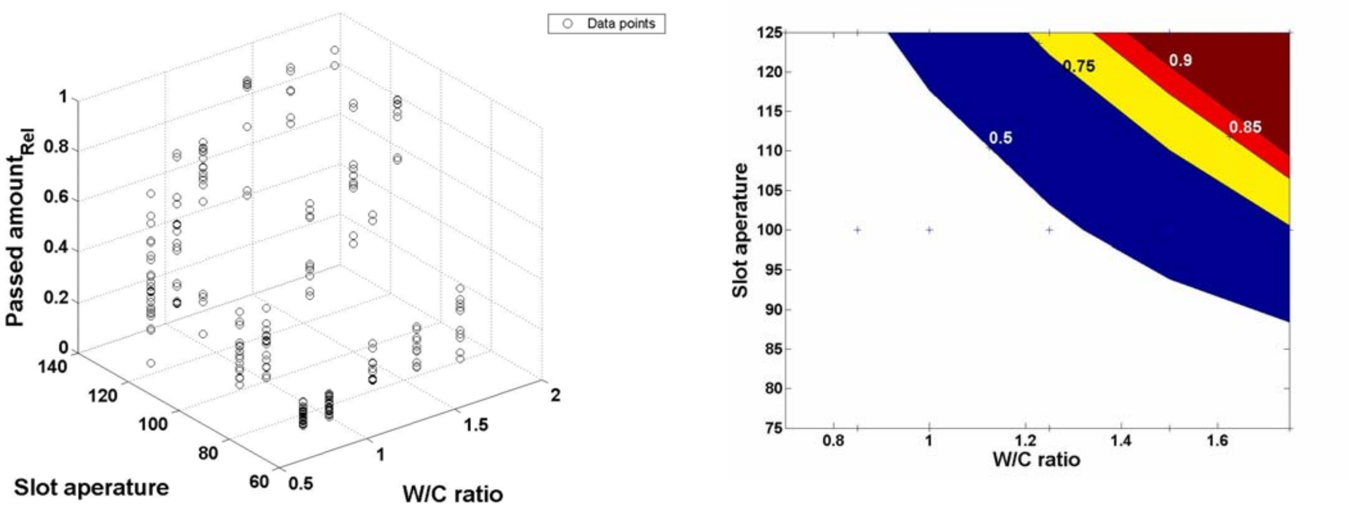


Figure 6.23, Illustration of the passed amount of mixture as a function of the W/C ratio and slot aperture. The figures show the results of all the performed filtration tests with cement based material (UF 12, UF 16 and IC 30). The passed amount of mixture through the slot is expressed relative to the available amount of mixture (used in the filtration apparatus, according to VU:SC 48). The marked dots in the right figure show performed measurements.

The right illustration in Figure 6.23 show the relationship between the W/C ratio and the required slot aperture in order to achieve a certain passed relative amount of mixture (between 50 and 100 % of the available amount of mixture). An increased W/C ratio and slot aperture is associated with a larger relative passed amount of mixture.

As an example, it can be mentioned that in order to maintain the W/C ratio as low as possible it is necessary to use a slot aperture of at least approximately 125 μm to obtain a relative passed volume of 75% or more (yellow field). A lower W/C ratio will naturally decrease the passed amount.

Alternatively, if the used W/C ratio increases to 1,75 the slot aperture should be approximately at least 100 μm in order to pass a relative amount of 75 % or more.

Relatively few tests have been made with mixtures containing higher W/C ratio than 1,5. This is basically because these mixtures have a bleeding that exceeds 5%. Unstable mixtures with a bleeding larger than 5 % have not been the focus for this study. Practically, in grouting operations the most commonly used W/C ratio is in the region of 0.7 to 1.5.

6.4.1 UF 12, Influence of grain size, grain size distribution, grain concentration and slot aperture

Figure 6.24 shows all the performed tests with UF 12 compared to the total amount of performed tests regarding cement based mixtures (UF 12, UF 16 and IC 30). Tests with UF 12 are colored blue, red and green dependent of used superplastisiser.

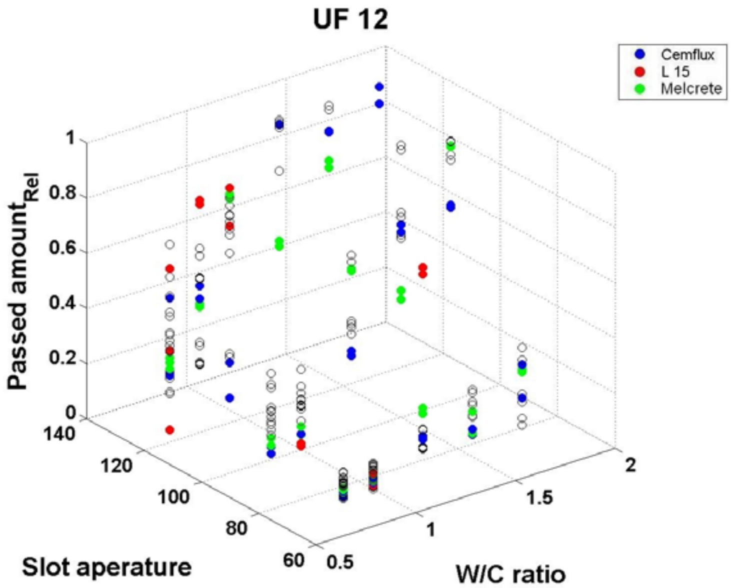


Figure 6.24, The figure shows the performed tests with UF 12 compared to all of the performed tests (with cement based mixture) done in this work.

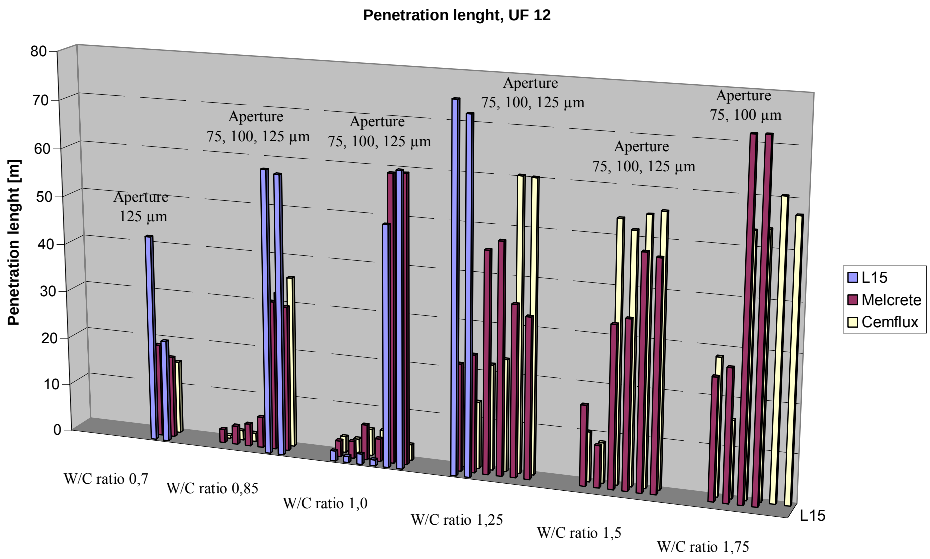


Figure 6.25, This figure is based on the filtration results in Figure 6.24. The passed amount of mixture is converted to a penetration length in an artificial slot volume. For all passed amounts see appendix B.

As can be seen from Figure 6.25 the penetration length of a certain mixture will vary between wide limits. The variation is between a few meters up to about 70 m. Performed tests with UF 12 showed that a W/C ratio of approximately 1,25 is necessary to achieve a penetration length of 5 m in the artificial slot volume with an aperture of 75 µm. If the W/C ratio is decreased to 0,7, a slot aperture of approximately 125 µm is necessary to achieve the 5m of penetration length.

The bleeding of the mixtures after filtration seems to vary between 0-8%. The tested W/C ratios are 0,7, 0,85, 1,0, 1,25, 1,5 and 1,75. Mixtures containing Cemflux as a superplastisiser show a lower bleeding compared to mixtures containing the superplastisiser Melcrete. The bleeding of mixtures containing L 15 is zero (or very close to zero) for all tested cases. The mixtures containing Melcrete and Cemflux at a W/C ratio of 1,75 exceed 5% of bleeding.

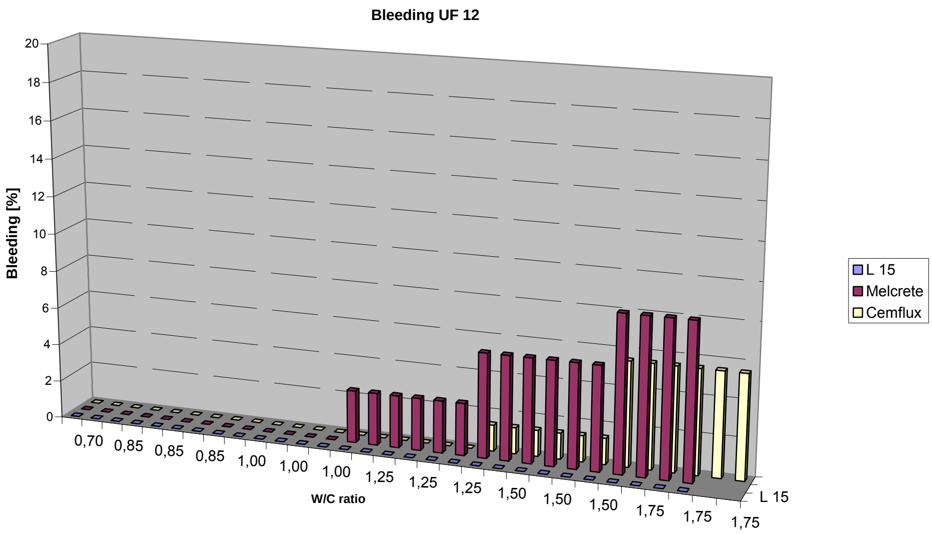


Figure 6.26, Bleeding measurements according to SS-EN 445. Used cement is Ultrafint cement 12 (UF 12). Used superplastisiser are Cemflux, Melcrete and L 15. Used W/C ratio are between 0,7-1,75.

$b_{req.}$ is evaluated according the two criteria of penetration length and bleeding of the passed mixture. Penetrated length shall be >5 m and the bleeding of the passed mixture shall be < 5%. The calculated penetration length is the ratio between the passed volume and filter area. b_{crit} is evaluated with the stipulation that all the amount of mixture shall pass the slot.

The priority in the performance of the filtration tests is that each slot aperture (with the beginning with the smallest of 75 µm) is tested with the used W/C ratios. If none of the used W/C ratios fulfil the requirements of $b_{req.}$ or $b_{crit.}$, the next slot aperture will be tested (for example 100 µm) in the same manner as the previous tested slot aperture. The bleeding of the passed amount of mixture has been measured in order to evaluate the quality of the passed mixture.

In order to fulfil the definitions of b_{crit} and b_{req} a vast number of combinations regarding the used slot aperture and W/C ratio can be chosen. Figure 6.27 shows the shaded area (numbered 1-3) within which the definitions b_{crit} are fulfilled.

The line that show a passed amount of 10 % or more, represents roughly a penetrated length of more than 5 m, which is one of the definitions regarding b_{req}. The shaded area (numbered 4-7) does not fulfil the definitions of b_{req}.

The shaded areas are also show areas that do not fulfil the bleeding requirements. The vertical line that represents the limit for acceptable bleeding is placed where a bleeding of more than 5 % was measured.

The area that fulfils the demands of b_{crit} and b_{req} are consequently given by the used slot aperture, W/C ratio and bleeding of the passed mixture.

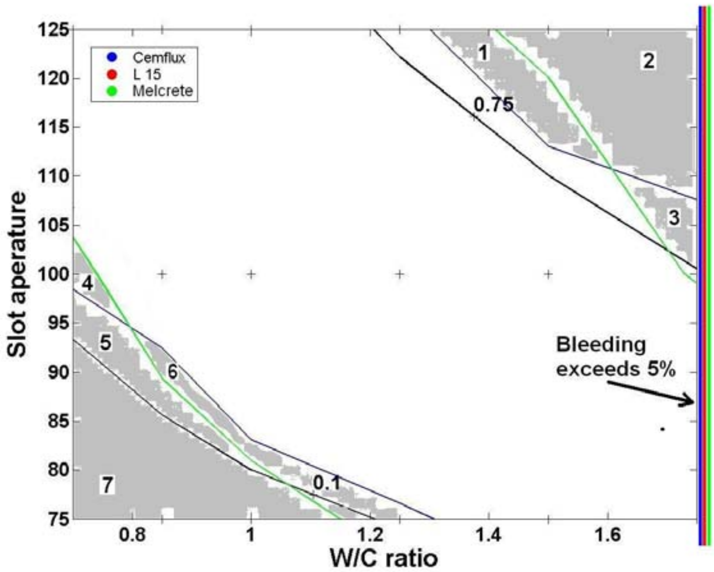


Figure 6.27, Evaluation of b_{crit} and b_{req} from the filtration experiments for the UF 12 cement. The figure shows the average value of all used mixtures (black line) compared to the values for UF 12 (with two types of superplastisisers, green line indicate use of Melamin and blue line is use of Cemflux). The marked dots in the figure show the performed measurements.

The results from the use of L 15 are not shown, because of the relatively few measuring values, see Figure 6.24. The method used in Figure 6.27 and similar, in order to evaluate the data, is a nonparametric local regression called LOESS that was introduced by Cleveland and Devlin (1988). LOESS has been implemented for several numerical packages and has been described by Cleveland (1993).

According to Figure 6.27 the UF 12 cement seemingly requires a larger slot aperture and higher W/C ratio in order to pass a relative amount larger than 75 %, compared to the average of the studied cements. The black line marked with 0,75 indicates the average value of the three used cement mixtures (UF 12, UF 16 and IC 30) that show a passed amount larger than 75 %. In Figure 6.27 it can also be seen that the shaded area will be different due to used superplastisiser (numbered 1-3). Only a minor difference can be seen between different superplastisisers when looking at passed amounts larger than 75 %.

It should be noted that relatively few measuring points form the basis for the interpolation of the lines which delimits the passed amount of 75% for each superplastisiser. The absolute delimiting values should therefore be treated with some precaution.

The limit of acceptable bleeding is exceeded in the case of using a W/C ratio of 1,75. The W/C ratio should according to these tests be ≤ 1,75 in the passed amount of mixture in order to fulfil the definitions of b_{crit} and b_{req}.

6.4.2 UF 16, Influence of grain size, grain size distribution, grain concentration and slot aperture

Figure 6.28 shows all the performed tests with UF 16 compared to the total amount of performed tests regarding cement based mixtures (UF 12, UF 16 and IC 30). Tests with UF 16 are colored blue, red and green depending on the used superplastisiser.

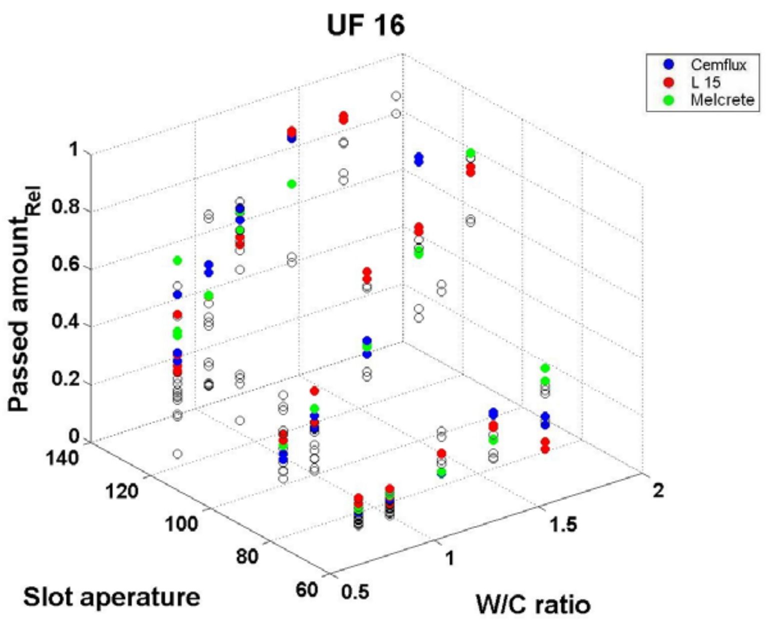


Figure 6.28, The figure shows the performed tests with UF 16 compared to all of the performed tests (with cement based mixture) done in this work.

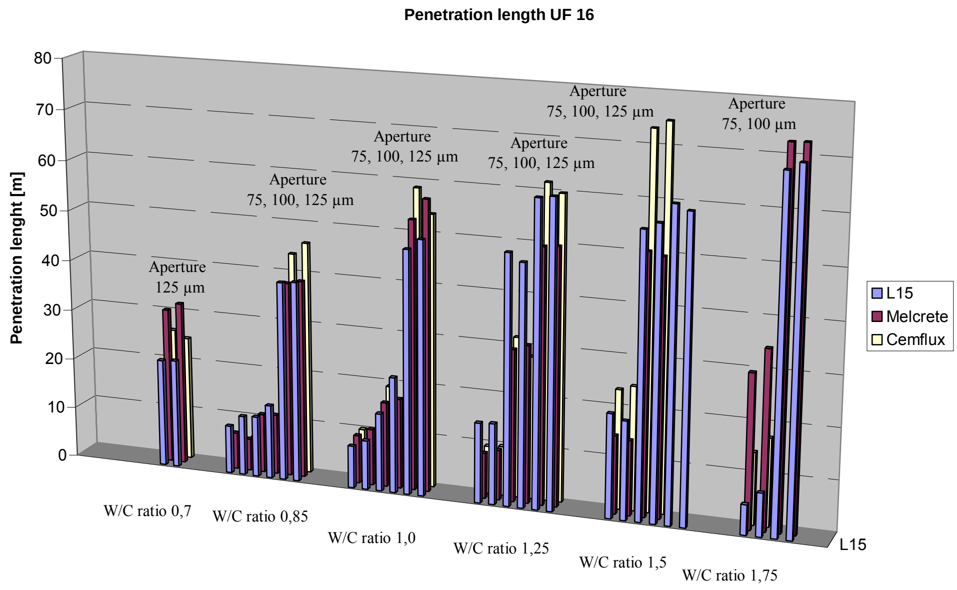


Figure 6.29, This figure is based on the filtration results from Figure 6.28. The passed amount of mixture is converted to a penetration length in an artificial slot volume. For all passed amounts see appendix B.

The bleeding of the mixtures seems to vary between 0-8%. The tested W/C ratios are 0,7, 0,85, 1,0, 1,25, 1,5 and 1,75. The mixtures containing L 15 at W/C ratio of 1,75 and mixtures with Cemflux at W/C ratio of 1,50 exceed the upper limit of 5% bleeding. Mixtures with Melcrete show a bleeding which is less than 5% for all tested cases. The bleeding values of the UF 16 mixtures is somewhat lower compared to the UF 12 mixtures.

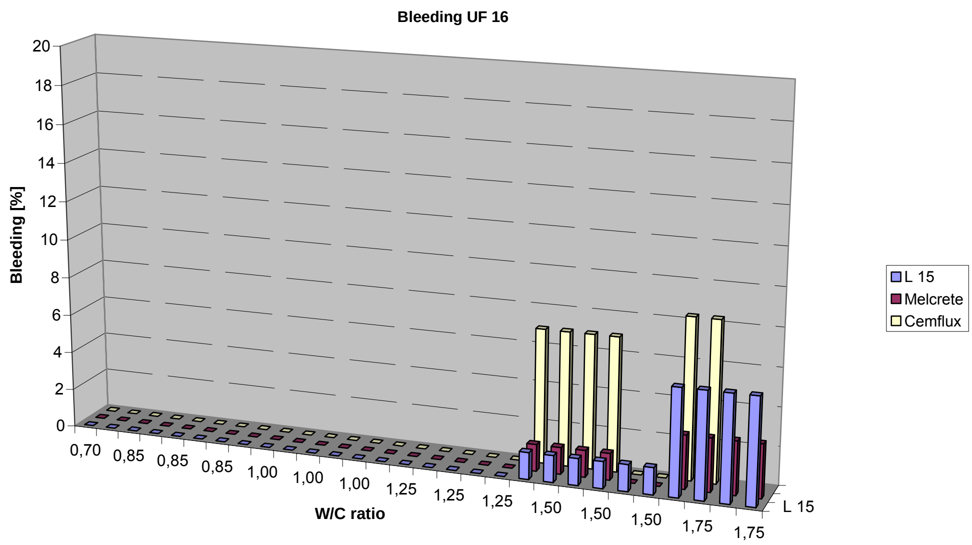


Figure 6.30, Bleeding measurements according to SS-EN 445. The cement used is UF 16. Used superplastisiser are Cemflux, Melcrete and L 15. Used W/C ratio are between 0,7-1,75.

Figure 6.32 show the shaded area (numbered 3,4,6,7) within which the definition of b_{crit} are fulfilled, regarding Cemflux. In the case of using L 15 and Melcrete the area will be defined by 1,2,3,5,6,7. The shaded area (numbered 9-11) does not fulfil the definitions of b_{req}.

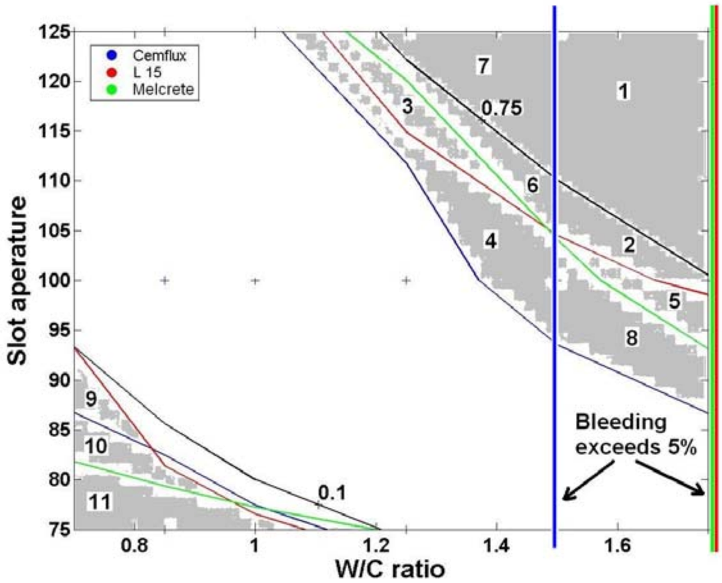


Figure 6.31, Evaluation of b_{crit} and b_{req} from the filtration experiments for the UF 16 cement.

According to Figure 6.31 the UF 16 cement seems to require a smaller slot aperture and lower W/C ratio in order to pass a relative amount larger than 75 %, compared to the average of studied cements (UF 12, UF 16 and IC 30). In Figure 6.32 it can also be seen that the shaded area will be different due to the superplastisiser used (numbered 1-8). The passed amount is consequently influenced by the used superplastiser. The use of Cemflux generates the largest passed amount at the smallest tested slot apertures and lowest tested W/C ratios (regarding a passed amount larger than 75%).

The limit of acceptable bleeding is exceeded in the case of using a W/C ratio of 1,50 (Cemflux). When using L 15 and Melcrete the limit is reached at a W/C ratio of approximately 1,75.

6.4.3 IC 30, Influence of grain size, grain size distribution, grain concentration and slot aperture

Relatively few measuring values are found in the region of 75-100 % passed amount of mixture, see Figure 6.32.

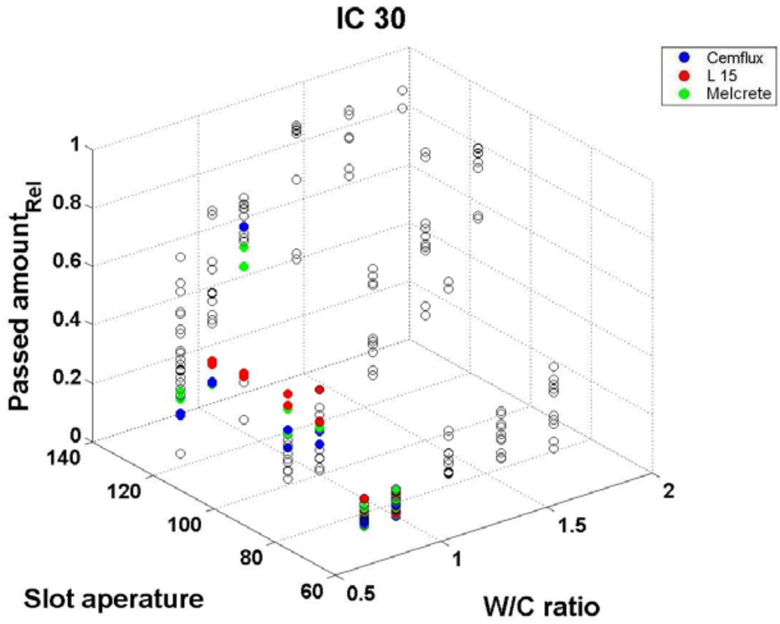


Figure 6.32, The figure shows the performed tests with IC 30 compared to all of the performed tests in this work (with cement based mixture) (Figure 6.23). The figure shows the values for IC 30 with three types of superplastisisers, green dots indicate use of Melamin, blue dots is use of L 15 and red dots is Cemflux. The marked dots in the right figure illustrate the performed measurements.

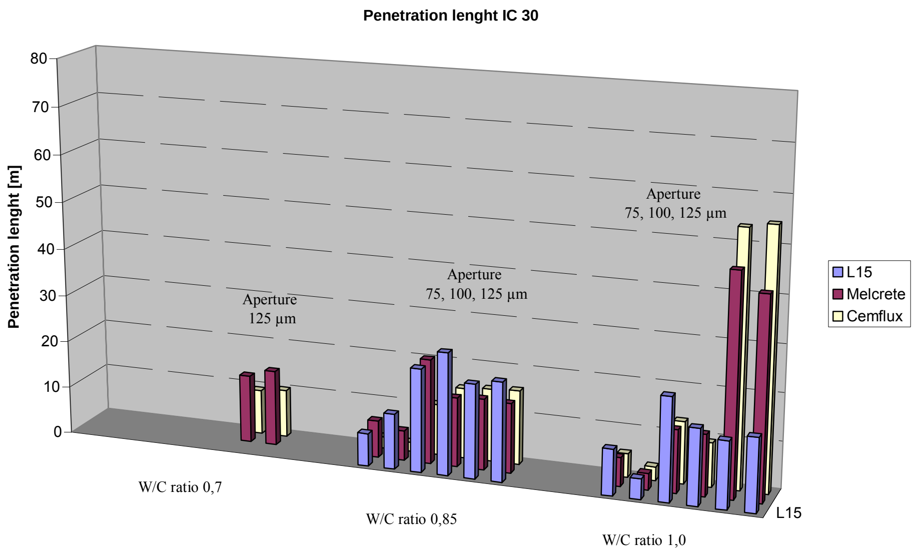


Figure 6.33, This figure is based on the filtration results from Figure 6.32. The passed amount of mixture is converted to a penetration length in an artificial slot volume.

A penetration length of more than 5m in a slot aperture of 75µm is achieved for mixtures with W/C ratio of 0,85 and containing L 15 and Melcrete. Penetration lengths (more than 5m) are achieved with mixtures containing Cemflux with a W/C ratio of 0,85 and a slot aperture of 100 µm.

The bleeding of the mixtures seems to vary between 0-20%. The tested W/C ratios are 0,7, 0,85 and 1,0. The mixtures containing L 15 at a W/C ratio of 0,85 and mixtures with Cemflux at W/C ratio of 1,0 exceed the upper limit of 5% bleeding. Mixtures with Melcrete and Cemflux with a W/C ratio of 0,85 show a bleeding less than 5 %.

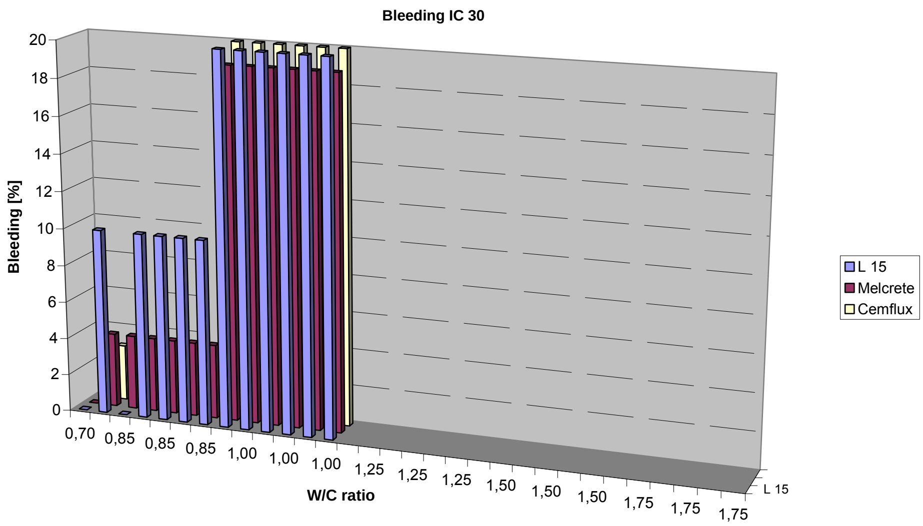


Figure 6.34, Bleeding measurements according to SS-EN 445. Used cement is Injekterings cement 30 (IC 30). Used superplastisiser are Cemflux, Melcrete and L 15. Used W/C ratio are between 0,7, 0,85 and 1,0.

Figure 6.35 shows that none of the tested mixtures containing Melcrete fulfil the definition of b_{crit}. The shaded area (numbered 2 and 3) does not fulfil the definitions of b_{req} for the case when using Melcrete. b_{crit} and b_{req} are not evaluated for the cases where Cemflux and L 15 are used, due to toofew measuring points in regions of 0-10% and 75-100% passed amount.

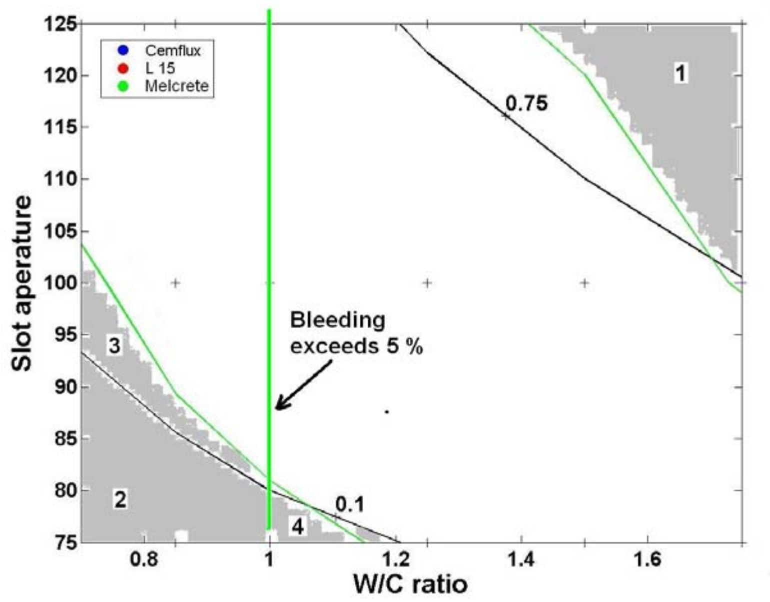


Figure 6.35, Evaluation of b_{crit} and b_{req} from the filtration experiments for the IC 30 cement.

6.4.4 Cem 2 and Cem 4, Influence of grain size, grain size distribution, grain concentration and slot or mesh aperture

The basic idea of sieving cement into the grain distributions of Cem 2 and Cem 4 was to investigate if these cement based grain curves have the same low filtration tendency as the inert material of the same grain curve (2 and 4). Performed filtration experiments indicate that it is not the case, see Table 6.18. The same available volume (~110 cm^3) of mixture was used in these experiments as for the rest of the inert based experiments. The mesh geometry was used in order to compare the results from the inert mixtures to the cement based mixtures.

Table 6.18, Comparison of the passed amount of inert material versus passed amount of cement based material. Used filter geometry is the mesh, aperture 75 μm and the mixtures W/C and W/S ratio is 0,7.

Inert grain curve	**Passed amount**	No.	Dev.	**Cement based grain curve**	**Passed amount**	No.	Dev.
	[%]				[%]		
2	83	3	1,0	Cem 2	29	3	1,6
4	68	3	2,6	Cem 4	19	3	1,3

The passed amounts (29 and 19 %) of the sieved cement based mixtures (Cem 2 and Cem 4) are based upon the average value from the use of three superplastisisers (L 15, Melcrete and Cemflux). No major difference could be identified between the mixtures containing the different superplastisisers and these specific mixtures (Cem 2 and Cem 4).

Compared to the UF 12, UF 16 and IC 30 cements the Cem 2 are a bit coarser and have a steeper grain size distribution than UF 12 and UF 16, but finer and with a steeper grain size distribution than IC 30. Cem 4 is the most finely grained of these cements, for the complete grain size distribution see Figure 5.16.

6.4.5 UF 12 fine and UF 12 coarse, Influence of grain size, grain size distribution, grain concentration and slot aperture

The performed tests indicate that the fine cement grains in the mixture influences the filtration tendency of the mixture. An increased amount of fine grains is achieved when a part of the coarser grains are removed from the cement powder. The opposite is the removal of fine grains which will increase the amount of coarser grains. According to these tests an increased amount of fine grains (<3µm) will cause a smaller passed amount of mixture (see Figure 6.36 compared to Figure 6.25).

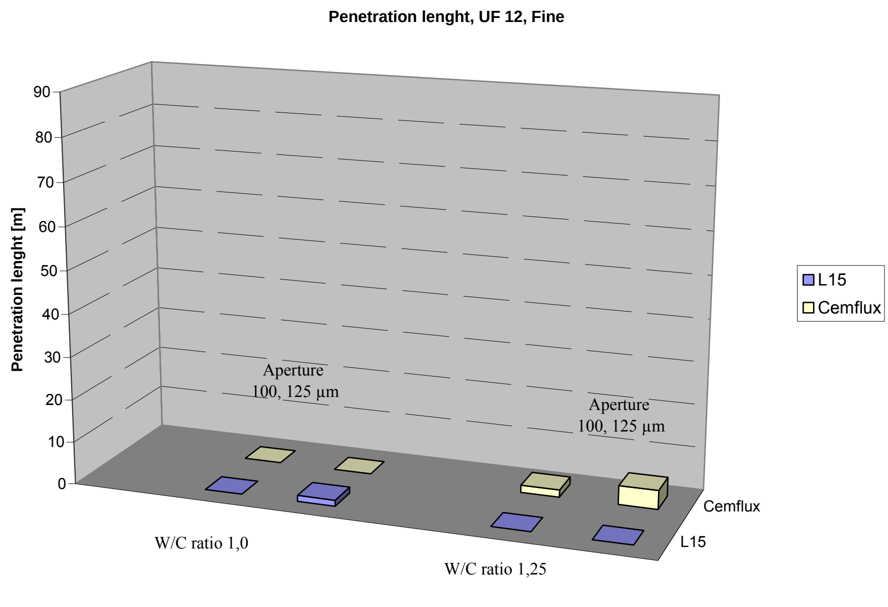


Figure 6.36, This figure is based on the passed amounts of UF 12 Fine in the filtration tests, see appendix B. The passed amount of mixture is converted to a penetration length.

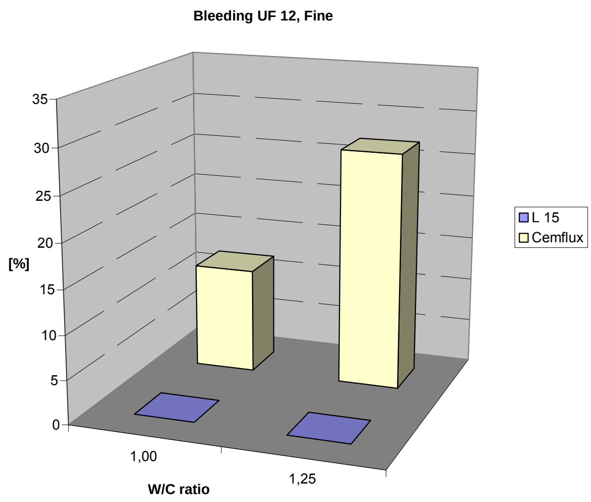


Figure 6.37, Bleeding measurements according to SS-EN 445. Used cement is sieved UF 12 cement (UF 12, Fine). Used superplastisisers are Cemflux and L 15. Used W/C ratios are between 0,85 and 1,25.

As can be seen from Figure 6.36 and Figure 6.37 it can be concluded that none of the tested mixtures containing the UF 12 fine cement, will fulfill the requirements of b_{req} or b_{crit}, see Table 6.19. These results regard the use of a maximum slot aperture of 125 μm. No major difference between the two types of superplastisisers (L 15 and Cemflux) could be seen.

Due to the few measuring values regarding UF 12 Fine and UF 12 Coarse, there is no point in producing graphs like Figure 6.35. An alternative way of showing the values of b_{crit} and b_{req} is presented in Table 6.19 and Table 6.20.

Table 6.19, Evaluation of the filtration tendency of UF 12, fine cement.

UF 12, fine	**Superplastisiser**	**b_{req}**	**W/C ratio**	**bleeding**		**b_{crit}**	**W/C ratio**	**bleeding**
		[μm]		[%]		[μm]		[%]
	L 15	>125	-	-		>125	-	-
	Cemflux	>125	-	-		>125	-	-

As can be seen from Figure 6.38 and Figure 6.36, there is a difference in the passed amount of mixture between the mixtures containing a major part of fine grains compared to the mixture containing a major part of coarser grains. There is also a difference in the used superplastisiser, the mixture containing the steric type of superplastisiser (Cemflux) seems to have the lowest filtration tendency of the two tested superplastisisers.

The difference in the passed amount (due to used superplastisiser) will according to performed filtration tests be smaller when the grain concentration gets lower (increased W/C ratio).

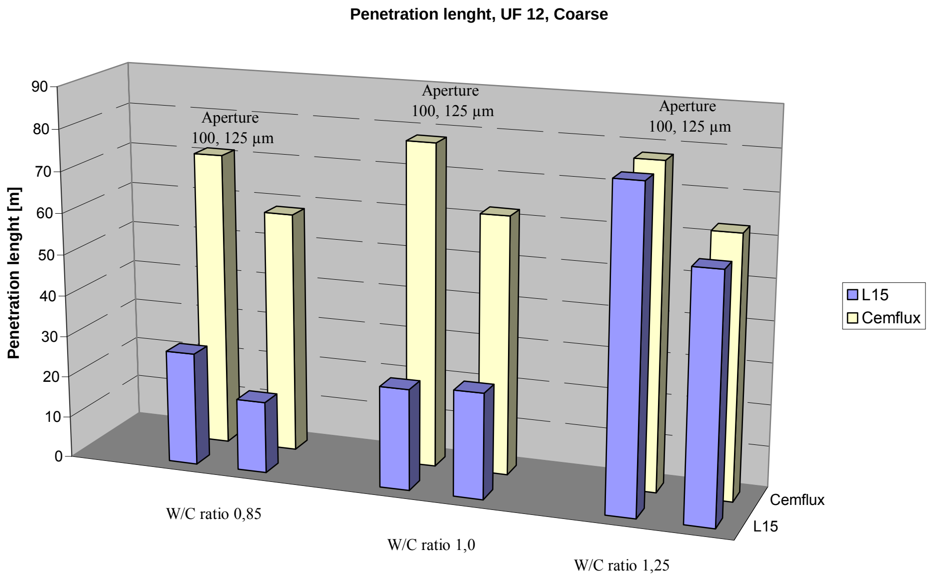


Figure 6.38, This figure is based on the passed amounts of UF 12 coarse in the filtration tests, see appendix B. The passed amount of mixture is converted to a penetration length.

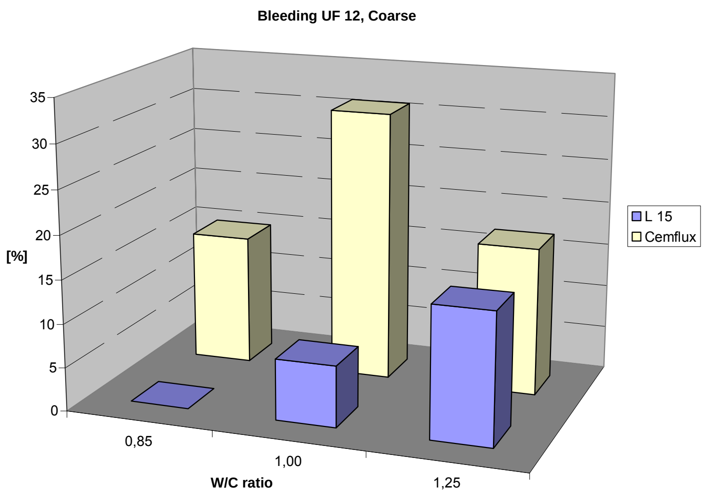


Figure 6.39, Bleeding measurements according to SS-EN 445. Used cement is sieved UF 12 cement (UF 12, coarse). Used superplastisisers are Cemflux and L 15. Used W/C ratio are between 0,85 and 1,25.

It should be mentioned that two slot apertures (100 and 125µm) are tested for the same initial mixture batch. Consequently the first slot aperture has been tested and then the other. In the performed tests the 100 µm slot has first been tested, then the 125 µm slot. This may influence the passed amount of mixture due to flocculation of grains in the mixture. It has been attempted to be reduced the influence of flocculation by continuously stirring during the testing of the first aperture (100µm). The two tests (100 and 125 µm) have been performed during a time of approximately 5 minutes.

As can be seen from Figure 6.38 and Figure 6.39 that most of the tested mixtures with the sieved cement (based upon UF 12) show that a major passed amount (penetration length) will also show a bleeding larger than 5%. The only mixture that has a major passed amount and a bleeding less than 5% is the UF 12, coarse mixture with a W/C ratio of 0,85 and the superplastisiser of L 15. For an evaluation of b_{crit} and b_{req}, see Table 6.20.

Table 6.20, Evaluation of the filtration tendency of UF 12, coarse cement.

UF 12, coarse	**Superplastisiser**	b_{req}	**W/C ratio**	**bleeding**		b_{crit}	**W/C ratio**	**bleeding**
		[µm]		[%]		[µm]		[%]
	L 15	<100	-	-		100	≥ 1,25	>5
	Cemflux	<100	-	-		<100	-	-

6.4.6 Influence of superplastisisers

A difference in the passed amount according to the time after mixing has been found between the studied superplastisisers. These tests show the results for 10 minutes or longer after mixing. The original used time is 0-5 minutes after mixing. The two types of electrostatic superplastisisers (L 15 and Melcrete) show deterioration a time after mixing, compared to the steric superplastisiser Cemflux, see Figure 6.40. The time dependent test is performed with the UF 12 cement. The UF 12 cement is supposed to be the cement that has the fastest setting and hydration time of the three used cement (UF 12, UF 16 and IC 30). This is because of its relatively small grain sizes (large specific surface of the grains in the dry powder). Large specific surface is supposed to speed up the setting and hydration of the mixture (Lidström, 2003), (Gartner 1987).

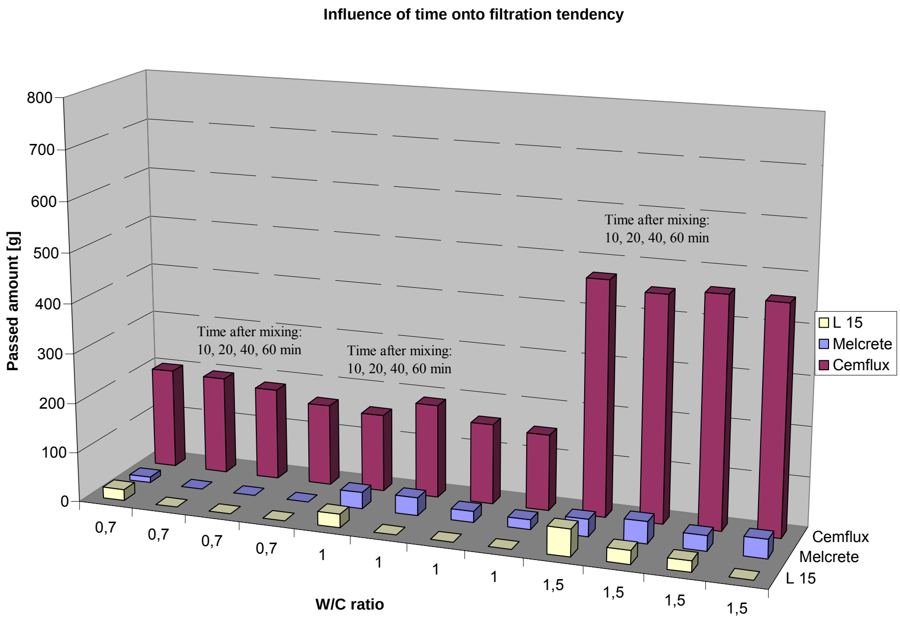


Figure 6.40, Influence of time on the passed amount through a 100 µm slot. The available amount of mixture is approximately 750 g. Used cement is UF 12.

The passed amount of mixture with a W/C ratio of 1,5 before filtration shows a relatively high bleeding after filtration (>5%). The difference in relative deterioration between the two types of superplastisiser (steric and electrostatic) can schematically be illustrated according to Figure 6.41.

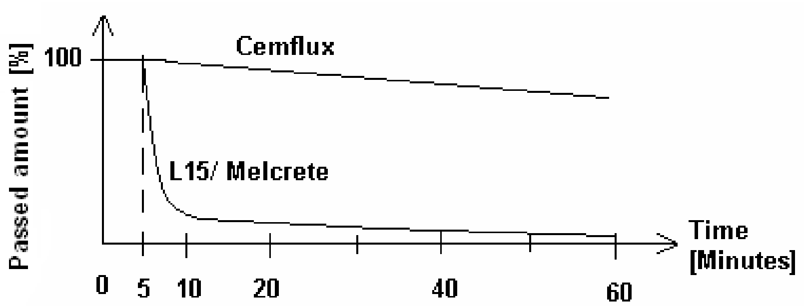


Figure 6.41, Illustration of the relative passed amount of mixture through used filter geometry related to the used type of superplastisiser (within 5 min after mixing), compared to the passed amount a certain time after the mixing.

As mentioned earlier in this work, the superplastisiser influence the rheology of the mixture. The superplastisiser is often used in order to reduce the yield- and viscosity value without increasing the W/C ratio of the mixture. The results from the rheological measurements , which are presented in Table 6.21 indicate that the three different used superplastisisers influence the yield- and viscosity values in different ways. Generally it can be seen that the steric superplastisiser (Cemflux) will decrease the yield- and viscosity values of the mixture to a larger extent compared to the electrostatic ones (L 15 and Melcrete).

The comparison is only performed for the W/C ratio of 1,0, the result may be different if other W/C ratios are studied. The measurements are performed 0-5 minutes after mixing.

Table 6.21, Results from the rheological measurement of the viscosity in the mixtures with W/C ratio of 1,0. No. concerns the number of performed measurements.

Cement	**UF 12**			**UF 16**			**IC 30**		
	Viscosity	std. dev.	No.	Viscosity	std. dev.	No.	Viscosity	std. dev.	No.
Superplast.	[Pas]	[-]		[Pas]	[-]		[Pas]	[-]	
L 15	0,11	0,03	5	0,09	0,02	7	0,03	0,01	7
Melcrete	0,15	0,02	7	0,10	0,01	7	0,02	0,003	7
Cemflux	0,03	0,02	5	0,03	0,002	7	0,01	0,001	7

Table 6.22, Results from the rheological measurement of the viscosity in the mixtures with W/C ratio of 1,0. No. concerns the number of performed measurements.

Cement	**UF 12 fine**			**UF 12 coarse**		
	Viscosity	std. dev.	No.	Viscosity	std. dev.	No.
Superplast.	[Pas]	[-]		[Pas]	[-]	
L 15	0,48	-	2	-	-	-
Melcrete	-	-	-	-	-	-
Cemflux	0,07	-	2	-	-	-

Table 6.23, Results from the rheological measurement of the yield value in the mixtures with W/C ratio of 1,0. No. concerns the number of performed measurements.

Cement	**UF 12**			**UF 16**			**IC 30**		
Superplast.	Yield value	std. dev.	No.	Yield value	std. dev.	No.	Yield value	std. dev.	No.
	[Pa]	[-]		[Pa]	[-]		[Pa]	[-]	
L 15	4,95	2,79	5	3,32	3,88	7	0,13	0,05	7
Melcrete	2,59	0,64	7	1,74	0,60	7	0,13	0,09	7
Cemflux	0,36	0,22	5	0,40	0,23	7	0,07	0,01	7

Table 6.24, Results from the rheological measurement of the yield value in the mixtures with W/C ratio of 1,0. No. concerns the number of performed measurements.

Cement	**UF 12 fine**			**UF 12 coarse**		
	Yield value	std. dev.	No.	Yield value	std. dev.	No.
Superplast.	[Pa]	[-]		[Pa]	[-]	
L 15	11,90	-	2	-	-	-
Melcrete	-	-	-	-	-	
Cemflux	0,72	-	2	-	-	-

The rheology for the UF 12 coarse was not possible to measure because of the large bleeding which occurred within the measuring gap in the plate-plate measuring system.

As can be seen when comparing the yield- and viscosity values Table 6.21 to Table 6.24) to the passed amounts (filtration tendency in Figure 6.24, Figure 6.28 and Figure 6.32) it is difficult to see any significant relationship between the rheology and the filtration tendency. The yield- and viscosity values of the mixture seem to vary within wide limits but not affect the filtration tendency. As an example it can be mentioned that the yield- and viscosity values of mixtures containing UF 12, UF 16 and IC 30 will according to performed rheological measurements (at W/ C ratio of 1,0), vary in the range between 5-0,1 Pa and 0,01-0,1 Pas, see Table 6.21 to Table 6.24.

6.4.7 Evaluation of measured values.

The quotient between the used slot aperture and the grain size expressed as d_{95} can be calculated. The comparison is made for a W/C ratio of 1,0.

Table 6.25, Relationship (quotient) between the used slot apertures and the grain size (d_{95}). Quotients which are shaded are the ones that fulfil the demand of $b_{req.}$ at a W/C ratio of 1,0.

		slot aperture		
Mixture	**d_{95} [µm]**	**75 µm**	**100 µm**	**125 µm**
UF 12	12	6,3	≥ 8,3[1]	10,4
UF 16	16	≥ 4,7	6,3	7,8
IC 30	30	≥ 2,5[2,3]	3,3[3]	4,2[3]
Cem 2	10	-	-	-
Cem 4	9	-	-	-
UF 12, fine	8	9,4	12,5	15,6*
UF 12, coarse	16	-	≥ 6,3[4,3]	7,8[4,3]

*quotient should larger than 15,6

[1] L 15 penetration length is less than 5 m at a W/C ratio of 1,0.
[2] Cemflux shows a penetration length less than 5 m at a W/C ratio of 1,0.
[3] Bleeding of the passed mixture is larger than 5 % at a W/C ratio of 1,0
[4] Melcrete is not included in the filtration test.

Table 6.26, Relationship (quotient) between the used slot apertures and the grain size (d_{95}). Quotients which are shaded are the ones that fulfil the demand of $b_{crit.}$ at a W/C ratio of 1,0.

		slot aperture		
Mixture	**d_{95} [µm]**	**75 µm**	**100 µm**	**125 µm**
UF 12	12	6,3	8,3	≥ 10,4[1]
UF 16	16	4,7	6,3	≥ 7,8[2]
IC 30	30	2,5	3,3	≥ 4,2[3,4]
Cem 2	10	-	-	-
Cem 4	9	-	-	-
UF 12, fine	8	9,4	12,5	15,6*
UF 12, coarse	16	4,7	≥ 6,3[2,4]	7,8[2,4]

* quotient should larger than 15,6

[1] Cemflux does not fulfill the demand of b_{crit}.at W/C ratio 1,0.
[2] L 15 does not fulfill the demand of b_{crit}.at W/C ratio 1,0.
[3] L 15 and Melcrete do not fulfill the demand of b_{crit}.at W/C ratio 1,0.
[4] Bleeding of the passed mixture is larger than 5 % at a W/C ratio of 1,0.

Cem 2 and Cem 4 could not be included in the analysis because the filtration tests are performed with a mesh geometry (75 µm) and with a smaller initial amount of mixture (110 cm^3).

Correlation tests of the results, like those done for the inert material, was not found relevant because of the bad correlation between the passed amount of inert material and the d_{95} and d´ value, see Table 6.18. The influence of the chemical reactions in the cement based mixtures will complicate the correlation even more.

The poor results (correlations) from the inert material caused the need for an alternative method to be tested to evaluate the cement based results. All performed cement based filtration experiments are evaluated with a multivariate analysis program called Simca©. The purpose of statistical modelling is to find the parameters and correlations which significantly influence the passed amount of material and the quality of the material that passes. The statistical modelling is constructed using a number of input variables (x-variables) and output variables (y-variables).

Table 6.27, Used variables in the statistical model.

Variable	Description	Remark
Input variables x variables	W/C ratio (-) Slot aperture (µm)	
Outpu variables y variables	Passed amount$_{rel}$ (-) W/C ratio$_{after}$ (-)	=Passed weight/ Sample weight

The used statistical model is constructed due to a linear model, were the influence of the input variable (x_i) on the output variable (y_i) is studied. In order to compare the different coefficient (a_i), the variables are normalised to comply with the following equation:

eq 6.2

$$y_i = a_0 + a_{W/Cratio} \cdot x_1 + a_{Slot\ \ aperture} \cdot x_2$$

The relative amount of material passed through the slot (Passed amount$_{rel}$) is calculated as the quotient between the used test weight of the volume (~490 ml) and the passed weight of mixture.

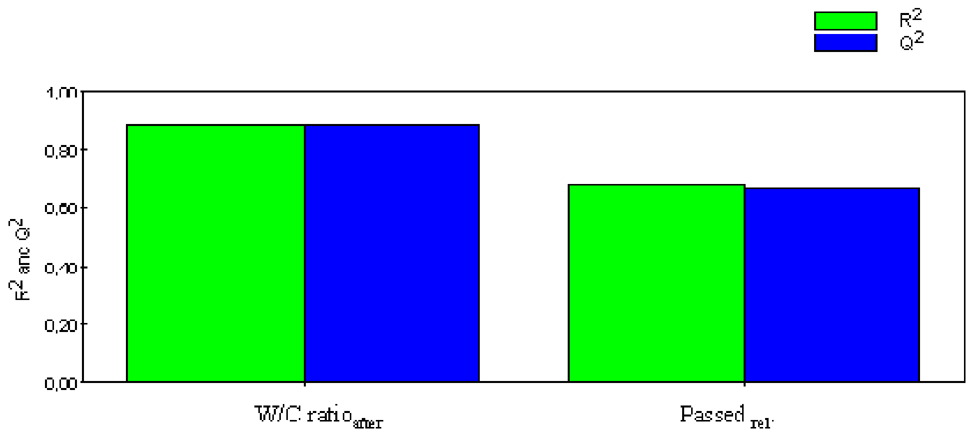


Figure 6.42, Correlation between used x- and y-variable in the used model. x-variables are W/C ratio and slot aperture. y-variables are passed$_{rel}$ and W/C ratio$_{after}$.

R^2 is a measure of how the x-variables are fitted to the used linear model. Q^2 is a measure of how sensitive the linear model is to a possible variation of the invariables (x-variables).

Both R^2 and Q^2 should be as close to 1,0 as possible, in order to have a model that in a good way describes the relationship between the x- and y-variables. W/C ratio$_{after}$ correlates fairly well with the x-variables (R^2=0,89). The fit of the measuring values of the passed amount (passed$_{rel}$) is a bit poorer (R^2=0,67). The Q^2 values for both y-variables are good (relative to the R^2 value), which indicates that the measuring values are of good quality and contain few outliers.

Description of the connection between the x- and y-variables can be made by illustrations like Figure 6.43. Figure 6.43 indicates that the passed amount (Pass. rel_{after}) is mainly affected by the used slot aperture. The W/C ratio after filtration is mainly due to the initial W/C ratio of the mixture. y_i concerns the spread of the variables Pass. rel_{after} and W/C $ratio_{after}$, the x_i concerns the spread of the variable's slot aperture and W/C ratio. Consequently Figure 6.43 concerns different measured data from the filtration tests on the cement based mixtures.

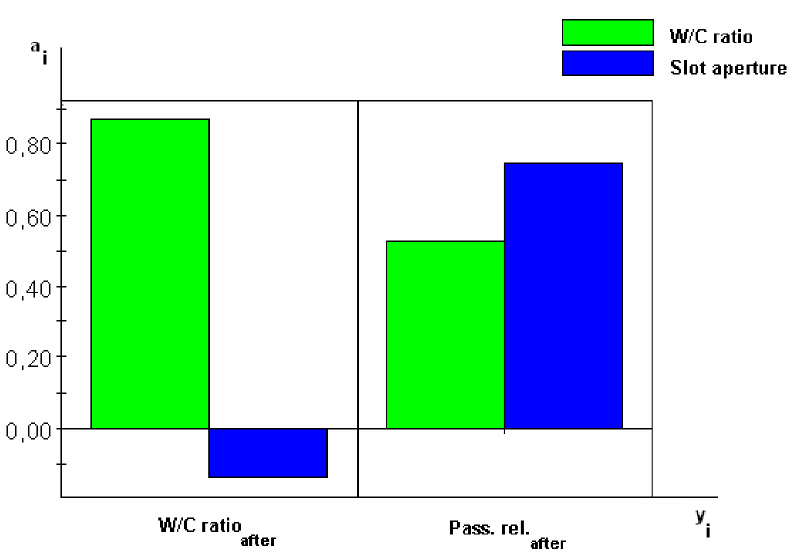


Figure 6.43, Influence of different x-variables on the y-variables.

a_i shows the influence of the x_i variable on the y_i variable with normalized x and y variables. The small negative influence of the slot aperture on the W/C $ratio_{after}$ indicates that a smaller slot aperture can cause a higher W/C ratio in the passed mixture.

Table 6.28, Used coefficicents in eq 6.2 which are illustrated in Figure 6.43.

	W/C ratio	Pass. rel.
Coefficient	[-]	[-]
a_0	3,36	1,05
$a_{W/C\ ratio}$	0,873	0,525
$a_{Pass.\ rel.}$	-0,142	0,746

In order to compare the measured values of passed amount$_{rel.}$ and W/C ratio$_{after}$ to the predicted values of the same, eq 6.2 is used, see Figure 6.44 to Figure 6.48.

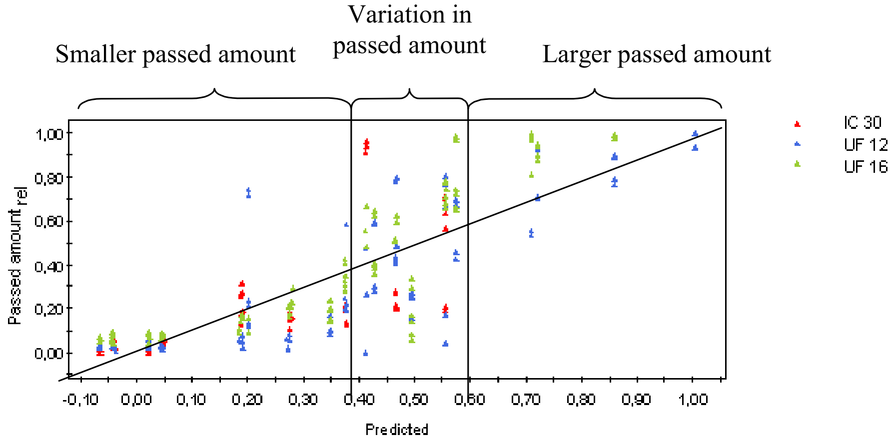


Figure 6.44, Statistical analysis of the passed amount of material through the slot as a function of cement type. *Predicted* concerns the predicted amount of mixture through the used slot aperture according to eq 6.2. The diagonal line across the figure marks the line at which the correlations (between predicted and measured values) should be placed along if the relationship between the measured and predicted would have been linear (according to eq 6.2**).**

The different colors in Figure 6.44 represent the three different types of cement used in the study. Red color represents IC 30, blue color is UF 12 and green is UF 16. The analysis has not taken any consideration to describe the cement in detail (e.g. chemical composition and grain size distribution). Figure 6.44 indicates that the predicted values are compared with the observed values (passed amount), a non-linear behavior can be detected.

The predicted and measured values should coincide along the diagonal line if the correlation is linear. The spread of predicted values is largest in the middle of the Figure 6.44. This non-linearity is one explanation to the relative low Q^2 value in Figure 6.42. The green marks (UF 16), seems generally to show a slightly larger passed amount than predicted, compared to the other cement types. The spread in the passed amount seems to be slightly larger for IC 30, compared to the other two cements.

As can be seen from Figure 6.44 the spread of correlation values is large in the middle of the graph (at approximately 50 % of predicted passed amount). This is basically because the required slot aperture to achieve a certain passed amount (slot apertures between b_{crit} and b_{min}) varies. Naturally the value of b_{crit} and b_{min} will also vary according to the mixture used. As an example, it can be mentioned that b_{req} varies within certain limits (between b_{crit} and b_{min}), see Figure 6.45. Consequently it can be stated that it is quite accurate to predict smaller and larger amounts of passed mixture according to the linear model (eq 6.2), but in the region between these two amounts (small and large) it is more difficult to predict the passed amounts of mixture (no linear model).

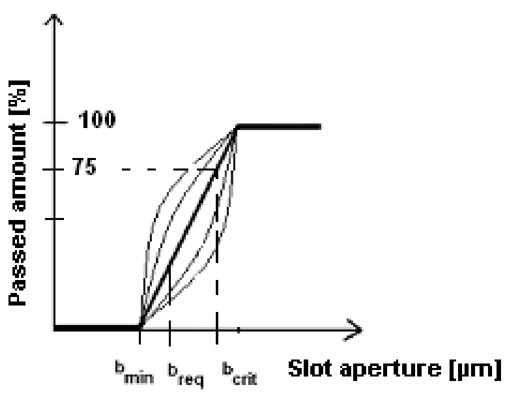


Figure 6.45, Evaluation of a slot aperture between b_{min} and b_{crit}, in order to pass a certain amount of mixture. The passed amount of mixture will consequently vary when using a certain slot aperture in combination with a certain mixture.

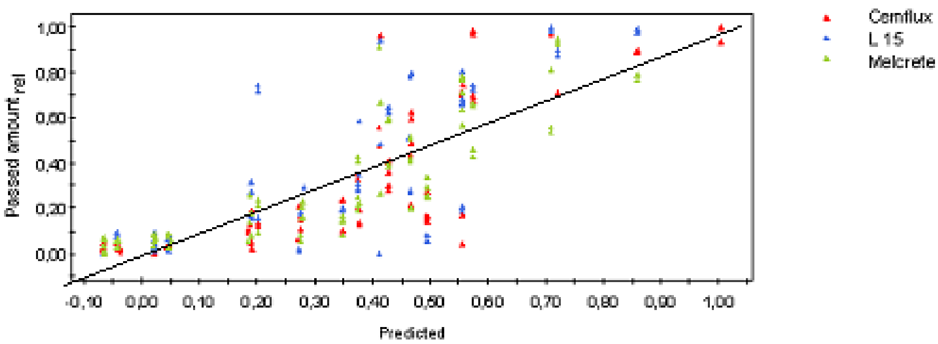


Figure 6.46, Statistical analysis of the passed amount of material through the slot as a function of the type of superplastisiser. *Predicted* concerns the predicted amount of mixture through the used slot aperture according to eq 6.2.

The different colors in Figure 6.46 represents the three different types of superplastisisers used in the study. Red color represents Cemflux, blue color is L15 and green is Melcrete. As can be seen in Figure 6.46 the spread of the measurements are largest in the middle of the figure, which is probably due to the same phenomenon as presented in Figure 6.44 and Figure 6.45.

The L15 (blue marked dots) seems to be slightly better than the other two types of superplastisisers, whereas the Melcrete (green marked dots) seems to be a bit poorer than the others. According to Figure 6.46 it seems that the influence of superplastisiser is not that influential as the used type of cement. This can be seen because of the dots of different colors are slightly more mixed up in Figure 6.46 than in Figure 6.44, where a more systematic difference can be seen.

Obviously the correlation between the observed and predicted value is not linear, but that will probably not affect the conclusion of the influence of used cement and superplastisisers.

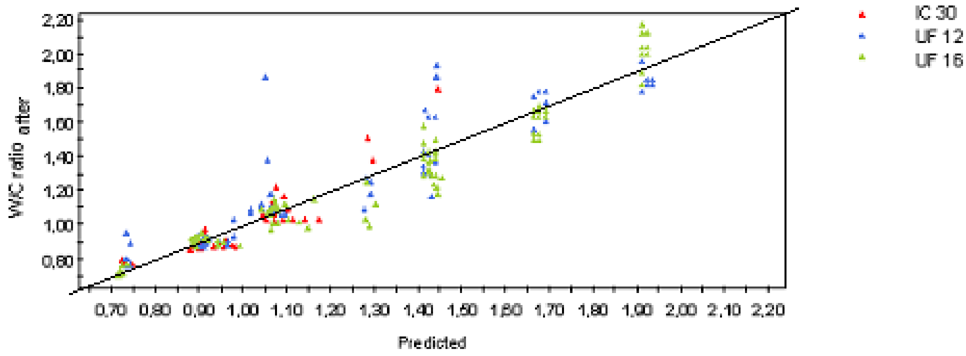


Figure 6.47, Statistical analysis of the W/C ratio after filtration, as a function of type of cement. *Predicted* concerns the predicted W/C ratio of the mixture after the mixture has passed the slot according to eq 6.2.

The correlation between predicted and observed values for the W/C ratio after filtration seems to be almost linear (the correlation coincides quite well with the diagonal line in Figure 6.47). No larger difference can be seen between different types of cement.

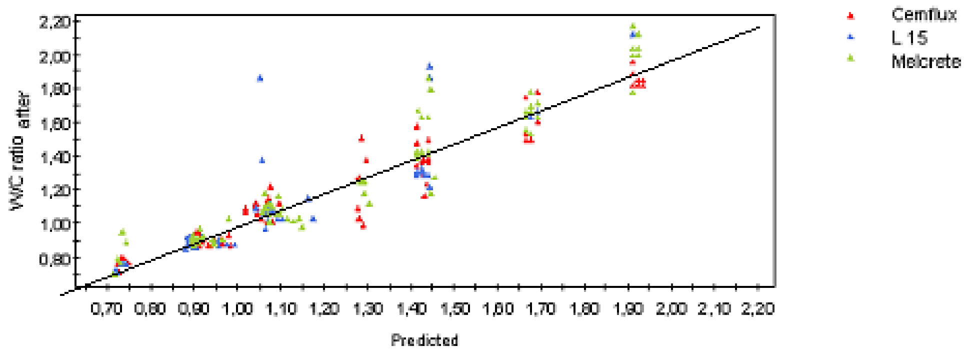


Figure 6.48, Statistical analysis of the W/C ratio after filtration, as a function of type of superplasticisers. *Predicted* relates to the predicted W/C ratio of the mixture after the mixture has passed the slot according to eq 6.2.

The superplastisiser consistent with Melcrete (green dots) seems to generate a slightly higher W/C ratio after filtration than predicted, especially when the filtration generates higher W/C ratio$_{after}$. The linear correlation between the observed and predicted value, seems to be rather good, even though some deviation can be seen in Figure 6.47 and Figure 6.48. The rather good linear correlation indicates that the W/C ratio before filtration is of great importance for W/C ratio after filtration. The earlier shown Figure 6.26, Figure 6.30 and Figure 6.34 indicate that an increased W/C ratio even will increase the bleeding of the mixture.

Table 6.29, Used coefficicents in eq 6.2 which are illustrated in Figure 6.44, Figure 6.46 to Figure 6.48.

	W/C ratio	Pass. rel.
Coefficient	**[-]**	**[-]**
a_0	0,365	-1,40
$a_{W/C\ ratio}$	1,06	0,5335
$a_{Pass.\ rel.}$	-0,0026	0,0115

The difference between the coefficients in Table 6.28 and Table 6.29 is because the coefficients in Table 6.28 are scaled and centered to a value between zero and one, while the coefficients in Table 6.29 are not scaled and centered.

6.5 Comprehensive conclusions cement based material

The filtration tendency of cement based mixtures is a result of a complex interaction of several parameters. The parameters can be summarized in two groups, one that concerns the physical parameters and the other that concerns the chemical parameters. The physical parameters are grain size, grain size distribution, concentration of grains in the mixture and the used slot aperture. The chemical parameters concern the reactions during the hydration of the cement grains. The physical and chemical parameters will generally interact with each other.

6.5.1 Influence of grain size, grain size distribution and slot aperture

The presented results all relate to the use of mixtures with W/C ratios between 0,7-1,75.

- Influence of grain size and grain distribution has been investigated by using seven types of cements. The cements are UF 12, UF 16 and IC 30, the other four cements are sieved to adhere to a certain grain size distributions, here called Cem 2, Cem 4, UF 12 fine and UF 12 coarse.

- The fine grained cement (UF 12) has a higher filtration tendency compared to the coarser grained cement (IC 30). Also UF 12 has a higher filtration tendency compared to UF 16 although they have almost the same grain size distribution and grain size. UF 16 has a somewhat steeper grain size distribution and a d_{95} value of 16 instead of 12 μm in the UF 12 cement. These results are all based on the same W/C ratio.

- According to filtration tests with the sieved cement Cem 2 and Cem 4 it can be said that it is not solely the grain size distribution that governs the filtration tendency of the cement based mixture. Cem 2 and Cem 4 represent grain size distributions which show the lowest filtration tendency regarding the inert mixtures. Comparison between the sieved cement of UF 12 fine and UF 12 coarse show that the fine grains in the cement have a negative influence onto the filtration tendency (higher filtration tendency).

- According to the performed filtration tests with cement based material, it may be concluded that it is advantageous for the penetrability to have a grain size distribution which not contain too many fine grains but neither too many coarser grains. The shape of the grain size curve should according to the performed tests be relatively steep (narrow grain size range). This result was the same as that obtained when testing the inert mixtures.

- Time dependent properties like surface- and pore water properties (the hydration process of the cement) can influence the passed amount of mixture through the filter. This has not been shown explicitly, but the results from the filtration tests give indications of a major influence of the chemical properties of the cement on the filtration tendency of the grout. The high filtration tendency of the mixture containing the UF 12, fine cement can be caused by flocculation of small grains into larger agglomerates. The risk of flocculation can increase as the specific grain surface increases (larger chemical reactivity in the mixture).

- The slot aperture has been shown in Figure 6.43 to be one of the most important parameter in order to predict the passed amount of mixture through the slot.

- To be able to fulfill the requirements of a penetration length of more than 5m and a bleeding of the passed amount of less than 5% (b_{req}), it is generally necessary to use a slot aperture of 95 µm and a W/C ratio >0,70 or a W/C ratio of 1,2 and a slot >75 µm. If all the available amount (b_{crit}) of mixture shall pass the filter, a slot aperture of 125 µm and a W/C ratio >1,25 or W/C ratio of 1,75 and a slot >100 µm has to be used (independent of the type of cement). Some of these combinations will show a bleeding of more than 5%.

- A larger spread of the passed amount can be shown in Figure 6.44 and Figure 6.46. The larger spread mainly occurs in the central part of the figures. This is probably due to the slot aperture used in combination with the mixture used (grain size, grain size distribution and W/C ratio etc.). Relative low spreads in the passed amount of mixture are shown in the region around slot apertures of b_{min} (small passed amount). In the transition zone from small to larger passed amounts of mixture (slot apertures in the region of b_{req}), the spread will, according to performed experiments, be larger. Lower spread is then shown at larger apertures than b_{crit} (large passed amounts). According to performed tests there seems to exist critical parameters that govern the passed amount of mixture through the used slot aperture (grain size, grain size distribution, W/C ratio, slot aperture etc.). When the values of these parameters increase or decrease the passed amount of mixture will be strongly affected.

- The ratio between the slot aperture and the grain size expressed as d_{95} is found to vary between 4-10 in order to fulfil the definition of $b_{crit.}$, which can be compared with a quotient between 3-4 when using the inert mixtures. The quotient shown for the cement based mixtures relates to a W/C ratio of 1,0. The W/S ratio used for the calculation of the quotient regarding the use of inert mixture is 0,7. The difference between the quotients could therefore have been different if the same grain concentration had been used for the comparison. It is likely to believe that the quotients between the slot aperture and d_{95} for the cement based mixtures would have been even larger if the evaluation had been performed at a lower W/C ratio e.g. 0,7. The W/C ratio of 1,0 was used because fairly stable mixtures (based upon the used cements) were achieved at this grain concentration (bleeding generally less than 5 %).

6.5.2 Influence of grain concentration

The presented results all relates to filter geometries with slot apertures of 75, 100 and 125 µm.

- Changes in the mixture's grain concentration causes differences in the passed amount of mixture through the slot. A higher W/C ratio will generally increase the passed amounts of mixture containing UF 12, UF 16 and IC 30

- When using fine grained cements e.g. UF 12 and UF 16, the W/C ratio can be kept higher without risk of major bleeding of the mixture compared to coarser grain cements (IC 30).

6.5.3 Superplastisisers

The presented results all relate to filter geometries with to slot apertures of 75, 100 and 125 μm.

- Some minor difference could be seen in the passed amount of mixture through the used slot apertures when comparing the steric and electrostatic superplastisisers. All tests in Figure 6.25 are performed within 5 minutes after mixing, during the time of major reactivity in the mixture (hydration process of cement grains).

- Performed filtration tests show that the passed amount of mixture decreased more rapidly with time when using electrostatic superplastisisers compared to use of the steric one, see Figure 6.40. Thus, the tests indicate that the repulsive effect between the grains of the electrostatic superplastisisers (L 15 and Melcrete) decline more rapidly than the steric one (Cemflux).

- Generally it can be seen that the steric superplastisiser (Cemflux) will decrease the yield- and viscosity values of the mixture to a larger extent compared to the electrostatic ones (L 15 and Melcrete).

7 Analysis of Results

7.1 General

It is hard and almost impossible to give any general advice that is valid for all types of grouting operations. Decisions regarding the composition of grout mixtures probably have to be based upon functional requirements of the grout properties (filtration tendency, rheology, bleeding, setting time etc.) and the properties of the grouted structure (water tightness, structural strength and durability). As discussed earlier in this work, the composition of the mixture is made by a number of compromises between different properties. For example, it can be mentioned that a high W/C ratio (low concentration of grains) will favour the penetrability (filtration tendency and rheology), but be a disadvantage to bleeding and setting time of the fresh grout and tightness of the grouted structure. Prioritising between different properties (fresh and hardened properties) has to be done for the individual grouting operation. Different grouting operations mean that different grout properties will be of more or less importance. Grouting of fine cracks will require a mixture with low filtration tendency and yield value. In another case, as for example when grouting cracks with high water pressure, a stiffer mixture (higher yield value) and a rapid setting time are desirable in order to avoid a rewind pressure and wash out of the mixture from the grout hole.

The main aim with this project is to map and explain the mechanisms of the freshly mixed grout which govern the plug formation and how to compose grout mixtures to fulfil the requirements of penetrability, see Figure 7.1.

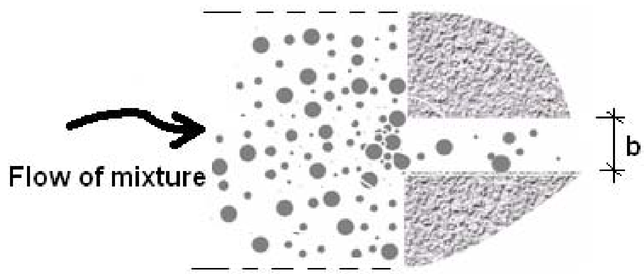


Figure 7.1, Flow and plug formation of a mixture at the entrance of an aperture of b.

In order to avoid plug formation the geometrical condition of the grains or agglomerates of grains have to be smaller then the aperture of b that is supposed to be penetrated by the mixture. In this work it has been shown that the grain size expressed as d_{95} can be compared to the aperture of b in order to analyse the aperture that is possible to be penetrated by the mixture. Theoretically it should even be possible to use the maximal grain size in relation to the aperture of b. Because of practical reasons it is hard to measure the maximum grain size in the mixture, due to the presence of oversized grains and agglomerates of grains.

7.2 Requirements of filtration tendency

Generally the filtration tendency governs fresh mixture property for the penetrability of cement based grout into fine cracks. The filtration tendency can be tested in the filter pump (prEN 14497), pressure chamber (VU:SC 48) or other similar devices like that described in (Eriksson 2003), which is also a type of pressure chamber. The basic idea of using measurement methods for filtration tendency is to identify the mesh or slot aperture when plug formation occurs at the mesh or slot entrance (measurement of the passed amount of mixture through the used slot or mesh aperture before plug formation occurs).

The requirements of the passed amount of mixture through a certain slot or mesh aperture should be related to e.g. the requirements of inflow of water into the tunnel. Consequently if the requirements allow a relatively high inflow of water (q), there is probably no use for a grout with a very low filtration tendency. The bigger cracks that have to be sealed will anyway be penetrated by the grout (provided that a grouting cement and ordinary grouting equipment has been used). On the contrary, if high requirements (low inflow of water) are set, it is of great importance to make the grout's filtration tendency as low as necessary to fulfil the requirements of inflow that were set and also seal the finer cracks.

The requirements of inflow into the tunnel can be translated to a required permeability of the grouted rock mass. Work done by Erikson and Stille (2004) show that grouting operations can be divided into three different classes depending on the desired permeability and sealing effect of the grouted rock mass (class 1, 2 and 3). The classes concern the sealing effects between 90% and higher. The sealing effect is defined by the quotient of the difference between the inflow in the ungrouted and the grouted structure divided by the inflow in the ungrouted structure.

eq 7.1

$$Sealing\ effect = \frac{q_{ungrouted} - q_{grouted}}{q_{ungrouted}} \quad [\%]$$

It can generally be said that if the grouting operation shall fulfil a sealing efficiency of more than 99% and a permeability of the rock mass of about 10^{-7}-10^{-8}, the filtration tendency of the grout must be regarded as an important parameter to fulfil this requirement. The same situation will occur if the requirement upon the sealing efficiency is lowered to 90-99%, but the permeability requirement is lowered to less than 10^{-8}. These two fictive examples are regarded to belong to the most difficult grouting operations, class 3 in the classification (Eriksson and Stille 2004).

If the grouted structure is exposed to for example one-sided water pressure in a concrete dam structure, the filtration tendency is generally of great importance. The cracks in the concrete structure can be due to e.g. overload, shrinkage, alkali aggregate reaction etc. all these damages cause generally relatively small crack apertures of approximately 0,05-0,5 mm. The cracks have to be penetrated a certain length, in order to resist the leaching of the cement paste through the dam structure (eg leach of chalk that deteriorates the strength of the cement paste). If only a short length of the crack is sealed (high filtration tendency of the grout) the grouting operation will show a lower resistance to leaching compared to a case where a grout with a lower filtration tendency is used.

The necessary penetration length (grouted zone) around a tunnel structure varies from between 5 to 25 meters depending on the desired sealing effect and the original inflow and pressure of water (Eriksson, 2002).

To fulfil the requirements of filtration tendency it is often of importance to pretest the mixtures properties, before using them in a real grouting operation. Pretesting can give valuable information on the variation of the mixture's properties (fresh and hard properties) due to variation of different variables such as used cement, W/C ratio, superplastisisers, additives etc. Pre-testing is suitable to perform in a laboratory environment were most of the conditions of importance for the grout properties can be controlled (such as surrounding air temperature, temperature of the mixing water, accurate amount of the substances in the mixture). The pretest will result in a number of recipes that result in different properties of the mixtures. Suitable mixtures can then be used in different grouting situations in order to fulfil the requirements of for example water tightness and structural strength, which in many cases are governed be the mixture's filtration tendency.

7.3 Main conclusions

Based on the performed tests and literature review presented in chapter 2 and 3, the following conclusions have been stated:

- Filtration tendency is a complex phenomenon which depends on several parameters. The parameters can also interact.

- The design of this work means that some caution should be taken regarding the conclusions. The work has mainly focused on the principals of filtration tendency.

- b_{crit} and b_{min} are two important parameters which have been discussed earlier. It can be questioned if there exists an unambiguous value of b_{crit}. The tests indicate that the evaluated b_{crit} will in many cases be dependent on the used amount of mixture in the test. The relative amount that passes through the used filter will therefore be related to the initial amount of mixture.

- b_{req} is a new concept which is introduced in order to show that the passed amount of mixture can be enough regarding grouting of cracks, although plug formation finally will occur.

- The used method of testing the mixtures filtration tendency is probably of importance. Important parameters are for example used initial volume of mixture, filter geometry and pressure gradient over the constriction.

In order to analyse the filtration tendency of a certain mixture, the filtration tendency can be expressed according to performed tests as a relationship between the aperture and the grain size expressed as d_{95}, see eq 7.2.

eq 7.2

$$b_{crit} > k \cdot d_{95}\,; \quad 2 \le k < 16$$
$$k = f(\textit{grain size}, \textit{grain size distribution}, \textit{superplastisiser}, W/C\ \textit{ratio}, \textit{chemical reaction}, \textit{geometry of aperture}, \textit{amount of mixture})$$

According to performed filtration tests with inert and cement based mixtures k should be larger than 2 in order to avoid plug formation. The value of k has in this study been found to vary between at least 2 to and up to more than 16, in order to pass a major part of the available amount of mixture (75-100%). The variation of k is due to a number of parameters of the mixture, see **eq 7.2**.

- Results from filtration tests with inert based mixtures using a slot aperture show that the value of k has to be **more than 12**. The results relates to the use of a slot aperture of 75 and 125 µm and a W/S ratio of 0,7.

- Results from filtration tests with inert based mixtures using a mesh aperture show that the value of k has to be not less than **3-4**. The results apply for the used mesh apertures of 36, 45 and 75 µm and a W/S ratio of 0,7.

- When using a monodisperse mixture (scaled-up tests) the value of k has been found to be between **2-3**. The result is applicable for a slot aperture and a W/S ratio of 1,3

- Results from filtration tests using cement based mixtures show that the value of k has to be between **4-10,** some results regarding very fine grained mixtures show that a value of k should be **more than 16.** The results are applicable for slot apertures of 75, 100 and 125 µm and a W/S ratio of 1,0.

In a similar way the value of k can be described for the case of using b_{req}.

eq 7.3

$$b_{req} > k \cdot d_{95}\,; \quad 2 \le k \le 8$$
$$k = f(\text{grain size, grain size distribution, superplastisiser,}$$
$$\text{W/C ratio, chemical reaction, geometry of aperture, amount of mixture})$$

The relative passed amount corresponds to roughly to 10 % of the available amount (490 cm^3) of mixture.

The parameters in **eq 7.2** and **eq 7.3** will influence the filtration tendency of the mixture (value of the aperture b_{crit} and b_{req}) in different ways. Lower filtration tendency of the mixture will decrease the value of b_{crit} and b_{req}. In Table 7.1 an approximation of the different parameters influence on the filtration tendency has been made. The degree of safety in the judgement is also estimated in Table 7.1.

Table 7.1, Description of parameters that influences the filtration tendency of the mixture.

Parameter	Influence	Degree of safety
Steep grain size distribution	Decreasing of k	Large
Higher W/C ratio	Decreasing of k	Large
Cement chemistry	Increasing of k	Large
Superplastisiser	Decreasing of k	?[1]
Filter geometry	?[2]	?[3]

[1] The influence and use of superplastisisers have not been the focus for this work. If the dosage and type of superplastisisers had been changed, the results may have been different. However, it can probably be stated that a correct use of superplastisisers will increase the mixtures penetrability.

[2,3] In this work, the influence of the filter geometry has found to be of importance. The results are however in some cases not that clear. More work has probably to be done in order to better analyze the influence of the used filter geometry on the mixture's filtration tendency.

In order to illustrate the influence of the factor k on the passed amount of mixture through an aperture of b_{crit}., it can be supposed that k can be related to b in the following way, see Figure 7.2.

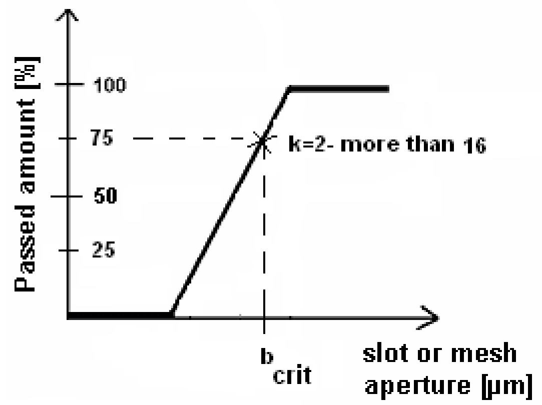


Figure 7.2, Example of the influence of k onto the slot or mesh aperture b. The smallest value of k=2 was found from the scaled-up filtration tests using a monodisperse mixture with slot geometry. The larger value of k >16 was found when using a fine grained cement based mixture and a slot geometry.

It should be noted that some of the mixture does not completely fulfil the definitions of b_{req} and b_{crit} (penetration length and bleeding), see Table 6.25 and Table 6.26. The mixtures containing coarser grains will generally show a bleeding of more than 5 % with regard to the passed amount of mixture.

This result only partly confirms the old rule of a thumb that recommends the grain size expressed as d_{95} of the dry powder to be smaller than 3 times the aperture that is supposed to be penetrated (Bergman 1970). The use of inert mixtures, that generated a d_{95} value smaller than between 3-4 has a better compliance to the rule of thumb (compared to the cement based mixtures). The old rule of thumb can be seen as a lower limit of the k value. The shown interval of k regarding b_{crit} and b_{req} show the complexity of the filtration tendency phenomenon.

Plug formation in the performed tests is probably due to cake filtration (Svarovsky, 1985). The plug of grains builds-up between two or more adjacent obstacles in the mixture’s flow path.

7.4 Detailed conclusions

In the following sections of this chapter a summary of the more detailed conclusions regarding the influence of grain size and grain size distribution, grain concentration and bleeding, rheology and superplastisisers and cement chemistry on the filtration tendency have been performed.

7.4.1 Grain size and grain size distribution in relation to the aperture

A more flattened grain size distribution will increase the amount of small grains. The smaller grains have an increased risk for flocculation (create agglomerates) that will increase the filtration tendency of the mixture. It can probably be stated regarding the use of modern micro cements for grouting purpose (grain sizes up to approximately d_{95} =30 µm), that the small grain sizes are of crucial importance in order to develop plug formation because of the increased risk of the smaller grains to flocculate into larger agglomerates, which will initialise plug formation at the slot entrance.

Tests performed by Eriksson 2002, indicated that the length (thickness) of the plugs in the filtration experiments are different due to the different cement used (grain size and grain size distribution). This was probably because the thicker plugs were made of grains with a sharper grain size distribution compared to thinner plugs consistent with a wider grain size distribution. A wider grain size distribution will consequently in a more effective way create a watertight plug, based on a smaller volume of grains. If the mixture had been consistent with a monodisperse grain size distribution, no plug formation would have occurred which could stop the flow of the liquid phase through the plug. The created plug had always been pervious to water.

The influence of used filter geometry has been investigated by using different slot and mesh apertures in the filtration experiments. The design (slot or mesh) and size of the filter geometry have in this work been found to be of importance for the measurement of the mixtures filtration tendency.

In the case of the mesh geometry a lot smaller initial amount of mixture had to pass each filter opening of b compared to the case with a slot geometry (when using the same available amount of mixture). According to the performed filtration tests, it is reasonable to believe that the probability of plug formation will increase if larger amounts of mixture pass the filter. The performed tests show however that larger penetration lengths were achieved in the case of using the slot geometry compared to the use of mesh geometry. The larger penetration length is probably due to differences in the build-up of grains around the entrance of the aperture.

In the scaled up experiment, that use larger grains and larger filter geometries show that the filtration tendency can be visualized. The location of the initialisation of the plugs along the slot in the scaled up model seems to be a stochastic process. Smaller plugs along the slot entrance will after a while create a plug that covers the whole slot length or a wide range of the slot length.

In the scaled up experiments, the influence of the grain shape is investigated by the use of irregular black (rombic shaped) and regular white (spherically shaped) grains. No differences, between the black and white grains were shown. However, there ought to be a difference due to the difference in shape between the black and white grains. The reason for this assumption is because the frictional forces between the grains, which become dominant when the concentration of grains become high, should be dependent at least to some extent on the grain shape.

In the case of using the mesh, a plug of grains can build-up in four directions around the aperture. This compares to the case of using a slot aperture where the build-up of grains can only be done in two directions. The abutment for plug formation against the wires (mesh) can also be better compared to the edges of the slot aperture. A better abutment can create a more stable plug formation, see Figure 6.8. Theory of grain build-up between to obstacles has earlier been studied by Matinet, 1998.

In an actual grouting situation in a fractured rock or concrete structure, the probable build-up of grains around the aperture will be two dimensional, which complies with the case of using a slot geometry, but that is subject for further research. Gustavsson 2004 showed that the cracks in a rock structure are barely plan parallel to each other and the borehole can strike the crack plane in any direction, see Figure 7.3.

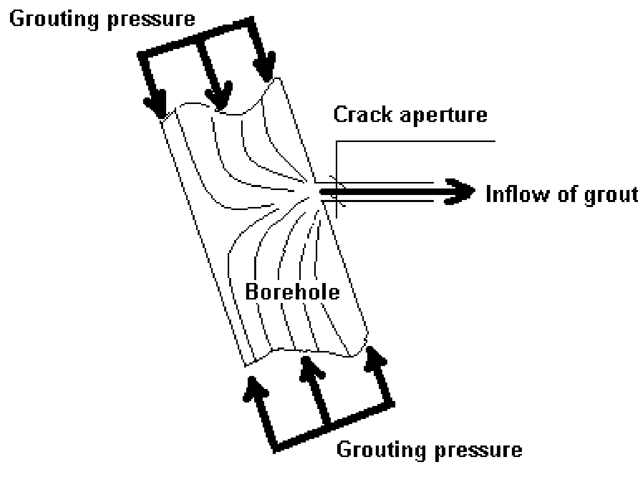


Figure 7.3, 2D figure of the borehole and an aperture. Illustration of inflow of grout from a borehole into an aperture in a rock or concrete structure.

Practically, it is probably that the parameter $b_{req.}$ is the most valuable in a grouting operation. This parameter may be evaluated in a pre-testing operation. $b_{req.}$ can be specified according to the desired penetration length (grouted zone) and the quality of the passed mixture through the filter (W/C ratio of the mixture after passing the filter). Basically, the $b_{req.}$ will be governed by the desired sealing effect and the permeability of the grouted structure. A high desired sealing effect and a low permeability would consequently mean that $b_{req.}$ is set to a low slot aperture in the filtration test.

7.4.2 Grain concentration and bleeding

The old way of dealing with penetration of small crack apertures was to start the grouting operation using a mixture with a high W/C ratio (W/C ratio of 3 and even higher). The grains would then create a plug (plug formation) that consists of a far lower W/C ratio than the original mixture. The position and extension of the plug (penetrated crack volume) will be uncertain. The W/C ratio should therefore be kept as low as possible from the beginning in order to achieve a durable grouting performance. This can be done by the use of a mixture with a suitable filtration tendency and rheology.

A low W/C ratio in the mixture is associated with a number of disadvantage properties of the mixture. For example can be mentioned that the W/C ratio will strongly affect the tendency for flocculation of grains in the mixture. A lower W/C ratio will increase the risk of extensive flocculation compared to a mixture with a higher W/C ratio.

The increased filtration tendency is mainly due to flocculation of grains into larger agglomerates which, for example, causes plug formation at the slot entrance. The speed of the chemical reactions (hydration of the cement grains) will be faster with a lower W/C ratio. Fast hydration can obstruct the rheology (increase of yield and viscosity) and lower the penetrability of the mixture. The negative influences of a low W/C ratio on the filtration tendency and rheology can in some cases be treated by the use of superplastisisers. The increased flocculation and chemical reactivity will cause a limited ability to rearrange the grains (compared to a high W/C ratio) within the mixture. The ability of rearranging the grains in the mixture has earlier been illustrated in Figure 3.12.

According to performed tests the filtration tendency is more influenced by an increased W/C ratio in fine grained mixtures compared to mixtures containing coarser grains. Higher W/C ratio will generally lower the filtration tendency to a larger extent than a similar increase of W/C ratio in a coarser mixture.

The bleeding is generally larger (at the same grain concentration) for the inert mixtures compared to the cement based mixtures. A large bleeding of the mixture can probably be related to a large possibility to rearrange the grains of different sizes within the mixture, because of the large distance between the grain surfaces in a mixture. The large distance can be caused by e.g. low grain concentration (high W/C ratio) or an inert material that will not react and create new material in the mixture (as in the case of cement hydratisation).

Depending on the cement chemistry, pore water chemistry and the surface chemistry in the grout mixture, a cement gel will be created around the grains in the mixture, see chapter 4. This gel will probably decrease the possibility of the grains to rearrange within the mixture and therefore probably also decrease the bleeding in the grout. The fine grained cements (for example UF 12 and UF 16) can be mixed to a W/C ratio of about 1,50 and still show a bleeding of less than 5%. According to performed tests, the mixtures consisting of coarser cement grains (for example IC 30) show a bleeding that exceeds 5% at an approximate W/C ratio of 1,0.

7.4.3 Grain concentration and numerical simulations

The numerical simulations of plug formation show that it is possible to see variations of grain concentration near the slot entrance depending on the initial grain concentration in the mixture and the slot aperture. The variation of grain concentration near the slot aperture can be used as an indication of the probability for a plug formation to occur. A large difference in flow velocities above and within the slot is, according to this numerical experiment, also a sign of increased probability for plug formation at the slot entrance. When the concentration gets high near the slot entrance, the flow velocity decreases within the slot compared to the grain flow velocity above the slot. If this is the case an accumulation of grains will occur at the entrance of the slot. In the current model the probability of plug formation near the slot entrance can be evaluated by using the grain concentration and the grain flow velocity as indicating parameters.

No increase of grain concentration near the slot entrance (no matter how long the simulations were run) could be seen for either the 5 or the 10 mm slot aperture cases with an initial grain concentration of 5 % in the mixture. For the 10 mm slot aperture, an increase of grain concentration near the slot entrance could be seen at an initial grain concentration in the mixture of 20 %. If the grain concentration was increased up to 30 % the mixture caused a significant increase of grain concentration near the slot entrance (plug formation is therefore likely to occur).

According to the performed numerical simulations, it is believed that the plug formation occurs when the inter-particle spacing becomes small (high grain concentration at the slot entrance). When this happens the dominant forces will be the frictional forces between the grains. This has also been stated by Wachem (2000).

This frictional force model "kicks in" when the local solid’s volume fraction becomes higher than about 50 %. Because of the direct dependence between the formation of a plug and the frictional force model it is very crucial to have a good model for the inter grain frictional forces. Moreover it might be feasible to set the “kick in” volume fraction lower than 50 %. It is likely to believe that the numerical simulation in the future can serve as a useful tool in order to predict filtration tendency of a grout mixture.

From the numerical simulations it can be concluded that some similarities between the filtration and scaled-up experiments were found. It should be stressed that at present the numerical model is far too crude to be qualitatively compared with the real experiments (filtration and scaled-up experiments). However, it is a first step towards a numerical simulation of a real grain mixture and in the far future maybe even a real grout mixture. More development is necessary in order to improve the algorithms and criteria for plug formation.

7.4.4 Rheology and superplastisisers

In order to achieve a good penetrability (filtration tendency and rheology) of the grout mixture, it is probable according to performed filtration tests, of importance to have a relatively low yield- and viscosity value of the mixture. The necessity of using superplastisisers is especially illuminated when using fine grained mixtures. The use of superplastisisers in the grout mixture will increase the possibilities to influence the fresh properties compared with only using cement and water in the mixture. The superplastisisers mainly influence the flow properties (fluid phase) in the mixture (viscosity and yield value). The addition of superplastisisers will decrease the yield and viscosity in the mixture. The yield- and viscosity values of the mixture seem to vary within relatively wide limits and still show a low filtration tendency (for the mixtures within these limits of yield- and viscosity values).

Results presented by Hansson 1997 show that it was difficult to predict the mixture's filtration tendency by the help of rheological measurements. Further development of rheological models in order to predict the mixture's penetrability has also been made by Eriksson 2002 (based upon a model developed by Hässler, 1991). This work also concludes that it is hard to predict the mixture's filtration tendency by the help of existing rheological measurements and models.

The superplastisisers also have an effect on the grains, (solid phase) because of the decreased risk of flocculation due to the creation of repellent forces between the grains. Superplastisisers can generally have a large influence on fine grained mixtures such as mixtures containing the UF 12 cement. The penetrability can decrease even though a cement with smaller grain sizes is used (compared to a coarser one). This is probably due to flocculation of the smaller grains into larger agglomerates which will initialise the plug formation.

There seems to be a difference between different types of superplastisisers (steric and electrostatic ones). Generally it can be seen that the steric superplastisiser (Cemflux) will decrease the yield- and viscosity values of the mixture to a larger extent compared to the electrostatic ones (L 15 and Melcrete). This has also been found by Fjällberg (2003). The repulsive effect of the electrostatic superplastisisers seems to decline more rapidly compared to the steric ones.

Commercially available cement based grout mixtures with addition of superplastisiser can generally achieve a yield value and viscosity of less than 2 Pa or 0,10 Pas, with a bleeding of less than 5 %.

The variation of yield- and viscosity values can according to performed rheological measurements (at W/ C ratio of 1,0 and UF 12, UF 16 or IC 30), be varied between 5-0,1 Pa and 0,01-0,1 Pas (depending on used grain size and grain size distribution).

A relatively high yield- and viscosity value of approximately 5 Pa and 0,1 Pas for UF 12 generated a relative passed amount of mixture of 30-40%. A relatively low yield- and viscosity value of approximately 0,1 Pa and 0,01 Pas for IC 30 generated a passed amount of mixture of 10-20%. These results are valid for the use of a slot aperture of 125 μm. Even in this case with the cement based mixtures, it can be seen that a low yield- and viscosity values will not always generate a low filtration tendency. This is probably because of the influence of the mixture's different grain sizes expressed, as for example, by d_{95}.

7.4.5 Cement chemistry

In reality the possibilities of affecting the chemical composition of the cement (based upon commercially available cements) are relatively small. The composition of the cement is to a wide extent governed by the raw material content in the cement manufacturing process. There is, however, some possibilities of affecting the cement reactions in the grout.

The gypsum reactions are probably one of the dominant early cement reactions which influence the filtration tendency of the freshly mixed grout. The added gypsum must balance the rapid aluminate reactions in the cement. A shortage of added gypsum will create false setting of the grout and consequently contribute to a bad penetrability (Bradley, 1986). An overdose of added gypsum will probably also influence the penetrability in a negative way. Other early cement reactions of interest are the silicate reactions, which affect the setting time of the mixture. W/C ratio is however probably one of the most influential parameter on to the setting time. Additives like retarders or accelerators can, of course, also control the setting time.

The results from the filtration experiments indicate that there is a difference between the filtration tendencies of a mixture consisting of inert or cement grains, although the grain concentration and grain size and grain size distribution are almost the same. It is probable to believe that the hydration process in the fresh grout mixture, influences the filtration tendency. Performed tests with cement based material indicate that a major part of fine grains in the mixture will increase the filtration tendency of the grout mixture.

The fine grained cements have a larger specific grain surface area compared to the coarser ones. The larger specific surface area will cause the speed of the early chemical reactions to increase. An increased chemical reaction speed will generally increase the risk for creation of agglomerates of grains in the mixture, which consequently will higher the filtration tendency.

8 Proposal to continued research

8.1 General

There are basically three main areas that are of interest to further develop in order to map and explain the mechanisms of the freshly mixed grout that govern the filtration tendency. The main areas are filtration tests, scaled-up experiments and numerical simulations. This work has mainly focused on filtration tests of inert and cement based grouts. The performed filtration tests of grouts can of course be expanded and developed by using, for example, several other grain size distributions, other types of cements and other test equipment. An alternative and complementary way of dealing with the filtration tendency is to further develop the scaled-up experiment and numerical simulations. The scaled-up experiment has primarily to be developed in terms of improvements of the test equipment (for example better defined used grain concentration and measurements of flow velocities within the model). The numerical simulation has to develop its equation and algorithms in order to handle flow of densely packed mixtures of grains and fluid.

Other fields of interest to further develop and investigate are grout mixer design, test equipment for field use (regarding filtration tendency) and optimisation of the used cement (physical and chemical properties). A larger field study of a grouting operation at, for example, a tunnel project can be performed in order to investigate the practical use of certain ideas about penetrability of cement based grouts.

8.2 Development of test equipment for laboratory and field use

The design of the measurement systems suitable for laboratory and field use is not that easy to predict. Both the systems should measure representative parameters like the passed amount through a filter (filtration tendency), yield value and viscosity, on a representative grout sample.

The influence of the used filter geometry, initial amount of mixture and applied pressure gradient over the filter geometry has to be further investigated in laboratory tests.

Testing of grout mixture on site is of importance to secure the quality of the used grout. Robust methods for testing are a subject for further development. It is desirable in many aspects to use insitu measurement systems in the mixing procedure of the grout. Measurement systems for filtration tendency and rheology could maybe be placed on the outlet of the stirring chamber for example.

8.3 Filtration experiments

The many parameters (W/C ratio, slot- and mesh geometry, grain size and grain size distributions, superplastisiser etc.) that were investigated in this work caused that a limited number of experiment were performed (in order to reduce the number of experiments). The statistical bases of the conclusions about the influence of different parameter onto the filtration tendency can in some times be a bit brief in the presented work. A larger number of experiments of each test combination should of course improve the statistical bases of the conclusions.

The inert and special produced cement material was very time consuming to produce. The inert and special produced cement materials available for testing have therefore been limited. It would be desirably to use a larger amount (volume) of mixture in order to investigate the influence of used initial amount in relation to the filtration tendency (the stochastic filtration process).

The influence of different gypsum additives is an interesting area to investigate. The rate of soluble and sparingly soluble gypsum is probably an important parameter to analyse when producing cements with low filtration tendency. The soluble gypsum commonly consists of $CaSO_4*2H_2O$ (semi hydrated gypsum) and the sparingly soluble gypsum can be $CaSO_4$ (Anhydrite). Experiments should be made varying the amount of these two gypsum types.

This study just focused on to the use of a certain cement clinker mineral (Portland clinker), it would, of coarse, be of interest to use for example aluminate cement or a portland cement with the addition of slag, fly ash or other additives in order to investigate their influence on to the mixture's filtration tendency.

The additional investigations regarding filtration tests will probably get even more valuably information of the influence of the mixture's ingredients and its potential risk of plug formation.

8.4 The physical model

The scaled-up experiment can be improved in numerous aspects. It would be desirable to run the experiment for a longer time. Moreover it is important to be able to control the volume fraction of grains at the model inlet. A mechanism for dropping the grains into the model at a controlled volumetric rate should be constructed. In addition the model should be rebuilt to make it easier to remove the grains at the bottom of the model after each test is finished.

Measurement of the pressure drop, volumetric flow (mixture of grains and fluid) and flow of grains should be done within the slot and nearby the slot entrance and outlet. These measurements would make it easier to do qualitative, and maybe even quantitative, distinctions between each experiment (influence of for example different grain concentrations and slot apertures). It would be interesting to do the experiment with different grain sizes distributions. The roughness of the crack should be altered. However it should be kept in mind that only one parameter at a time should be changed. If all of the above improvements of the experiments where realized the repetitiveness of the experiment could be checked, which would imply that the statistical and random nature of the experiment could be verified in a more rigorous manner.

It should be kept in mind that it is of limited interest to simulate formation of mono sized grains. Cohesive and interlocking are also neglected, which is probably is of major importance for the plug formation in a real grout mixture. The present type of experiment will probably anyhow improve the understanding of how the mixture's physical properties, e.g., the grain concentration, grain size and grain shape, influence the probability of plug formation.

8.5 The numerical model

The numerical simulations performed in this work can be regarded as a first step towards a useful tool of predicting the risk of high or low filtration tendency for a certain mixture. In this work the attention is focused on the frictional forces between the grains. The frictional forces in combination with the slot geometry are probably two of the most important of a vast number of parameters, which affect the filtration tendency of a real grout. Major efforts should be devoted in order to get the frictional model as close to reality as possible. The frictional model used in this work is a semi-empirical model. Therefore, the success of the model is to a large extent dependent on good experiments.

The success of the numerical models is probably to a large extent dependent on good experimental results in order to get high-quality correlation for the different quantities that are modelled. To improve the numerical model even more additional transport equations for the frictional and adhesive forces between the grains could be programmed. A function for the mass flow of grains, as a function of time through a given cross-section of the model should be programmed in order to get a quantitative measure on whether a plug is forming.

When this is done the focus of attention should be turned to the other parameters like the time dependent properties (hydration of cement grains) and boundary conditions. The no-slip boundary conditions used for the granular phase are not correct, hence a partial slip boundary condition, which would be closer to reality, should be implemented. Equations for chemical reactions in the mixtures and time dependent rheological models should be implemented. The interphase momentum transfer correlations need to be improved, i.e., the momentum transfer between grains and the fluid phase.

A grout mixture is a non-spherical non-elastic multicomponent grain mixture. Preferably 3D simulations instead of 2D simulations should be performed. Simulations of complex geometries are probably necessary in order to predict the initialisation of the plug formation. Lagrangian simulations (instead of the performed Eulerian simulations) of such a mixture will probably require an order of magnitude increase in computational power and a difficult extension of the granular dynamics theory.

In an iterative way, the numerical model for penetrability of grout mixtures could be improved and give results closer to reality.

8.6 Mixer design

Further investigation has probably to be made in order to analyse the influence of the mixer design on the filtration tendency. It is of importance for the rheology and filtration tendency to quantify the rate of dispersion of the grout. A measuring system for the dispersion rate would certainly make it easier to optimise the mixer design and the procedure of the mixing process.

8.7 Field tests

The field test ought to be performed in a tunnelling project. Parameters of the grout mixture like grain size, grain size distribution, grain concentration and the use of superplastisisers could be tested at different grouting situations. Suitable inspection methods shall be used to evaluate the results. Examples of methods can be drilling of inspection holes in the grout curtain, measuring the loss of water under a certain pressure head in a number of holes. The major problem with this kind of field measurements is however the various conditions of the rock along the tunnel (as for example various crack apertures, rock type and ongoing inflow of water). The field tests can probably illuminate the properties of the fresh grout mixture that are of major influence in the actual grouting operation. Different grouting operation will probably always highlight different grout properties as the most important.

9 References

1. Alemo J (2004), *Injection and sealing of cracks*, Report from the Rehabcon project, Vattenfall Utveckling AB, Älvkrleby, Sweden.

2. Alemo J, Hansson P. (1997) *Cementitious injection grouts - penetration ability. Selected research studies from scandinavia – Dedicated to professor Göran Fagerlund on his 60th anniversary*. Lund Institute of Tech. Report TVBM-3078. pp 1-4.

3. Alemo J,Johansson N, Bronner N (1988). *Urlakning av cementinjektering*. Vattenfall FUD-rapport. Råcksta, Sweden.

4. Arenzana L, Krizek R.J, Pepper S.F (1989). *Injection of micro fine cement suspensions into fine sands*. International Conference on Soil Mechanics and Foundation Engineering 12. Rio De Janeiro 1989. pp 1331-1334. Balkema Rotterdam 1989.

5. Axelsson M, Nilsson J (2002). *Sealing of narrow fractures in rock with use of silica sol. An introductory study on material characteristic and behaviour as a grout*. Chalmers University o Technology, Göteborg, Sweden.

6. Axelsson M, Turesson S (1996). *Laboratorieförsök och projektutvärdering, Glödbergstunneln*. Examansarbete 96/12, Dept. Of Soil and Rock Mechanics, Royal Institute of Technology, Stockholm.

7. Barnes P (ed). Structure and performance of cements. Applied Science 1983, pp 270-275.

8. Batchelor, G.K. (2001). *An introduction to fluid mechanics*. Cambridge Mathematical Library, Cambridge. ISBN 0-521-66396-2.

9. Bergman , S.G.A. Lindman, K. Söderman, P (1970). *Injekteringsmedels inträngning i sand och tunna spalter*. BFR-rapport R 45:1970, Stockholm.

10. Bernstone C, Thorsell P.E, Elforsk rapport, (2001). *Uppgradering av Forshuvuddammen, en demonstrationsstudie av oförstörande undersökningsmetoder och cementinjektering*.

11. Betonghandboken, *Material*, utgåva 2 (1994), Svensk Byggtjänst, Stockholm.

12. Bradley G, Howarth I M. (1986). *Action and rate to cement hydration*. Cement Concrete Aggregates. pp 68-75.

13. Brandberger M, Dahlmalm T, Eriksson M, Stille H (1998) Styrande faktorer för täthet kring en förinjekterad tunnel, Division of Soil and Rock Mechanics, Department of Civil and Environmental Engineering, Royal Institute of Technology, Stockholm, Sweden.

14. Brantberger & Nelson, (1998) Hallandsås Ridge Railway Tunnels- Development of pre-grouting methology, proc. Underground Constructions in Modern Infrastructure, Stockholm, Sweden, (Franzé, Bergdahl & Nordmark eds.), Balkema, Rotterdam, ISBN 90 5410 964 5.

15. Chen Z, Narayan S.P, Yang Z, Rahman S.S (2000). *An experimental investigation of hydraulic behaviour of fractures and joints in granitic rock*. Int. J. Rock Mech. & Min. Sci. 37, pp 1061-1071.

16. Cleveland W.S, Devlin S.J. *Locally-Weighted Regression: An Approach to Regression Analysis by Local Fitting*, Journal of the American Statistical Association, 83:596-610, 1988.

17. Cleveland W.S. *Visualizing Data*. Hobart Press, Summit, New Jersey, U.S.A., 1993

18. Committee on Grouting, 1980, *Preliminary Glossary of Terms Relating to Grouting*. Journal of the Geotechnical Engineering Division, ASCE. 106 (GT7): 803-815.

19. Crawford A.M, G. Graskopf, (1984) *Groutability ratio for filter blocking of joints in rock*, Department of Civil Engineering University of Toronto, Canada.

20. Crowe, C., Sommerfeld, M. and Yutaka, T. (1998). MULTIPHASE FLOWS with DROPLETS and PARTICLES. CRC Press, New York. ISBN 0-8493- 9469-4.

21. Cumberland D J (1985), Crawford R.J. Handbook of powder technology, Volume 6, *The Packing of particles*, Elsevier.

22. Dalmalm, T., (2004), Grouting Prediction Systems for Hard Rock- based on active design. Doctoral . Thesis, Division of Soil and Rock Mechanics, Department of Civil and Environmental Engineering, Royal Institute of Technology, Stockholm, Sweden.

23. De Paoli, B Bosco, B Granata and Bruce D.A, (1992). *Fundamental observations on cement based grouts,* Proc. ASCE Conferance, Grouting Soil Improvement and Geosynthetics, New Orleans, L.A, Feb. 25-28, 2 Volumes, pp. 474-485.

24. *Dershowitz W, Doe T, (1997). Analysis of Heterogeneously Connected Rock Masses By Forward Modelling of Fractional Dimension Flow Behaviour*, Int. J. Rock Mech. & Min. Sci 34:3-4, Paper No. 061, Elsevier.

25. Eklund D (2000 b), Försök med modifierad mätgeometri, Vattenfall Utveckling AB, Älvkarleby.

26. Eklund D, (2000), *Reologisk mätmetodik för injekteringsbruk*, Vattenfall Utveckling AB, Älvkarleby.

27. Eklund, D., Alemo, J.,(2001). Penetration *Ability of Cementitous Grouts*. Proc. to the 4´th Nordic Rock Grouting Symposium, Sweden Rock Engineering Research (SveBeFo), Stockholm, Sweden. pp 41-50. ISSN 1104-1773.

28. Emmelin, A, Eriksson M, Åsa Fransson (2004), *Characterisation, design and execution of two grouting fans at 450 m level, Äspö HRL*, Swedish Nuclear Fuel and Waste Management Co, Stockholm, Sweden.

29. Enwald H, Peirano E, Almstedt (1996), A-E, *EULERIAN TWO-PHASE FLOW THEORY APPLIED TO FLUIDIZATION*, Int. J. Multiphase Flow Vol 22., pp 21-66.

30. Eriksson L., Johansson E., Kettaneh-Wold N. and Wold S. (1999) *Introduction to Multi- and Megavariate Data Analysis using Projection Methods (PCA & PLS)*, UMETRICS, Umeå, Sweden.

31. Eriksson M, Brantberger M, Dalmalm T, Stille H (1999). *Separations- och filtreringsstabilitet hos cementbaserade injekteringsmedel*. Rapport 3065. Dept. Of Soil and Rock Mechanics, Royal Institute of Technology, Stockholm.

32. Eriksson, M, (2002) Prediction of grout spread and sealing effect, a probabilistic approach, Doctoral Thesis, Royal Institute of Technology, Stockholm, Sweden.

33. Eriksson, M, (2003) *A method for measuring and evaluating the penetrability of grouts,* Grouting and Ground treatment, Royal Institute of Technology, Stockholm, Sweden. Proc. Of the third international conference, New Orleans, USA, 2004.

34. Eriksson, M, Stille, H (2002a), A Method for measuring and evaluating the penetrability of grouts, Royal Institute of Technology, Stockholm, Sweden.

35. Eriksson, M. (1999). Model for Prediction of Grouting Results- Spreading Sealing Efficiency and Inflow. Lic. Thesis, Division of Soil and Rock Mechanics, Department of Civil and Environmental Engineering, Royal Institute of Technology, Stockholm, Sweden.

36. Eriksson, M, Stille H (2004). *Injektering i hårt berg,* Department of Civil and Environmental Engineering, Royal Institute of Technology, Stockholm, Publisher SveBeFo, Sweden.

37. Ferraris C F, Gaidis J M. (1992) Connection between the Rheology of Concrete and Rheology of Cement Paste. ACI Materials Journal., vol 88, no 4, pp 388-393.

38. Fjällberg L (1998) Superplasicerarnas inverkan på cementpastans hydratations och flytförmåga. CBI rapport. ISRN CBI/R-98/1-SE. Stockholm, Sweden.

39. Fjällberg L, Lagerblad B (2003), *Cement based injection grouts- different types, cement reactions, setting time and flowability*. CBI rapport. ISRN CBI/R-2003/1-SE. Stockholm, Sweden.

40. Fransson Å (1999) Grouting Predictions Based on Hydraulic Tests of Short Duration, Department of Geology, Chalmers university of technology, Göteborg, Sweden.

41. Fransson Å (2001) Characterisation of Fractured Rock for Grouting Using Hydrogeological Methods, Department of Geology, Chalmers university of technology, Göteborg, Sweden.

42. Gartner E.M, Gaidis J.M, Grace W.R et al (1987), *Hydration Mechanisms I.* Materials Science of Concrete I, The American Ceramic Society, Westerville, USA.

43. Graton L.C, Fraser H.J (1935). *Systematic packing of spheres with particular relation to porosity and permeability*. The journal of geology, No 8, Part I, vol XLIII.

44. Gu Ping, Xie Ping, (1994) Beaudoin JJ, Jolicoeur C. Investigation of the retarding effect of superplasticizers on cementhydration by impedance spectroscopy and other methods. Cement and Concrete Research. v 24 n 3, pp 433-442.

45. Gustavsson G (2004). *Ett nytt angreppssätt för bergbeskrivning och analysprocess för injektering*, Väg och Vattenbyggaren nr 4, Department of Geology, Chalmers university of technology, Göteborg, Sweden.

46. Gylling B, (1997). *Development and Applications of the Channel Network Model for Simulation of Flow and Solute Transport in Fractured Rock*. Doctoral thesis, Department of Chemical Engineering and Technology, Royal Institute of Technology, Stockholm, Sweden.

47. Hakami E (1995) Aperture distribution of rock fractures. PhD Thesis, Division of Engineering Geology, Royal Institute of Technology, Stockholm, Sweden.

48. Håkansson U. (1993). Rheology of Fresh Cement-Based Grouts. Ph D Thesis, Dept. Of Soil and Rock Mechanics, Royal Institute of Technology, Stockholm.

49. Hansson P, (1995) *Provningsmetoder för cementbaserade injekteringsmedel och deras användning*, Vattenfall Utveckling AB, proc. of Vann og frostsikring av trafiktunneler- Injektion og kledning, Trondheim.

50. Hansson P, (1998). *Materialmässiga aspekter på injektering*, Vattenfall Utveckling AB, PM UC98:5, Älvkarleby.

51. Hansson P, (2000) *Ny proportioneringsmetodik för betong*, Vattenfall Utveckling AB, Älvkarleby.

52. Hansson P, Eklund D, (2001) *Filtreringsstabilitet –State of the art*, Vattenfall Utveckling AB, Älvkarleby.

53. Hansson P. (1994). Cementinjektering- Handbok för injekterare, Elforsk rapport 94:12, Sweden.

54. Hansson P. (1995b). Filtration Tendency of Cement Grouts for Injection of Concrete Structures. Proceedings: IABSE Symposium San Francisco 1995 - Extending the Lifespan of Structures. pp 1199 – 1204.

55. Hansson P. (1996). F*iltration Tendency of Cementitious Injection Grouts with Low W/C-Ratio*. Conference paper of " Concrete in the Service of Mankind - Concrete Repair, Rehabilitation and Protection" Dundee, Scotland. pp 303-311.

56. Hansson P. (1997). *Cementbaserade injekteringsmedels inträngningsförmåga – mätning och modellering*. Nordiskt symposium i berginjektering, 1998, Finland.

57. Hjertström S, Pettertsson S Å, (2004), *Ny kunskap vid dispergering av mikrocement*, Bergsprängningskommitténs diskussionsmöte BK 2003, Stockholm, Sweden.

58. Hestroni G (1982), Handbook of Multiphase Systems, McGraw Hill, New York.

59. Hiroshi Uchikawa, (1994) Ultra-Fine Cements for Special Applications. Advanced cement based material 1994;1:150-154, Japan.

60. Houlsby A.C (1990). Construction and design of cement grouting, John Wiley and Sons, Inc.

61. Hässler, L. (1991). Grouting of Rock- Simulation and Classification. Ph D Thesis, Dept. Of Soil and Rock Mechanics, Royal Institute of Technology, Stockholm.

62. International Federation of Societies of Cosmetic Chemists (1997), IFFSCC, Monograph Number 3, An Introduction to Rheology, Weymouth, England.

63. Jansson T (1998). Calculation models for estimation of grout take in hard jointed rock. Doctoral Thesis, Royal Institute of Technology, Stockholm, Sweden.

64. Johnson D. E. (1998) *Applied Multivariate Methods for Data Analysts*, Duxbury Press, ISBN 0-534-23796-7.

65. Justnes H, Nygaard E.C (1995), *Technical calcium nitrate as a set accelerator for cement at low temperatures*, Cement and Concrete Research, Vol. 25, No. 8, pp. 1766-1774.

66. Kinoshita M, Okada K (1999), *Performance of methacrylic water-soluble graft polymers with different types of cement*. RILEM, PRO 5, s 34-47, 1999.

67. Larsson E, (1997). *Groundwater flow through a natural fracture: Flow experiments and numerical modeling*. Licentiate Thesis, Department of Geology, Publ. A82, Chalmers University of Technology, Göteborg, Sweden.

68. Lidström L, Westerberg B, (2003), *Fine Ground Cement in Concrete- Properties and Prospects*. ACI Materials Journal, V 100, No 5, 2003.

69. Lohmander, S (2000), The influence of Particle Shape of Coating Pigments on their Packing Ability and on the Flow Properties of Coating Colours, Ph D Thesis, Dept. Of Pulp and Chemistry and Technology, Royal Institute of Technology, Stockholm.

70. Marinet P (1998), Flow and clogging mechanisms in porous media with application to dams, Doctoral Thesis, Royal Institute of Technology, Stockholm, Sweden.

71. Mezger, T, (2000), Proc. Rheo-Workshop 2000, Paar Physica, Stuttgart, Germany.

72. Mitchell J, (1970) In-place treatment of foundation soils. Proc. ASCE Jour. of SMFE, paper 7035, pp 117- 152.

73. Mork, J H (1994), Effekt av sementens forhold mellom gips og hemihydrat på den ferske betongens reologi, Dr.Ing. Avhandling 1994:04, Norges Tekniska Högskole, Trondheim, Norge.

74. Nguyen Q.D (1986), Measuring the flow properties of yield stress fluids, Department of Chemical Engineering, the University of Adelaide, Adelaide, South Australia 5001, Australia.

75. Nied R, Horlamus H (2000). *Ullmann´s Encyclopedia of Industrial Chemistry*, Sixth Edition. Wiley-VCH Verlag GmbH, Weinheim, Germany.

76. Nonveiller E (1989), Grouting Theory and Practice, Elsevier Science Publishers B.V, ISBN 0-444-87400-3, Amsterdam, Netherlands.

77. Nordqvist A, Tsang Y.W, Tsang C.F, Dverstorp B, Andersson J (1992). *A variable aperture fracture network model for flow transport in fractured rocks*, Water Resources Research, vol. 28, No 6, pp1703-1713.

78. Palmqvist K (1983), Tätning av bergtunnlar –Injekteringsutförande och resultat, BeFo 66:1/83, Sweden.

79. Panton, R.L. (1996). INCOMPRESSIBLE FLOW (2:nd ed.). John Wiley & Sons, Inc., New York. ISBN 0-471-59358-3.

80. Pettersson S Å, Molin H (1999) Grouting & Drilling for grouting, Atlas Copco Craelius AB, Sweden.

81. Powers T.C et al. Capillary continuity or discontinuity in cement-pastes. Journal of the PCA Research and Development Laboratories, May 1959, pp 38-48.

82. Ramachandran V.S (1998), Malhotra V.S, Jolicoeur C, Spiratos N, *Superplasticisers: Properties and application in concrete, CANMET, s 404.*

83. Saaidi S (2004), *Numerical and Physical Simulation of Grout Penetration*, Master Thesis, Department of Thermo and Fluid Dynamics, Chalmers University of Technology, Sweden.

84. Schwarz, L.G., (1997), *Roles of rheology and chemical filtration on injectability of microfine cement grouts*, Doctoral Thesis, Northwestern University, Evanstone, Illinois, UMI number 9814310.

85. Shaw D J (1980). Introduction to Colloid and Surface Chemistry. Third Edition. Butterworth-Heinemann Ltd.

86. Stille H, (2001). Grouting- Research work and practical application, Proc. "4 th. Rock grouting symposium", Stockholm, Sweden, 2001.

87. Svarovsky L. Handbook of Powder Technology. Solid-Liquid Separation Process and Technology. Elsevier 1985.

88. Taylor H.F.W, (1990). Cement Chemistry, Academic Press, ISBN 0-12-683900-X, Great Britain.

89. Tsang Y.W, Tsang C.F, (1989). *Flow channeling in a single Fracture as a two-dimensional Strongly Hetrogeneous Permeable Medium*. Water Resources Res 25(9), 2076-2080.

90. Umetrics (2000) *Simca-P 8.0 User Guide and Tutorial*, UMETRICS, Umeå, Sweden.

91. VAST (Kraftverksföreningens Utvecklingsstiftelse) (1991) Betong i vattenkraftsanläggningar, Stockholm, Sweden.

92. Vovk A I, Osenkova N N, Usherov Marshak A -V, Falikman V R. (1989). Kinetics of the evolution of heat and structure-making during hydration of 3CaO·SiO2 in the presence of polymethylenepolynaphthalenesulfonates. Colloid J USSR., pp 492-495.

93. Wachem, BGM, (2000) *Derivation, Implementation and Validation of Computer Simulation Models for Gas-Solid Fluidized Beds*, Doctoral Thesis, Delft University of Technology, Holland.

94. Widman R, (1993) *Grouting in Rock and Concrete*. Proc. of the international conference on grouting in rock and concrete, 1993, Salzburg, Austria.

95. Wieker, W, R. Herr (1989). *Zu einigen Problemen der Chemie des Portlandzements*,
Z. Chem.,29 JG.,Heft 9, s 321-327.

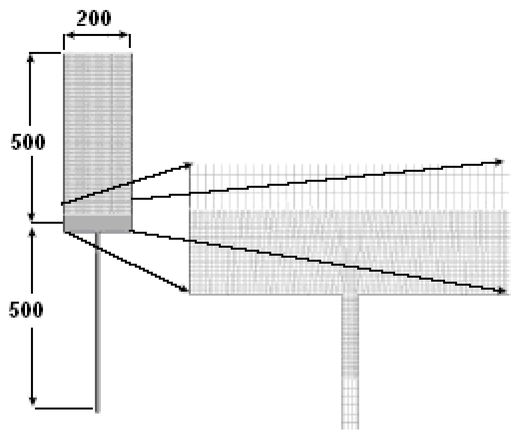
96. Yamada K, Takahashi T, Hanehara S, Matsuhisa M.(2000), *Effects of the chemical structure on the properties of polycarboxylate-type superplastisiser*. Cement and Concrete Research, Vol 30, No. 2, pp 197-207.

97. Yang M, Neubauer C M, Jennings H M. (1997). Interparticle Potential and Sedimentation Behaviour of Cement Suspensions. Advanced Cement Based Materials 1997;5:1-7. pp 1-6.

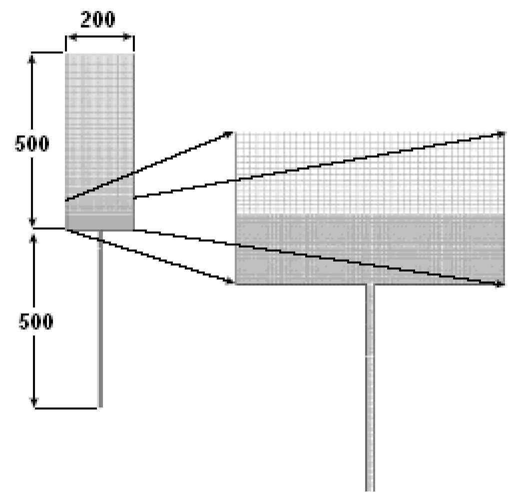
98. Zimmerman R.W, Kumar S, Bodvarsson G.S (1991). *Lubrication theory analyses of the permeability of rough-walled fractures*, Int. J. Rock Mech, & Min. Sci. & Geomech Abstr. Vol. 28, No. 4, pp 325-331.

APPENDIX

1 Numerical Model Plots

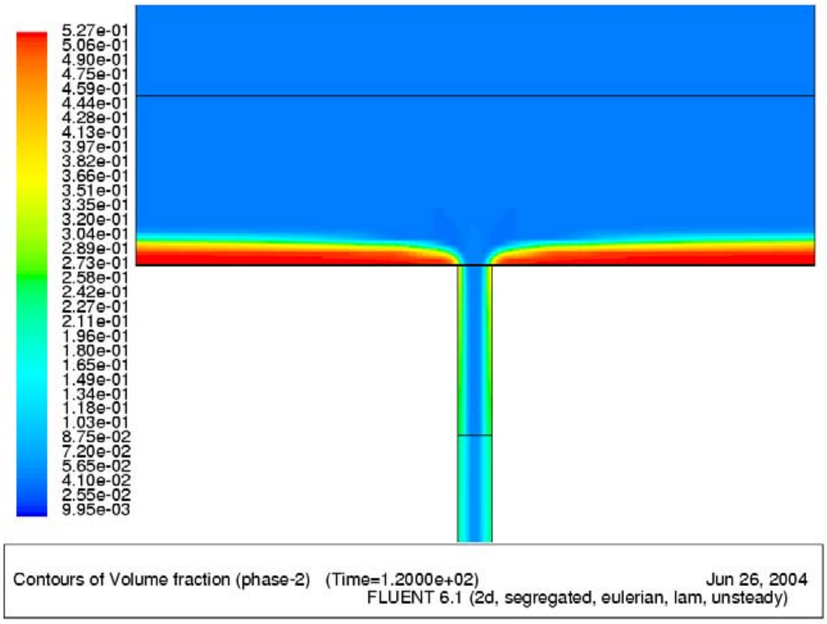


(a)



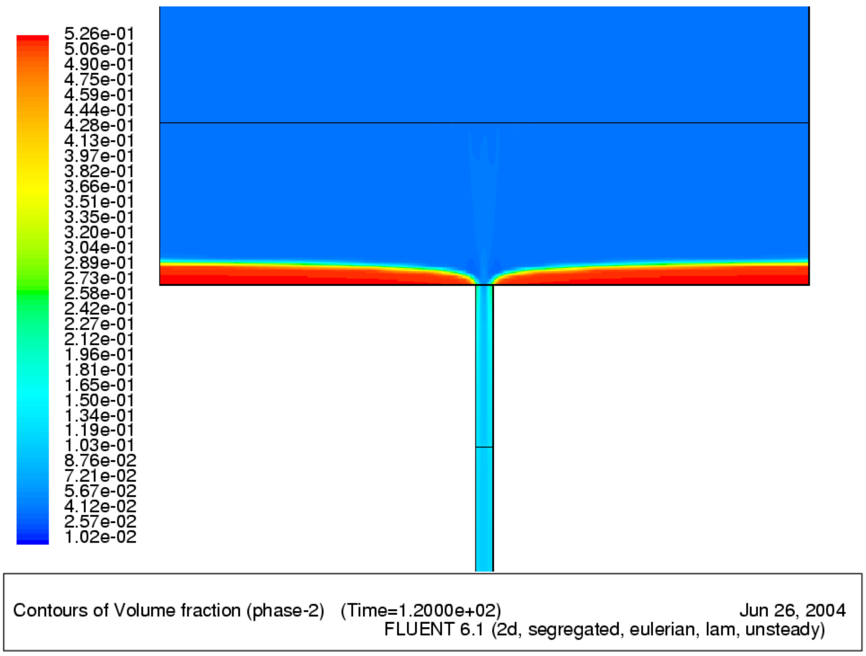
(b)

Figure 1.1, Computational grids of the simulated cracks, (a) 10 mm crack and (b) 5 mm crack. The geometry of the simulated slot aperture has the same measures as the physical model.



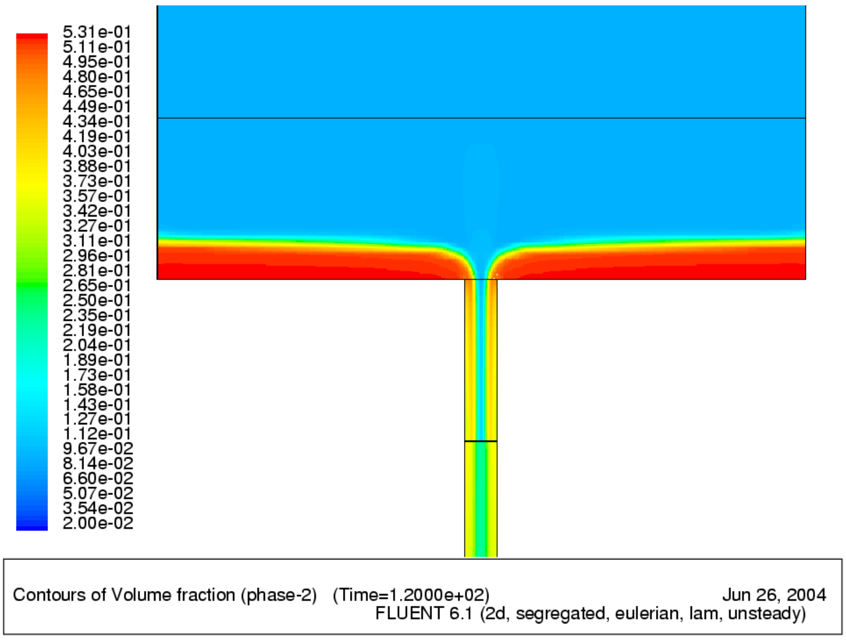
10 mm slot aperture.

Figur 1.1, Transient numerical simulation results at t=120 s, 5% grain volume concentration.



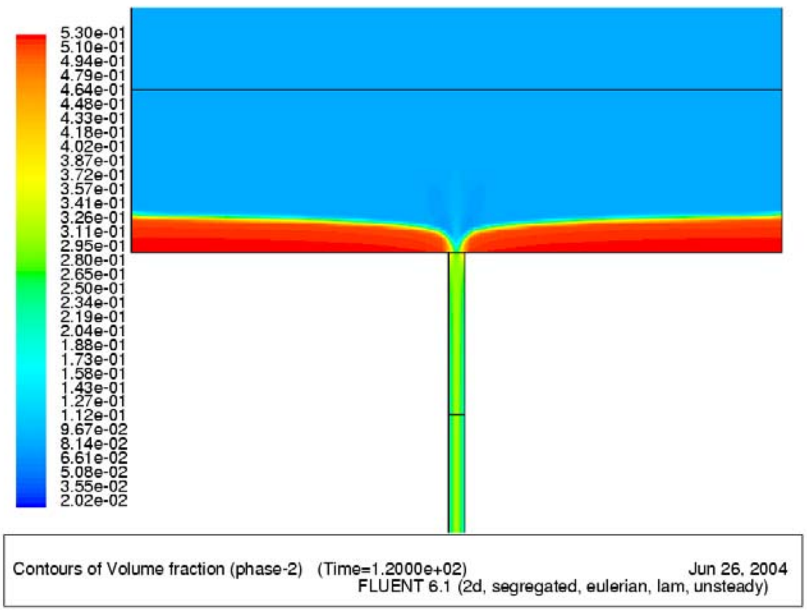
5 mm slot aperture.

Figur 1.2, Transient numerical simulation results at t=120 s, 5% grain volume concentration.



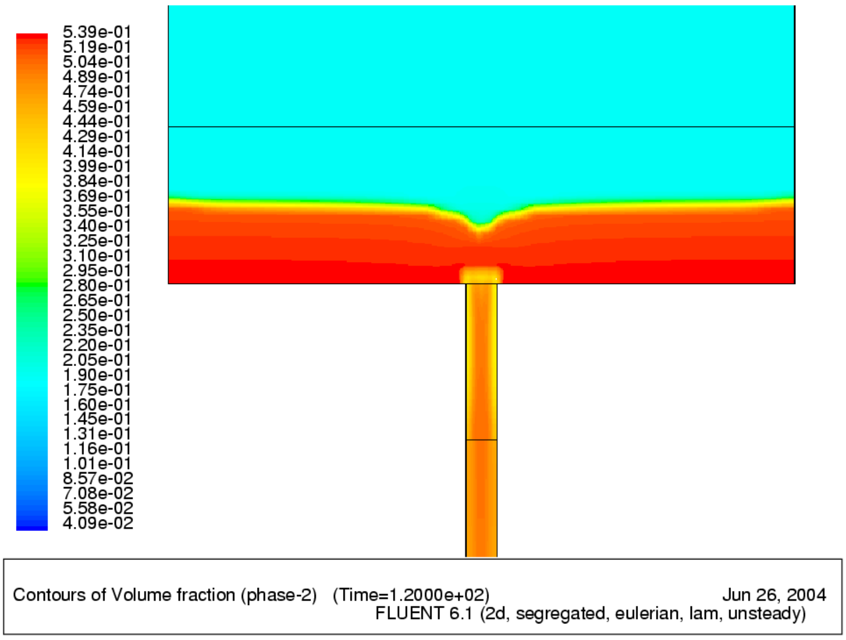
10 mm slot aperture.

Figur 1.3, Transient numerical simulation results at t=120 s, 10% grain volume concentration.



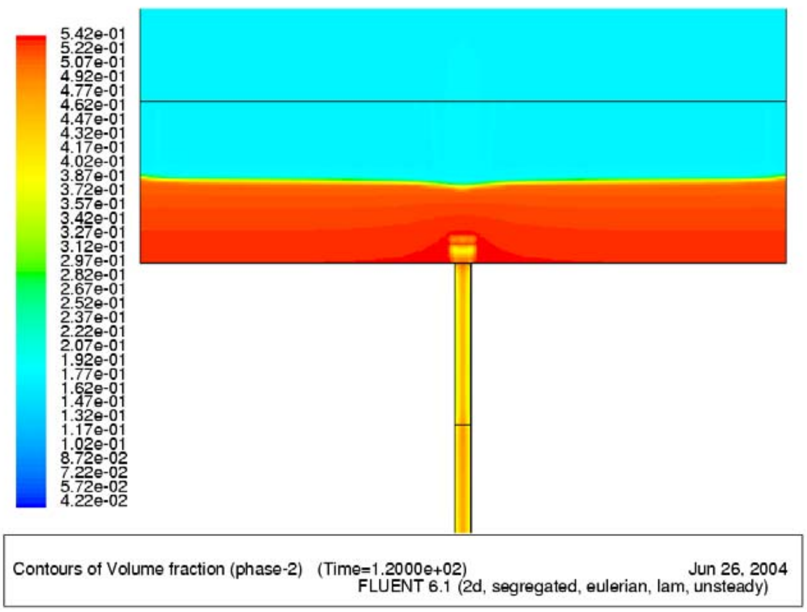
5 mm slot aperture

Figur 1.4, Transient numerical simulation results at t=120 s, 10% grain volume concentration.



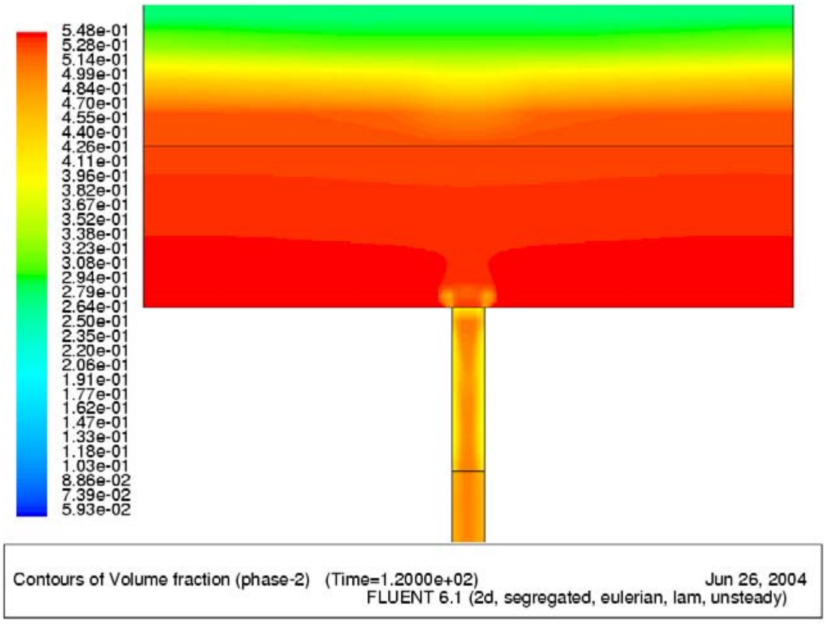
Slot aperture 10mm

Figur 1.5, Transient numerical simulation results at t=120 s, 20% grain volume concentration.



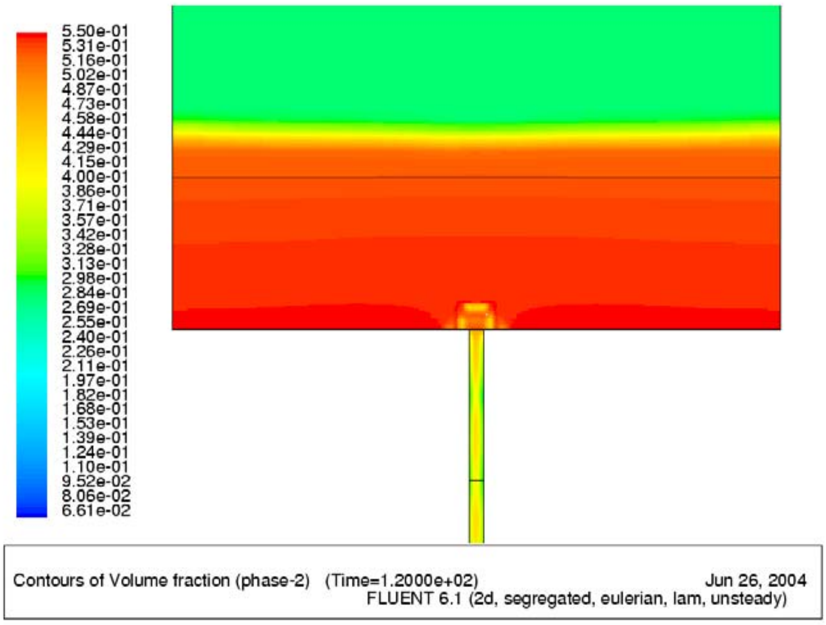
Slot aperture 5 mm

Figur 1.6, Transient numerical simulation results at t=120 s, 20% grain volume concentration.



Slot aperture 10 mm

Figur 1.7, Transient numerical simulation results at t=120 s, 30% grain volume concentration.



Slot aperture 5 mm

Figur 1.8, Transient numerical simulation results at t=120 s, 30% grain volume concentration.

2 FILTRATION EXPERIMENTS

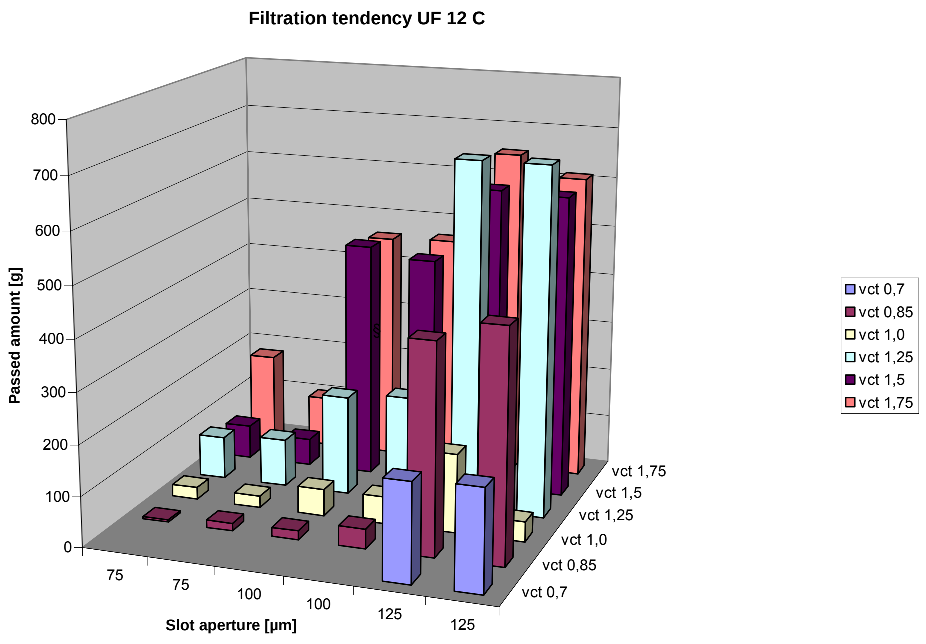


Figure 2.1, Filtration experiments with Ultrafint cement 12 (UF 12). Used W/C ratio is 0,7-1,75. Used superplastisisers is Cemflux (C). Slot apertures 75, 100 and 125 µm.

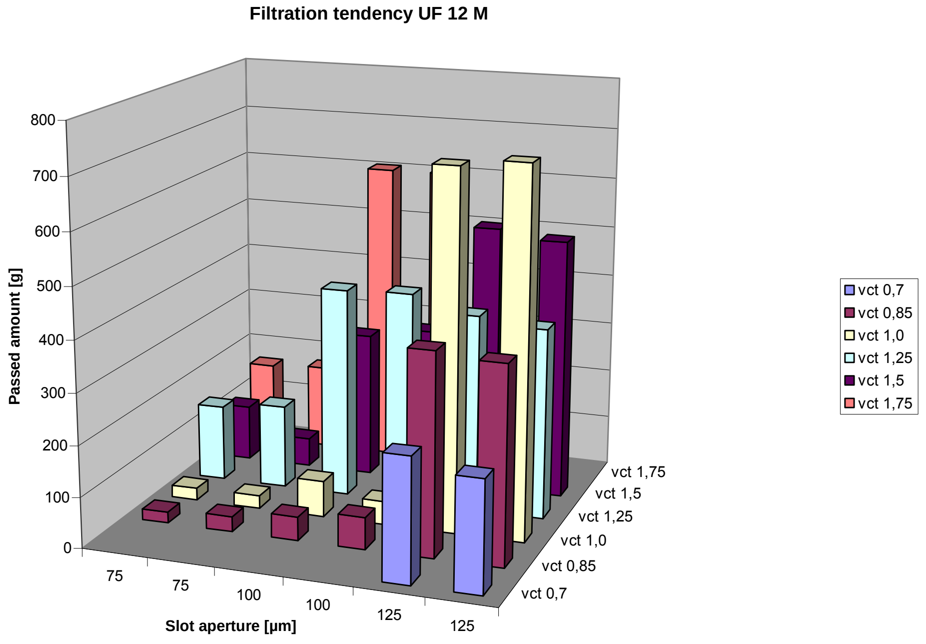


Figure 2.2, Filtration experiments with Ultrafint cement 12 (UF 12). Used W/C ratio is 0,7-1,75. Used superplastisisers is Melcrete (M). Slot aperture 75, 100, 125 µm.

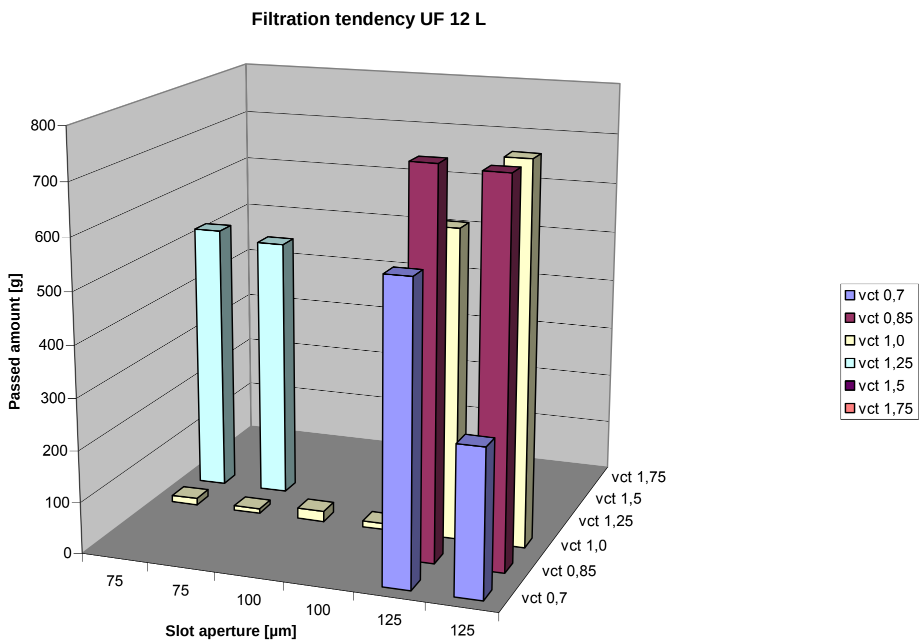


Figure 2.3, **Filtration experiments with Ultrafint cement 12 (UF 12). Used W/C ratio is 0,7-1,25. Used superplastisisers is L 15 (L). Slot aperture 75, 100, 125 µm.**

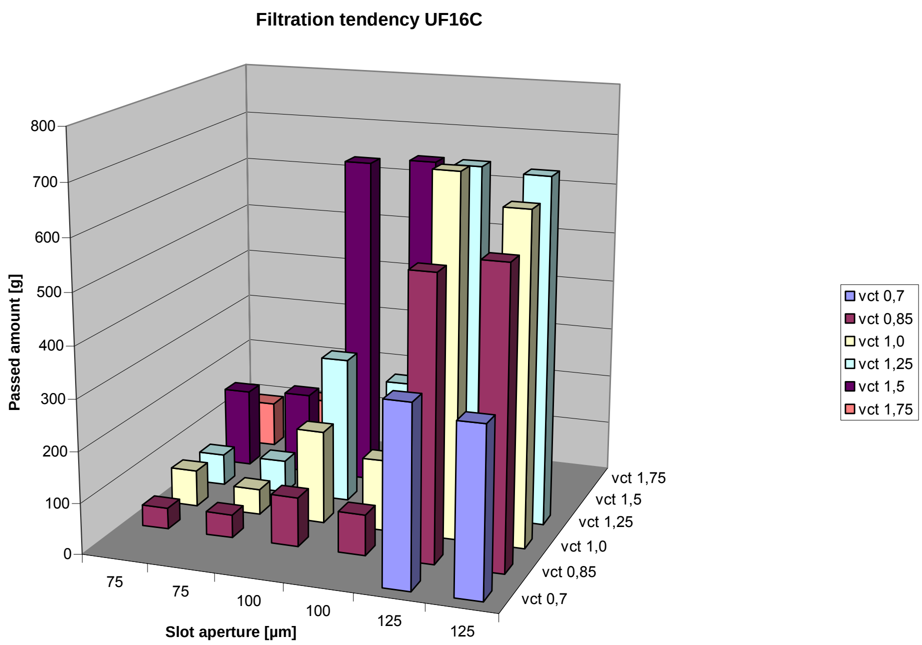


Figure 2.4, **Filtration experiments with Ultrafint cement 16 (UF 16). Used W/C ratio is 0,7-1,75. Used superplastisisers is Cemflux (C). Slot apertures 75, 100, 125 µm.**

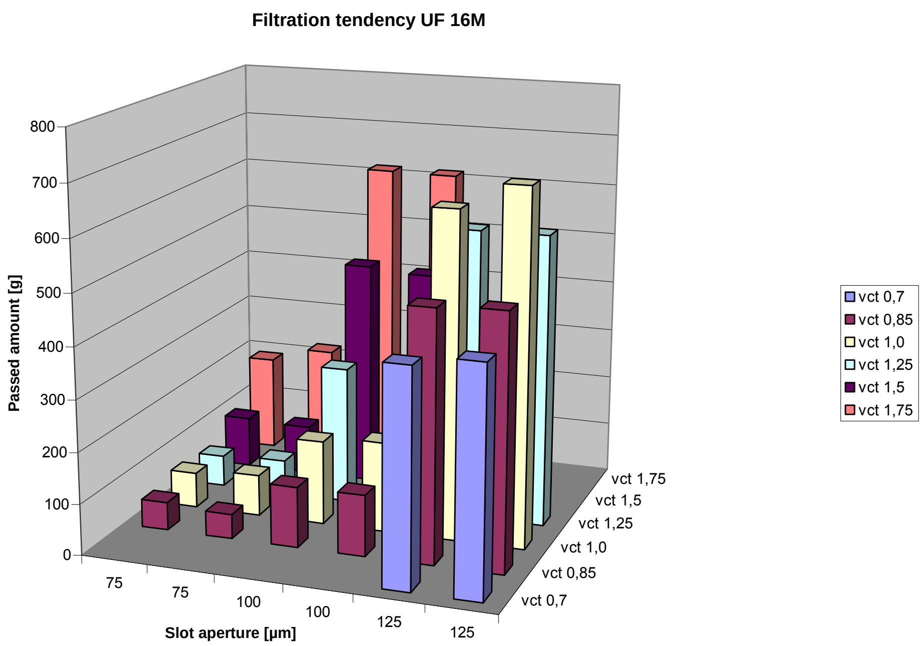


Figure 2.5, **Filtration experiments with Ultrafint cement 16 (UF 16). Used W/C ratio is 0,7-1,75. Used superplastisisers is Melcrete (M). Slot apertures 75, 100, 125 µm.**

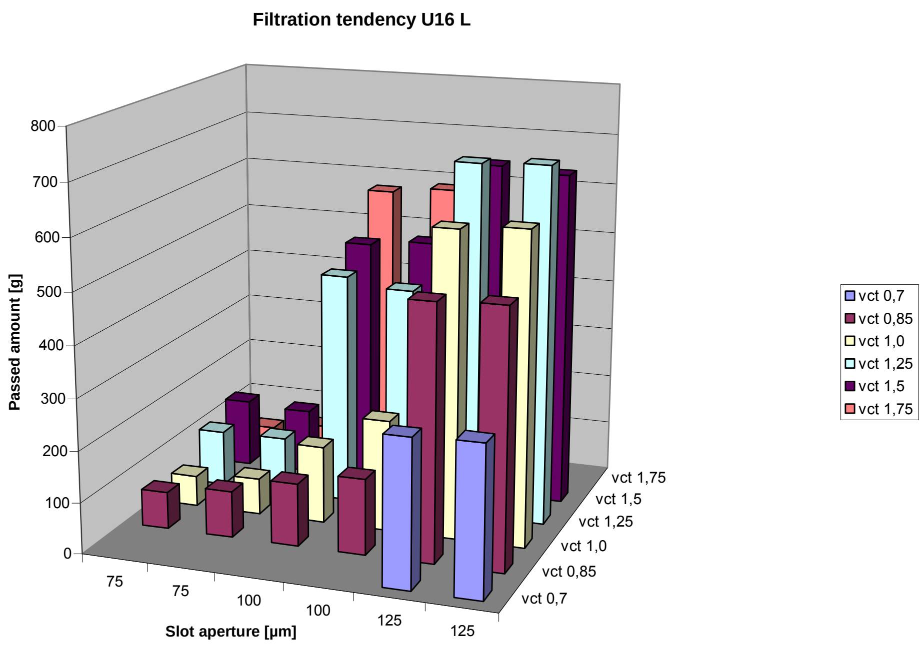


Figure 2.6, Filtration experiments with Ultrafint cement 16 (UF 16). Used W/C ratio is 0,7-1,75. Used superplastisisers is L 15 (L). Slot aperture 75, 100, 125 µm.

Filtration tendency IC 30 C

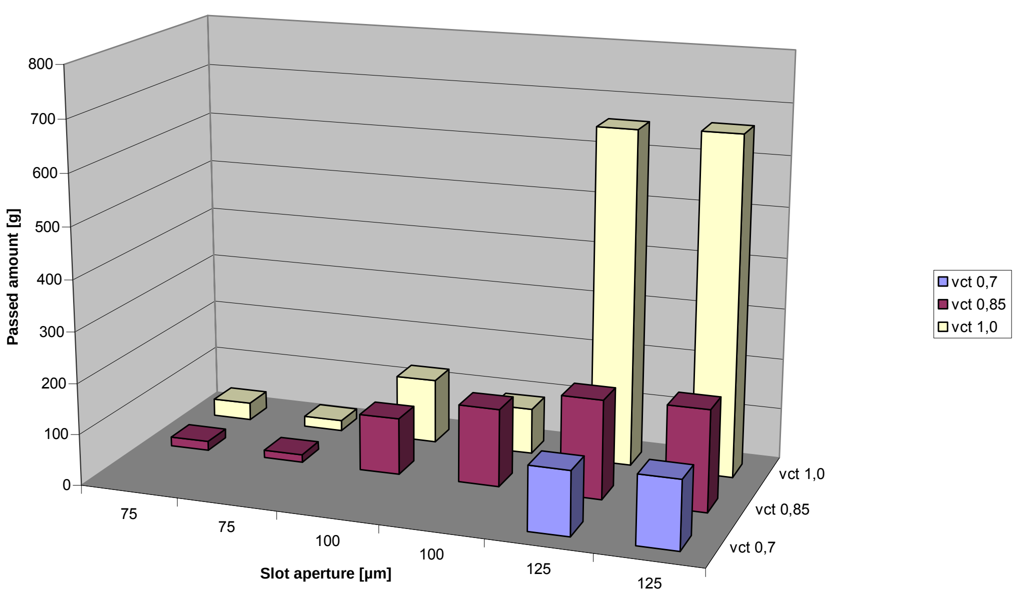


Figure 2.7, **Filtration experiments with Injekteringscement 30 (IC 30). Used W/C ratio is 0,7-1,0. Used superplastisisers is Cemflux (C). Slot aperture 80, 100, 125 µm.**

Filtration tendency IC 30 M

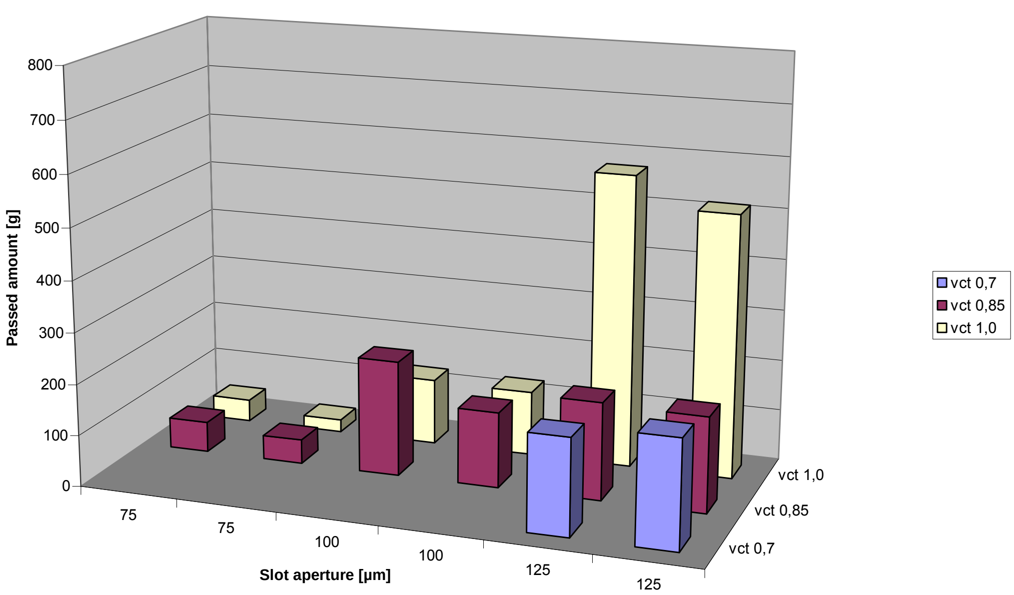


Figure 2.8, **Filtration experiments with Injekteringscement 30 (IC 30). Used W/C ratio is 0,7-1,0. Used superplastisisers is Melcrete (M). Slot aperture 75, 100, 125 µm.**

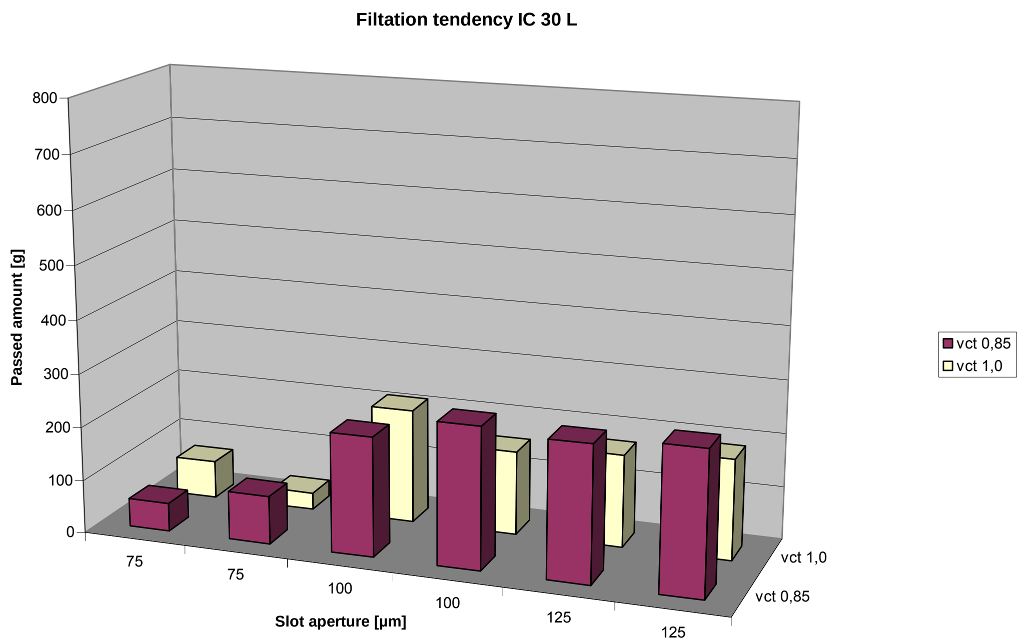


Figure 2.9, **Filtration experiments with Injekteringscement 30 (IC 30). Used W/C ratio is 0,85-1,0. Used superplastisisers is L 15 (L). Slot aperture 75, 100, 125 µm.**

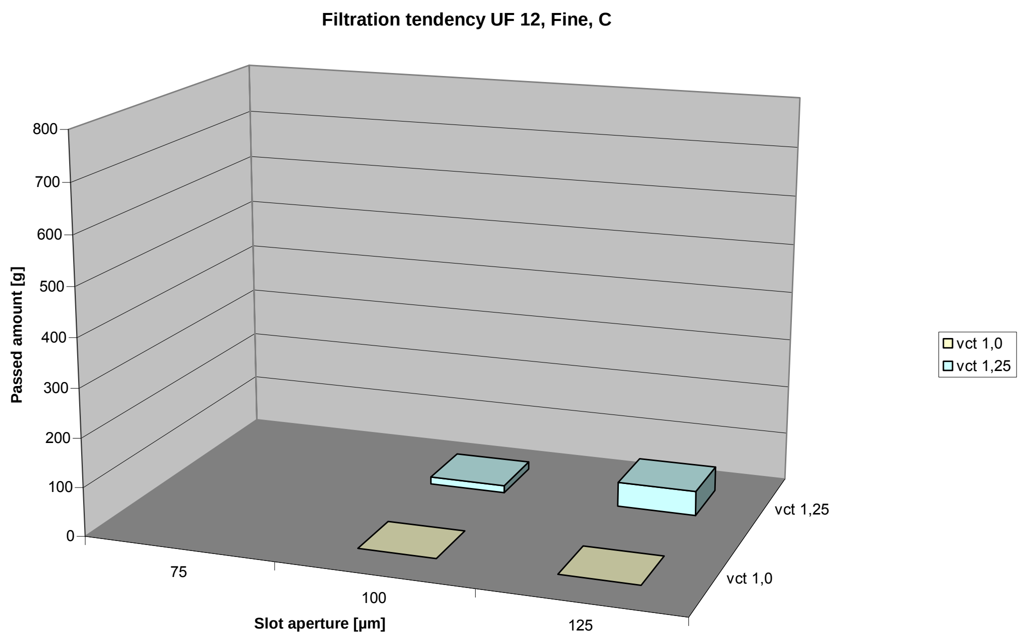


Figure 2.10, Filtration experiments with sieved Ultrafint cement 12 (UF 12, Fine). Used W/C ratio is 1,0-1,25. Used superplastisisers is Cemflux (C). Slot aperture 100, 125 µm.

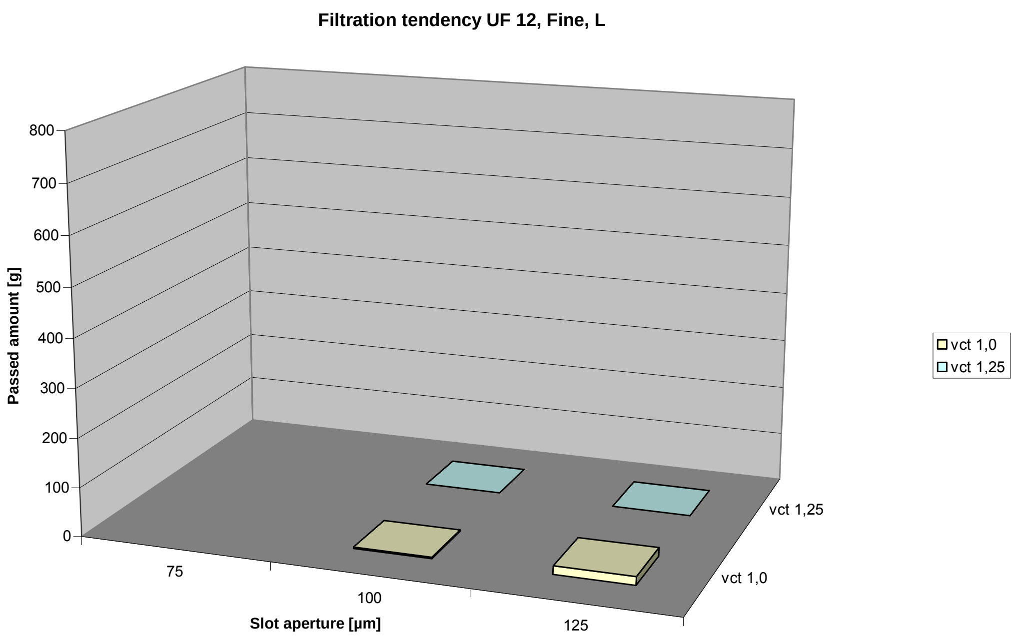


Figure 2.11, Filtration experiments with sieved Ultrafint cement 12 (UF 12, Fine). Used W/C ratio is 1,0-1,25. Used superplastisisers is L 15 (L). Slot aperture 100, 125 µm.

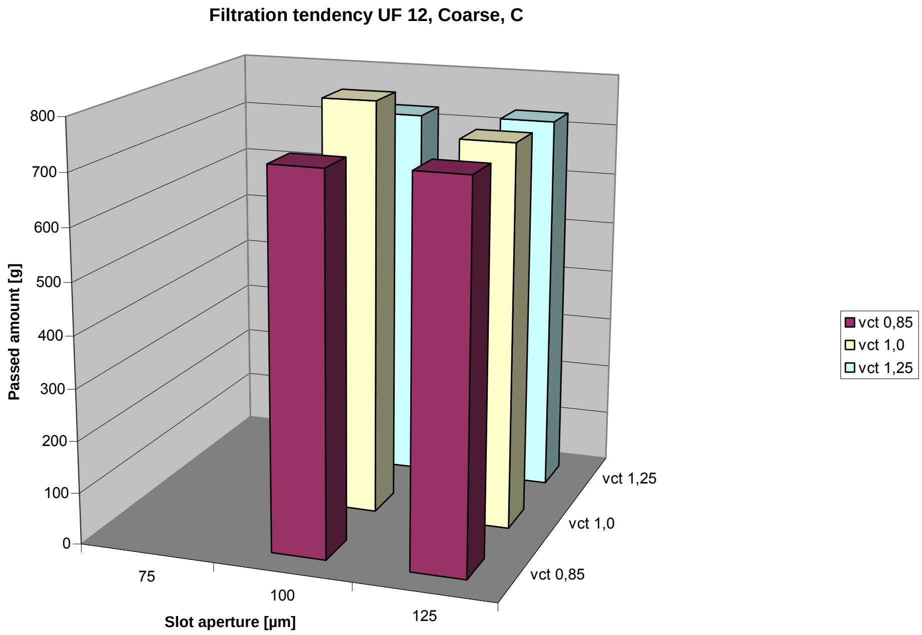


Figure 2.12, Filtration experiments with sieved Ultrafint cement 12 (UF 12, Coarse). Used W/C ratio is 0,85-1,25. Used superplastisisers is Cemflux (C). Slot aperture 100, 125 µm.

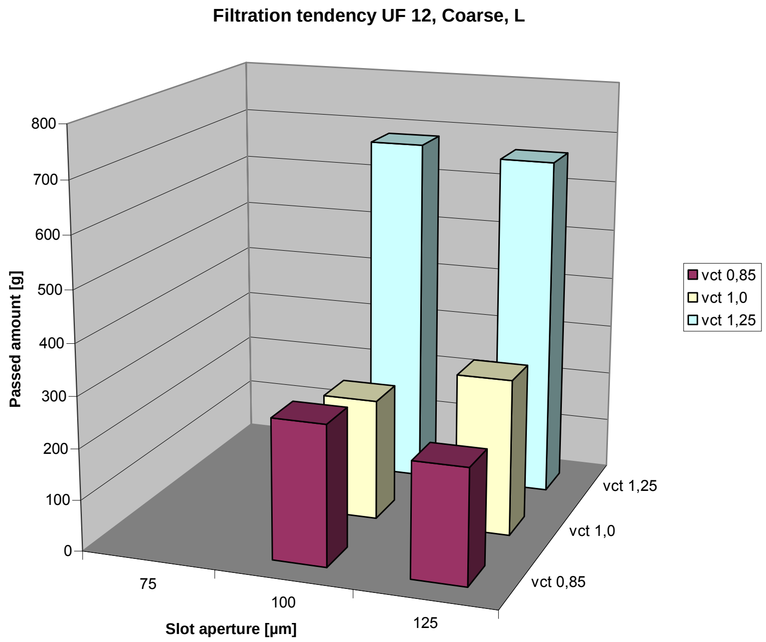


Figure 2.13, Filtration experiments with sieved Ultrafint cement 12 (UF 12, Coarse). Used W/C ratio is 0,85-1,25. Used superplastisisers is L 15 (L). Slot aperture 100, 125 µm.

3 CHARACTERISATION OF GRAIN CURVES (RRSB)

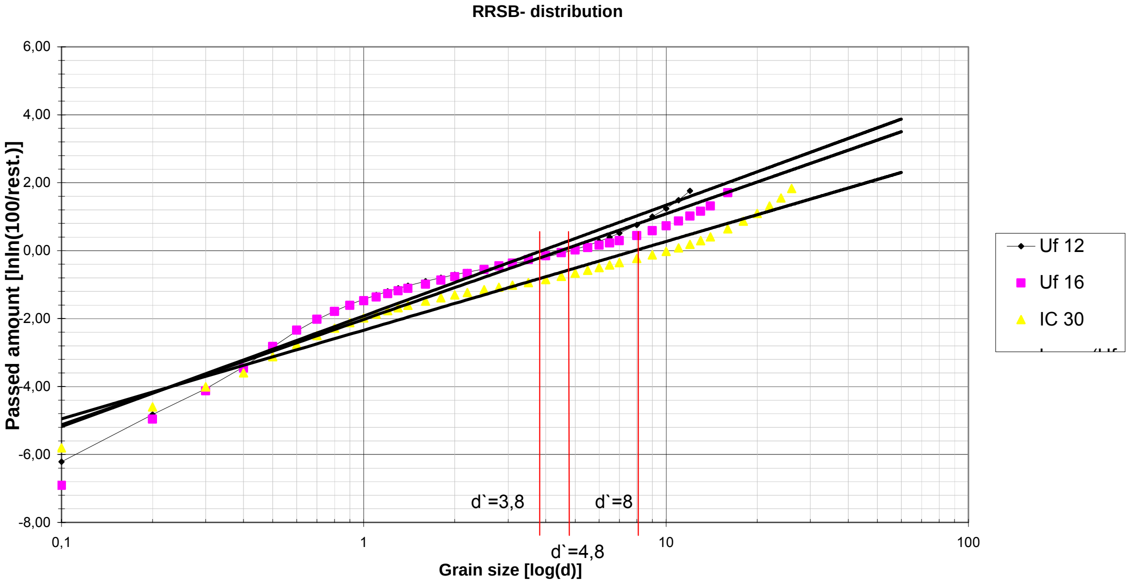


Figure 3.1, Calculation of the d` value of UF 12, UF 16 and IC 30, according to the RRSB distribution.

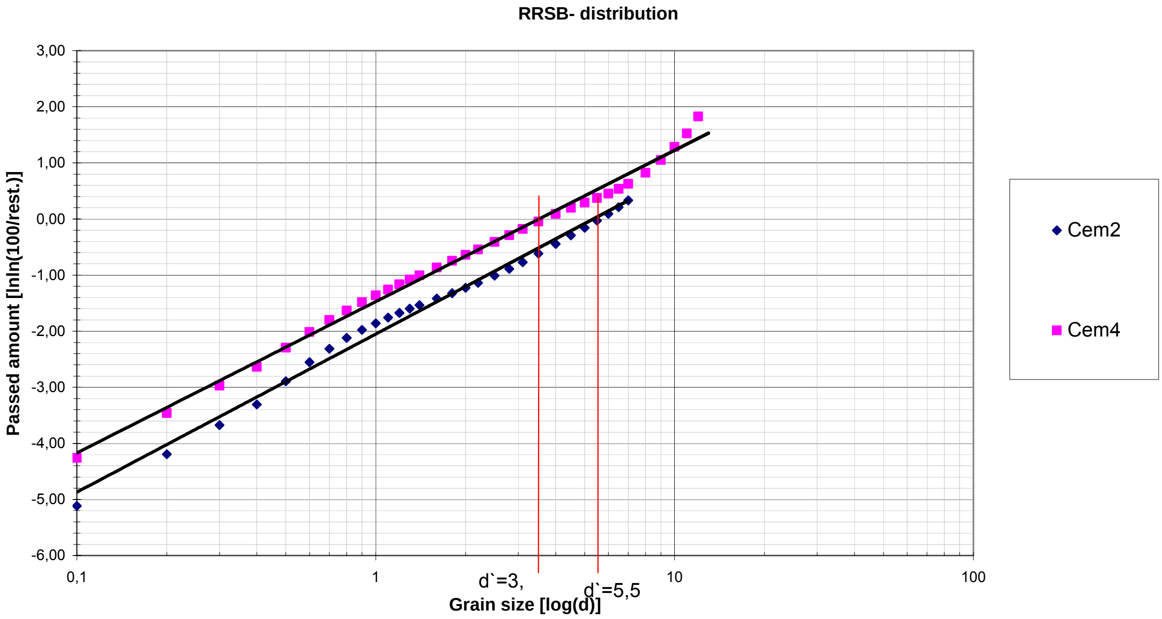


Figure 3.2, Calculation of the d` value of Cem 2 and Cem 4 according to the RRSB distribution.

4 EVALUATION OF FLOWREGIME IN THE SLOT

In order to evaluate the flow regime (laminar or turbulent) that occurs in the slot during the filtration experiments, calculation of Reynolds number has been performed. The flow regime is of importance to establish in order to evaluate the filtration results (passed amount of mixture). If laminar flow is established during flow of mixture through the slot, the influences from the flow regime can be assumed to be neglected. Turbulent flow can influence of the passed amount of mixture because of creation of eddies at the entrance of the slot aperture and entrapment of air in the mixture that shall pass the slot. None of these phenomenons are desirable to occur in order to analyse the filtration tendency of the grout in a proper way.

Reynolds number can be defined as:

eq 4.1

$$\mathrm{Re} = \frac{U \cdot L}{\nu}$$

U is the characteristic velocity of the mixture and L is a characteristic length of the flow. ν is the kinematic viscosity of the mixture.

The kinematic viscosity is defined according to:

eq 4.2

$$\nu = \frac{\eta}{\rho}$$

According to textbooks regarding fundamental flow mechanics, it can be assumed that a turbulent flow regime is achieved in a non circular pipe (for example a slot) if the Reynolds number exceeds approximately 2300. Measured viscosity (η) of the mixture in the region of 0,06 Pas and a density (ρ) of approximately 1550 kg/m3, the kinematical viscosity can be calculated according to:

eq 4.3

$$\nu = \frac{0{,}06}{1550} = 3{,}9E^{-5}\ m^2 / s$$

The velocity to achieve a turbulent flow regime:

eq 4.4

$$U_{turbulent} = \frac{\mathrm{Re}_{turbulent} \cdot \nu}{L}$$

When using a slot aperture (characteristic flow length) of 75 µm, the flow velocity to cause turbulent flow is:

eq 4.5

$$U = \frac{2300 \cdot 3{,}9E^{-5}}{75E^{-6}} = 1196 m/s$$

An alternative way of calculate the critical flow velocity is to base the calculations upon the slot length (thickness of the steel plate), which is 500 µm. This calculation will give the critical velocity according to:

eq 4.6

$$U = \frac{2300 \cdot 3{,}9E^{-5}}{500E^{-6}} = 179 m/s$$

A flow rate of 179 m/s of the mixture through the slot, would make that the available content in the container (490 cm^3 mixture), would be empty in much less than one second during the filtration experiments, which is not the case in any performed filtration experiment. The required measured time to empty the container is approximately in the region of one minute or more. The used pressure gradient of 30 kPa in the filtration experiments to press the mixture through the slot geometry, will most likely not give rise to a turbulent flow regime. The flow rate can be considered according to these calculations to be laminar during all performed filtration experiments.

5 AIR SIEVING OF INERT MATERIAL

Air sieving of material is a process of classification the size and distribution of the grains in a dry powder. The powder is introduced into a gas flow, and the grains are primarily subject to two forces,

1. The resistance, or the drag force caused by the gas flowing around the grains.

2. The mass force. The mass force acting on the grains can be gravity or centrifugal forces.

Depending on the magnitude and direction of these forces, grains move along different trajectories and can thus be collected separately after leaving the classification zone in the apparatus. An exact calculation of the forces acting on grains and thus a calculation of trajectories, and ultimately, of the cut size, is not possible in most cases. The prevailing flow in the separation zone of a classifier is too unsteady and inhomogeneous. The effect of feed (concentration, grain size distribution), friction preclude the description of temporal and local velocity patterns in the classification zone. Theoretical considerations must therefore be based on simplified models.

In this work is the air classifier used as a sorter. In sorting, a homogenous powder is separated into components of more or less uniform material. The grains are separated according to density or grain shape. Air classification is used predominantly in the fine and very fine range, for effective separation of grain sizes between 300 µm down to 1 µm. The capacity of a classifier is generally characterized by the quantity of fines that can be obtained. The quantity of fines is not a fixed value, because the output of fines depends on the grain size distribution of the input powder and on the cutting point that is set. A large amount o fines in the original powder will generally lower the produced amount of sieved powder. A typical procedure of air sieving a dry powder can be like the following steps:

1. A product of the desired fineness is removed.

2. A relatively coarse-grained product is dedusted.

3. A small amount of oversize material is separated.

4. Fractions with a definite upper and lower limit are produced.

5. Grain size and grain distribution of the produced powder is analyzed. The used equipment in this work for analysation of the grain size and grain distribution is Cilas 850 GR.

The used laborotory classifier with the name Alpine 100 MZR was supposed to be suitable for separation of this dry dolomite and cement powder into the specified grain curves.

Some technical data for the classifier 100 MZR:

- Steplessly adjustable separating rang of d_{97} =2-80 µm.

- Can be used for materials up to a Mohs´ hardness of 5.

- Capacity of the classifier:
  - minimum charge approximately 50 g or 1-2 dm^3.
  - maximum feed capacity approximately 2-6 kg.

- High precision cut of even in the ultrafine range < 10 µm

- Variable parameters to be set in the separation process are the wheel speed (2200-20000 rpm) and the flow rate of air through the wheel.

For further information of air sieving of dry powder material, see (Nied R, 2000)

6 SEDIMENTATION VELOCITY AND OIL VISCOSITY

Table 6.1, Measurement of the sedimentation velocity for the black and white grains.

Measurement	v_{Black} [m/s]	v_{White} [m/s]
1	$3.8*10^{-4}$	$3,9*10^{-4}$
2	$4,1*10^{-4}$	$3,7*10^{-4}$
3	$3,7*10^{-4}$	$3,9*10^{-4}$
4	$3,6*10^{-4}$	$4,0*10^{-4}$
5	$3,8*10^{-4}$	$3,5*10^{-4}$
mean	$3,8*10^{-4}$	$3,8*10^{-4}$

Table 6.2, Measurements for calculation of oil viscosity.

Measurement	Time [s]	Oil temperature [C^o]
1	98,3	20,2
2	97.6	20.2
3	98,5	20,3
4	99,0	20,3
5	98,7	20,3
mean	***t* = 98,4**	**T_{Oil} = 20,3**

7 DENSITY OF CEMENT AND INERT BASED MIXTURES

Table 7.1, Density measurements (kg/m^3) of cement based mixtures (before filtration).

W/C ratio	UF 12	UF 16	IC 30	Cem 2	Cem 4	UF 12, fine	UF 12, coarse
0,7	1624	1650	1625	1450	1525	-	-
0,85	1558	1557	1559	-	-	-	1580
1,0	1481	1494	1484	-	-	1475	1475
1,25	1389	1399	-	-	-	1395	1398
1,50	1346	1355	-	-	-	-	-
1,75	1306	1292	-	-	-	-	-

Table 7.2, Theoretical densities of the mixtures, based of a compact density of 3100 kg/m^3. All used cements are supposed to have the same compact density (before filtration).

W/C ratio	Density
0,7	1662
0,85	1578
1,0	1512
1,25	1431
1,50	1372
1,75	1327

Table 7.3, **Density measurements (kg/m^3) of cement based mixtures, after filtration through a slot geometry with an aperture 75, 100 and 125 µm.**

Mixture	**UF 12**			**UF 16**			**IC 30**		
	Density	**No.**	**Std. dev.**	**Density**	**No.**	**Std. dev.**	**Density**	**No.**	**Std. dev.**
W/C ratio 0,7	[kg/m^3]	[-]	[-]	[kg/m^3]	[-]	[-]	[kg/m^3]	[-]	[-]
75 µm	-	-	-	-	-	-	-	-	-
100 µm	-	-	-	-	-	-	-	-	-
125 µm	1588	9	40,31	1656	9	13,90	1605	90	14,61
W/C ratio 0,85									
75 µm	1500	8	18,8	1517	10	23,2	1469	8	20,0
100 µm	1536	4	25,62	1552	6	12,11	1564	6	8,01
125 µm	1536	6	8,80	1552	6	2,58	1561	6	15,30
W/C ratio 1,0									
75 µm	1447	12	65,1	1459	12	14,3	1296	10	81,1
100 µm	1480	6	8,37	1493	6	26,03	1495	6	8,37
125 µm	1478	6	12,14	1488	6	21,13	1503	6	9,31
W/C ratio 1,25									
75 µm	1406	6	14,97	1398	6	25,45	-	-	-
100 µm	1383	4	21,79	1402	6	13,66	-	-	-
125 µm	1386	4	24,96	1402	6	19,92	-	-	-
W/C ratio 1,50									
75 µm	1349	4	14,36	1356	6	13,20	-	-	-
100 µm	1349	4	18,43	1355	6	14,14	-	-	-
125 µm	1341	4	13,77	1348	2	3,54	-	-	-
W/C ratio 1,75									
75 µm	1300	4	20,41	1295	6	17,32	-	-	-
100 µm	1299	4	21,75	1286	4	17,50	-	-	-
125 µm	1318	2	-				-	-	-

Mixture	UF 12, Fine			UF 12, Coarse		
	Density	**No.**	**Std. dev.**	**Density**	**No.**	**Std. dev.**
W/C ratio 0,7	[kg/m^3]	[-]	[-]	[kg/m^3]	[-]	[-]
75 µm	-	-	-	-	-	-
100 µm	-	-	-	-	-	-
125 µm	-	-	-	-	-	-
W/C ratio 0,85						
75 µm	-	-	-	-	-	-
100 µm	-	-	-	1550	2	14,14
125 µm	-	-	-	1568	2	38,89
W/C ratio 1,0						
75 µm	-	-	-	-	-	-
100 µm	-	-	-	1495	2	0
125 µm	1495	1	-	1468	2	17,68
W/C ratio 1,25						
75 µm	-	-	-	-	-	-
100 µm	1375	1	-	1383	2	24,75
125 µm	1395	1	-	1410	2	28,28
W/C ratio 1,50						
75 µm	-	-	-	-	-	-
100 µm	-	-	-	-	-	-
125 µm	-	-	-	-	-	-
W/C ratio 1,75						
75 µm	-	-	-	-	-	-
100 µm	-	-	-	-	-	-
125 µm	-	-	-	-	-	-

Table 7.4, **Density measurements (kg/m^3) of inert based mixtures (before filtration)..**

W/S ratio	Mix 1	Mix 2	Mix 3	Mix 4	Mix 5	Mix 6
0,6		1590	1628	1536	1556	1607
0,7	1588	1569	1555	1575	1551	1543
0,8	1527	1534	1518	1510	1511	1529
1,4	-	1349	1335	-	-	-

Table 7.5, **Theoretical densities of the inert mixtures, based of a compact density of 2850 kg/m^3. All used mixtures are supposed to have the same compact density (before filtration).**

W/S ratio	Density
0,6	1683
0,7	1618
0,8	1564
1,4	1370

Table 7.6, **Density measurements (kg/m^3) of inert based mixtures, after filtration through different filter geometries, W/S ratio 0,7.**

	Slot aperture		Mesh/ slot aperture		Mesh aperture		
	75 µm	125 µm	45 µm	75 µm	36 µm	45 µm	75 µm
	Density	Density	Density	Density	Density	Density	Density
Mixture	[kg/m^3]	[kg/m^3]	[kg/m^3]	[kg/m^3]	[kg/m^3]	[kg/m^3]	[kg/m^3]
2	1400	1500	1500	1570	-	1477	1557
3	-	1100	-	1293	-	-	-
4	1563	1573	1600	1607	-	1533	1610
5	1550	1540	1193	1477	-	-	1590
6	1450	1487	1053	1277	-	-	-