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Dispersion of cement-based grout with ultrasound and conventional laboratory dissolvers

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Abstract

In any underground facilities especially tunnels, it is essential to seal the area against water ingress and leakage of reserved materials. Grouting is a common method used to seal rocks around tunnels, successful grouting reduces the duration and cost of the construction, guarantees better working environment and higher safety, minimizes the maintenance and most important decreases the corresponding environmental hazards significantly. Achieving a sufficient grout spread is one of the prerequisites for a successful and efficient sealing, the penetration of a grout is defined as the length of how far grout penetrates in the rock through fractures from a bore hole. Chemical grouts and cement-based grouts are the prevailing ones among the grouting materials. Despite the better penetrability of chemical grouts, they are unfavorable to use due to environmental hazards associated to them, whilst cement-based grouts are more convenient to use because of their low cost and low environmental impact.

The major drawback with cement-based grouts is their limited ability to penetrate the very narrow fractures which is directly related to their filtration tendency which is defined as the tendency of cement grains to agglomerate and build an impermeable filter cake during the flow. Many previous studies investigated the factors that affect the filtration tendency. They drew different conclusions and suggested various methods to improve the penetrability of cement-based grouts.

The mixing method is one of the factors that have a great influence on the penetrability of the grout. An effective mixing method improves the dispersion of cement particles in the mixture, thus the penetrability of the grout. As it is known from previous studies, the finer the cement particles the harder to disperse. Grouts based on micro-fine cement (< 30 µm) are essential for the development of grouts that can seal very narrow fractures (20-50) µm compared to (70-80) µm at the present.

In this study, the dispersion efficiency of three different mixing methods was evaluated, a conventional lab dissolver equipped with 90-mm disk, a conventional lab dissolver equipped with R/S system and an ultrasound UP400St device. Two cement types, INJ30 and UF12, that are similar in chemical composition but differ in degree of milling were tested. Dispersion was tested with filter pump.

The results showed that the conventional lab dissolver equipped with 90-mm disk is ineffective method. The conventional lab dissolver equipped with R/S system is a better method compared to the 90-mm disk but still not effective enough especially when it comes to grouts based on ultra-fine cement (UF12). The ultrasound dispersion is not only the best method between the three methods in comparison, but even more stable and reliable. The best result obtained was grout based on UF12 passing through the 54 µm filter. This could mean that fracture aperture down to 55 µm now can be sealed. This is a significant improvement but there is still a marginal for further improvements.

In combination with the dispersion efficiency of different dispersion methods, the study investigated the effect of additives on dispersion in particular and penetrability in general. Results showed that additives do not directly contribute to better dispersion, but they are necessary for better spread since they affect the flow properties.

Keywords

ultrafine cement grout; microfine cement grout; dispersion; additives; ultrasound dissolver; laboratory dissolver

Sammanfattning

I underjordiska anläggningar, särskilt tunnlar, är det viktigt att täta området mot vatteninträngning och läckage av lagrad material. Injektering är en vanlig metod som används för att täta bergen runt tunnlar, framgångsrik injektering minskar konstruktionens underhåll och kostnad, garanterar bättre arbetsmiljö och högre säkerhet, och reducerar miljöfaror. Att uppnå en tillräcklig spridning av injekteringsmedel är en av förutsättningarna för en framgångsrik och effektiv tätning. Inträngning av ett injekteringsbruk definieras som längden på hur långt bruket tränger in i berget genom sprickor från ett borrhål. Kemiska injekteringsmedel och cementbaserade bruk är de vanligaste bland injekteringsmaterialen. Trots den bättre spridningen av kemiska injekteringsmedel, är de ogynnsamt att använda på grund av miljöfaror som är förknippade med dem. Cementbaserade bruk är mer praktiska att använda på grund av deras låg kostnad och låg miljöpåverkan.

Den största nackdelen med cementbaserad bruk är deras begränsade förmåga att tränga igenom de mycket smala sprickorna. Det är direkt relaterade till deras filtreringstendens. Cementpartiklar har en benägenhet att agglomerera och bilda en ogenomtränglig filterkaka under flödet. Många tidigare studier undersökte faktorerna som påverkar filtreringstendensen och de drog olika slutsatser och föreslog olika metoder för att förbättra inträngningen hos cementbaserade bruk.

Blandningsmetoden är en av faktorer som har en stor påverkan på brukets inträngningsförmåga. En effektiv blandningsmetod förbättrar dispergering av cementpartiklar i blandningen, och därmed inträngnigsförmågan hos injekteringsbruket. Det är känt från tidigare studier att finare malda cementpartiklar är svårare att dispergera. Bruk baserade på mikrofin cement (<30 µm) är viktiga för utveckling av bruk som skulle kunna täta mycket smala sprickor ner till 20 till 50 µm jämfört med 70 till 80 µm idag.

I denna studie utvärderades dispergering hos tre olika blandningsmetoder, en konventionell laboratorieblandare utrustad med 90-mm skiva, en konventionell laboratorieblandare utrustad med R/S system och en ultraljudapparat UP400St. Två cementtyper, INJ30 och UF12, med samma kemisk sammansättning men skiljer sig i malningsgrad har användes för att tillverka bruk. Dispergeringen har testats med filterpump.

Resultaten visade att den konventionella laboratorieblandaren utrustad med 90-mm disk är en ineffektiv metod. Den konventionella laboratorieblandaren utrustad med R/S systemet är en bättre metod jämfört med 90-mm disk men är fortfarande inte tillräckligt effektiv, särskilt när det gäller bruk baserade på ultrafint cement (UF12). Ultraljud är inte bara den bästa metoden mellan de tre metoderna i jämförelse, den är även mer stabil och pålitlig. Det bästa resultatet som erhölls var injekteringsbruk baserat på UF12 som passerade genom 54 µm-filtret, vilket innebär att sprickor ner till 55 µm kan tätas nu. Det kan anses som en betydande förbättring och det finns fortfarande utrymme för ytterligare förbättringar.

I samband med dispergerings effektiviteten hos olika dispergerings metoder, undersöktes också effekten av tillsatser på dispersionen och inträngnigsförmåga. Resultaten visade att tillsatser inte direkt bidrar till en bättre dispergering, men de är nödvändiga för längre spridning i bergssprickor eftersom de påverkar flödesegenskaperna.

Nyckelord

ultrafint cementbruk; mikrofin cementbruk; dispergering; tillsatser; ultraljud apparat; laboratorieblandaren

Preface

This thesis is a part of a project with Dr Almir Draganović, that was carried out in 2019 at Division of Soil and Rock Mechanics, Department of Civil and Architectural Engineering, Royal Institute of Technology. The work has been generously financed by Swedish Rock Engineering Research Foundation (BeFo) and Development Fund by the Swedish Construction Industry (SBUF).

My decision to be part of this project was based on my great interest in underground constructions especially tunnels, my desire to gain deeper knowledge in this filed and most important my belief that combining the theoretical knowledge with practical experience gives the best learning outcomes.

Working two months in the lab with mixing and testing cement-based grouts was not an easy task, but my interest and the constant support from my supervisor facilitated the whole work and kept me motivated. So, I would like to express my gratitude to my supervisor Dr. Almir Draganović for initiating the project, constant support, guidance and rewarding comments. I have learned a lot about the topic from him. Further acknowledgements are dedicated to Division of Soil and Rock Mechanics, they had a great influence on my choice through their courses, seminars, discussions and advices. Special thanks to Royal Institute of Technology for giving me the opportunity to pursuit my goals.

Finally, i would like to thank my Wife and child for their love, patience and understanding, Success in this project would definitely not have been possible without their infinite support.

Stockholm, April 2020

Antranik Karamanoukian

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1. Introduction

1.1. Problem description and relevance

Grouting is an essential part of underground constructions. The main purpose of grouting is to reduce the water ingress and to create a watertight zone as much as possible. In recent years the water sealing has become crucial and the requirements have continuously been increased due to more sensitive locations, advanced technology and increased knowledge. The water inflow requirement is often expressed as a maximum allowable amount of water ingress to the tunnel. In Sweden the requirement varies between 0.5 and 10 liter/min/100 meters of tunnel (Dalmalm, 2004) and the more sensitive the area, the higher the demand. The requirement is usually governed by two factors, how dense population and infrastructure exist on the surface, secondly how sensitive the area is to fluctuation in ground water levels (Norwegian Tunneling Society, 2011). A successful grouting gives many advantages and benefits both long-term and short-term. A lower water ingress results in better working environment and smooth tunneling progress, lower risk for corrosion of different installation that exist in the tunnel, stabilization of some weak zones, prevents drainage of wells and the most important one prevents the lowering of the ground water level. Thus, prevents the settlement of the ground surface caused by consolidation especially when the ground consist of or contains layers of clay which is very sensitive to consolidation. Surface settlements can be crucial in urban area with dense population and infrastructure.

Grouting is basically injection of a liquid material into rock fractures and channels using a pump and through drilled holes (Eklund, 2005). There are two types of grouting process, pre-grouting which is performed at the tunnel face before any blasting or excavation, the second one is post-grouting which is used to seal fractures in already excavated rocks (Tolppanen and Syrjänen, 2003).

Normally grouting is performed as pre-grouting with complementary post grouting if needed (Dalmalm, 2004). In Nordic countries, pre grouted tunnels have been standard (Stille and Palmström, 2011). The reason behind this preference is that pre-grouting is more efficient due to the possibility to use higher grout pressures, with extended grout spread and penetration as a result (Dalmalm, 2004). Post-grouting should be limited and used only if needed due to higher cost and poorer results compared to pre-grouting (Dalmalm, 2004). According to Tolppanen and Syrjänen (2003) the cost for post-grouting can be higher 3 to 10 times more than the cost for pre-grouting.

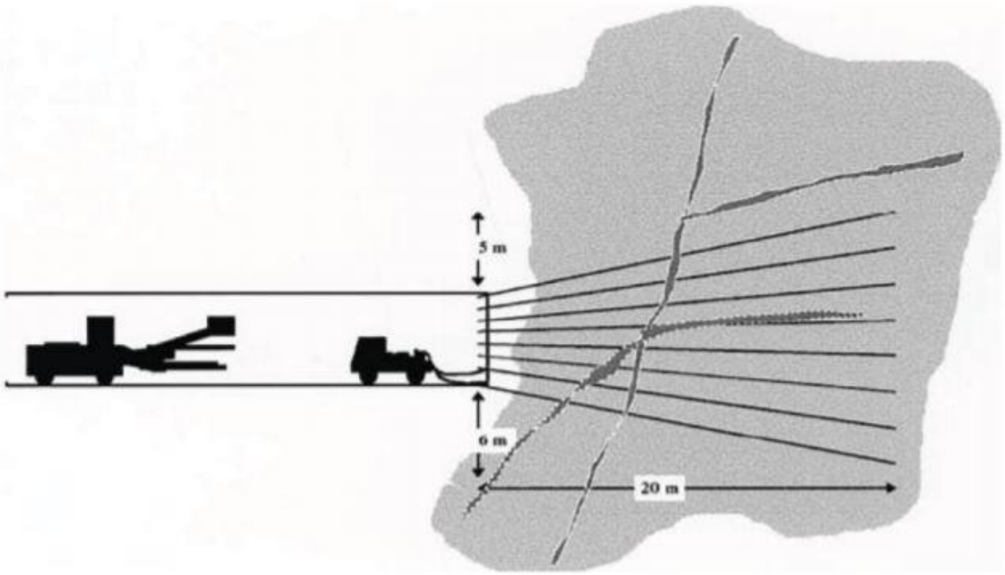

Figure 1. The principle of pre-grouting (Tolppanen & Syrjänen, 2003). From (Akbar and Al-Naddaf, 2015)

When it comes to grouting material, we have mainly two categories, solution grouts (chemical grout) e.g. epoxies, polyurethanes and acrylates, and suspension grout e.g. cement based grout. Each type has pros and cons. The main advantage of chemical grouts is that they have high penetrability that gives them the ability to seal even the very narrow cracks and channels due to their low viscosity without any suspended solids. It makes them very similar to the water (Tolppanen and Syrjänen, 2003). But on the other hand, their high cost and harmful impact on environment make them less desirable. Hallandsås Tunnel is a clear example of chemical grout's harmful impact both on environment and human health (Weideborg et al., 2001), so the use of chemical grouts should be kept at the minimum as a complementary or for very special cases where the other types of grout are not efficient enough.

In contrast to the chemical grout, cement-based grout which contains suspensions of grains in water has a lower penetrability (Tolppanen and Syrjänen, 2003), with a limited ability to penetrate fine cracks due to its content of solid grains (Eklund, 2005). When it comes to the other aspects, the cement-based grout is considered to be good choice due to its low cost, compatibility with the environment and predictable durability (Eklund, 2005).

The cement-based grout basically is a mix of water and cement in a given water to cement ratio by weight and it is signed by w/c (Draganovic, 2009). Previously, the used cement was coarse grained with small specific surface area of around 300 m2/kg (Holt, 2008). It was intended mainly for concrete manufacturing and therefor it is not suitable for grouting of fine cracks (Eklund, 2005). The constant development of grouting materials and techniques led to introduce the fine-grained cement with large specific area of around 1500 m2/kg which is known as micro cement (Akbar and Al-Naddaf, 2015). It is suitable for grouting of fine cracks in rock.

To achieve a successful and efficient grouting, the grout has to fulfill different criteria and many aspects should be taken into account such as grouts bleeding, hardening time, penetrability, chemical resistance, penetration lenght and durability. These different aspects have been tested, discussed and detailed by Nonveiller (1989), Houlsby (1990), Dalmalm (2004), Eriksson and Stille (2005) and Draganovic (2009). The main focus in this thesis is the penetrability of the grout and how it can be improved by better dispersion, so the other aspects and properties of grout mixture are not covered.

As it is mentioned above the penetration length is one of the most important aspects that affects the efficiency of the grouting process. Penetration length is defined as the length of how far a grout penetrates in the rock through fractures from a bore hole (Draganovic, 2009).

If we assume that the fracture has a constant aperture, the penetration length depends on water pressure, grout pressure, the wideness of the aperture and the yield value of the grout (Gustafson and Stille, 1996). In other words, the penetration length is a function of the accumulated friction between the grout and the fracture walls where aperture of the fracture is assumed to be constant along the entire length (Draganovic, 2009). In reality, parallel fractures with constant aperture are never the case and for that reason Gustafson and Stille (2005) came up with improvement on that relationship which makes the calculation of the penetration more realistic. They estimated the cracks opening as a stepwise constant over a range and called it a harmonic mean for the aperture.

The accumulated friction is not the only factor that influence the penetration length, there is another factor of great importance which is the filtration tendency. Filtration can be divided into chemical filtration and mechanical filtration which is known as plug formation (Schwartz, 1997).

The chemical filtration causes the smaller grains to be filtered due to physiochemical properties (Eklund, 2005) and it is not covered in this thesis. The mechanical filtration which is known as plug formation or plug building is when particles of the suspension block the grout route (Dalmalm, 2004). According to Draganovic (2009) plug formation occur when cement grains build a stable arch over a fracture constriction during penetration. Lower filtration tendency means that a larger amount of mixture will pass the filter in the test equipment, meanwhile higher filtration tendency will obviously signify a smaller passed amount of mixture (Eklund, 2005).

As it is shown in the figure 2, the plug formation can occur at the entrance of the crack (contact between the bore hole and the fracture) Alt.1 or even at the contractions Alt.2

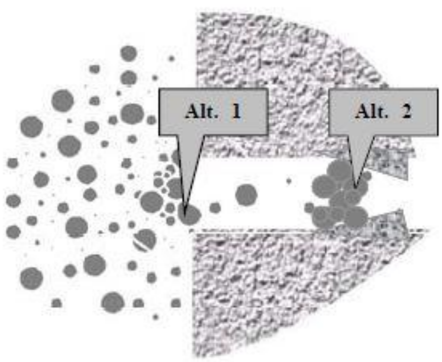

Figure 2. Illustration of plug building Hansson 1994. From Eklund 2006

Many researchers have studied the penetrability of the grout. They even invented many methods to test the penetrability in order to understand the factors that affect the filtration tendency and thus the penetrability, but they came up with different and sometimes even contradictory conclusions.

A sand column test was performed by Schwartz (1997) for the purpose of studying the penetrability and filtration of microfine cement. In this type of test, the penetrability of the grout is measured through the penetration length in the sand or by the amount of the grout that passes the sand column with a possibility to examine the outflowing grout and built filter cake. The author drew the conclusion that the grains of size between 0.4 to 4 μm had the worst penetrability which means that they filtered first, possibly due to flocculation. Grains of size larger than 4 μm do not contribute much to the filtration. So, any grout mixture with high proportion of these particles (between 0.4 and 4 μm) has poorer penetrability, thus flocculation is a key factor that affects the filtration.
The same test was performed also by Axelsson et al. (2009). They studied the filtration effect on penetrability simply by comparing the penetrability results from the test with calculated penetrability based on Gustafson and Stille's (2005) equation. They concluded that the filtration mechanism in the sand is not the same as in the rock fractures, thereby the sand column test is not a proper method to evaluate the penetrability.

Hansson (1995) tested the penetrability using a filter pump. In this method the aperture reduction was represented by the grout suction through a mesh of thin woven steel wires. He claimed that the penetrability of the grout is strongly related to the tendency of the particles to agglomerate and form a filter cake that blocks the grout path and reduces the penetrability. According to him the filter cake formation in the filter pump is very similar to filter cake formation in the fractures. The author found that filtration stability is highly dependent on w/c ratio. Increasing of w/c ratio increases the filtration stability and thus also penetrability. Eriksson and Stille (2003) studied w/c ratio effect on penetrability using another test device called penetrability meter. This device is very similar to the filter pump with one main difference, they replaced the manual pump with a pressure chamber that presses the grout through the same filter. They found the w/c ratio effect on penetrability is not significant. Both the filter pump and penetrability meter are widely used tests in Sweden for estimation of grout penetrability in field and laboratory.
Others also argued about the w/c ratio effect, Hjertström (2001) and Eriksson et al. (1999) confirmed the w/c ratio effect on penetrability but they used another testing method called NES.
Eklund (2005) claimed that the increased w/c ratio is combined with increased porosity of hardened grout, something considered as negative. Bleeding and sedimentation can be another negative result of increased w/c ratio according to Draganovic (2009).

NES is another test that can be used to test penetrability of the grout. The method was developed by Sundberg (1997). In this method a rock fracture is represented by the slot between two steel plates. Many researchers used this method to test different factors that affect penetrability. Hjertström (2001) claimed that the pressure has an impact on the penetrability. According to him higher pressure means better penetrability and the same idea was confirmed by Eriksson et al. (1999). Their conclusions were obtained by tests using NES method. On the other hand, Eriksson and Stille (2003) concluded that the high-pressure effect on penetrability is not significant, but this conclusion was based on the tests using penetrability meter.

Draganovic (2009) developed another test device called short slot. The idea in this new method was to develop a test that covers all the aspects and requirements that affect the penetrability. According to Draganovic (2009) the short slot meet the all requirements that are needed to study the plug formation problem, it has the possibility to vary the pressure and geometry of the constriction, possibility for erosion during penetration and allows us to examine the filter cake. By performing number of tests Draganovic and Stille (2011) confirmed some of the previous conclusions. They concluded that higher w/c ratio gives better penetrability and a higher grouting pressure reduces the plug formation.

There are many other tests besides the aforementioned ones that have been used to study the same issue, PenetraCone developed by Axelsson (2009), Pressure chamber test (Widmann, 1996), High pressure clogging test (Nobuto et al., 2008).

The researchers attempt to study the penetrability and determine the factors that affect the plug formation. Thus, penetrability was accompanied with different suggestion about how to improv it. As it is mention earlier some of them suggested to change the mixing recipe by using higher w/c ratio, others suggested to change the grouting pressure, etc.

One can notice that sometimes there is contradiction between conclusions drawn by different researchers. They performed tests by different test methods which gives rise to one of the major problems, the comparability of the results. How we can compare results from two different test methods with different procedures, settings and standards?
There is an imperative need of having common parameters and standardization to some extent. The parameters b_{min} and $b_{critical}$ are used to describe the penetrability suggested by Eriksson and Stille (2003) represent a good solution for comparability problem. Parameter b_{min} is defined as the largest aperture that the grout can penetrate at all, while $b_{critical}$ is defined as the smallest aperture that the grout can penetrate without filtration. Another option can be the k ratio suggested by Eklund and Stille (2008) which represents a ratio between aperture of the groutable fracture and d95.

There are other suggestions and solutions about how one can improve the penetrability. One of these methods is the use of additives.
There are many types of additives that one can add to the mixture depending on the purpose. There are superplasticizers, retarders, accelerators, swelling and stabilization agents.

Superplasticizers are the most widely used additives that improve penetrability. According to Eklund (2005) they improve the rheology properties, both viscosity and yield value, thus enhance the penetrability and flow characteristics of the grout. Dalmalm (2004) also confirmed the positive effect of using superplasticizers on grouting works. According to him the grout with a superplasticizer can penetrate and reach further before hardening begins. Superplasticizers are basically liquid additives that work mainly in two different mechanism based on the active substance that contain. The melamine-naphthalene based superplasticizers give an electrostatic stabilization, while the acrylic-carboxyl based superplasticizers give a steric stabilization (Dalmalm, 2004).

This type of additives is mostly used in combination with fine-grained cement and many researchers tested and studied the efficiency of adding superplasticizers on the penetrability of the grout. Draganovic (2009) performed many tests to see the effect of adding superplasticizers on fine-grained cement mixtures using the short slot method. He drew the conclusion that additives have a positive effect on penetrability of the grout based on fine-grained cement, but the flocculation is still considerably high. Vovk (1989) stated that adding a superplasticizer to the mixture does not break the flocculation completely. So, there is a positive effect of adding superplasticizers, but it is not sufficient. This effect is highly dependent on grain sizes and distribution, amount of alkali, sulphate and celite (Eklund, 2005).

Grinding the cement is another method that is used in order to improve the penetrability of the grout. Many researchers tried to establish a relation between the maximum grain size and opening of the fracture. One of them is Hansson (1995), he drew a conclusion based on empirical experiments that a good penetrability is achieved when the fracture opening is 3 times larger than the maximum grain size. Other studies showed that to ensure better penetrability the fracture opening should be 10 times larger than the maximum grain size (Ranata-Korpi, et al., 2008).

Theoretically the finer cement particles should result better penetrability but in reality, this relation is partly true. According to Hjertström (2001) grout with cement INJ30 (d_{95} =30 µm) has better penetrability than grouts with both UF16 (d_{95} of 16 µm) and UF12 (d_{95} =12 µm) based on laboratory tests. Other studies showed also that milling the cement grains until a certain point gives better penetrability. Further milling after that point gives a negative impact on penetrability.

Eklund and Stille (2008) studied penetrability of the grout based on different grain size and found that the k ratio ($b_{groutabe}/d_{95}$) for the fine-grained cement must be higher than the k ratio for course-grained cement to meet the same requirements. Figure 3 shows the results of Eklund and Stille (2008) study. One can notice that milled cement to grains size of d_{95} between approximately 20 and 30 µm has the best penetrability (smaller b_{crit}) and further milled cement (smaller than 20 µm) has considerably lower penetrability (higher b_{crit}).

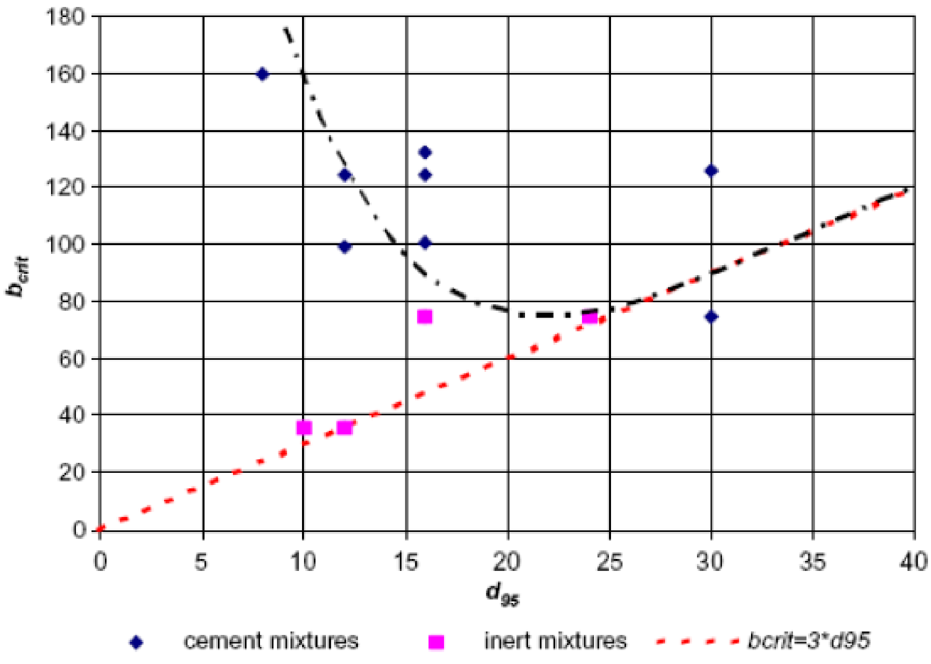

Figure 3. The relation between b_{crit} and d_{95} .From Eklund and Stille (2008)

Draganovic (2009) confirmed the same concept by testing the penetrability of different grain size cement using the short slot. He concluded that the penetrability of grout based on fine-grained cement is considerably lower due to higher hydration and flocculation. According to him, the hydration process where gel particles are developed at the surface of the cement grains increases their sizes and thus decreases their penetrability. The higher flocculation of the finer grains is explained by higher interaction between the particles.
Draganovic and Björk (2014) confirmed the same finding, that the INJ30 has a better penetrability than UF12. Hansson (1995), Hjertström and Petersson (2006) drew the same conclusion, that grouts with very fine-grained cement such as UF16 and UF12 are accompanied with higher hydration and flocculation.

The table 1 shows a comparison between penetrability of four different grouts based on different cement types, ANL (coarse-grained cement), INJ30, UF16 and UF12 expressed by $b_{critical}$ and k ratio. It is clear that grout based on cement INJ30 has much better penetrability than the grout based coarse-grained cement ANL, and grouts with further milled cement, UF16 and UF12, have a worse penetrability than the grout with INJ30.

Table 1. Summarized penetrability of the grouts based on coarse and fine-grained cement from Draganovic (2009)

	ANL, w/c=0.5	INJ30, w/c=0.6	UF16, w/c=1.0	UF12, w/c=1.2
$b_{critical}$ [µm]	250	65	125	250
k ($b_{critical}/d_{95}$)	2	2	8	21

Beside grinding of the cement and using superplasticizers, grout mixing technique also affects the penetrability.
The mixing technique is of great importance especially when the cement is fine-grained or/and different additives are used in the mixture. As it is mention before the fine-milled cement has a higher tendency to flocculate. An efficient mixing technique that can reduce this problem.

Many researchers studied the effect of different mixing devices and concluded that the relation between the penetrability and mixing technique does exist. Eriksson et al. (1999) and Dalmalm (2004) among others expressed the importance of mixing method for the penetrability of freshly mixed grout and how the dispersion of the cement particles is dependent on the type of mixer and its performance.
Axelsson and Turesson (1996) examined the effect of mixing efficiency on filtration and found that longer mixing time and more effective mixers are required for micro-fine cement than the coarse cement.

Hjertström and Petersson (2004) recommended to use higher rotational speed in the colloidal type of mixer to achieve better penetrability of grout based on fine-grained cement. Toumbakari et al. (1999) made a comparison between the results from mechanical mixing with a stirrer at 2400 rpm and combination of ultrasonic dispersion with stirrer at 300 rpm. The grout that they tested was based on ordinary Portland cement (coarse cement) and he found that the combination of ultrasound dispersion with mechanical mixing at low rotational speed is more efficient than only mechanical mixing with high rotational speed.

So, the penetrability of the grout is a complicated process and it is related to many different aspects. Many researchers developed many different tests to study the penetrability and determine the factors that affect it. They came to various conclusions and gave different weight to the same factors.
In general, further milling until a certain point improves the penetrability but the finer the cement the harder to disperse. Then additives and efficient mixing methods are substantial to use in combination for grout based on very fine cement.

1.2. Objectives and scope of work

The main objective of this study is to test the ultrasound technique for dispersion of micro-fine cement grout. As mentioned above ultrasound was tested for dispersion of coarse cement grout in combination of mechanical mixing and showed a good result but it has never been tested on micro-fine cement grouts. Today the best result is achieved using cement with d_{95} around 20 to 30 µm for sealing fractures with aperture of approximately 70 µm. For smaller aperture chemical grouts are used. If the filtration tendency and flocculation problem can be solved using ultrasound dispersion, then grouts based on micro-fine cement can be further improved and used to seal fractures with aperture about 20-30 µm. Then, the need for the undesirable chemical grouting could be reduced. This study evaluates the efficiency of ultrasound dispersion in comparison to conventional laboratory dissolver equipped with either a 90-mm disk or a rotor-stator system.

1.3. Dispersion with ultrasound

Ultrasonic waves can cause a phenomenon known as cavitation in liquids. During the sonication of liquids at high intensities, sound waves that propagate through the liquid media generate alternating high and low-pressure cycles with various rates depending on the frequency. During the low-pressure cycles, the sound waves creates small vacuum bubbles in the liquid. The volume of these bubbles increases as they absorb energy until they reach a certain point at which they cannot longer absorb energy so they collapse violently during the high pressure cycle resulting in high temperature, high pressure and jet streams (Hielscher, 2005).

Suslick (1998) defined the cavitation as the formation, growth, and implosive collapse of bubbles in a liquid. According to him the violent collapse of the bubbles results in intense local heating (approximately 5000 K), high pressure (around 1000 atm) and liquid jets of up to 400 km/h velocity.

Cavitation can also occur in suspensions which contains solid material. Jet streams and their associated shock waves have a wide range of applications with variety of purposes. According to Hielscher Ultrasound Technology, a german company that designs and manufacturers ultrasound devises, the application of ultrasound include blending, homogenizing, deagglomerating, dispersing, emulsifying, dissolving, particle size reduction, etc. It is used in different industries such as cosmetic industry, food industry, paints and pigments, nanomaterials dispersion, chemicals and oils.

Detailed description of the cavitation process in liquids and its applications can be found in many publications such as Suslick (1990, 1998, 1999) and Hielscher (2005). The ability to disperse the agglomerated particles in different suspension is of interest and it is addressed in this study. The next figure illustrates the particles agglomeration problem and expected effect of the ultrasound cavitation

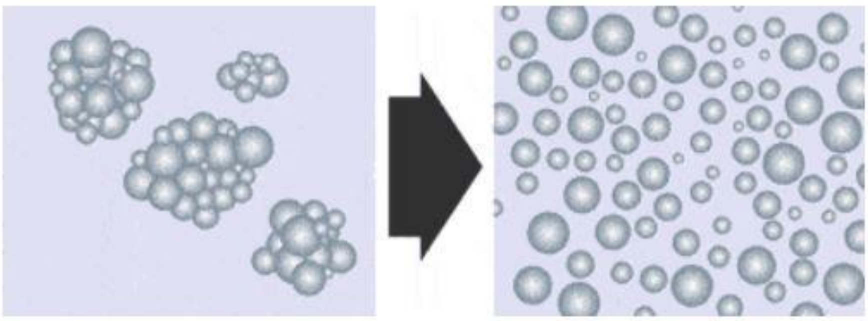

Figure 4. The picture in the left shows agglomerated particles and the picture to the right shows anticipated effect of ultrasound dispersion. From Hielscher website.

Despite the various applications of ultrasound in different domains, it is rarely used for the dispersion of cement grouts. Therefore, the published studies about ultrasound usage to disperse cement grouts are limited and hard to find. Toumbakari et al. (1999) and Miltiadou-Fezans and Tassios (2013) tested the ultrasound dispersion of coarse cement grouts but in combination with a mechanical mixer. Toumbakari et al. (1999) evaluated the efficiency of mixing procedures on the injectability of the grouts based on coarse-grained cement, by comparing the result obtained from using a mechanical mixer at high rotational speed (2400 rpm) with the result obtained from using combination of ultrasound dispersion and mechanical mixer at low rotational speed (300 rpm). The study showed that the combination of ultrasound with mechanical mixer at low rotational speed is more efficient than the use of only mechanical mixer at high rotational speed. Grout mixed using the combination showed better penetrability and rheology. The results obtained from these two studies do not confirm the ability of ultrasound to disperse grouts based on fine-grained cement since they used coarse cement (d_{95} of approximately 60 µm). This type of coarse cement is relatively easy to disperse compared to microfine cement grouts.

However, there are many published studies that confirm the efficiency and ability of ultrasound to disperse and homogenize other materials than grouts. The duration and amplitude of ultrasound are highly dependent on the purpose and material. There are no fixed settings that can be applied on every material. Table 2 shows how different amplitude can be used for different purposes.

Table 2. General recommendations for amplitudes from Hielscher (2005)

Process	Amplitude
Cleaning	0.5 to 2 micron
Intensive Cleaning	10 to 20 micron
Dispersing/Deagglomeration	10 to 30 micron
Emulsifying	20 to 60 micron
Primary Particle Reduction	40 to 120 micron

Zou et al. (2015) investigated with varied ultrasonication energy the dispersion of Carbon nanotubes (CNTs) in cement pastes and found that the ultrasonication could effectively improve the aqueous dispersion of surface functionalized CNTs with the aid of a polycarboxylate-based cement.

Peters (2016) studied the influence of power ultrasound on setting and strength development of cement suspension and as a part of his study he investigated the effect of power ultrasound on workability of Portland cement suspensions. The author verified the homogenizing and dispersing effect of power ultrasound for cement suspensions. Sedimentation experiments and cyro-SEM images clearly showed that the dispersion of very small particles (< 1 µm) was much better after applying the power ultrasound.

A study on the effect of ultrasonication on viscosity and heat transfer performance of multi-wall carbon nanotube-based aqueous nanofluids done by Garg et al. (2009) assured the positive effect of ultrasound on dispersion. They found that the ultrasonication has twofold effect on CNT depending on the processing time. Below the optimum processing time, ultrasonication gives better dispersion. But once the optimum time has been reached a further sonication causes increased breakage rate of the nanotubes. The same process can be used for improving viscosity, which increases with sonication time until a certain value and start to decrease thereafter. Similar confirmation came from Alrekabi et al. (2016), they investigated also the effect of ultrasonication on dispersion and mechanical performance of multi-wall carbon nanotube-cement mortar composites.

Ultrasound also was tested to disperse nanosized silica and alumina in distilled water and glycerol Schilde et al. (2011). In this study the efficiency of different dispersing devices such as kneader, dissolver, 3-roller mill, disk mill, stirred media mill and ultrasonic homogenizer was evaluated and compared. Adio et al. (2016) studied also the ultrasound dispersion of Al2O3–glycerol nanofluid. Both studies yielded similar results that confirm the positive and effective impact of ultrasound on dispersion.

Hielscher Ultrasound Technology, the german company that designs, and manufacturers ultrasound devises, provides many other examples of successful ultrasound dispersion such as ultrasonic dispersion of polishing agents (CMP), ultrasonic dispersion of Graphene, etc.

There are numerous studies that confirms the efficiency of ultrasound dispersion. The method has been tested on different materials with sizes of micro to nano. Some of them with addition of stabilizers and others without. Different measuring methods were used to evaluate the dispersion depending on the application. All the tests regardless of the differences yielded the same conclusions that ultrasound can be used to disperse material and it is more efficient than the conventional mixers.

2. Material and Methods

2.1. Grout material

In this study two types of cement were used, INJ30 with d_{95} of 30 µm and UF12 with d_{95} of 12 µm produced by Cementa. Both cements have the same chemical composition since they are produced form the same cement. They are produced from Anlägningscement which is Portland cement CEM I 42,5 N - SR 3 MH/LA. The only difference between the two types is the degree of milling.

Both INJ30 and UF12 according to technical data sheet from Cementa are sulphate resistant, chromate reduced and low alkaline cement that are produced to be used as injection cement.

There are two reasons behind the choice of using these two types of cement. The first one is the fact that the finer the cement particles, the more difficult the dispersion is. This was confirmed by Eklund and Stille (2005) who found that the penetrability is improved by milling the cement to between 20 and 30 µm and further milling results in decreased penetrability as it is illustrated in figure 3. Others such as Draganovic (2009), Draganovic and Björk (2014) also confirmed that grout with INJ30 has better penetrability than grout with UF12 due to fact that grout with INJ30 cement is relatively easier to disperse than grout with UF12 cement. Since this is the first time that Ultrasound us used to disperse micro-fine cement, so it is more convenient to start testing the ultrasound on INJ30 first and gain some experience about the new technique before applying it on hard dispersed UF12.

The second reason is that these two cements were widely used and tested in many earlier studies and this facilitates the comparison of the results.

2.2. Grout recipe

For INJ30 a water to cement ratio w/c of 0.8 was used. The reason for using this w/c ratio is that it is the most often used recipe used to seal underground construction in Sweden and thus the most relevant to study. For UF12 a water to cement ratio w/c of 1.2 was used.

For both INJ30 and UF12, iFlow additive from Sika was used. The recommended dosage is approximately 0.3 to 0.5% of the cement weight. In this study the dosages for INJ30 and UF12 were 0.4% and 0.5% respectively. According to Sika iFlow has both electrostatic and steric effect on cement particles and it improves both rheology and dispersion of the grout.

Both INJ30 and UF12 were mixed using tap water with temperature between 13 to 14 °C.

2.3. Dispersion equipment and process

To evaluate the efficiency of ultrasound dispersion, one need to compare results obtained from ultrasound dispersion with results obtained from other dispersion methods carried out under the same settings and conditions. In this study three different dispersing methods were used. The first one is the laboratory dissolver DISPERMAT CV-3 from VMA-GETZMANN equipped with 90-mm disk. The second one is the same laboratory dissolver but equipped with a rotor-stator (R/S) system and the third one is ultrasound device UP400St from Hielscher equipped with the sonotrode H22. A detailed description of the mixing procedure with each of these methods are presented in the following sections (2.3.1 to 2.3.3). The next figure shows the three different dispersion methods used in this study

Figure 5. a) DISPERMAT CV-3 dissolver equipped with 90-mm disk, b) DISPERMAT CV-3 dissolver equipped with rotor-stator system, c) Ultrasound sound UP400St device

2.3.1. Dispersion with DISPERMANT CV-3 dissolver equipped with 90-mm disk

DISPERMANT CV-3 is a conventional mixer that widely used in laboratories and can be equipped with different types and sizes of modular dispersion and fine grinding systems.

With 90-mm disk, movement of the disk's vanes at a high velocity creates high- and low-pressure areas in front of and behind the vanes, and according to VMA, the alternating stress acting on the agglomerates in these areas facilitate the dispersion in addition to the smashing impact of hitting the larger agglomerates by the edges and surfaces of the vanes. So, the greater part of the dispersion occurs at the surface of the dissolver disk, but some partial dispersion could also achieve by shear forces between the mixture layers due to their flow at different velocity.

To achieve the best dispersion results there are some recommendations from VMA regarding the geometry of the container/bucket, the diameter of the disk, the peripheral velocity and the distance between the disk and the bottom of the container/bucket. In this type of mixers, the speed of the mixing shaft is controlled and should be increased until no standing material can be seen at the wall of the container and a laminar rolling flow pattern obtained. At a certain speed a part of the disk becomes visible, this is a sign that a doughnut-like flow pattern is obtained which means that the maximum mechanical power possible is transferred to the mixture. So, it is very important to optimize the mechanical power input, so the highest rotational speed thus greatest peripheral velocity is obtained without disturbing the doughnut flow pattern.

According to VMA the doughnut-like flow develops because the mixture is accelerated outwards from the edges of the disc and when it hits the wall of the container, the jet/flow is split into two parts. One is going downwards and flows back to the middle of the disk along the bottom of the container and rises and hits the disk again. The second one is going upwards with the same circular path restricted by the gravity and rheology of the mixture. The next figure shows the recommended geometry by VMA and the flow pattern

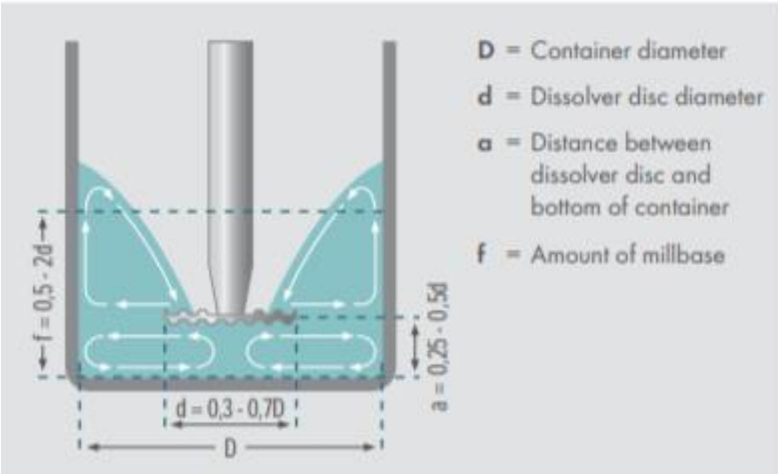

Figure 6. Recommended setup from VMA-GETZMANN

Draganovic and Björk (2014) performed a series of tests using the same dissolver, grout batch and recipe, they did a comparison between the disk and R/S system. Their conclusion was that to achieve a proper mixing, the whole grout mixture should be in motion and in case of the disk, the doughnut-like flow indicates that the whole grout is in motion.

In this study the bucket that was used had a volume of 10 l and a diameter of D=240 mm and the disc diameter d=90 mm which means that d=0.375D. The height of the grout was approximately f=98 mm which means f=1.09d and the distance between the disk and the bottom of the bucket was a=32 mm which means that a=0.356d. So the tests setup were within the recommended range of values.

The weighted cement was added to the water gradually while a mixing rod on a screwdriver was used to pre-mix the grout for approximately 30 to 45 seconds. After the pre-mixing the grout was mixed for 4 minutes at 2000 or 6000 rpm. Two different rpm were tested. After mixing the grout was agitated for additional 10 minutes at 700 rpm. The total mixing time was about 15 minutes, and this is normally the minimum time required to prepare and inject the grout in the field. So performing the measurements after 15 minutes makes the results more representative and realistic than the results obtained directly after 4 minutes mixing.

In tests with additive, iFlow was added after 1 minute of mixing. The total batch volume was approximately 4.4 l. Figure 7 a) shows the doughnut-like flow where part of the disk is visible. Figure 7 b) shows the screwdriver equipped with a mixing rod that was used for pre-mixing.

Figure 7. a) the doughnut-like flow of the grout at a mixing speed of 2000 rpm b) the mixing rod and screwdriver that was used for pre-mixing

2.3.2. Dispersion with DISPERMANT CV-3 dissolver equipped with R/S system

The dissolver is the same but equipped with other dispersion system called rotor-stator R/S system. This system consists of a rotating metal shaft (the rotor) which exists inside a stationary metal sheath (stator) that contains slots or holes. The dispersion process takes place through mechanical tearing, shear fluid forces and cavitation.

The flow pattern is this system is totally differing from the disk system and the dispersion occurs through a different mechanism. The rapid rotation of the metal shaft (the rotor) generates a suction effect which draws both the liquid and solid particles into the gap between the rotor and stator. Then centrifugal forces throw the material out through the holes/slots in the stator. The main dispersion takes place in the small gap between the rotor and stator by high shearing forces due to an extreme alteration in velocity there. The open-configuration of the rotor-stator system in combination with the high-speed motion of the mixture caused by the system ensure that the mixture/product is repeatedly recirculated. The flow pattern and shearing process of the R/S system are illustrated in figure 8.

As mentioned earlier according to Draganociv and Björk (2014), the whole amount of grout being in motion is an essential prerequisite for proper mixing and in case of R/S system it is achieved when the boiling effect takes place.

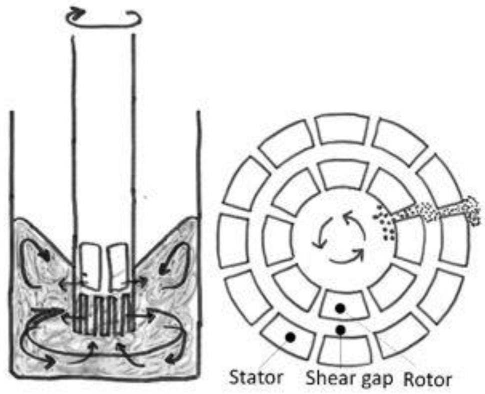

Figure 8. Illustration of the flow pattern and shearing process of the R/S system. From Draganović et al. (2020)

The same bucket and batch volume used with the 90-mm disk was also used with the R/S system, the distance between the bottom of the bucket and the R/S shaft was 17 mm. The dispersion process was also divided to pre-mixing with a rod on a screwdriver for approximately 30 to 45 seconds, mixing for 4 minutes at 10000 rpm and agitation for 10 minutes at 4000 rpm for grouts without additives and at 3000 rpm for grouts with additives. The difference in the rotational speed during the agitation is due to better rheology when the additives are used. In figure 9 one can see the boiling effect that was achieved at 10000 rpm

Figure 9. The boiling effect using the R/S system at a speed of 10000 rpm

2.3.3. Dispersion with the ultrasound device UP400St

Ultrasound device UP400St, from the German company Hielscher (figure 5 C), is designed to be used mainly in the lab, but also can be used for production of small quantities. The device has great technical features and is also suitable for the use in demanding environments according to Hielscher.

Some of the technical features of UP400St:

- It operates at 24 KHZ frequency
- It has a powerful 400 watts ultrasonicator
- It can be operated continuously 24h/7d under full load
- Amplitude (from 20 to 100%), pulse (from 10 to 100%) and time of sonication can be controlled
- Equipped with a colored touch screen for easy controllability
- It has pluggable temperature sensor
- It has integrated SD-card for automatic data recording
- It is compatible with sonotrodes of a diameter range from 3 to 40 mm
- It can be used for the sonication of sample volumes from 5 to 4000 ml
- It has an automatic calibration when needed

The above-mentioned features among others makes this device not only suitable for dispersion, but for various application also such as emulsifying, wet milling, extraction, homogenizing, sonochemical processes, degassing, cell disruption and disintegration.

Dispersion with ultrasound is based on phenomenon known as acoustic cavitation in liquids which is described in detail in section 1.3. Since the material is dispersed by shock waves and jet streams there is no specific flow pattern (see figure 10) as in the other two methods.

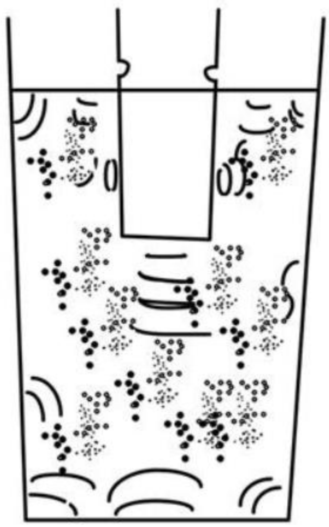

Figure 10. illustration of dispersion process with ultrasound. From Draganović et al. (2020)

As it is mentioned earlier, this the first time that ultrasound was used for dispersion of microfine cement grouts and for that reason we lack a reference guide regarding the appropriate sample size, amplitude and sonication time. The only available reference guide is the general recommendation for amplitudes from Hielscher (2005) that suggests using amplitude of between 10 to 30 microns for dispersion and deagglomeration purposes as it is shown in table 2.

To establish a proper setup for the tests with ultrasound device and better understanding for the whole sonication process, several trials were performed using only water. The idea was to observe the air bubbles, jet streams and flow pattern using various amplitudes and different submerged depths of sonotrode. During the next series of trials, a small amount of cement was added to the water to observe the cement particles motion during the process and how the dispersion works. Figure 11 c1) shows one of the trials with only water and c2 shows another trial after adding a small amount of cement.

In this study the ultrasound device was equipped with sonotrode H22. The sonotrode is made of titanium and has length of 100 mm and tip diameter of 22 mm and is suitable for samples from 100 to 2000 ml and was suitable for this study.

Three different sample volumes were used in the study. The first one was approximately 435 ml in a 600 ml vessel, the second one was approximately 860 ml in a 1 liter vessel and the last one was approximately 2 liters in 2.5 liters vessel. Using different volumes with different geometry was important to the understanding and evaluation of the sonication process. According to Hielscher (2005) “the sonicated sample volume and the time of exposure at a certain intensity have to be considered to describe a sonication process in order to make it scalable and reproducible”. In other words, the final result of a sonication process is a function of the sample volume, intensity and the time of exposure, this relation is expressed in Hielscher (2005) as the follow

$$\text{Result} = f\,(E\,/V)$$

where: V is the sample volume
E is the product of the power output (P) and the time of exposure (t)
$E[Ws] = P[W]*t[s]$

This means that changes in the parameter configuration can change the result.

Beside the different volumes, the tests were performed with different amplitudes and sonication times. Before sonication the grout samples were pre-mixed with a spoon for approximately 30 seconds. During sonication the sonotrode was submerged 45 mm into the grout. In figure 11 a) one can see the grout condition after only pre-mixing with a spoon for 30 seconds. It is obvious that the grout was not well dispersed. Picture in b) shows the grout condition after the sonication process. The improvement in the grout dispersion is easily noticeable.

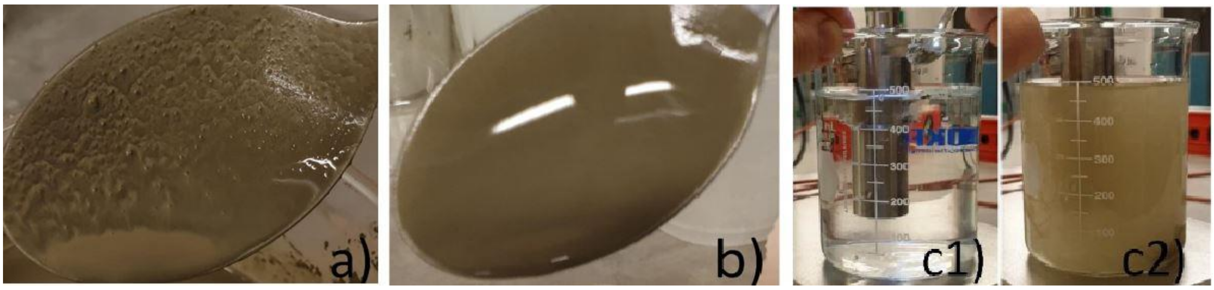

Figure 11. a) shows the grout after pre-mixing for 30 seconds with a spoon, b) shows the grout after sonication, c1) shows a test with only water and c2) shows a test with water and small amount of cement

2.4. Filter pump as a method to test dispersion efficiency

There are different devices and methods that can be used for measuring the penetrability of freshly mixed grout such as the filter pump, sand column, short slot, NES method, etc.

The choice of a test method is governed and effected by many factors such the aim of the study, the available time frame, the simplicity of the method, how representative the results are etc.

In this study the filer pump was used as a method to test dispersion efficiency. Filter pump is an instrument developed by Per Hansson in 1995 for measuring filtration stability. The method can be used both in laboratory and on site and follows the European standard EN 14497. It consists of a metal tube with diameter of 25.6 mm and length of 583 mm and a metal rod. The rod ends with a handle and is attached to a rubber piston at the other side. A cup holder holds the filter which is a mesh of thin woven steel wires. Different sizes of filter can be used. In this study filters of sizes between 200 to 42 µm were used. Figure 12 illustrates the filter pump's outline.

Filter pump is relatively easy to use compared to other methods. The measurement does not require long time to perform and gives a fair assessment of the grout's dispersion. The poorly dispersed and agglomerated particle cannot pass through the filter, while well dispersed particles do.

According to the European standard EN 14497, the filter pump should be immersed in 1-liter grout. It is recommended to keep the tip of the filter pump at the half depth of the grout. The grout is drawn/sucked by pulling the handle to the full stroke or until a flow stop is achieved due to plug formation at the filter. The sucked grout is pumped out into a measuring vessel to obtain the volume. The maximum grout volume that can be sucked by the filter pump is 300 ml which indicates that there is no filtration tendency. The measured volume of passed grout is used as a relative measure of filtration tendency of the grout thus the dispersion of the particles.

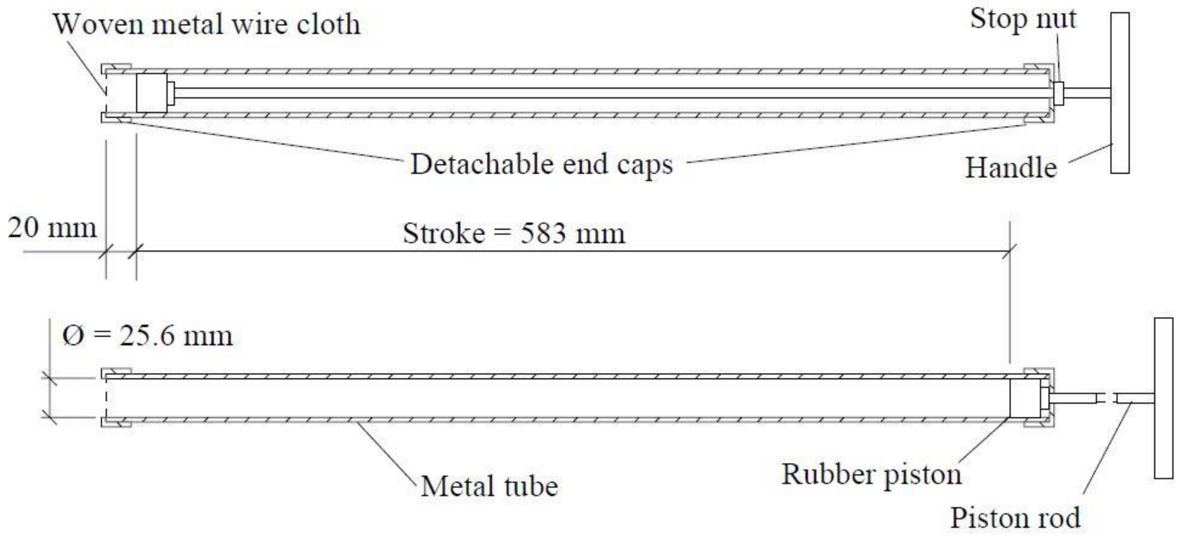

Figure 12. Filter pump's outline. From Eklund (2005)

It is important that the sucking pressure is kept uniform/constant as much as possible during the test. The pump should be easy to draw while the grout passes through the filter without forming a filter cake. But once the filter cake starts to build the resistance became higher and in short time a complete filter cake is built. See figure 13 b), in this case there is no need to use an excessive sucking pressure otherwise there is a risk for pressing the plug through the filter. It is also important to keep the sucking pressure similar for all tests, and avoid any variance caused for example by tiredness.

In the tests with DISPERMANT CV-3 dissolver equipped with 90-mm disk or R/S system, the batch volume was approximately 4.4 l. After the mixing for four minutes and agitation for ten minutes the grout was directly poured into 4 different sample vessels 1 l each and tested using 4 different filter pumps with various filter sizes. See figure 13 c). Using four filter pumps was very effective from a time perspective. It reduced the overall time needed for testing, and it minimized the waiting time between the measurement on the same grout batch which is also important. A visual inspection was also done as a complementary step for the test with filter pump. The whole remaining grout in the mixing bucket was poured out and the bottom of the bucket was visually observed, in case of any sediment at the bottom. An eventual presence of sediment is a sign of poor dispersion and mixing. For the same reason after each filter pump test the remaining grout was poured out from the sample vessels and the bottom of the vessel was observed.

In the tests with Ultrasound, the measurements with filter pump were performed directly from the sample vessel. The remaining grout was also poured out and the bottom of the vessel was examined. It is important to highlight that three different sample volumes were used as it is mentioned before. Two of them were less than 1 liter (435 ml and 860 ml) the recommended sample volume for measurement with filter pump according to the European standards EN 14497. The maximum suction volume for the filter pump is 300 ml which means that the samples of 435 ml were 1.5 times more than the maximum capacity of the filter pump and the samples of 860 ml were almost 3 times more than the maximum capacity of the filter pump. Moreover, neither disturbance nor negative effect of that issue were noticed during the test. Therefore, the samples were considered as sufficient for performing the test and for the aim of this study.

It is important to know that after each test the filter pumps were cleaned carefully both outside and inside, lubricated with WD-40 Multispray and new filters were mounted. Figure 13 a) shows a new filter mounted on filter pump. The cleaning was also applied on sample vessels, measuring vessels, mixing bucket and all equipment that were used in the test. Careful cleaning is substantial to ensure that the same quality of measurements was maintained for all the tests, and to exclude any errors related to human or/and equipment that can affect the results.

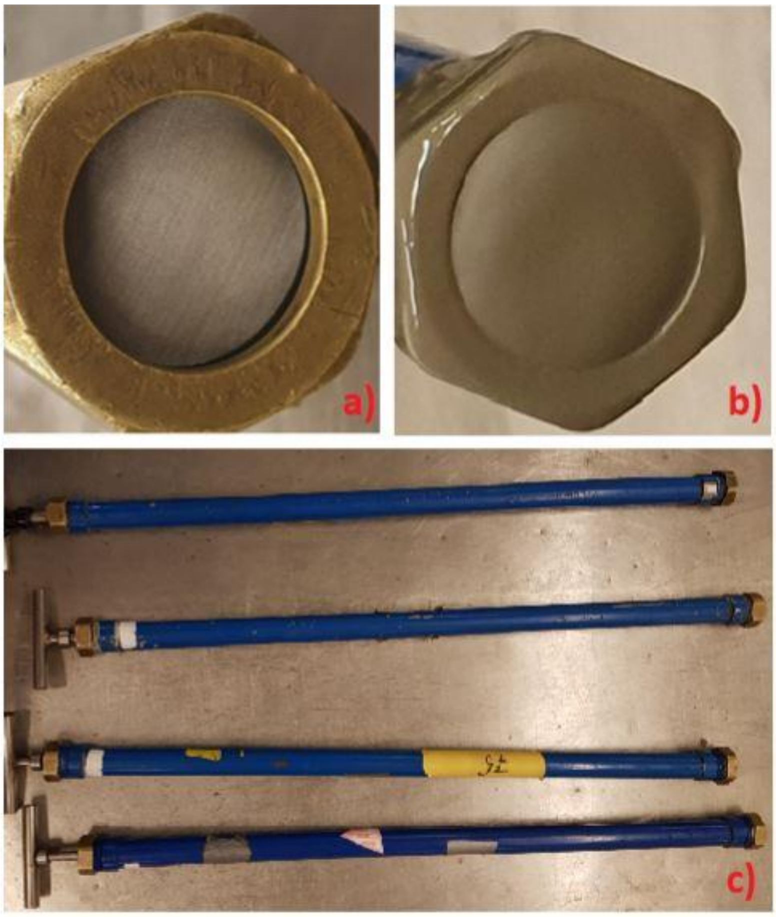

Figure 13. a) filter pump with clean filter, b) filter pump with filter cake and c) the four filter pumps used in this study

2.5. Rheology test

There are two main factors that affect the penetrability of freshly mixed grout. The first one is the rheology (flow properties), and the second one is the filtration tendency. According to Eklund (2005) both should be optimized in order to achieve a good penetrability of the grout mixture. As it is explained earlier in section (1.1.) adding superplasticizers is the most widely used method to improve the rheology properties, thus enhance the penetrability of the grout mixture.

The main focus of this study is the dispersion effect on penetrability of the grout, but due to fact that rheology is also related to the penetrability, it is relevant to the overall objective of this study to do rheology of some tested mixtures.

Performing rheology tests on both INJ30 and UF12 grouts gives us a better understanding of the additives effect on dispersion and penetrability.

2.5.1. Rheology test with TA AR2000 Rheometer

TA AR2000 is an advanced rheometer from TA Instrument that includes controlled shear stress, controlled shear rate and interchangeable temperature control, and is suitable for the most demanding rheological applications. It can be used with wide range of viscosities from like water liquids to polymer melts and has the ability to apply a full range of shearing motions such as steady, oscillatory, startup and cessation.

This device was used to test the rheology of INJ30 according to DIN-53019. The figure 14 a) shows this rheometer

2.5.2. Rheology test with Brookfield DV-II+ Programmable Viscometer

Brookfield DV-II+ Programmable Viscometer is a device that designed to measure fluid viscosity at given shear rates. The viscosity is defined as a fluid’s resistance to flow. According to Brookfield, the manufacturer, the principal of operation for this device is to drive a spindle that is immersed in the test fluid through a calibrated spring. By measuring the spring deflection with a rotary transducer, the viscous drag of the fluid against the spindle is determined.

The device was used to test the rheology of UF12 grout according to DIN-53019. The figure 14 b) shows the Brookfield DV-II+ Programmable Viscometer that was also used in this study.

Figure 14. a) TA AR2000 Rheometer b) Brookfield DV-II+ Programmable Viscometer

3. Results

The results for INJ30 and UF12 grouts are presented in two different sections and each section is divided into subsections that shows the results from corresponding methods which was tested in this study.

3.1 Results for INJ30

3.1.1. Dispersion achieved with DISPERMANT CV-3 dissolver equipped with 90-mm disk

The results for this method are presented in table 3 and as can be seen from the table, this method was not effective in dispersing of the tested grout. In tests without an additive (iFlow), direct stoppage occurred even with the largest filter of 154 µm. No improvement at all was noticed when the additive was added and increasing of the rotational speed to 6000 rpm improved the dispersion slightly.

Table 3. Dispersion of cement grouts based on IN30 mixed with DISPERMANT CV-3 dissolver equipped with 90-mm disk

Mixture	**Additive**	**Dispersion**		**Grout volume passed through filters**							
		Mixing 4 min	Agitation 10 min	54 µm	62 µm	77 µm	91 µm	104 µm	122 µm	132 µm	154 µm
No.	[%]	[rpm]	[rpm]	[ml]	[ml]	[ml]	[ml]	[ml]	[ml]	[ml]	[ml]
1	None	2000	700			≈0	≈0	≈0			
2	None	2000	700						≈0	≈0	≈0
3	0.4	2000	700				≈0	≈0	≈0	≈0	
4	0.4	2000	700					≈0	≈0	≈0	≈0
5	0.4	2000	700					≈0	≈0	≈0	≈0
6	0.4	6000	700					25	25	25	75
7	0.4	6000	700					20	60	60	100

3.1.2. Dispersion achieved with DISPERMANT CV-3 dissolver equipped with R/S system

The results for this method are presented in table 4. As can be seen from the table, this method gave significant improvement on dispersion of the grout and was much more effective than the 90-mm disk. In tests without an additive, the grout passed the 91 µm filter with full volume of 300 ml in all four measurements and even passed the 77 µm filter with full volume of 300 ml in two out of five measurements. In tests with additive, the results were surprisingly slightly worse so adding an additive did not improve the dispersion in contrast to what was expected.

One can see from the table that in tests with an additive, the grout passed the 104 µm filter with full volume of 300 ml in all three measurements. The grout also passed the filter 91 µm with full volume of 300 ml only in two out of six measurements and the 77 µm filter in one out of five measurements so the additive did not improve the penetrability of the mixture. As it is mentioned before, rheology tests were also performed to understand the effect of additives on the mixture. Two rheology tests were performed, one on a sample taken from mixture number 3 (without additive) and another test on a sample taken from mixture number 8 (with additive). The results from rheology tests are presented in section 3.1.4.

Table 4. Dispersion of cement grouts based on INJ30 mixed with DISPERMANT CV-3 dissolver equipped with R/S system

Mixture	Additive	Dispersion		Grout volume passed through filters							
		Mixing 4 min	Agitation 10 min	54 µm	62 µm	77 µm	91 µm	104 µm	122 µm	132 µm	154 µm
No.	[%]	[rpm]	[rpm]	[ml]	[ml]	[ml]	[ml]	[ml]	[ml]	[ml]	[ml]
1	None	10000	4000			200	300	300	300		
2_a	None	10000	4000			300	300				
2_b						300	300				
3_a	None	10000	4000		20	260	300				
3_b						220					
4	0.4	10000	3000					300	300	300	300
5	0.4	10000	3000	≈0	40	150	300				
6_a	0.4	10000	3000			50	150				
6_b						50	125				
7_a	0.4	10000	3000			75	210	300			
7_b							210				
8	0.4	10000	3000		20	300	300	300			

3.1.3. Dispersion achieved with the ultrasound device UP400St

Three different sample volumes (435 ml, 860 ml, 2 l) were used to test the dispersion with ultrasound UP400St. The device came without standard sample vessel or any recommendation about the geometry of the sample vessel. Therefore, we performed some tests with different sample volumes with different vessel, to investigate if the variance in volume and geometry affects the dispersion outcome. Tables 5, 6 and 7 show the results obtained using the 435 ml, 860 ml and 2 l sample, respectively.

From table 5 one can see that different sonication times, amplitudes and even depths of sonotrode were tested. The best result obtained was the grout passing the 77 µm filter with full volume of 300 ml and it was achieved with different combinations of amplitudes and durations. The reason behind testing different combinations for the same sample volume (435 ml) is that to find most optimal setup with respect to specific energy. It was relevant to explore the possibility of getting the best dispersion result with the minimum required specific energy. Amplitude of 20 µm and sonication time of 2 minutes was sufficient to achieve a good result where the grout passed the 77 µm filter with a full volume of 300 ml in two out of 3 tests, increasing the amplitude to 30 and 50 µm in combination with the same sonication time of 2 minutes gave the same result (the grout passed the 77 µm filter with a full volume of 300 ml in 7 out of 9 tests) and enhanced the stability and repeatability of the method. Of course, higher amplitude and longer sonication time was accompanied with higher specific energy and even higher grout temperature at the end of sonication process. The consequences are discussed later in the respective section.

Neither the higher amplitude (up to 90 µm) nor the longer sonication (up to 3.5 minutes) time was sufficient to achieve a better dispersion and make the grout passes through the 62 µm filter. Only small amount of the grout passed through it.

Table 5. Dispersion of cement grouts based on INJ30 with ultrasound device UP400St, sample volume 435 ml

Mixture	**Additive**	**Dispersion**						**Grout volume passed through filters**	
		Depth of sonotrode	Amplitude	Time	Total energy	Specific energy	Grout temp.	62 µm	77 µm
No.	[%]	[mm]	[µm]	[min]	[Wh]	[Ws/ml]	[°C]	[ml]	[ml]
1	0.4	45	20	1	1.13	9.34	18		190
2	0.4	45	20	1.5	1.76	14.59	19		250
3	0.4	45	20	2	2.32	19.26	20		300
4	0.4	45	20	2	2.34	19.40	21		300
5	0.4	45	20	2	2.30	19.07	21		230
6	0.4	45	30	2	2.41	19.96	21		300
7	0.4	45	30	2	3.15	26.10	23		300
8	0.4	45	30	2	2.48	20.54	23		300
9	0.4	45	30	2	2.49	20.60	23	10	
10	0.4	45	30	2	2.47	20.46	-		160
11	0.4	45	50	2	3.88	32.12	-		175
12	0.4	45	50	2	4.06	33.59	26		300
13	0.4	45	50	2	4.03	33.39	29		300
14	0.4	45	50	2	4.13	34.16	26		300
15	0.4	57	50	2	5.97	49.43	-		300
16	0.4	45	50	2.75	5-59	46.25	30		300
17	0.4	45	50	3.5	7.37	61.00	32		300
18	0.4	45	50	3.5	7.55	62.53	31	50	
19	0.4	45	50	5	9.90	81.98	37		300
20	0.4	45	65	2	5.23	43.26	27		300
21	0.4	57	75	2	7.45	61.71	38		300
22	0.4	45	75	2	5.90	48.87	28		300
23	0.4	45	75	3.5	14.93	123.53	45	50	
24	0.4	45	90	3.5	17.83	147.55	51	45	

Fewer tests were performed using the 860 ml sample. The reason for that is the experience and knowledge gained from the previous tests with ultrasound. The finding the optimum settings (best penetrability with minimum specific energy) was important but not the only goal that need to be explored. There was a need to explore the effect of the sample vessel geometry also.

Results from tests using the 860 ml sample are presented in table 6, one can see from the table that a repeatable dispersion was achieved using an amplitude of 60 µm and sonication time of 2 minutes where the grout passed the 77 µm filter with a full volume of 300 ml in 3 out of 3 tests. Due to limited time frame only one more test with lower amplitude of 30 µm was performed. Despite the fact that the grout could not pass the 77 µm filter with a full volume, still considerable amount of the grout managed to pass through the filter. It means that it is possible that any amplitude between 30 and 60 could be sufficient enough to have a full volume of 300 ml passing through the 77 µm filter.

Table 6. Dispersion of cement grouts based on INJ30 with ultrasound device UP400St, sample volume 860 ml

Mixture	**Additive**	**Dispersion**						**Grout volume passed through filters**
		Depth of sonotrode	Amplitude	Time	Total energy	Specific energy	Grout temp.	77 µm
No.	[%]	[mm]	[µm]	[min]	[Wh]	[Ws/ml]	[°C]	[ml]
1	0.4	45	60	2	4.76	19.93	23	300
2	0.4	45	60	2	4.76	19.93	22	300
3	0.4	45	60	2	4.96	20.78	22	300
4	0.4	45	30	2	3.66	15.34	21	250

The same was applied even on the 2-l sample. Totally only five tests were performed to investigate the relation between the geometry of the sample vessel and the required amplitude. Results obtained from the 2-l sample are presented in table 7. One can notice from the results that the required amplitude to obtain a good dispersion similar to the 435 and 860 ml was much higher, and this higher amplitude gave considerable rise for the specific energy. The reason behind this increase is probably the geometry between the sample vessel and the sonotrode, where the distance between the sonotrode and the sample bottom in this case was probably too long.

Table 7. Dispersion of cement grouts with ultrasound device UP400St, sample volume 2 l

Mixture	**Additive**	**Dispersion**						**Grout volume passed through filters**		
		Depth of sonotrode	Amplitude	Time	Total energy	Specific energy	Grout temp.	77 µm	91 µm	102 µm
No.	[%]	[mm]	[µm]	[min]	[Wh]	[Ws/ml]	[°C]	[ml]	[ml]	
1	0.4	45	60	2	4.76	8.57	23	20		
2	0.4	45	80	3	9.30	16.76	22		150	300
3	0.4	45	90	4	13.72	24.70	25		250	300
4	0.4	45	90	7	23.99	43.20	28	300	300	
5	0.4	45	90	7	23.89	43.02	30	300	300	

3.1.4. Rheology measurements

Two rheology tests were performed using the TA AR2000 Rheometer that follow the standard DIN-53019. The samples were taken from the batches mixed using DISPERMANT CV-3 dissolver equipped with R/S system (mixture no.3 without additive and mixture no.8 with additive). The filter pump tests showed that additives did not improve dispersion of the grout and sometimes even give poorer results.

Results from rheology test are presented in figure 15. Yield stress and plastic viscosity were estimated using Bingham model. From the results one can see that adding an additive (iFlow in this study) gave a significant improvement on the rheology of the grout. The yield stress dropped off from 6.06 Pa in case of grout without additive to 1.27 Pa for the grout with additive. The plastic viscosity dropped off also from 15.11 mPas in grout without additive to 7.24 mPas in the grout with additive.

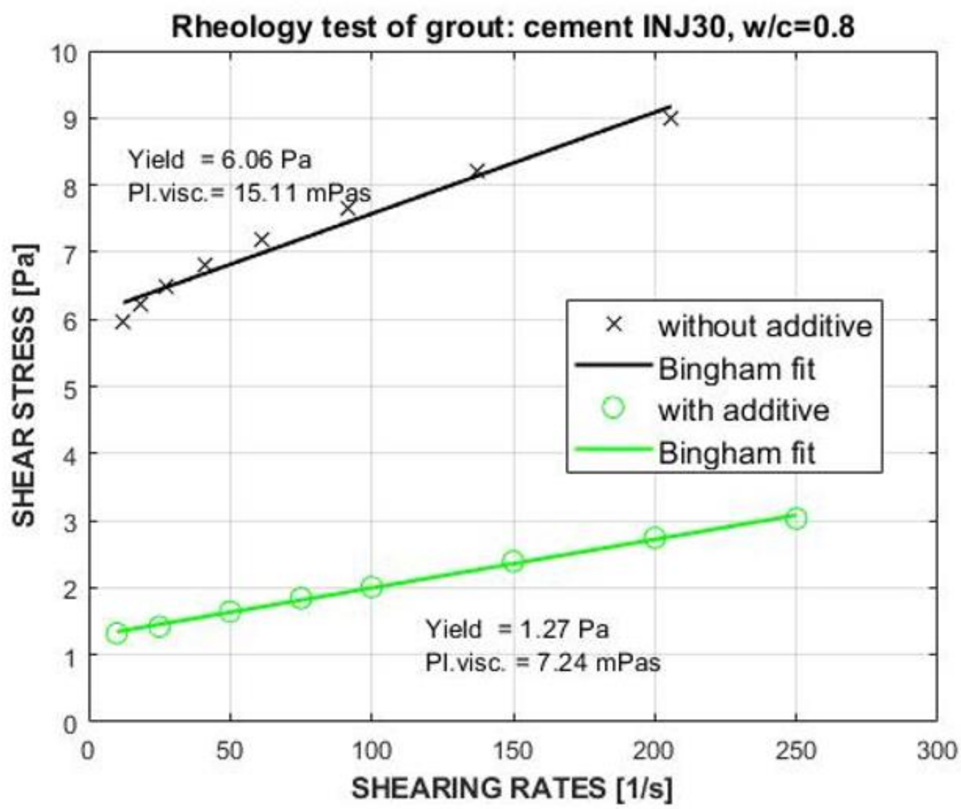

Figure 15. Rheology measurements with TA AR2000 Rheometer (cup r=15 mm, bob r=14 mm) for two samples, one with additive and another without additive

3.2 Results for UF12

3.2.1. Dispersion achieved with DISPERMANT CV-3 dissolver equipped with R/S system

The results tested with this method are presented in table 8. As it can be seen from the table, seven measurement were performed on five mixtures. Four of them without additives and three with additives. This method showed very poor dispersion results for both cases with and without additives, where the grout failed to pass with a full volume of 300 ml through the 154 µm filter and even the 200 µm filter.

Table 8. Dispersion of cement grouts based on UF12 mixed with DISPERMANT CV-3 dissolver equipped with 90-mm disk

Mixture	Additive	Dispersion		Grout volume passed through filters					
		Mixing 4 min	Agitation 10 min	104 µm	122 µm	132 µm	154 µm	104 µm	200 µm
No.	[%]	[rpm]	[rpm]	[ml]	[ml]	[ml]	[ml]	[ml]	[ml]
1_a	None	6000	700					80	150
1_b								100	175
2_a	None	6000	700					60	125
2_b								75	105
3	0.5	6000	700		20	20	40	26	
4	0.5	6000	700			40	50	70	110
5	0.5	6000	700			30	40	50	75

3.2.2. Dispersion with DISPERMANT CV-3 dissolver equipped with R/S system

The results for this method are presented in table 9. As it can be seen from the table, the dispersion results were much better than the disk. In tests without an additive, the cement grout passed the 104 µm filter with a full volume of 300 ml in three out of three tests, and even considerable amount passed the 91 µm filter in all three tests.

Tests with additive showed that the dispersion was not improved by the additive, on the contrary the dispersion became somewhat worse. The grout passed the 104 µm filter with a full volume of 300 ml in three out of six tests, and even failed to pass the 122 µm with a full volume of 300 ml in two out of six tests. Note that samples from mixture no.1 (without additive) and mixture no. 7 (with additive) were used to perform rheology measurements.

Table 9. Dispersion of cement grouts based on UF12 mixed with DISPERMANT CV-3 dissolver equipped with R/S system

Mixture	Additive	Dispersion		Grout volume passed through filters					
		Mixing 4 min	Agitation 10 min	77 µm	91 µm	104 µm	122 µm	132 µm	154 µm
No.	[%]	[rpm]	[rpm]	[ml]	[ml]	[ml]	[ml]	[ml]	[ml]
1	None	10000	4000		260	300	300	300	
2	None	10000	4000	100	260	300	300		
3	None	10000	4000		260	300	300	300	
4	0.5	10000	3000			75	240	290	300
5	0.5	10000	3000			225	300	300	300
6	0.5	10000	3000			300	300	300	300
7	0.5	10000	3000	80	250	300	300		
8	0.5	10000	3000	100	160	210	260		
9	0.5	10000	3000	175	300	300	300		

3.2.3. Dispersion achieved with the ultrasound device UP400St

In case of UF12, only one sample vessel was used (865 ml) in contrary to tests with INJ30 where three different sample volumes were used (435, 860 and 2000 ml). The results for dispersion with ultrasound are presented in table 10 for case with an additive and table 11 on case without an additive. As it can be seen from the table, the total 19 tests were performed and the focus this time was to achieve the best dispersion results at minimum specific energy.

In tests with an additive, the best dispersion result was grout passing the 54 µm filter with a full volume of 300 ml in 3 out of 5 tests.The amplitude was 60 µm and 2 minutes of sonication. Different combinations were tested to find the optimum settings, same amplitude but longer sonication time, same sonication time but higher amplitude, higher amplitude and longer sonication time. In all previous mentioned combinations, the cement grout passed through the 54 µm filter with a full volume of 300 ml as it was achieved with 60 µm amplitude and 2 minutes sonication. But the grout failed to pass through the 43 µm filter. In all previous combinations the specific energy was much higher than the one with 60 µm amplitude and 2 min of sonication. Lower amplitude was also tested but it gave worse dispersion results, where the cement grout failed to pass through the 54 µm filter but managed to pass through the 62 µm filter with a full volume of 300 ml.

In tests without an additive, the cement grout passed through the 62 µm filter with a full volume of 300 ml in 2 out of 3 tests. So, in case of UF12 the dispersion results without using an additive were poorer compared to the dispersion results with an additive where the cement grout passed through the 54 µm filter.

Table 10. Dispersion of cement grouts based on UF12 with additive 0.5% using ultrasound device UP400St, sample volume 865 ml

Mix.	Dispersion						Grout volume passed through filters					
	Depth of sonotrode	Amp.	Time	Total energy	Specific energy	Grout temp.	43 µm	54 µm	62 µm	77 µm	91 µm	122 µm
No.	[mm]	[µm]	[min]	[Wh]	[Ws/ml]	[°C]	[ml]	[ml]	[ml]	[ml]	[ml]	[ml]
1	45	60	2	26.72	26.72	23					300	300
2	45	60	2	25.90	25.90	23			300		300	
3	45	60	2	24.11	24.11	23		25	200			
4	45	60	2	26.09	26.09	23		210	300			
5	45	60	2	25.57	25.57	23		300	300			
6	45	60	2	25.58	25.58	24		300	300			
7	45	60	2	25.52	25.52	25	120	300				
8	45	60	4	51.80	51.80	31	125	300				
9	45	80	2	34.69	31.69	26	120	300				
10	45	80	4	66.19	66.19	35	130	300				
11	45	95	5	91.04	91.04	41	130	300				
12	45	40	2	16.87	16.87	22		150	300			
13	45	40	2	18.74	18.74	23		180	300			

Table 11. Dispersion of cement grouts based on UF12 without additive using ultrasound device UP400St, sample volume 865 ml and 2 min sonication

Mix.	Dispersion					Grout volume passed through filters							
	Depth of sonotrode	Amp.	Total energy	Specific energy	Grout temp.	43 µm	54 µm	62 µm	77 µm	91 µm	122 µm	132 µm	154 µm
No.	[mm]	[µm]	[Wh]	[Ws/ml]	[°C]	[ml]	[ml]	[ml]	[ml]	[ml]	[ml]	[ml]	[ml]
1	45	60	6.15	25.61	22	≈0	≈0						
2	45	60	6.19	25.78	21		≈0	≈0					
3	45	60	5.99	24.95	25							300	300
4	45	60	5.88	24.49	24				300	300			
5	45	60	6.28	26.13	23		230	300					
6	45	60	5.82	24.22	25		250	300					

3.2.4. Rheology measurements

Two rheology tests on this grout were performed using the Brookfield DV-II+ Programmable Viscometer that follow the standard DIN-53019. The samples were taken from the batches mixed using DISPERMANT CV-3 dissolver equipped with R/S system (mixture no.1 without additive and mixture no.7 with additive). The results from rheology tests are presented in figure 16.

The dispersion tests showed that additives have a relative low influence on dispersion, but results obtained from rheology tests showed that additives have a significant impact on rheology of the grout. As it can be seen from the figure 16, the yield stress dropped off from 5.44 Pa in case of grout without additive to 2.47 pa for the grout with additive, and plastic viscosity dropped off from 17.51 mPas in case of grout without additive to 6.55 mPas for the grout with additive. Yield stress and plastic viscosity were estimated using Bingham model.

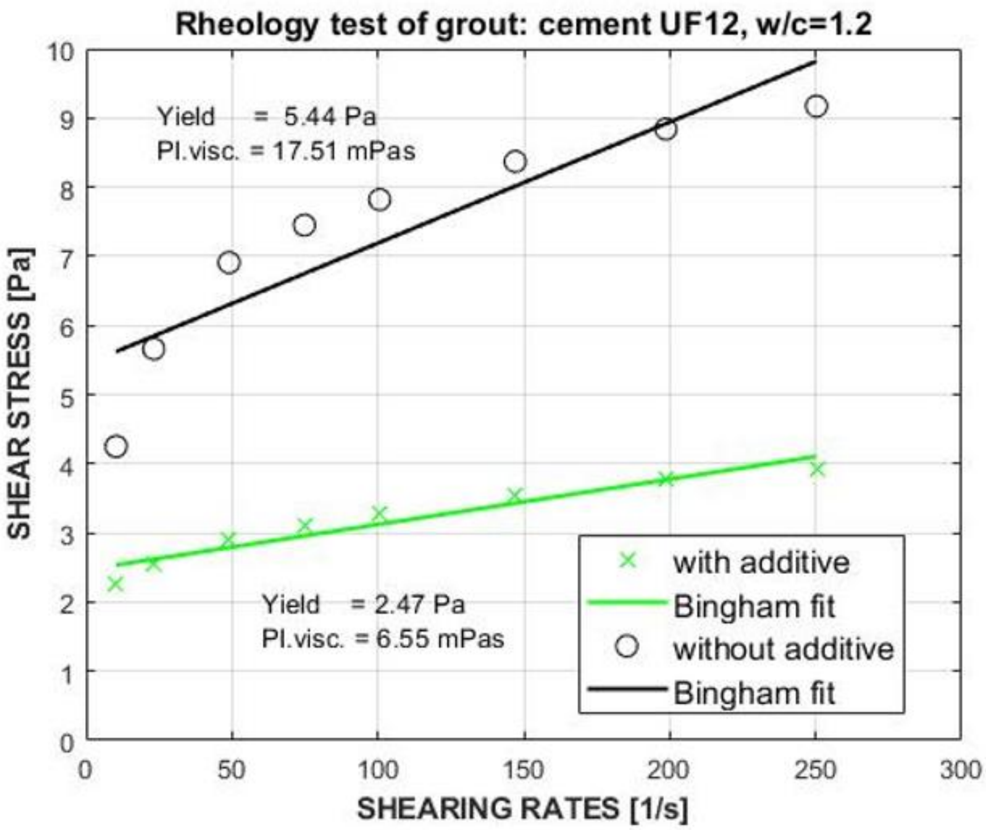

Figure 16. Rheology measurements with Brookfield DV-II+ Programmable Viscometer (DIN-87 spindle: cup r=6.41 mm, spindle r=5.91 mm) for two samples, one with additive and another without additive

3.3. Summary of the results

To facilitate the comparison between the different dispersion methods, summarized results of best dispersion achieved with the respective method for both INJ30 and UF12 grouts are presented in table 12. The results from both INJ30 and UF12 grouts clearly show that the mixing method has a significant influence on the penetration ability of the grout, the conventional laboratory dissolver equipped with R/S system much efficient than the 90-mm disk, but ultrasound gave the best dispersion results. Additives had a limited effect on dispersion.

Table 12. Summary of the results achieved with respective method for both INJ30 and UF12

Dispersion method		Additive	Best dispersion achieved
INJ30			
DISPERMANT CV-3 with 90-mm disk	Mixing for 4 min at 2000/6000 rpm Agitation for 10 min at 700 rpm	Without additive	Could not even pass the 154 µm filter
		With 0.4 % iFlow	Could not even pass the 154 µm filter
DISPERMANT CV-3 with R/S system	Mixing for 4 min at 10000 rpm Agitation for 10 min at 3000/4000 rpm	Without additive	The 91 µm filter with good repeatability but even the 77 µm filter
		With 0.4 % iFlow	The 91 µm filter with good repeatability but even the 77 µm filter
UP400St with H22 sonotrode	860 ml sample 60 µm amplitude 2 min sonication 45 mm sonotrode depth	Without additive	No tests were performed
		With 0.4 % iFlow	The 77 µm filter
UF12			
DISPERMANT CV-3 with 90-mm disk	Mixing for 4 min at 2000/6000 rpm Agitation for 10 min at 700 rpm	Without additive	Could not even pass the 200 µm filter
		With 0.5 % iFlow	Could not even pass the 200 µm filter
DISPERMANT CV-3 with R/S system	Mixing for 4 min at 10000 rpm Agitation for 10 min at 3000/4000 rpm	Without additive	The 104 µm filter
		With 0.5 % iFlow	The 104 µm filter
UP400St with H22 sonotrode	865 ml sample 60 µm amplitude 2 min sonication 45 mm sonotrode depth	Without additive	The 62 µm filter
		With 0.5 % iFlow	The 54 µm filter

4. Discussion

As it can be seen from table 12, the DISPERMANT CV-3 equipped with 90-mm disk yielded the worst results between the three tested methods. Better results were achieved using the DISPERMANT CV-3 equipped with R/S system and the best results were obtained by ultrasound device UP400St.

This study showed that the DISPERMANT CV-3 equipped with 90-mm disk was not efficient at all, where grout based on INJ30 failed to pass through the 154 µm filter (>5 x d_{95}) and grout based on UF12 failed to pass through the 200 µm filter (>16 x d_{95}). These results could be of great importance due to fact that the dispersion mechanism and flow pattern in this method is similar to colloidal mixer used in the field where the dispersion is achieved by shear forces between the mixture layers due to their flow at different velocity and by shearing in contact with the surface of the dissolver disk.

Dispersion using the R/S system was much better than the disk, where grout based on INJ30 passed through the 77 µm filter (>2.5 x d_{95}) and grout based on UF12 passed through the 104 µm filter (>8 x d_{95}). This better dispersion is probably a result of better shearing process where the dispersion process takes place through mechanical tearing, shear fluid forces and cavitation. For INJ30 the results were slightly under 3 x d_{95} very close to the empirical relation that Hansson (1995) found which states that a good dispersion is achieved when the opening is 3 times larger than the maximum grain size. This means that probably there is no marginal for further improvement when it comes to INJ30 because 3 large particles in the suspension are enough to initiate arch building thus plug formation. When it comes to UF12, the R/S system yielded better results than the disk but still far from 3 x d_{95} which means that there is a considerable potential for further improvement.

It is important to highlight that this study did not include different setup for dispersion with DISPERMANT CV-3 equipped with disk and R/S system. The setup was based on recommendation from the supplier, literature study and instructions from Dr Draganović based on his own experience. The nonexistence of sediment at the bottom of mixing bucket was considered as an indication of a proper mixing setup.

The dispersion results using ultrasound were absolutely the best between the three methods, where grout based on INJ30 passed through the 77 µm filter (>2.5 x d_{95}) and grout based on UF12 passed through the 54 µm filter(>4.5 x d_{95}) in tests with an additive. For INJ30 the results were similar to results obtained by DISPERMANT CV-3 equipped with R/S system due to the same explanation mentioned above but one could notice that dispersion with ultrasound was more stable and repeatable to some extent.

The most efficient dispersion was achieved in 860 ml samples using amplitude of 60 µm for 2 min sonication time, but regardless the required energy similar results were also possible to achieve using both lower and higher amplitudes which means that dispersion with ultrasound is not so sensitive to amplitude. For UF12 the results were significantly better not only compared to dispersion with DISPERMANT CV-3 equipped with R/S system but even compared to previous studies. The results were better by approximately 100% (from 104 µm filter down to 54 µm filter), which makes the results (>4.5 x d_{95}) close to Hansson's empirical relation (3 x d_{95}) but still a small marginal for further improvement does exist. Results obtained from this study confirm with no doubt the ability of ultrasound to disperse cement particles.

This is the first time that ultrasound was used to disperse grouts based on microfine cement. The setup for dispersion with ultrasound was based on recommendations from the manufacturer, few available references and mostly our own experience gained from performing large number of pilot tests (tests with water and tests with small amount of cement) prior the actual tests. In general, the method was simple to use and easy to monitor and control the process. The setup is highly dependent on many factors such as the sample volume, sonotrode depth and geometry of sample vessel. So, there is no single setup (amplitude and sonication time) that can be applied on different sample volume and yield the same result. This was confirmed in the tests with ultrasound on grout based on INJ30 where the required amplitude to disperse the 2 l sample was much higher than the required amplitude to disperse the 860 ml and 435 ml samples.

This study showed that dispersion with ultrasound is highly affected by the geometry between the sonotrode and sample vessel especially the energy demand. Tests with ultrasound showed that the larger the distance between the sonotrode and the sample vessel bottom, the higher energy is required. This is an issue for further research.

It was noticeable that increased amplitude and/or sonication time was accompanied with higher specific energy and grout temperature. Higher specific energy is directly related to the cost. In this study the effect on cost was neglectable due to that the tested sample was relative small and the increase in sonication time was about couple of minutes, but in the filed the effect on cost could be higher because of large batches used. Higher grout temperature could contribute to a lower dispersion because of its influence on hydration. A higher grout temperature increases the hydration which means stronger bonding between cement particles. This type of effect was not observed during this study, but it is possible to occur in the field. So, a high specific energy or/and a high grout temperature could be an issue for using this method in the field.

This study also showed the limited effect of additives on dispersion. The additive (iFlow in this study) did not improved the dispersion in neither the disk nor R/S system but had a relative positive effect on dispersion in tests with ultrasound. However, rheology measurements showed that additives had a significant impact on the rheology properties of the grout both yield stress and plastic viscosity. Good rheological properties are directly associated with longer penetration distance for the grout, which is considered to be an important factor to achieve a high sealing efficiency. So, despite the low impact of additives on dispersion, additives are important and have a positive effect on the final result of the whole sealing process.

It is important to distinguish between the penetrability of the grout and measured dispersion with filter pump. They are not the same thing but closely related to each other. The better dispersion the better penetrability.

5. Suggestions for future studies

This study is the first time that ultrasound technology was used to disperse grouts based on microfine cement. The study showed that ultrasound has the potential to be used as a dispersion method, and gave the positive result required to carry on to the next steps.

In this study two different cement were tested, INJ30 and UF12, but both have the same chemical composition since they are produced form the same cement. The only difference between the two types is the degree of milling. So, to assess the robustness of this method it is important to test it with different cement types that varies in chemical composition and even different types of additives should be tested.

The overall aim for testing ultrasound dispersion is to develop a grout that can seal the very narrow fractures (20-30) µm, compared to (70-80) µm at the present. Therefore it is important to test the efficiency of ultrasound to disperse even finer milled cement for example d_{95} =6 µm.

Having good results in the lab does not mean necessary that this method suitable for field use. Many questions need to be answered and investigated. The device used in this study had a limited capacity because it is designed to be used for laboratory purposes but in the field much larger volumes are needed up to 100 l per batch. There are powerful ultrasound devices available at the market that have much larger capacity. The multiple ultrasound devices can be installed also as cluster, but to assess the suitability of these devices for field use, deeper study is needed.

The questions that need to be considered:

- Time aspect, it is important to investigate how the productivity is affected?
- How much energy is needed for dispersion? Energy consumption is an important factor in the field.
- For how long the dispersion effect will last? In this study the measurements were performed directly after the sonication but in the field, time is needed to pump the grout through the fractures.
- How does the high temperature affect the grout flow and rheology properties?
- How simple and convenient it is to use in the field? For example, cleaning of the equipment is important.

6. Conclusion

This study clearly showed that the conventional lab dissolver equipped with 90-mm disk is not suitable for dispersion of grouts based on microfine cement. This method could not disperse neither grout based on INJ30 with d_{95} of 30 µm nor grout based on UF12 with d_{95} of 12 µm. The dispersion mechanism and flow pattern in this method is very similar to the colloidal mixer used in the field which makes the efficiency of this type of mixers questionable. There is a need to investigate this issue.

The conventional lab dissolver equipped with R/S system is much better method for dispersion compared to 90-mm disk system. Using the R/S system, grout based on INJ30 passed through the 77 µm filter (approximately 2.5 x d_{95}). This is probably the best result that could be achieved using INJ30. Using the same system, grout based on UF12 passed through 104 µm (approximately 8 x d_{95}) which is far from the optimum results. So, the results from this method confirmed the fact that the finer the cement the harder to disperse. This method could be used to disperse grout based on fine cement until approximately 30 µm, but for grouts based on very fine cement, the efficiency of this method is lower.

Dispersion with the ultrasound device UP400St yielded the best result between these three tested methods. With ultrasound, grout based on INJ30 passed through the 77 µm filter which is principally the same results achieved with the R/S system. As it is mentioned before, probably this is the limit for INJ30, but ultrasound showed better repeatability and lower variation than the R/S system. Using the same method, grout based on UF12 passed through the 54 µm filter (4.5 x d_{95}), which is considerably better than the achieved results with other two methods or presented in literature. This means that fractures down to 55 µm can be sealed compared to (70-80 µm) today. Still there is a marginal for more improvements especially if ultrasound is combined with further milling of cement particles.

This study clearly showed that ultrasound has the potential to be used as a dispersion method especially for grouts based on microfine cement with $d_{95} < 30$ µm. Ultrasound dispersion can be considered as a step forward in development of grout that can seal the very narrow fractures dawn to 30 to 40 µm.

Additives are important to achieve better penetration length even though their direct effect on dispersion is limited. Both good dispersion and flow properties (rheology) are essential to achieve a good sealing.

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