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Energy efficient fibre composites recycling

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Abstract

In this project, an investigation will be performed about how to improve thermal properties of recycled composite material. First, a literature study was performed about the potential techniques to improve the heating efficiency of the composites. Heating techniques, fibres and possible fillers were investigated. Secondly, an experimental method was set with the material available. In the laboratory a precedent work was performed on the thermal conductivity of polyamide 12 reinforced with glass fibres. The conductivity of polyamide 12 reinforced with carbon fibres is measured using the same experimental method to compare the thermal conductivity. In theory, carbon fibres have a better thermal conductivity than glass fibres, this was confirmed by the experiments performed. During the recycling of thermoplastic fibre composites the scrap will be grinded. Therefore, the thermal conductivity of small pieces of carbon fibre composites was measured, the thermal conductivity is reduced due to the increase of air fraction and the shortening of the fibres. The thermal conductivity of small pieces of glass fibre composite was investigated in the previous work, by mixing the grinded pieces of carbon fibre composite and the small pieces of the glass fibre composite the effect on the thermal conductivity was investigated. It was noticed that using smaller pieces of grinded material allows to reduce the air fraction between the bigger pieces and increase the thermal conductivity.

Sammanfattning

I detta projekt kommer en undersökning att göras om hur man effektivt kan återvinna fiberförstärkta termoplaster. Först genomfördes en litteraturstudie om de potentiella teknikerna för att förbättra kompositernas uppvärmningseffektivitet. Uppvärmningstekniker, fibrer och möjliga fyllmedel undersöktes. Därefter genomfördes experiment med tillgängligt material. I laboratoriet hade tidigare ett arbete genomförts för att mäta värmeledningsförmågan hos polyamid 12 förstärkt med glasfibrer. Ledningsförmågan hos polyamid 12 förstärkt med kolfibrer kommer här att mätas med samma experimentella metod för att jämföra värmeledningsförmågan. I teorin har kolfibrer bättre värmeledningsförmåga än glasfibrer, detta bekräftades av de utförda experimenten. Under återvinning av kompositer av termoplastfibrer males restmaterialet ner. Därför mättes värmeledningsförmågan hos små bitar av kolfiberkompositer, värmeledningsförmågan minskades på grund av ökningen av luftfraktion och förkortningen av fibrerna. Värmeledningsförmågan hos små bitar av glasfiberkomposit undersöktes i det föregående arbetet, genom att blanda de slipade bitarna av kolfiberkomposit och de små bitarna av glasfiberkomposit undersöktes effekten på värmekonduktiviteten. Det noterades att användning av mindre bitar av slipat material gör det möjligt att minska luftfraktionen mellan de större bitarna och öka värmeledningsförmågan.

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Contents

1	Introduction	1
1.1	Issues	1
1.2	Aim	2
1.3	Social and ethical considerations	2
2	Fibre composite	3
2.1	Recycling of fibre composites	3
2.2	Fibre composites and their thermal properties	5
2.3	Heating techniques	8
2.4	Welding efficiency	10
3	Experimental method	13
3.1	Material	14
3.2	Equipment	15
3.3	COMSOL model	15
3.4	Measuring	17
4	Results	20
4.1	Thermal conductivity of the commingled CF/PA12 composite	20
4.2	Thermal conductivity of the consolidated CF/PA12	20
4.3	Thermal conductivity of chips of consolidated CF/PA12	21
4.4	Thermal conductivity of GF/PA12 chips with commingled fibres of CF/PA12	22
4.5	Thermal conductivity of GF/PA12 chips with grinded consolidated CF/PA12	22
5	Analyse and discussion	24
5.1	Comparison between the theoretical and experimental thermal conductivity	24
5.2	Comparison of the thermal conductivity of the consolidated and the commingled CF/PA12	25
5.3	Thermal conductivity of the chips and the grinded CF/PA12	25
5.4	Thermal conductivity of GF/PA12 chips with added CF/PA12	26

5.5	Potential errors	27
6	Conclusions	28
6.1	Future work	28
7	Acknowledgements	30
8	References	31

1 Introduction

1.1 Issues

Composite materials such as fibres reinforced polymers are very high-performance materials with high mechanical properties and a low weight. They are used in domains where high specific properties (properties/weight) are needed. 30% of composite materials are used for automotive applications and 20% in the aerospace field. Nowadays, composites are more and more used for the sport, leisure time, aeronautics applications and for wind turbines [1]. They are especially used in the sector of transports as their low weight allow less consumption of fuel and less emission of CO₂ during the period of use. It has been reported that the annual emissions of greenhouse gases due to the transport sector represent 14% and 24% of the CO₂ emission [2]. By reducing the weight of the vehicles, the direct emissions can be reduced, but if a whole life cycle analysis is performed it can be noticed that fibre reinforced composites are less favourable than aluminium in the automotive industry especially because of the poor recycling efficiency of composites [3]. Therefore, it is important to treat this subject and find new techniques with maybe new types of materials more efficient for the recycling. Improving the already existing ones would also be a solution. Moreover, new legislations are created about the recycling and the end of life of materials. One of those law is for example the EU-directive for end-of-life vehicles which set objectives about reuse and recycling of the materials used in the vehicles, this enters in a circular economy logic used by more and more country in the world [4]. Recycling is a great challenge because there are many steps that are playing a great roll in the recycling chain. First, scrap must be available and then collected, after this the product must be recycled and reprocessed and at least there has to be a market available for the recycled materials [1].

There are a lot of recycling techniques in the field of composites materials due to the large panel of composites and their varied applications. Those techniques have different degrees of efficiency depending on the composite type. In any case it is difficult to recycle composites because of their heterogeneous nature with the matrix and the fibres [1].

1.2 Aim

The aim of the project is to search for a technique that will lead to an efficient recycling of fibre composite thermoplastics by reducing the energy needed. First of all, a literature review was performed in order to set an experimental method. The type of matrix and fibres used was investigated to understand the thermal behaviour of composites. Research was performed on the heating techniques to find an efficient way to melt the thermoplastic matrix depending on the different materials that could be used. By reading articles about the welding efficiency some potential fillers to increase the melting ability were identified. After the literature study, an experiment was set up with the material available. A previous work was performed in the lab about the thermal conductivity of a glass fibres and polyamide 12 composite (GF/PA12). It was concluded that the thermal conductivity was small, and the composite was transparent to microwave heating [5]. The aim of the project is to study if the use of carbon fibres instead of glass fibre have an influence on the thermal conductivity of the composite with a polyamide 12 matrix. Later the thermal conductivity of a mixing from small pieces of GF/PA12 and CF/PA12 are going to be tested.

1.3 Social and ethical considerations

Recycling has a big ethical and social impact, both in a good or a bad way. As mentioned before recycling plays a central role in a circular economy logic. In order to keep a good social and ethical aspect while recycling, some aspects need to be taken into account. First of all, recycling materials demand energy and not every materials can be recycled or they may lose some quality [6]. Therefore, recycling should not be an excuse to produce more waste. Secondly, the wastes form the developed countries are sometimes recycled in the undeveloped ones [6]. In those countries the social and safety conditions of workers are not always respected and this may lead to a huge social and ethical impact. In conclusion recycling can be a good solution but it has to be done in the right way, it should not be an excuse to produce more wastes and it should be recycled as much as possible in the region from which the scrap materials are coming.

2 Fibre composite

2.1 Recycling of fibre composites

There is a large panel of recycling techniques for composite materials due to the diversity of materials in this class. This review is going to be based on fibres composite with a polymeric matrix only. First of all the recycling techniques depend mainly on the type of the polymeric matrix. It can either be a thermoplastic or a thermoset matrix. The main recycling methods can be seen in Fig. 2.1.

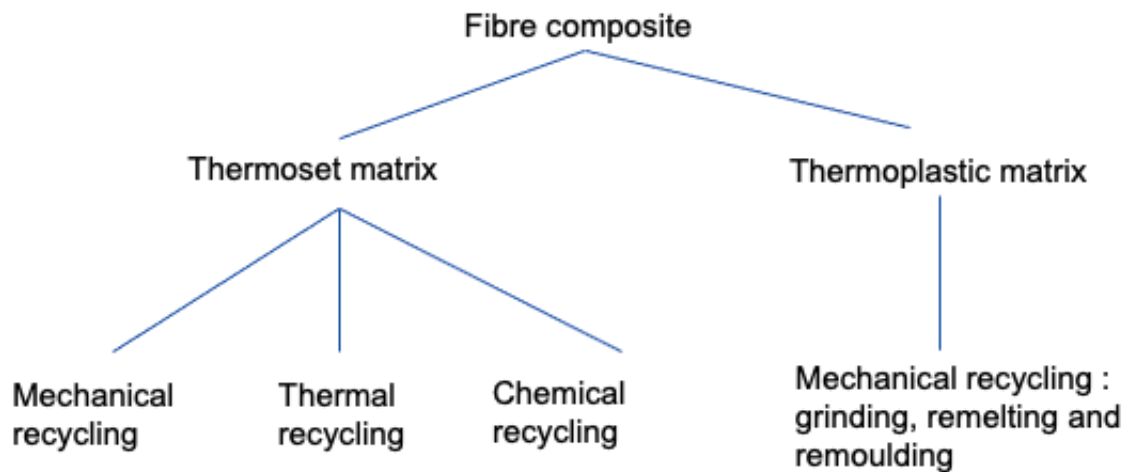


Figure 2.1: Recycling techniques for fibre reinforced polymers

2.1.1 Recycling of thermoset composites

There are three main recycling methods of thermoset fibre composites (see Fig. 2.1): the mechanical, the thermal and the chemical processes [7]. The use of a thermoset matrix reduces the ability to recycle composites because the polymer cannot be remelted and remoulded. Techniques to separate fibres from the matrix have been thought up.

First of all, the mechanical recycling of a fibre reinforced thermoset consists of grinding the scrap into small pieces, the aim of this step is to cut the material in order to separate the fibres from the matrix. After the cutting step it can be separated into a fibre-rich part or a matrix-rich part [1]. The recycled fibres could be used to replace virgin fibres, but the mechanical properties of the recycled

fibres are lower [8]. Furthermore, the grinding of the composite requires a lot of energy.

The chemical recycling of thermoset fibres composites can be performed using two techniques. The first one is the chemical depolymerisation of the matrix. Using this recycling process, the fibres and the monomers previously used are regenerated [1]. The dissolution process is called the solvysis process and different types of solvent can be used. One limitation of this chemical process is that the chemical products used depend on the composition of the matrix. Therefore, on a large scale it can be challenging if all the different composite scraps are mixed together [1].

Finally, the purpose of the thermal recycling process is to degrade the organic matrix of the composite by applying heat. The product obtained from the degraded matrix can be used as energy [7]. Unfortunately, even though with this process the entirety of the fibres are collected, a diminution of strength due to the high temperature heating can be observed [1].

2.1.2 Recycling of thermoplastic fibre composites

One of the main advantages of the thermoplastic matrix is the ability to be remelted. Therefore, it will be easier to reshape and remould the composite. The recycling technique used for the thermoplastic composites is a mechanical recycling method, the composite is grinded, remelted and then reshaped by injection moulding, compression moulding or extrusion [7]. By using a thermoplastic matrix, the entire composite can be recycled without separating both constituents. Therefore, the losses are going to be reduced. Grinding of the fibres will shorten the fibres and a loss of strength can occurs [9]. If this recycling technique can be very interesting to minimise the losses of scrap materials the recycling can be improved by reducing the energy and the cycle time needed. The use of more efficient heating techniques and conductive materials can reduce the cycle time, the cost, and the energy needed to reprocess the material. Therefore, during this project it was chosen to study the new promising recycling techniques for fibre composites with a thermoplastic matrix.

2.2 Fibre composites and their thermal properties

During this project fibre composites made from glass or carbon fibres with a polyamide 12 matrix are going to be studied. For the aim of the project a composite with a good thermal conductivity is being investigated.

2.2.1 Glass Fibre

Glass fibre are a type of fibre commonly used fibres in composites. Their advantages and disadvantages are listed in table 2.1.

Table 2.1: Advantages and disadvantages of glass fibres [10, 11]

Advantages	Disadvantages
High strength	Low stiffness
Tolerance to high temperatures	Moisture sensitive
Resistant to corrosive environment	Abrasive
Low price	Low thermal conductivity
	Non electrical conductive
	Transparent to electromagnetic signals

The important properties to heat up efficiently the material are the thermal conductivity for the classical oven, the electrical conductivity for the induction heating and for microwave heating the material must absorb microwaves, these techniques are going to be described in section 2.3. The thermal conductivity of the E-glass fibre in the fibre direction is 10-13 W/mK [12]. Table 2.1 gives an insight of the poor ability of glass fibres to be heated by the techniques listed above.

2.2.2 Carbon Fibre

Carbon fibres are a material commonly used in fibre composites, their advantages and disadvantages are listed in the Table 2.2.

Table 2.2 gives an insight of the ability of carbon fibres to participate efficiently to the heating of the composite, they have a good thermal conductivity, a good electrical conductivity and they can absorb microwaves. Furthermore, due to their high price it may be interesting to recycle them. The thermal conductivity of carbon fibres varies depending on the type of fibres. The PAN-based carbon

Table 2.2: Advantages and disadvantages of carbon fibres

Advantages	Disadvantages
High strength	High price
High modulus	Birttleness
High temperature tolerance	
High corrosion resistance	
Electrical conductivity	
Thermal conductivity	
Microwave absorption	

fibres, the most used type, have a thermal conductivity of 7-70 W/mK in the fibres direction [12]. The PITCH-based carbon fibres have a higher thermal conductivity, 530-1100 W/mK [13].

2.2.3 Thermoplastic matrix

In the past, the most used matrix for fibre reinforced polymers were the thermoset matrix. Fibre reinforced thermosets have many advantages compared to the thermoplastic composites. Among them the price, the thermal resistance and the smaller melt viscosity which make them cheaper and easier to produce [10]. Nowadays the recycling of materials is more and more demanded, therefor the composites with a thermoplastic matrix which can be remelted and reshaped are becoming more interesting. Moreover, there is other advantages of using thermoplastic matrix, the process time is reduced, the toughness is increased and there can be less hazard for the health during the production [10]. Most of the usual thermoplastics do not have a good thermal and electrical conductivity [13]. Furthermore, thermoplastics are usually transparent to microwave heating [14]. The thermoplastic matrix used in the project is the polyamide 12 (PA 12), the chemical structure is represented in Fig. 2.2. The thermal conductivity of PA 12 is 0.21-0.31 W/mK [12]. PA 12 belongs to the aliphatic polyamide class, which are engineered thermoplastics. These types of plastics have high thermal properties and mechanical properties [15]. PA 12 has a good strength, hardness, and stiffness. These efficient properties are due to the ability of the amide function to create strong hydrogen bonds.

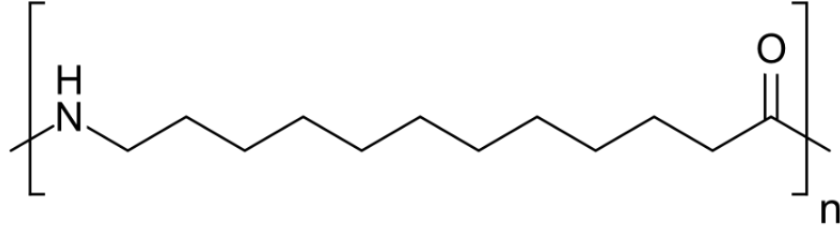


Figure 2.2: Chemical structure of PA12 [16]

2.2.4 Thermal properties

The thermal properties of composite can be predicted using equation 1 to 4 [12]. The density of the composite can be calculated using Eq. 1, ν_m and ν_f are the volume fraction of respectively the matrix and the fibres, ρ_m and ρ_f are their respective density. The specific heat capacity can be calculated with Eq. 2 using the specific heat capacity of the matrix C_{pm} and of the fibres C_{pf} . The thermal conductivity in the longitudinal direction can be calculated using Eq. 3 and the thermal conductivity in the transverse direction can be calculated using Eq. 4, k_m is the thermal conductivity of the matrix, k_f is the thermal conductivity of the fibres. In order for Eq. 2, 3 and 4 to be valid some assumptions have to be made, the matrix have to be isotropic and the fibres have to be transversely isotropic [12].

$$\rho_c = \nu_f \rho_f + \nu_m \rho_m \quad (1)$$

$$C_p = \frac{\nu_f \rho_f C_{pf} + \nu_m \rho_m C_{pm}}{\nu_f \rho_f + \nu_m \rho_m} \quad (2)$$

$$k_l = \nu_f k_f + \nu_m k_m \quad (3)$$

$$k_t = \frac{k_f k_m}{\nu_m k_f + \nu_f k_m} \quad (4)$$

2.3 Heating techniques

In order to economise energy and increase the efficiency of recycling a heating technique that would need less time and energy to melt the thermoplastic matrix is needed. Several techniques were identified; the conductive/convection heating (consist of a classical oven commonly used), the infrared heating, the induction heating, and the microwave heating. All those techniques work differently and will be more or less efficient depending on which materials are inside the composite.

2.3.1 Classical oven

Classical oven with resistances that heat up the air and lead to a convection heating of the surface of the material are not very efficient as an heating technique [17]. The heating process is very slow and demands a lot of energy. In addition, polymers do not have a good thermal conductivity which is approximately $0.1-0.5 \text{ Wm}^{-1}\text{K}^{-1}$, the higher is the thermal conductivity the faster the heating process will be [13]. Therefore, other types of heating techniques, more recent, can be investigated in order to decrease the time and the energy demand while recycling thermoplastic composites.

2.3.2 Infrared

The infrared heating does not need any medium to transfer the energy, it is a transported by radiation. This technique can be a good alternative the heat up efficiently the composite compared to the convection/conductive heating. Indeed, the polymers have a low thermal conductivity. Using infrared heating the heat is directly transferred to the composite and up to 50% of energy can be saved [17]. Furthermore, the radiation can be focused on a certain point which lead to a local heating, it also has a high-power density [18]. The infrared can either be short or medium wave emitters. Some experiences found during the literature study have been carried on the influence of the wavelength used, the fibres and the matrix for an infrared heating [17]. A sample of fibre reinforced composite was heated up from the top side and a temperature measurement was performed on the top and bottom surface, no temperature change has been noticed between

the two top surfaces but a higher temperature gradient between the two surfaces has been noticed while using the medium wave emitter [17]. This can indicate that the medium waves heat the surface, and the rest of the material is heated by convection while the short wave will penetrate the surface and the sample will be volume heated. The heating of a composite with carbon fibres is faster than with glass fibres but it will lead to a higher temperature gradient, the heating rate also depend on the matrix chosen [17].

2.3.3 Microwave

The purpose of microwave heating is the interaction of the microwaves with the material itself directly as shown in Fig 2.3 [19].

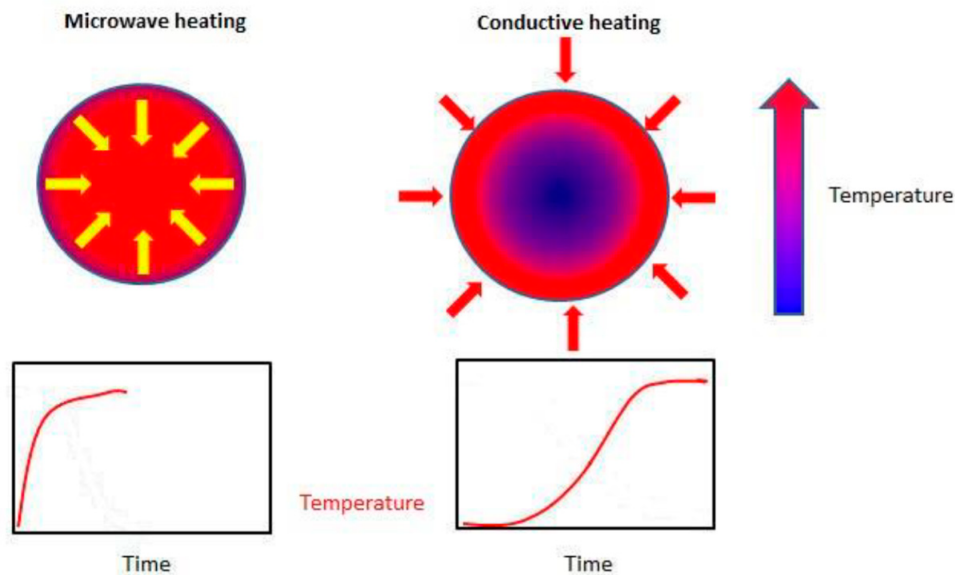


Figure 2.3: Microwave heating compared with the conductive heating [19]

The material can either reflect, transmit and/or absorb the microwaves [20]. If the material reflects the waves, it will not be heated, if it is transparent the waves will pass through the material without heating it, and if it is absorbent the waves are going to be absorbed and converted into heat. The ability of a material to absorb the microwaves and convert them into heat is related to the dielectric properties of the material. A relation between the dielectric loss factor ϵ'' , which represent the ability of the material to dissipate the energy absorbed, and the dielectric constant ϵ' , which represent the ability of the material to be polarised

by an electric field, is given in Eq. 5 [20]. The higher $\tan\delta$ is the higher the ability of the material to be heated will be. Glass fibres have a small loss tangent, they are transparent to microwave heating [21]. In a study performed by S. Joshi and S. Bhudoila about the cycle time of carbon fibre epoxy curing, has shown that the curing time was reduced by 2.5 times using the microwave curing instead of an autoclave curing and two times faster than an oven curing [22]. Therefore, microwave heating for composite with carbon fibres can be more efficient than classical heating techniques.

$$\tan\delta = \frac{\epsilon''}{\epsilon'} \quad (5)$$

2.3.4 Induction heating

When an electricity conductive material is exposed to an alternative magnetic field, an eddy current is induced inside the material [23]. The restive losses of the Eddy current will heat the material up. Therefore, the composite with conductive fibres such as carbon fibres can be heated up directly using this technique [24]. The fibres such as glass fibres and the thermoplastic matrix are not conductive materials, and they cannot be heated up this way. Moreover, it was noticed that there is an increase in temperature only when the fibres are forming closed loops, unidirectional composites cannot be heated up this way, fibre junctions are needed [23]. The heat could also be generated by dielectric losses, the matrix acts as a dielectric between the carbon fibres, a high-volume fraction of fibres will increase this phenomenon [24]. A study performed by P. Bengtsson has shown that it is possible to heat up woven carbon reinforced Nylon up to its process temperature using induction heating and it helps to considerably reduce the cycle time [25].

2.4 Welding efficiency

A lot of studies have been performed to improve the welding efficiency of fibre composites with a thermoplastic matrix. If the welding efficiency is improved this means that the thermoplastic matrix will melt faster. The studies done in this field will give insights on how the heating techniques or the material can be improved

to higher the melting efficiency.

2.4.1 Fillers to improve the thermal conductivity

In order to improve the thermal conductivity some fillers can be added to the composite. In Table 2.3 the potential materials with a high thermal conductivity are listed [13].

Table 2.3: Divers thermal conductivity for eventual fillers

Material	Thermal conductivity [W/mK][13]
Carbon nanotube	2000-6000
Pitch based carbon fibres	530-1100 (in the fibres direction)
Copper	483
Silver	450
Graphite	100-400 (in plane)
Boron nitride	250-300
Beryllium oxide	260
Aluminium	204
Aluminium nitride	200
Nickel	158
Silicon carbide	60-120
Carbon black	6-174
PAN-based Carbon fibre	8-70 (in the fibres direction)
Aluminium oxide	20-29

Some studies have shown that the thermal conductivity of composite containing carbon nanotube can be increased a lot. A study performed by Wang et al. have shown that adding 3 wt% of carbon nanotube to a glass fibre/polymer composite can increase the thermal conductivity by 150% [26].

2.4.2 Fillers to improve the microwave absorption

In order to improve the microwave welding of thermoplastics some materials that can be used as implants were found in the literature. Such materials are ceramic materials, water and polar liquids, metallic materials, ferrite, carbon and

conducting polymers [24]. Carbon nanotubes which have shown a great thermal conductivity can also absorb microwaves [27].

3 Experimental method

A precedent project done by R. Andolfi about the conductivity of glass fibres with a polyamide 12 matrix (GF/PA12) has shown that this composite has a poor thermal conductivity, which means that a lot of energy will be needed in order to recycle it [5]. Moreover, it was noticed that GF/PA12 composite is transparent to microwave heating techniques and could not be remelted this way [5]. After a literature study and a discussion about the instruments and materials available in the lab it was chosen to study the thermal conductivity of a composite with carbon fibres and a polyamide 12 matrix (CF/PA12) to investigate the influence of the fibres on the thermal conductivity of the composite. Using the results and the same program on COMSOL from the previous study, the thermal conductivity of the CF/PA12 composite is going to be measured. Later, different mixing between the two composites are going to be performed in order to investigate the influence on the thermal conductivity. Several experiments are going to be performed, here are the steps listed below.

1. Thermal conductivity of the commingled CF/PA12 composite
2. Thermal conductivity of the consolidated CF/PA12
3. Thermal conductivity of chips of consolidated CF/PA12
4. Thermal conductivity of GF/PA12 chips with commingled fibres of CF/PA12
5. Thermal conductivity of GF/PA12 chips with grinded consolidated CF/PA12

Each of these experiments are going to be described in the sections 3.4. The experimental set-up is represented in Fig.3.1. The composite will be heated by conduction with the bottom plate of hot press and the thermal conductivity will be evaluated. The specific requirements for each experiments are going to be described in the following sections. In order to analyse the measurement a software of finite element modelling COMSOL Multiphysique will be used [28]. The material needed is presented in more detail in section 3.1 and the equipment in section 3.2. The measuring procedures are going to be precise in each section from 3.4.1 to 3.4.5.

3.1 Material

A woven fabric of commingled composite containing 48% volume of carbon fibres and 52 % volume of polyamide 12 (CF/PA12) will be used. As mentioned in section 2.2.3 polyamide 12 is a thermoplastic polymer. The initial material used in this study is a commingled CF/PA12 composite. A commingled composite is a mixing of filament from carbon fibres and polyamide 12 producing a yarn and in this case woven into fabric [29]. This fabric can be consolidated by melting the thermoplastic matrix made out of PA12, this is called the reinforcement step and it will create a rigid sheet of consolidated CF/PA12 composite.

First of all, the commingled CF/PA12 composite will be used, later it will be consolidated. The thickness of the commingled composite is 0.92mm. After the consolidation the composite sheets will have a thickness of 0.34 mm, 0.51 mm and 0.74 mm for respectively 1, 2 and 3 layers of commingled composites. An already consolidated composite with 50% volume glass fibres and 50% volume polyamide (GF/PA12) will be used to cut into a plate of 20x20 cm, and different chips of 2cm, 1.5 cm and 1 cm will be used. This composite is 1 mm thick. The properties of carbon fibres and the PA12 are listed in Table 3.1.

Table 3.1: Specifications for carbon fibres and PA12

	Density [g/cm]	Specific heat capacity [J/gK]	Melting point [°C]
Carbon fibres [30]	1.75	0.73	-
Polyamide 12 [31]	1.34	2.1	180

Using Eq. 1 and 2 the properties of the CF/PA12 composite can be calculated. The properties of the two composites used are listed in Table 3.2

Table 3.2: Specifications for CF/PA12 and GF/PA12 composites

	Density [g/cm]	Specific heat capacity [J/gK]	Melting point [°C]	Fibres content [%]
CF/PA12	1.54	1.35	180	48
GF/PA12	1.85	1.016	180	50

3.2 Equipment

In order to measure the thermal conductivity of the different types of composites, an infrared camera is used to measure the temperature evolution on the surface of the composite. The thermal camera measure the temperature at a specific part of the composite and the measurement will be reported by hand every 1 seconds of 5 seconds depending on the speed of the thermal evolution, the camera can be seen in Fig. 3.1.

A hot press is used to consolidate the commingled composite and to heat the different samples during the measurement process. The hot press used have a top and a bottom plate that can be heated up. The bottom plate can be lifted in order to apply a pressure between the top plate and the bottom plate for the consolidation step for example. The hot press used can be seen in Fig. 3.1.

An oven is necessary to heat up the composites before the consolidation step up to $230^{\circ}C$.

A metal plate with adhesive cork will be used for the chopped materials, the adhesive cork delimits a square of $10 \times 10 \text{ cm}^2$ with a high of 2 mm .

Some metal weight will be needed in order to make sure the specimen measured are flat.

A ladder, some tape, plastic boxes and metal weight are needed to do the experimental set up shown in Fig. 3.1.

On Fig. 3.1 The experimental set up is represented. The infrared camera is fixed in a such way it can measure the temperature evolution of the composite sheet that will be putted on the bottom plate of the hot press.

3.3 COMSOL model

The model used in order to model the heat flux emitted by the hot press is represented in Fig. 3.2. This is the same model used in the project performed before [5]. The heat flux is assumed to come only from the bottom plate. The black lines are the thermal insulation conditions, this way the corners are not influencing the model, h is the thickness of the composite. The transfer coefficient

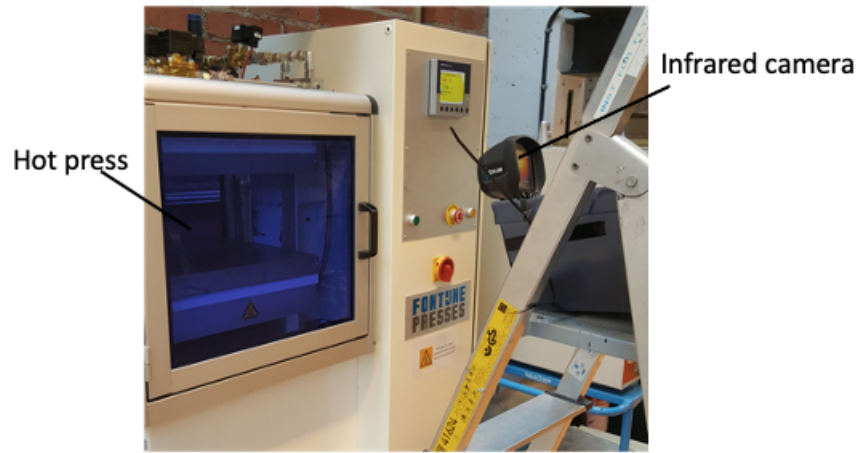


Figure 3.1: Experimental set up

between the composite and the hot plate has been chosen as $500 \text{ W/m}^2\text{K}$ due to the non-perfect contact between the bottom plate and the composite. The specific heat capacity and the density of the material are needed, in measuring 3.4.1 to 3.4.3 the values represented in Table 3.2 can be used. In measuring 3.4.4 and 3.4.5 the density and the specific heat capacity will be calculated using Eq. 1 and 2. With this model the same temperature time evolution as the one measured during the experiments is going to be created by varying the thermal conductivity in order to obtain the same behaviour. The thermal conductivity that will lead to the identical thermal behaviour will be the thermal conductivity of the composite.

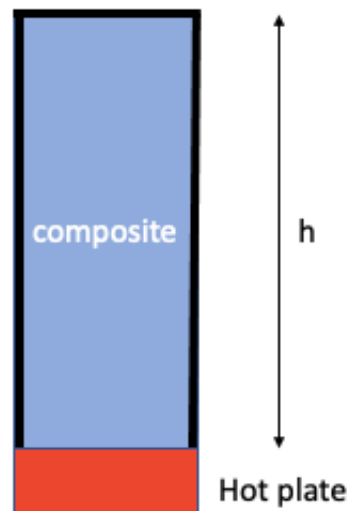


Figure 3.2: Model used in COMSOL. The blue part is the composite, the black lines are the isolation lines and the red plate is the hot plate.

3.4 Measuring

3.4.1 Thermal conductivity of commingled CF/PA12

Four measurements were performed in order to determine the thermal conductivity of the commingled CF/PA12 composite. Square of $20 \times 20 \text{ cm}^2$ of commingled composite were cut, the thickness of one layer is 0.92 mm . The first two measurements were performed with only one layer of commingled composite. This layer was placed on the bottom plate of the hot press previously heated up to 150°C . During the first measurement it was noticed that the contact between the composite and the hot plate was not uniform, for the second measurement the layer was placed more carefully to improve the contact between the layer and the hot plate. The last two measurements were performed using the same procedure but with two layers place on top of each other, the thickness is 1.84 mm . The temperature as a function of the time was reported every 5 seconds. Based on these results a temperature-time curve will be created using COMSOL by varying the thermal conductivity until a similar curve of the one measured with the infrared camera was obtained.

3.4.2 Thermal conductivity of consolidated CF/PA12

The commingled CF/PA12 composite was consolidated with the following procedure. First it was heated up during 20 minutes at 230°C , then hot pressed at 90 kN first during 5 minutes at 170°C and kept under the hot press while cooling up to 150°C . This procedure was used to create consolidated square of $20 \times 20 \text{ cm}^2$ with one, two and three layers of composite. The consolidated composite sheet with one layer is 0.34 mm thick. The one with 2 layers is 0.51 mm thick. The sample with 3 layers is 0.78 mm thick. Then, measurements were performed putting the sheet on the hot plate previously heated up at 150°C . Some problems were encountered because the composite was not very flat and the thermal camera did not always measured the zone in contact with the hot plate. The temperature as a function of the time was reported every 1 second. Based on these results a curve will be created using COMSOL by varying the thermal conductivity until a similar time-temperature curve was obtained.

3.4.3 Thermal conductivity of pieces of consolidated CF/PA12

The CF/PA12 consolidated with 1 layer of composite was cut in small squares of $1.5\text{ cm} \times 1.5\text{ cm}$ for the first two tests. After this the composite was cut in very small irregular pieces (grinded) and the measurements were performed again. In order to place them on the hot plate a thin metal plate where a square of $10 \times 10\text{ cm}^2$ was delimited by an adhesive cork (2 mm thick) was used. In both case 13 g of the small composite pieces were used. For the analyse with COMSOL a thickness of 2 mm was set. The metal plate is placed on the hot plate previously heated up at $150\text{ }^{\circ}\text{C}$, the thickness of the metal plate was neglected due to the high conductivity and the small thickness but has to be taken into account as a potential source of errors. Metal weights were put on both side of the metal plate to avoid the bending and a bad contact with the hot press. The temperature as a function of the time was reported every 5 seconds. Based on these results a curve will be created using COMSOL by varying the thermal conductivity until a similar time-temperature curve was obtained.

3.4.4 Thermal conductivity of GF/PA12 chips with commingled fibres of CF/PA12

The conductivity of small pieces of 13 g of $1\text{ cm} \times 1\text{ cm}$ GF/PA12 composite has been measured in a precedent study [5]. The idea of this measurement is to see how the addition of grinded particles of CF/PA12 are influencing the thermal conductivity.

First of all, 1.3 g of CF/PA12 commingled fibres were added to the 13 g of $1\text{ cm} \times 1\text{ cm}$ GF/PA12 chips, this is the 1:10 weight ratio. Secondly, 2.6 g CF/PA12 commingled fibres were added to the 13 g of $1\text{ cm} \times 1\text{ cm}$ GF/PA12 chips, this is the 2:10 weight ratio. The experimental set up is similar to the one described in section 3.4.3. Using Eq. 1 and 2 the density and the heat capacity are calculated (see Table 3.3), this values will also be used in section 3.4.5. The temperature as a function of the time was reported every 5 seconds. Based on these results a curve will be created using COMSOL by varying the thermal conductivity until a similar time-temperature curve was obtained.

During the first measurement it was noticed that the metal plate used was bending

Table 3.3: Reevaluation of the density and heat capacity depending on the weight ratio

weight ratio CF/PA12:GF/PA12	Density	Specific heat capacity
1:10	1.81	1.047
2:10	1.79	1.071
3:10	1.77	1.095
4:10	1.72	1.145

when being in contact with the hot plate. Therefore, the first measurement needs to be evaluated very carefully because for every other measurement with the metal plate metal, weights were used to maintain the plate flat and in contact with the hot plate.

3.4.5 Thermal conductivity of GF/PA12 chips with grinded consolidated CF/PA12

Finally, the thermal conductivity of 13g of 1 *cm* x 1 *cm* pieces of GF/PA12 mixed with the grinded CF/PA12 consolidated composite (measurement 3 and 4 in section 3.4.3) was measured. Four different weight ratios were tested. For the first weight ratio 1:10, 1.3 *g* of consolidated grinded CF/PA12 were added to the 13 *g* of GF/PA12 chips. For the second weight ratio 2:10, 2.6 *g* of consolidated grinded CF/PA12 were added to the 13 *g* of GF/PA12 chips. For the third weight ratio 3:10, 3.9 *g* of consolidated grinded CF/PA12 were added to the 13 *g* of GF/PA12 chips. For the fourth weight ratio 4:10, 5.2 *g* of consolidated CF/PA12 were added to the 13 *g* of GF/PA12 chips. The same experimental set up used was similar as the one in section 3.4.3. The Density and the specific heat capacity for the COMSOL model have to be adjusted using the values calculated in Table 3.3. The temperature as a function of the time was reported every 5 seconds. Based on these results a curve will be created using COMSOL by varying the thermal conductivity until a similar time-temperature curve was obtained.

4 Results

In this chapter the results from the experiments explained in section 3 are going to be presented.

4.1 Thermal conductivity of the commingled CF/PA12 composite

The thermal conductivity of the commingled CF/PA12 is $0.18 \pm 0.07 \text{ W/mK}$ using the value in Table 4.1.

Table 4.1: Thermal conductivity commingled CF/PA12 composite, one layer correspond to measurement 1 and 2, two layers correspond to measurements 3 and 4.

	Thickness [mm]	T_{min} [K]	T_{max} [K]	Thermal conductivity [J/mK]
1	0.92	310.15	384.15	0.11
2	0.92	317.55	391.15	0.12
3	1.84	316.85	376.15	0.25
4	1.84	308.15	-	0.25

4.2 Thermal conductivity of the consolidated CF/PA12

During the measurements performed on the consolidated CF/PA12 a lot of problems were encountered because of the difficulty to obtain a flat sheet. The thermal camera was not measuring the part of the composite the most in contact with the hot plate. It was noticed watching the videos of the measurements that the measurement 9 from Table 4.2 was the only measure where the part of the composite in direct contact with the hot plate was measured. For the other measurements it can be seen on the videos that the heat is spreading from the part of the composite in contact with the hot plate to the corner of the composite. Therefore, it is not relevant to do a mean and a standard deviation for those results. The value of 1.2 W/mK can be kept for the transverse thermal heat conduction value.

Table 4.2: Thermal conductivity of consolidated composite. Measurements 1 to 3 was consolidated with one layer CF/PA12 composite, 4-6 with two layers and 7-9 with three layers.

	Thickness [mm]	T_{min} [K]	T_{max} [K]	Thermal conductivity [J/mK]
1	0.34	321.25	406.15	0.06
2	0.34	314.25	411.15	0.4
3	0.34	307.15	414.15	0.4
4	0.51	310.45	407.15	0.075
5	0.51	318.45	415.15	0.27
6	0.51	320.05	407.15	0.09
7	0.74	311.55	416.15	0.15
8	0.74	320.35	416.15	0.1
9	0.74	312.25	418.15	1.2

4.3 Thermal conductivity of chips of consolidated CF/PA12

The thermal conductivity of 13 g of 1.5 cm x 1.5 cm chips of consolidated CF/PA12 from the measurement represented in Table 4.3 is $0.06 \pm 0.01 \text{ W/mK}$. The thermal conductivity of 13g of grinded CF/PA12 pieces calculated from the values in Table 4.3 is $0.08 \pm 0.01 \text{ W/mK}$.

Table 4.3: Thermal conductivity of consolidated composite chips of 1.5cm x 1.5cm for measurement 1 and 2. Thermal conductivity of grinded CF/PA12 composite for measurement 3 and 4.

	T_{min} [K]	T_{max} [K]	Thermal conductivity [J/mK]
1	303.65	307.55	0.07
2	375.15	376.15	0.05
3	307.55	380.15	0.07
4	304.75	384.15	0.09

4.4 Thermal conductivity of GF/PA12 chips with commingled fibres of CF/PA12

The thermal conductivity, from measurements in table 4.4 of the mixing between commingled fibres CF/PA12 and consolidated GF/PA12 with a weight ratio of 1:10 is 0.05 W/mK . The thermal conductivity for the 2:10 weight ratio is $0.043 \pm 0.003 \text{ W/mK}$.

Table 4.4: Thermal conductivity of 1 cm x 1 cm chips of GF/PA12 with some commingled fibres of CF/PA12

	weight ratio CF/PA12:GF/PA12	T_{min} [K]	T_{max} [K]	Thermal conductivity [J/mK]]
1	1:10	324.15	367.15	0.05
2	1:10	307.45	366.65	0.05
3	2:10	327.35	352.55	0.045
4	2:10	306.05	366.45	0.04

4.5 Thermal conductivity of GF/PA12 chips with grinded consolidated CF/PA12

For this part of the measurement different temperatures behaviour were noticed depending on the part measured. This leads to very different results in the thermal conductivity for the same weight ratio. The thermal conductivity of the weight ratio of 1:10 is $0.069 \pm 0.017 \text{ W/mK}$. The thermal conductivity for the 2:10 weight ratio is $0.074 \pm 0.016 \text{ W/mK}$. The thermal conductivity of the 3:10 weight ratio is $0.043 \pm 0.003 \text{ W/mK}$. Finally, the thermal conductivity of the 4:10 weight ratio is $0.039 \pm 0.004 \text{ W/mK}$.

Table 4.5: Thermal conductivity of GF/PA12 chips with reinforced particles of CF/PA12

	weight ratio CF/PA12:GF/PA12	Tmin	Tmax	Thermal conductivity
1	1:10	303.95	383.15	0.085
2	1:10	300.65	366.35	0.052
3	2:10	313.55	383.15	0.058
4	2:10	308.65	387.15	0.09
5	3:10	305.35	392.15	0.04
6	3:10	304.15	381.15	0.045
7	4:10	308.45	380.15	0.042
8	4:10	308.55	376.15	0.035

5 Analyse and discussion

The thermal conductivity of the GF/PA12 consolidated composite found in a previous study was 0.25 W/mK for big square sheets of $20 \text{ cm} \times 20 \text{ cm}$ [5]. The thermal conductivity of smaller chips of $1.5 \text{ cm} \times 1.5 \text{ cm}$ was 0.073 W/mK and the chips of $1 \text{ cm} \times 1 \text{ cm}$ used in this study were 0.051 W/mK [5]. With these values the results obtained from section 4 are going to be discussed and analysed.

5.1 Comparison between the theoretical and experimental thermal conductivity

The theoretical thermal conductivity of CF/PA12 and GF/PA12 can be calculated using Eq. 4 (see Table 5.1). It can be noticed that in theory the thermal conductivity of CF/PA12 and GF/PA12 composites in the transverse direction do not change a lot, but it has a great difference in the fibres direction. The tests were performed by heating the composite in the transverse direction.

Table 5.1: Theoretical thermal conductivity of composites

	CF/PA12	CF/PA12
$k_m [\text{W/mK}]$	0.31	0.31
fibre content [-]	0.48	0.5
$k_{fl} [\text{W/mK}]$	70	13
$k_{ft} [\text{W/mK}]$	7	1.3
$k_t [\text{W/mK}]$	0.59	0.6
$k_l [\text{W/mK}]$	49.72	6.66

Comparing the results found in Table 4.2 about the thermal conductivity of the fibre consolidated CF/PA12 it can be noticed that the thermal conductivity is higher than expected, it can be due to the fact that not only the transverse conductivity is taking part of the heating process, but the fibres are contributing in the longitudinal direction. Furthermore, the model was done by only tacking into account the heat produced by the bottom plate, but the top plate of the hot press was also heating the ambient air. These assumptions that the upper surface of the composite was isolated from heat can be a source of errors.

5.2 Comparison of the thermal conductivity of the consolidated and the commingled CF/PA12

It can be noticed that the thermal conductivity of the commingled CF/PA12 and the consolidated CF/PA12 composites are not the same. It could be explained by the voids occupied by air in the co-mingled sheets. This can be verified using Eq. 4 with the thermal conductivity of the reinforced CF/PA12 ($k_c = 1.2 W/mK$) and the air thermal conductivity ($k_{air} = 0.026 W/mK$ [32]). The content of air in one layer can be evaluated using the difference of thickness between the commingled and the consolidated layer. The thickness of the consolidated layer is 0.34 mm while the thickness of the commingled layer is 0.92 mm. It leads to an air volume fraction of 0.64, applying the rule of mixture in a transverse direction the thermal conductivity found is $0.04 W/mK$, this value is also smaller than the value found in the experiments but as mentioned before the fact that not only the bottom plate is heating up the material can be a source of errors.

5.3 Thermal conductivity of the chips and the grinded CF/PA12

The thermal conductivity of the chips and the grinded CF/PA12 reported in Table 4.3 can be compared. The grinded composite is showing a small increase in the thermal conductivity compared to the $1.5\text{ cm} \times 1.5\text{ cm}$ chips, it can be due to the fact that less air is present between the composite particles. Furthermore, the decrease of thermal properties with the increase of air fraction is not huge, it can give an insight about the contribution of the longitudinal conductivity. Compared to the thermal conductivity of the $1.5\text{ cm} \times 1.5\text{ cm}$ glass fibres chips ($0.073 W/mK$) a very small increase has been noticed. In both measurement 13 g of chips were taken in order to do the measurements, due to their different densities the volume change between the two material is not negligible. The 13 g of CF/PA12 represent a bigger volume of chips and was therefor thicker and less in contact with the hot plate, it would have been better to take the same volume of chips in order to compare the values.

5.4 Thermal conductivity of GF/PA12 chips with added CF/PA12

The thermal conductivity of $1\text{ cm} \times 1\text{ cm}$ glass fibres chips mixed with fibres of commingled CF/PA12 was reported in Table 4.4. The change in the thermal conductivity as a function of the added weight ratio can be seen in Fig. 5.1. With a weight ratio of 1:10 no increase in the thermal conductivity was noticed. For the 2:10 weight ratio a decrease in the thermal conductivity was noticed. The non-consolidated fibres are taking a lot of volume fraction reducing the contact with the hot plate. It would have been better to start the measurements with a smaller weight ratio because it can be noticed that with an increase in the weight ratio of 2:10 the thermal conductivity is dropping.

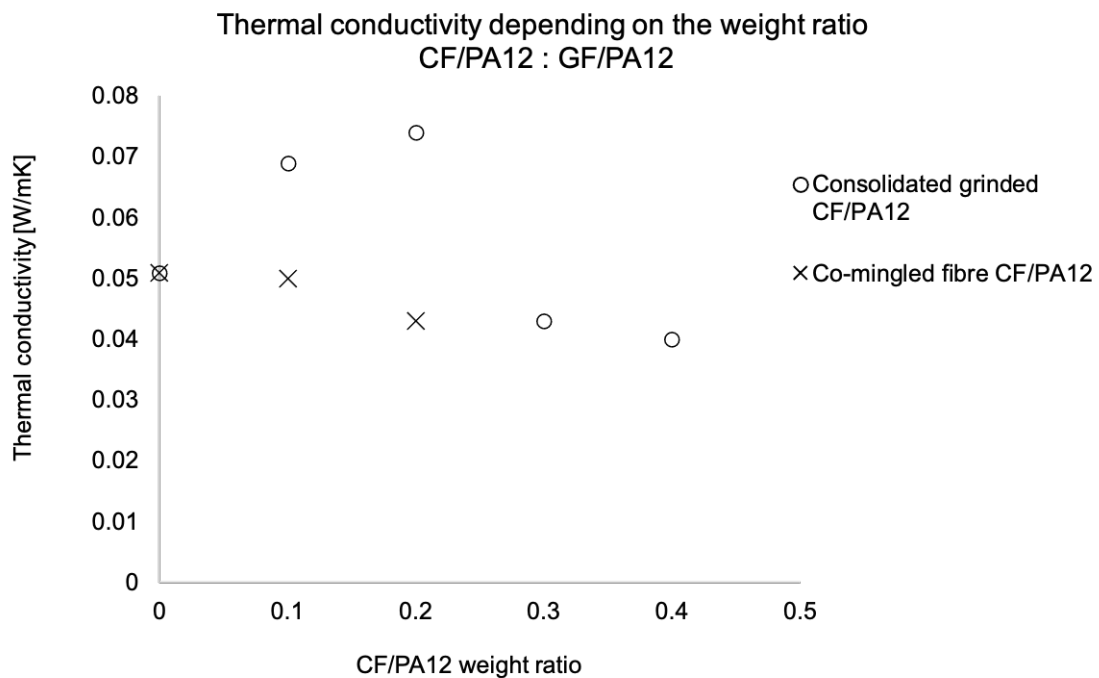


Figure 5.1: Influence of the commingle fibres and the grinded consolidated CF/PA12 composite on the GF/PA12 chips

The thermal conductivity of the $1\text{ cm} \times 1\text{ cm}$ GF/PA12 pieces mixed with grinded CF/PA12 was reported in Table 4.5. Up to a weight ratio of 2:10 an increase in the thermal conductivity was observed, this is certainly because the grinded particles of CF/PA12 are filling the gap previously occupied by air. From the weight ratio of 3:10 the volume starts to considerably increase, decreasing the surface of contact

between the composite and the hot plate which results in a decrease in the thermal conductivity.

5.5 Potential errors

There are some error sources that can be identified. First, the COMSOL model could have been improved, it does not consider the heat coming from the top plate. The infrared camera was not directly perpendicular to the surface and a bit far away. The use of weight to maintain the specimen flat increase the role of the longitudinal thermal conductivity and only the transverse conductivity was considered in the model. However, the thickness of the composite sheets were very small compared to their surface, a 1D heat transfer case can be considered avoiding big errors.

6 Conclusions

This work has brought to a lot of conclusions about the thermal conductivity of different fibre composites and the potential recycling efficiency. A list with all the conclusions that can be done is presented below.

- The thermal conductivity of fibre reinforced polymers with carbon fibres has shown an increase compared to the composites with glass fibres. The thermal conductivity measured was the transverse one, in this direction the effect of the fibres is smaller.
- By cutting the composite into smaller pieces to model more realistic conditions of recycling, it was noticed that the air fraction have an impact on the thermal conductivity by decreasing it. By filling the air between chips of GF/PA12 with smaller pieces of CF/PA12 the thermal conductivity was increased.
- The thermal conductivity of the commingled and the consolidated CF/PA12 composite were compared, a smaller thermal conductivity was obtained for the commingled material, this can be related to the higher air fraction. In conclusion to improve the efficiency of heating the quantity of air fraction must be avoided as much as possible.
- The use of better thermal conductive fibres is a good way to increase the thermal conductivity.
- The fact that mixing carbon fibre and glass fibre materials has increased the thermal conductivity of the simple glass fibre material is a good point because scrap material may be mixed with each others.

6.1 Future work

Some further works may be done on the subject. First the COMSOL model could be improved to be more realistic. The carbon fibres have shown a good ability to improve the thermal conductivity of the composite, a next experiment could be performed to see if the material can be melted using heating techniques such as microwave or induction. Another experiment could be performed by introducing

carbon nanotubes which are showing a very high thermal conductivity and an ability to be heated by microwave and induction.

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