

The effects of alkali-silane treatment and filler materials on the tensile and water absorption properties of hemp fibre reinforced polypropylene.

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Declaration

I declare that this dissertation is my own unaided work. It is being submitted for the degree of Master of Science in Engineering at the University of the Witwatersrand, Johannesburg. It has not been submitted before for any degree or examination at any other university.

(Lance Daniel Paiken)

_____ day of _____ (year) _____

Abstract

Research was undertaken to determine the effects that multi-walled carbon nanotube and glass flakes have on treated fibre composites. The fibre chosen was initially kenaf fibre to expand on work done by Asumani *et al.* [1], however, due to a number of complications, the fibre was changed to hemp. The previous research investigated the effects of a treatment using NaOH and Silane on kenaf fibre reinforced polypropylene and the current work uses the same treatment on hemp fibre reinforced polypropylene. The properties that were investigated were tensile strength, tensile modulus as well as water absorption.

Hemp fibre was purchased in long strands. The fibres were then manufactured into hemp fibre mats with a density of $350 \pm 20 \text{ g/m}^2$. The fillers were mixed into the matrix using a shear mixer. It was found that the carbon nanotubes and glass flakes did not mix perfectly inside the matrix which led to agglomeration of the filler material. These agglomerates led to holes being created inside the composite as matrix cannot penetrate to the centre of the agglomerates resulting in empty space. The composite plates were manufactured using a combination of the film stacking technique and compression moulding. The process was adapted from that of Asumani *et al.* [1]. Four different fibre weight fractions were investigated, namely: 15%, 20%, 25% and 30%. Composites containing 2% carbon nanotubes and composites containing 350 nm thick glass flakes at a concentration of 8% were investigated at all fibre weight fractions. The effect of variation in filler concentration was investigated at 30% fibre weight fraction.

Tensile tests as well as water absorption tests were performed on the composites. The treated fibre composites achieved better results than the untreated fibre composites for all fibre weight fractions when analyzing the tensile strength and for 15%, 20% and 25% when analyzing water absorption properties. The 30% treated fibre composites behaved poorly in the presence of water due to the number of fibres exposed on the surface of the composite. The carbon nanotubes increased the tensile strength of the matrix. The carbon nanotubes, however, may reduce the strength of the interfacial bonding between the fibres and the matrix and so the strength remains constant with increasing fibre weight fraction. Therefore, at low fibre weight fractions there was an increase in strength relative to treated fibre composites containing no carbon nanotubes. At high fibre weight fractions there was a decrease in strength relative to treated fibre composites containing no carbon nanotubes. Glass flakes improved the tensile strength slightly, for all fibre weight fractions. The highest tensile strength achieved used 350 nm thick glass flakes at a concentration of 12%. At high fibre weight fractions, there appears to be an improvement in water absorption properties using 350 nm glass flakes at a concentration of 8%. No significant improvement on the water absorption properties resulted from the carbon nanotubes or other glass flake sizes or concentrations.

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Nomenclature

Symbol	Description	Unit
F	Force	N
ε	Strain	
W	Width	mm
L	Length	mm
b	Breadth	mm
t_c	Centre Thickness	mm
t_e	Edge Thickness	mm
σ	Stress	MPa
E_{secant}	Secant Modulus	GPa
E_{linear}	Linear Modulus	GPa
m	Mass	kg
t	Time	hours
W_f	Fibre Weight Fraction	%
β	Rate of Water Absorption	/hour

1 Introduction

Natural fibre composites are receiving a great deal of interest due to the fact that they are more environmentally friendly than fully synthetic composites. Using natural fibres in areas which do not require high load bearing capabilities instead of fibres such as glass would be an important step forward in composite manufacture. Due to this environmental drive thermoplastic materials have become a favourite within industry. Polypropylene is one of the most commonly used thermoplastic materials. It is low cost, easily recycled and has reasonable mechanical properties [2, 3].

A disadvantage of using polypropylene in this application is that natural fibres and polypropylene don't readily bond to one another due to a difference in hydrophilicity [4-6]. The fibres are hydrophilic meaning they attract water. We can attribute the hydrophilic nature of the fibres to the hydroxyl groups on the surface of the fibres. These hydroxyl groups are able to absorb the water easily due to their ability to form hydrogen bonds both as donor and acceptor [7]. Polypropylene is hydrophobic meaning it repels water. This difference in hydrophilicity inhibits good chemical bonding between the fibres and the matrix. Work done using natural fibres as reinforcement for thermoplastic polymers found that the resulting mechanical and water absorption properties are poor [4-6, 8, 9]. These properties need to be improved if the composite is to replace traditional composites such as those reinforced by glass fibres in certain applications.

The poor properties are a result of the difference in hydrophilicity between the natural fibres and the matrix system [4-6]. This incompatibility leads to difficulties in ensuring effective fibre-matrix interface bonding, which in turn causes ineffective load transfer between the reinforcing material and the matrix. Additional work has consequently been conducted to find a fibre treatment which would allow the matrix and natural fibres to bond better with one another. Various fibre treatments have been done on natural fibre composites yielding promising results [1, 10-24]. One such treatment done by Asumani *et al.* [1] resulted in improvements of up to 100% in some mechanical properties of kenaf fibre composites. This treatment has two stages, a sodium hydroxide (NaOH) treatment followed by a silane treatment. The NaOH is used to clean the fibres of debris exposing more cellulose and increasing the number of reaction sites which increases the likelihood of the silane bonding to the fibres. The silane is used as a coupling agent and improves the bond between the fibres and the matrix system. The mechanical properties are though, still inferior to those of glass fibre reinforced composites. A possible method to enhance the properties still further is the use of filler materials.

Unfortunately it was not possible to source the same non-woven kenaf fibre mat used by Asumani *et al.* [1]. Attempts were made to manufacture the mat in-house but suitable kenaf fibre could not be obtained. Good hemp fibre could be readily purchased, however, and so hemp fibre was investigated instead. As with kenaf, untreated hemp has a difference in hydrophilicity between the fibre and the matrix system leading to poor mechanical properties [5, 6]. Studies have shown that similar treatments have improved the properties of hemp fibre composites [10-13]. These improvements are not as high as those achieved by Asumani *et al.* [1], however. The properties of the hemp fibre reinforced composites are still below that of glass fibre reinforced composites. The treatment described by Asumani *et al.* [1] may be able to improve these properties further and adding fillers such as multi-walled carbon nanotubes (MWCNT) and glass flakes may improve the properties to the level of glass fibre. This topic is the subject of the present investigation.

2 Objectives

- Manufacture non-woven hemp fibre mats with consistent properties.
- Manufacture hemp fibre reinforced composites across a range of fibre weight fractions using a similar production method to that set out by Asumani *et al.* [1].
- Manufacture composite plates using fibres that have not undergone the treatment of Asumani *et al.* [1].
- Manufacture composite plates using fibres that have undergone this treatment.
- Manufacture composite plates using treated fibres and multi-walled carbon nanotubes across a range of carbon nanotube concentrations.
- Manufacture composite plates using treated fibres and glass flakes across a range of glass flake sizes and concentrations.
- Test the tensile properties of each composite.
- Test the water absorption properties of each composite.
- Compare the results obtained.

3 Literature survey

3.1 Hemp

For the past number of years a large amount of time and money has been invested into finding greener approaches to all aspects of life. From motor manufacture to energy production, people are looking into less environmentally damaging methods.

Synthetic fibre composites manufactured using glass fibre, which is the most widely used synthetic fibre in the world [25], are very difficult to dispose of at the end of their lives. Environmental authorities are therefore scrutinizing the use of these composites [10]. The synthetic composites are difficult to dispose of as they are closely interconnected and relatively stable and are therefore difficult to separate and recycle [10].

Natural fibres, such as flax, hemp, jute, and kenaf have received considerable attention as an environmentally friendly alternative for the use of glass fibre [26]. These natural fibres are cheap, abundant, have a low density, show less concern with health and safety (e.g. skin irritation), give less abrasive wear to processing equipment such as extruders and moulds and have great mechanical properties when compared to their density and price [5, 10, 25, 26]. One of the most appealing properties of natural fibres is that they are biodegradable and the demand for renewable raw materials is increasing [5, 10, 25, 26]. Many companies are looking to produce non-woven products like mats for insulation, filters, geotextiles and car/vehicle composites based on natural fibres such as hemp fibres [27]. The mechanical properties of a number of different natural fibres are shown in Table 1.

Table 1 Mechanical properties of a number of natural fibres

		Hemp	Jute	Flax	Kenaf
Density	Kg/m ³	1480 [28]	1460 [28]	1400 [28]	1500 [29]
Elastic Modulus	GPa	70[28]	10-30 [28]	60-80 [28]	40 [29]
Tensile Strength	MPa	550-900 [28]	400-800 [28]	800-1500 [28]	350-600 [29]
Elongation at Break	%	1.6 [28]	1.8 [28]	1.2-1.6 [28]	2.5-3.5 [29]

The fibres of the hemp plant are among the longest and strongest in the plant kingdom [30]. Hemp has many agricultural benefits as it can easily be grown organically. Hemp quickly grows up to five metres in height with dense foliage which blocks weed growth. This means herbicides are not needed and the field is weed free for the next crop. Unlike cotton, hemp does not have a high water requirement. The hemp

plant has a deep tap root system which enables the plant to take advantage of deep subsoil moisture, thus requiring little or no irrigation [27].

There are two principal types of fibres in hemp – bast or long fibres and hurds or inner short fibre. Bast fibres account for 20-30% of the stalk. There are two different types of bast fibres, primary and secondary [31]. Primary bast fibres make up approximately 70% of the fibre and are among the strongest plant fibres known. The secondary bast fibres make up the remaining percentage. They are shorter, higher in lignin and less valuable [31]. An image of hemp under cultivation is presented in Fig. 1.



Fig. 1 Hemp under cultivation [31]

With all these advantages it would seem that natural fibres, such as hemp, would be extremely popular. There are, however, a number of disadvantages to hemp that need to be addressed to utilize these fibres to their full potential. These disadvantages include poor interfacial adhesion between the cellulose fibres and the thermoplastic matrix, limited thermal stability, poor fibre separation and dispersion within the composite and high moisture absorption as a result of the hydrophilic nature of the fibres [5, 10, 25, 26].

3.2 Polypropylene

Polypropylene is a thermoplastic matrix. Polypropylene has a linear structure based on the monomer C_3H_6 [3]. The structure of the polypropylene monomer is presented in Fig. 2. Most polypropylene used is highly crystalline and geometrically regular (i.e. isotactic). This is in contrast to amorphous thermoplastics, such as polystyrene, PVC and polyamide where radicals are placed randomly (i.e. atactic) [3]. Polypropylene has an intermediate level of crystallinity between low density polyethylene (LDPE) and high density polyethylene (HDPE) and has higher working temperatures and tensile strength than polyethylene [3].

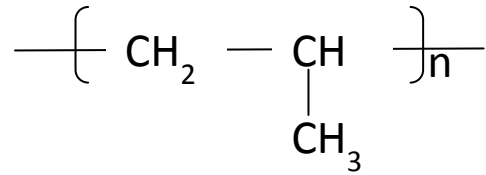


Fig. 2 Organic structure of polypropylene [32]

Polypropylene has many uses both in industry and in consumer goods since it is versatile and has low cost. It can be used both as a structural plastic and as a fiber [33]. As a plastic it is used to make items such as dishwasher-safe food containers since it doesn't melt below 160°C [32]. Other applications include packaging, cassette holders, pipes, boat hulls, seat shells and automotive parts e.g. battery cases and bumpers [3]. Polypropylene is also useful in that it doesn't soak up water as a result of its hydrophobic nature. This makes it ideal for uses where it will be constantly subject to moisture. Due to the thermoplastic properties of polypropylene it can be easily recycled, however, this degrades the properties of the polypropylene [3]. Recycling is a major advantage when selecting a matrix material as it reduces costs both environmentally and financially [3].

The mechanical properties of Polypropylene are shown in Table 2

Table 2 Mechanical Properties of Polypropylene

Density	Kg/m ³	910 [2]
Elastic Modulus	GPa	1.3 [2]
Tensile Strength	MPa	30 [2]
Elongation at Break	%	>30 [2]

It is evident from Table 1 and Table 2 that natural fibres have a much higher tensile strength when compared to polypropylene. The addition of natural fibres into polypropylene should, therefore, vastly improve the mechanical properties of the plastic material. However, as mentioned previously, natural fibres are polar and hydrophilic in nature [7] while polypropylene is non-polar and hydrophobic in nature [5, 6]. This difference in hydrophilicity leads to poor interfacial bonds, which results in composite materials with lower mechanical properties than expected [5, 6]. The interfacial bonds between the fibres and matrix can be improved by either modifying the surface of the fibres to make them more compatible with the matrix, or by modifying the matrix with a coupling agent that adheres well to both the fibres and matrix [5, 6].

3.3 Natural fibre composites

Considerable research has been done on a range of natural fibre reinforced composites, using various fibre treatments to improve the interfacial bonding between the fibres and the matrix. Improvement in a range of composite properties have been achieved using treatments such as alkali [1, 10-14], silane [1, 10-15], benzoylation [14, 16-18], acetylation [14, 15, 19-22], sodium chlorite [14, 23, 24], as well as a few other treatments [14].

An alkali treatment uses NaOH to clean the fibres of debris exposing more cellulose and increasing the number of reaction sites which increases the likelihood of the silane and matrix bonding to the fibres [34]. The silane is used as a coupling agent which improves the bonds between the fibres and the matrix system [35].

The alkali and silane treatments are the most widely used treatments for natural fibre reinforced composites. These fibres are used to reinforce thermoplastic polymers such as polypropylene and thermoset polymers such as polyester and epoxy resin. A combination of both treatments has achieved significant improvements as seen by Asumani *et al.* [1]. They used compression moulding techniques to manufacture composites using kenaf fibre mats and polypropylene. The kenaf underwent an alkali treatment followed by a silane treatment. Improvements in tensile strength and modulus of up to 100% were achieved using NaOH with a concentration of 6% and 3-aminopropyltriethoxysilane with a concentration of 5% by weight (weight of silane relative to weight of fibres). This shows promising improvements in mechanical properties but they are still inferior to that of glass fibre reinforced composites. The other fibre treatments have not achieved the same level of improvements as the silane and alkali combination used by Asumani *et al.* [1].

Studies have been done using treated hemp fibre to reinforce polypropylene and polyester [10-13]. These treatments were performed and tested separately. The studies used a range of NaOH concentrations and silanes to perform the treatments. All the studies showed that the interfacial bonds between the hemp fibre and the matrix have improved as a result of the treatments and so mechanical properties have improved. This improvement varies according to the type of silane used and the concentration of the chemicals. Mehta *et al.* [10] used treated hemp fibres to reinforce unsaturated polyester resin (UPE). They achieved one of the largest improvements using the silane treatment when compared to these other studies. Mehta *et al.* [10] saw an improvement of 50% in tensile strength as a result of a silane treatment using 1% γ -methacryloxypropyltrimethoxy silane. Suardana *et al.* [13] used treated hemp fibres to reinforce polypropylene. They achieved one of the largest improvements using the alkali treatment when compared to these other studies. Suardana *et al.* [13] saw an improvement of 35% using NaOH at a concentration of

4%. Significant improvements were seen using these treatments on hemp fibre composites, however, the improvements are less than those achieved by Asumani *et al.* [1].

It is possible that the improvements are not as large as seen by Asumani *et al.* [1] since the treatments were done separately. The effects of the treatments may be compounded to achieve better results. Therefore following the method set out by Asumani *et al.* [1] may achieve similar results using hemp as achieved using kenaf. Another cause for the difference may also be a result of the variation in silanes used and variation in NaOH concentrations. Finally it may be that hemp fibre will not be able to achieve the same level of improvements as kenaf due to the composition of the fibres themselves. The alkali treatment removes a certain amount of hemicellulose, lignin, wax and oils covering the external surface of the fiber cell wall [36]. This increases the number of reaction sites for the coupling agent and the matrix to bond [1]. The weight percentage of lignin and hemicellulose present in hemp fibre are 6.06% and 10.7%, respectively [37]. In contrast the weight percentage of lignin and hemicellulose present in kenaf are 14.7% and 19.21%, respectively [38]. There is more lignin and hemicellulose that can be removed from the kenaf fibre and so the effect of the treatment may be more significant.

Improvements in the interfacial adhesion between the hemp fibre and the polypropylene matrix using the process set out by Asumani *et al.* [1] can be expected. The extent to which the mechanical properties will improve as a result of the improved interfacial adhesion may not be as large as seen by them. This can only be confirmed through experimental tests.

Results archived by Asumani *et al.* [1], Mehta *et al.* [10] and Suardana *et al.* [13] are still lower than that of glass fibre composites therefore investigation into an additional reinforcement, fillers such as carbon nanotube and glass flakes, needs to be done as well.

3.4 Fillers

3.4.1 Carbon nanotubes

Carbon nanotubes have been around for the past 60 years. They only really burst into the scientific world in the early 1990's due to work done by Iijima [39, 40]. The nanotubes he prepared were far more perfect than had previously been produced catalytically, and differed from them by being all-carbon structures, closed at both ends and not "contaminated" with catalyst particles [39]. Since their discovery, they have attracted a great amount interest in research fields and in industrial applications, owing to their impressive thermal, electrical, and mechanical properties [40].

The properties of the carbon nanotubes depend on their structure and size. Carbon nanotubes are hollow cylindrical shapes made out of graphite. These nanotubes can be single walled tubes (SWNT) or multi-

walled tubes (MWNT). SWNTs are prepared by addition of a metal catalyst such as Co-Ni to graphite electrodes resulting in fine tubes [39]. By laser evaporation a high yield of SWNTs can be produced with uniform diameters [39]. Multi-walled carbon nanotubes are prepared by arc evaporation and chemical vapour decomposition (CVD) [40]. An example of Multi-walled carbon nanotubes prepared by CVD is presented in Fig. 3. Multi-walled carbon nanotubes are concentric single wall tubes. These concentric multi-walled nanotubes are held together through secondary Van der Waals forces [41].

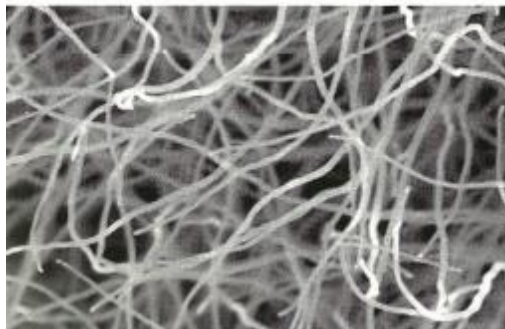


Fig. 3 High resolution scanning electron microscope image of MWNTs produced using CVD [41]

Carbon nanotubes have been used to reinforce matrix systems thereby forming composite materials. Much research has been done using carbon nanotube composites [42-44]. Prashanta *et al.* [42] investigated the effect MWNTs have on polypropylene. A commercially available carbon nanotube master batch of 20% by weight was used. Carbon nanotube concentrations ranging from 1% to 5% by weight were then manufactured from this master batch using a twin screw extruder. The carbon nanotubes used were 9-11nm in diameter and 1.2 μm in length. The composites were manufactured using compression moulding. The results showed an improvement of almost 20% in tensile strength and roughly 28% in tensile modulus using a concentration of 1% and roughly 35% in tensile strength and 52% in tensile modulus using a concentration of 5%. The difference in improvement on the tensile strength between the 4% and 5% concentrations is roughly 2% indicating a possible drop off in improvements above 5%. This trend is not evident for the tensile modulus. These results indicate significant improvement in the mechanical properties of polypropylene using carbon nanotubes.

Even after two decades of research into using carbon nanotubes as reinforcements, their full potential has not been reached. This is due to the difficulties associated with dispersing the entangled carbon nanotubes during processing and poor interfacial interaction between the carbon nanotubes and the polymer matrix [43].

The dispersion problem of carbon nanotubes is different to that of more conventional reinforcements such as carbon fibre and spherical particles since carbon nanotubes have diameters in the nano-scale and their

aspect ratios are much higher (>1000) leading to extremely large surface areas. Additionally the commercially available carbon nanotubes are usually supplied in the form of heavily entangled bundles [43].

Fig. 4 shows a uniform distribution of 0.1 % by volume of carbon fibre and carbon nanotubes inside a cube with a volume of 1 mm^3 . The number of carbon nanotubes is 1.7 million times larger than the number of carbon fibres creating a larger reinforcement surface area [43]. This increase in surface area causes difficulty when trying to uniformly disperse the carbon nanotubes without creating agglomerates. After production carbon nanotubes are held together in bundles consisting of 50 to a few hundred individual carbon nanotubes by Van der Waals force. This is illustrated in Fig. 5. It has been proven that these bundles and agglomerates result in diminished mechanical and electrical properties of composites as compared with theoretical predictions [43]. The two observations indicate the reasons why the uniform dispersion of carbon nanotubes is so difficult and important. This dispersion needs to be addressed.

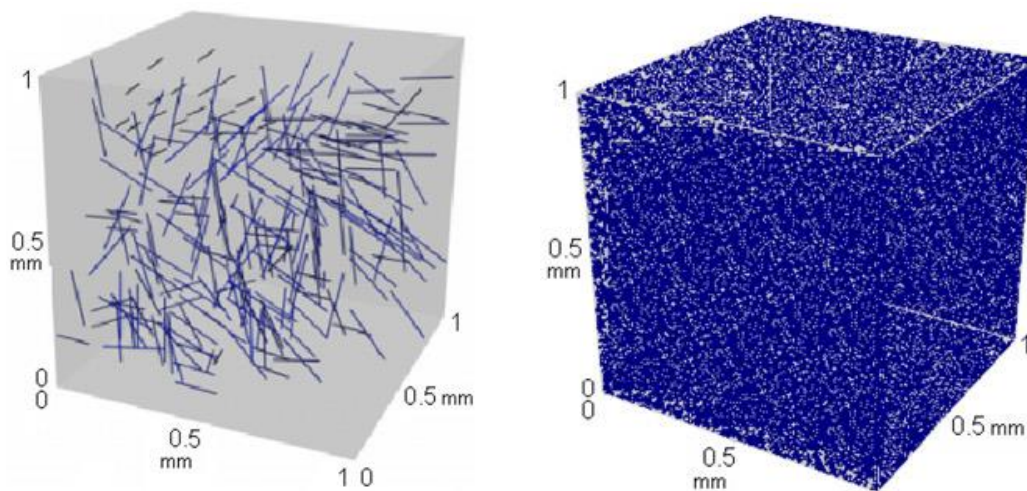


Fig. 4 Distribution of reinforcement at 0.1 vol% in a volume of 1 mm^3 Left: Carbon fibre \ Right: Carbon nanotubes [43]

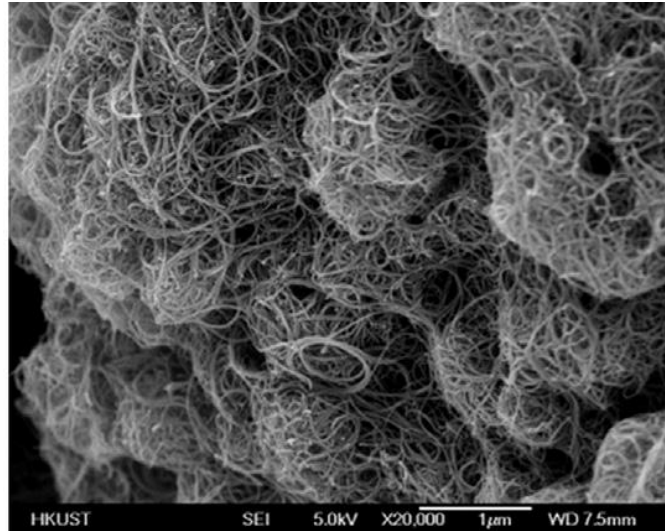


Fig. 5 SEM image of entangled multi-walled carbon nanotube agglomerates [43]

Carbon nanotube reinforced composites have achieved significant improvements in strength [42-44]. This improved strength is highly dependent on the dispersion of the carbon nanotubes [43]. It is therefore possible to improve the strength of a thermoplastic matrix, such as polypropylene, by adding carbon nanotubes [42]. Provided good quality dispersion can be achieved, using carbon nanotubes as a filler material in natural fibre composites should improve the overall mechanical properties due to the improved properties of the matrix. The overall strength will only improve if the interfacial adhesion between the fibres and the matrix is strong. This interfacial adhesion should be improved using one of the treatments explained above.

3.4.2 Glass flakes

Glass flakes are used as a reinforcing agent for many plastic products. The resulting composite material, properly known as flake-glass reinforced plastic (FGRP) or flake-glass reinforced epoxy (FGRE) is called glass flake in popular usage [45]. Glass flakes were originally designed to be mixed with other materials to enhance their characteristics. Glass flakes are not a chemical additive that provides a molecular reaction to improve properties, but are however shaped particles that reinforce and strengthen on a structural and microscopic level. Compared to glass fibers, glass flakes are stronger but when both are combined the synergy creates even greater strength, toughness, and stiffness than could be achieved by using only fibers or flakes alone [46].

Glass flake is formed when glass is extruded into an ultra-thin tube that is then crushed. Glass is unlike polymers in that, even as a flake, it has little crystalline structure. The properties of glass in its softened stage are very much like its properties when extruded into flake. Glass flakes are useful because of their

high surface area to weight, better known as “aspect ratio”. However, the increased surface area makes them much more susceptible to chemical attack [45]. A magnified image of glass flakes is presented in Fig. 6.

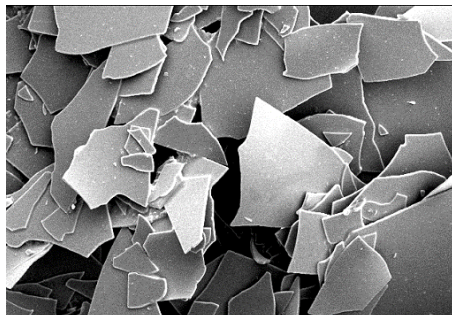


Fig. 6 Magnified image of glass flakes [47]

Only a few papers have been published on the effect of glass flakes in a polymer matrix. Broughton *et al.* [48] found that the effectiveness of the glass flake reinforcement is strongly dependent on the interfacial adhesion between the glass flake and the surrounding polymer matrix. These researches therefore investigated the effects of different surface treatments on the mechanical properties. One of the treatments used aminosilane as a coupling agent. Two concentrations were investigated namely 0.05% and 0.28% by weight. The untreated glass flakes significantly improved the stiffness of the polypropylene however reduced the overall tensile strength by 20%. The aminosilane treatments improved both the stiffness and the strength when compared to the untreated glass flakes. The treatment using 0.28% aminosilane achieved the highest stiffness and strength indicating improved interfacial bonding. The strength achieved was similar to pure polypropylene. The improved properties increase with increasing silane percentage.

As stated very little has been published using glass flakes to reinforce plastic materials. Glass Flake Limited is a company who specialize in manufacture and development of glass flakes. They are widely recognized as an innovator in this technology [49]. The company claims that the glass flakes can successfully be used as reinforcement for thermoplastics, such as polypropylene. They claim that the glass flakes can improve the overall tensile strength and modulus, flexural strength and modulus and reduce the permeation of liquid through the polymer [49]. Since the glass flakes have large aspect ratios the problem of dispersion is similar to that of the carbon nanotubes. Therefore it is important to achieve adequate dispersion of the glass flake filler to achieve the best results. For continuity, the same technique should be used for both fillers. Provided good quality dispersion can be achieved, the use of glass flakes as a filler material for natural fibre composites should be able to improve the overall mechanical and water absorption properties due to the improved properties of the matrix.

3.5 Shear mixing

As mentioned previously, one of the major obstacles when using a nano-filler such as carbon nanotubes or glass flakes is the dispersion of the nano-filler in the matrix. The properties of the nano-composites depend largely on this dispersion [43]. Therefore it is important to obtain a good quality dispersion. A number of different techniques can be used to achieve the desired dispersion and they can be split into two categories, mechanical and chemical [40]. The mechanical techniques involve, ball mixing, ball milling followed by sonication and shear mixing. The chemical techniques involve using surface functionalization of carbon nanotube to improve their chemical compatibility with the target medium [40].

The shear mixing technique is most widely used for the dispersion of the nano-fillers. Many authors have achieved good dispersion of the carbon nanotubes using this technique [40, 44, 50, 51]. It is important to note that Katihabwa *et al.* [44] found that at high carbon nanotube concentrations in silicone rubber (10 % by weight and higher) the carbon nanotubes begin to form bundles and the dispersion deteriorates.

The shear mixing process is performed as follows: The matrix is melted at temperatures above the melting point (for polypropylene this would be 210°C which is 50°C higher than the melting point) and mixed using two rotating screws. These screws are presented in Fig. 7. The temperature is much higher than the melting temperature to ensure the adequate melting of the matrix within the short period of time. The filler is then added to the matrix and the two are mixed as a result of the shear created by the screws. Fluid undergoes shear when one area of fluid travels with a different velocity relative to an adjacent area [52]. The velocity of the fluid at the outside diameter of the screw is higher than the velocity at the centre. This velocity difference creates the shear required to evenly disperse the filler inside the matrix. The matrix/filler mix is removed and cut into small pieces while warm.

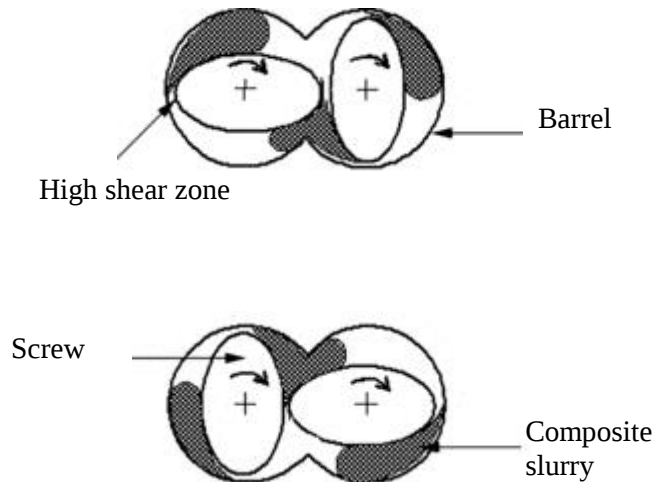


Fig. 7 Rotating screws for shear mixing [53]

The pieces of matrix/filler need to be turned into flat sheets to use in the compression moulding process. This is done by a method known as extrusion.

3.6 Extrusion

This process involves placing the pieces of matrix/filler mix created through the shear mixing process into a feed hopper connected to a heating chamber [54]. When the matrix/filler pieces reach the liquid state due to the heat, a piston or screw forces the plastic through a die. The extruded material is flattened using a rolling pin and thin sheets are created. The dimensions of the die and speed of the screw determine the dimensions of the sheets [55]. An example of the extruder without the rolling pin is illustrated in Fig. 8.

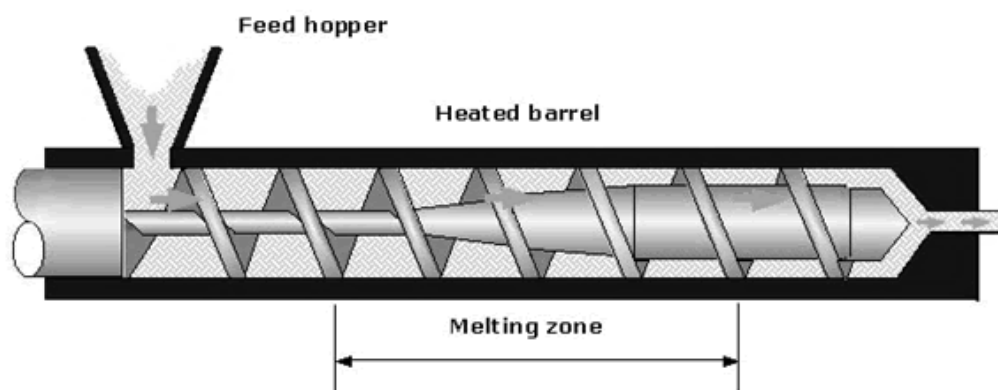


Fig. 8 Extruder [54]

3.7 Compression moulding using film stacking

The film stacking technique is suggested by its name. The fibres and matrix are stacked one on top of the other until the desired amount of material is used. This stack of fibre and matrix is also known as the material charge and a side view is presented in Fig. 9. A release agent is used to ensure that the composite can be released from the mould after manufacture without damage to either the composite or the mould. Two layers of PTFE on the outside of the material charge are often used in this role. A similar technique is used by Jespersen *et al.* [56]. Jespersen *et al.* [56] provides a clear description of this process.

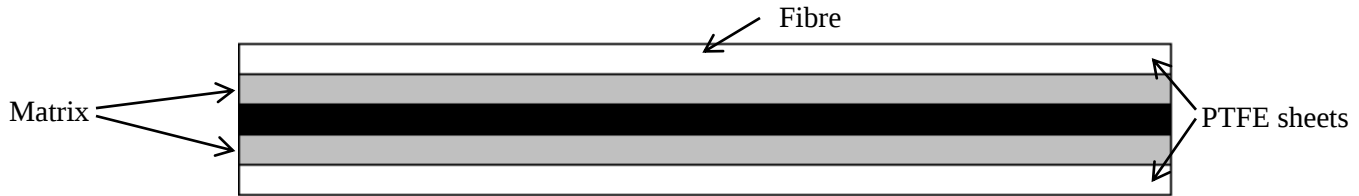


Fig. 9 Film stacking technique

Compression moulding is a technique used to manufacture composite material whereby the material charged is heated in an oven while pressure is being applied. When the matrix system has melted it begins to flow and due to the pressure it is forced through the fibres creating the composite material. The processing temperature needs to be above that of the melting temperature of the matrix such that the matrix is not too viscous to flow through the fibres. The temperature must not exceed the temperature at which chemical alteration of the fibres occurs. When the matrix used is a solid at room temperature, like polypropylene, compression moulding is preferred over other techniques. Processes such as injection moulding require a liquid matrix to inject through the fibres and so polypropylene would require constant heating to remain as a liquid at room temperature. An example of a compression moulding setup is presented in Fig. 10.

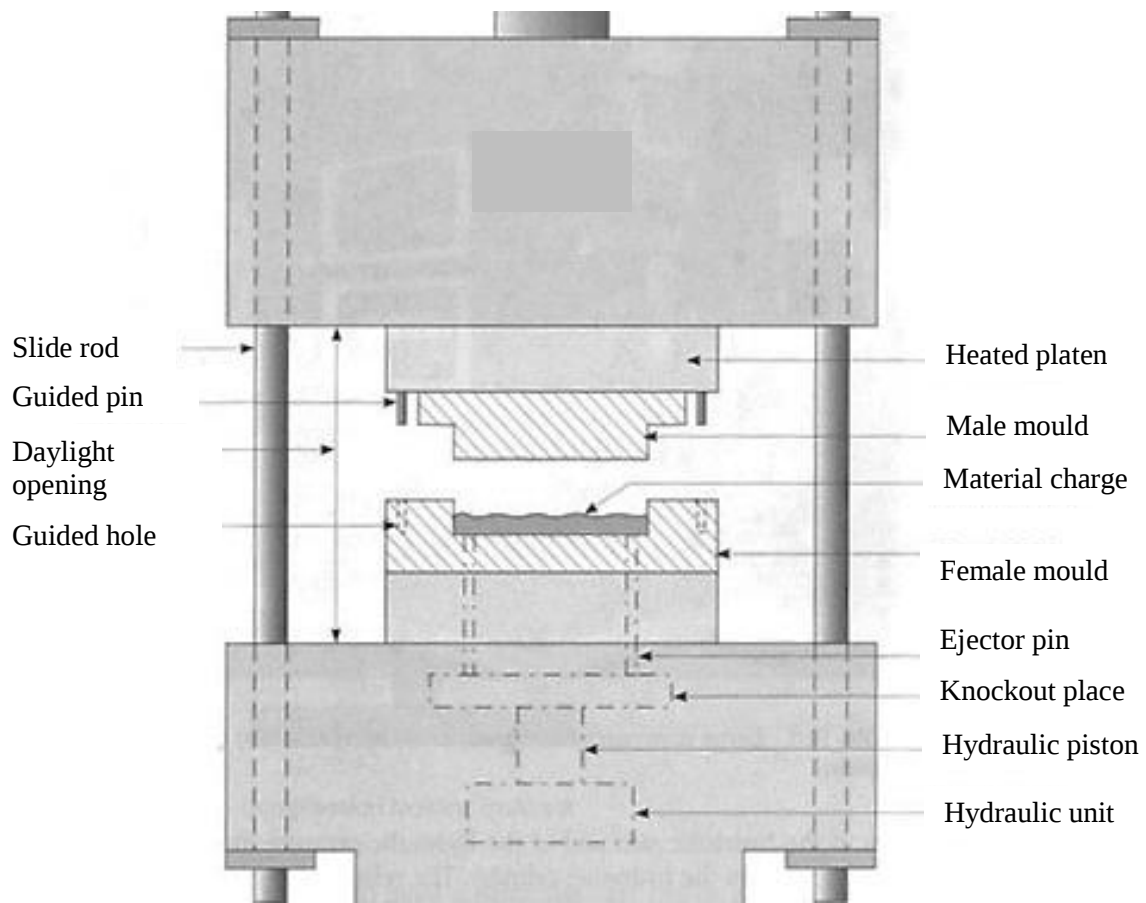


Fig. 10 Compression moulding setup [57]

4 Apparatus

4.1 Manufacturing materials

4.1.1 Hemp mat

Industrial hemp fibres, used for plumbing purposes, were purchased from a local plumbing store. The hemp fibres were purchased in long strands as presented in Fig. 11. The hemp fibres were then cut into short lengths, of roughly 5mm, so that the mats could be manufactured.



Fig. 11 Industrial hemp fibre [58]

The mats were manufactured using a process developed by the author which is discussed in the section 4.2. The mats had a density of $350 \pm 20 \text{ g/m}^2$. The variation in the density arose as a result of the manufacturing process. An example of a mat straight after manufacture is presented in Fig. 12. The circular mat was then cut to the rectangular size required in the moulding process. The final mat is presented in Fig. 13.



Fig. 12 Hemp fibre mat straight after manufacture



Fig. 13 Hemp fibre mat cut to size

4.1.2 Polypropylene (matrix)

The matrix used for the composite material was polypropylene. The polypropylene was acquired from Safripol in the form of pellets.

Polypropylene is a thermoplastic and therefore it can be melted many times during production. This allowed for the use of the rheomixer to disperse the multi-walled carbon nanotubes and glass flake fillers into the matrix. The rheomixer process and its results will be discussed later.

4.1.3 Multi-walled carbon nanotubes

Multi-walled carbon nanotubes were purchased from Sabi-Nano cc. They were produced using the CVD production method and had a typical diameter of 15 nm according to the supplier. The purity of the multi-walled carbon nanotubes was greater than 97%.

4.1.4 Glass flakes

Glass flakes were donated by Glass Flake Limited. Three different glass flake thicknesses were investigated. The thicknesses were suggested by the donor. The company also provided advice regarding manufacture, for example, the optimum weight percentage to use inside the polypropylene for each flake thickness. The thicknesses under investigation were 100 nm, 350 nm and 750 nm thick glass flakes.

The glass flakes as supplied had no surface treatment. A surface treatment was therefore required for the glass flakes to bond better with the polypropylene matrix. As recommended by Glass Flake Limited the glass was coated using 3-aminopropyltriethoxysilane. This process is explained in section 5.1.

4.1.5 Chemicals

A number of different chemicals were used for the treatment of the fibres. These chemicals were purchased from Merck (Pty) Ltd and are as follows: distilled water, methanol, NaOH, acetone and acetic acid (glacial).

The 3-aminopropyltriethoxysilane of 98.07% purity was purchased from Southern Chemicals.

4.1.6 Release agents

Sheets of PTFE were used as release agents. These sheets were purchased from Quadrant Poly-Hi Solidur SA (Pty) Ltd. The sheets were 0.5mm thick and placed on either side of the composite charge. This allowed easy release of the composite from the mould. The sheets could be reused for multiple processing runs until the point where they became distorted.

After a few composite plates were manufactured it was found that the excess matrix, which flows through the gaps between the female and male parts of the mould, tended to stick to the walls of the ram. A further release agent was consequently needed to separate the mould without damaging the composite material. The release agent used for this was “ease release 200” which is manufactured by Mann Release Technologies and was purchased from AMT composites. This specific release agent was selected since its maximum operating temperature is higher than the maximum temperature inside the mould.

4.2 Equipment for manufacture

4.2.1 Manufacture of non-woven hemp fibre mat

It was not possible to acquire non-woven hemp fibre mats at the required density. Therefore methods of manufacturing the mats in-house needed to be determined. A few methods were investigated and the final method chosen was the water pipe method. This method will be discussed in detail in the following section. Explanations of the other methods are presented in Appendix A.

This method uses the concept of turbulence inside a pipe as well as at the inlet to randomly disperse the fibres to achieve a uniformly distributed non-woven mat. At the inlet, the water flows into the pipe illustrated by Fig. 14. This flow is similar to a fountain and creates vortices in the corners which mix the fibres. Once water is pumped into the pipe, the fibres are forced up to the top of the pipe and collected on the mesh. An image of the mesh is presented in Fig. 15.

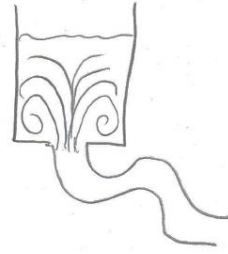


Fig. 14 Water flow in pipe (not drawn to scale)

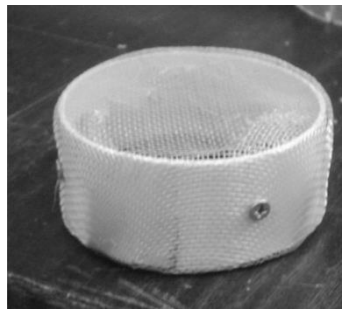


Fig. 15 Small scale test mesh to collect fibres

Initial testing on the water pipe method was done on a small scale due to the fact that a large scale test would require large piping which was not readily available. The rig for the small scale test is presented in Fig. 16.

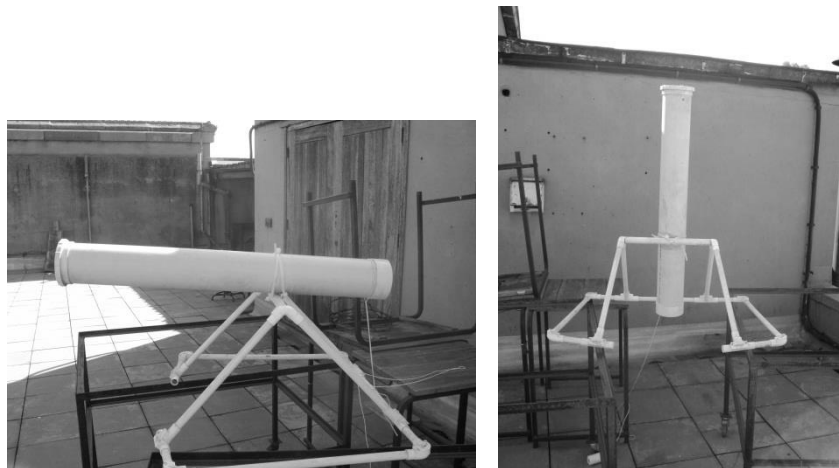


Fig. 16 Small scale test of water pipe. Left: vertical position \ Right: horizontal position

The manufacture of the mat was performed using the following approach: Firstly the fibres were pre-mixed in water. This is needed since the fibres cling to one another when dry and also tend to float. A jet of water at the base of the pipe was initiated while the pipe is standing vertically. The premixed

water/fibre mixture was immediately loaded into the pipe while the water was flowing. The water/fibre mix began to rise, forcing the fibres up towards the top of the pipe. The turbulence creates further mixing of the fibres. The constant mixing of fibres throughout the process and the continuous reduction in fibre volume fraction prevented agglomeration of the fibres. The mesh to catch the fibres was inserted some distance inside the pipe to avoid fibres being drawn towards the edge of the mesh at the pipe exit. This prevented a systematic variation in density as occurred when using compressed air. The top end was closed off and the pipe was flipped so that the originally open end was now at the bottom. The end was opened and the water was allowed to flow out at a flow rate caused by gravity. The reason for closing off the top end while flipping the pipe is that the water must flow straight down through the mesh. If the pipe is not perpendicular, the lateral component of the gravity vector could affect the distribution of the fibres.

The method yielded good results, as presented in Fig. 17, and so manufacture of the full scale equipment was justified. Drawings of this equipment are presented in Appendix B.

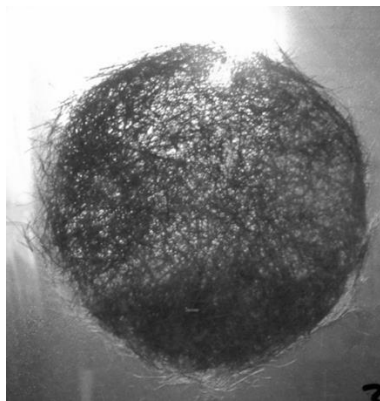


Fig. 17 Small scale test using an aerial density of 350g/m^2

The full size equipment was similar to that of the small scale test, however, due to the large pipe dimensions a clamp was placed around the pipe. Steel axles were welded onto either side of the clamp and then placed inside large bearings. This mechanism allowed the pipe to rotate. A photograph of the set-up is presented in Figs 18 and 19. The mesh to catch the fibres was inserted 150mm from the exit of the pipe.

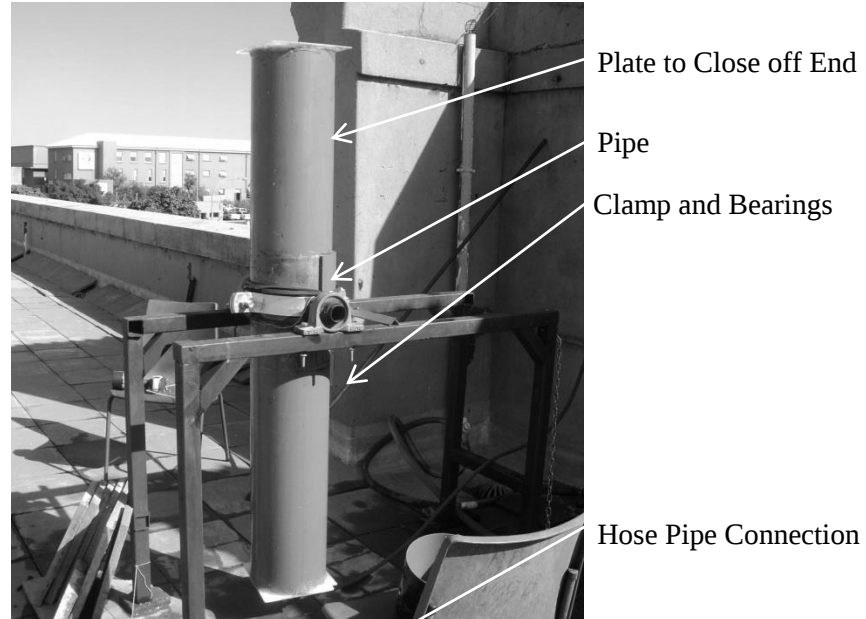


Fig. 18 Equipment for manufacturing the hemp fibre mats vertical



Fig. 19 Equipment for manufacture of hemp fibre mats horizontal

Commissioning tests using the full-scale equipment then commenced. A number of different hemp fibre densities were investigated. The uniformity of the mats increased with increasing densities. As a consequence, the highest density mat, 350 g/m^2 , was used in the remainder of this work. The resulting variation in fibre concentration was consistent with industrially produced mats. This is evident when comparing Figs. 20 and 21.



Fig. 20 Hemp fibre mat at a density of 350 g/m²

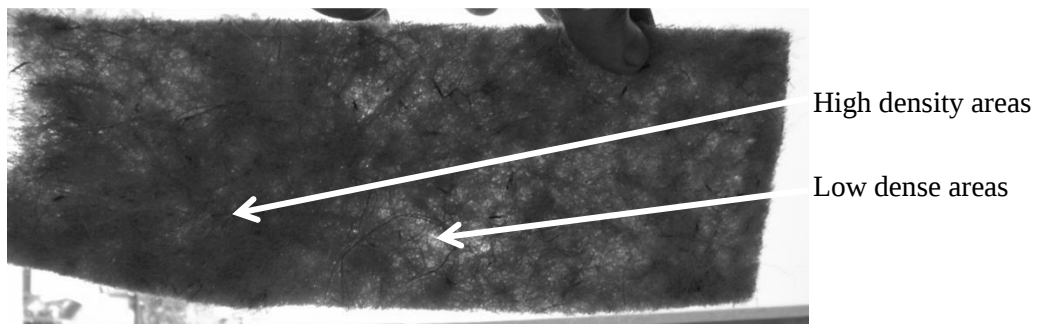


Fig. 21 Industrially manufactured non-woven fibre mat

4.2.2 Haake Polylab

The Haake Polylab consists of a controller which powers and controls two different pieces of equipment, namely a shear mixer and a single screw extruder with a die. The controller can only power one item at a time. The shear mixer is used to adequately disperse the fillers inside the polypropylene matrix and strips of this mixture are then extruded through the die onto a conveyer belt. The thickness and width of the strips vary according to the speeds of both the extruder and the conveyer belt. The strips can be used to manufacture the composite plates. The mixing process using the Haake Polylab is discussed in the experimentation section.

Time on the Haake Polylab used for this work was purchased from the Chemistry Department at the University of Johannesburg. The Rheomex is manufactured by Thermo Scientific and this specific model

is the Haake Polylab OS Rheo Drive 7 with the Rheomex and single screw extruder attachments. These attachments are presented in Figs. 22 and 23.



Single screw extruder

Extruding Die

Fig. 22 Haake Polylab with extruding die used in the present work

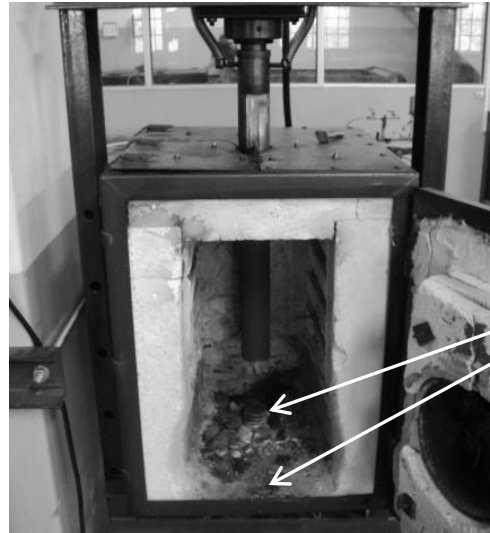


Shear mixer

Fig. 23 Haake Polylab with shear mixer attached used in the present work

4.2.3 Compression oven

Composite plates were manufactured using the compression moulding technique. The mould is heated to the melting temperature of the matrix and pressure is simultaneously applied. This is achieved using the oven presented in Fig. 24. The mould is placed in between the steel rods and the oven is turned on. The top rod is forced down using a hydraulic cylinder thereby compressing the mould. The oven simultaneously heats the mould to the required temperatures. The maximum force that can be applied by the piston is 20 tons.



Steel rod

Fig. 24 Compression oven

4.2.4 Mould

The mould was modeled according to moulds used previously for compression moulding composites and manufactured in the laboratory workshop. A new mould was manufactured since the current moulds available did not allow for composite sheets to be manufactured to the required size of 170 x 130 mm. These dimensions allowed for tensile test samples to be cut from one sheet and water absorption sample to be cut from a second sheet. A larger mould was investigated to allow all test samples to be cut from a single sheet, however, the dimensions of the oven limited the size of the mould.

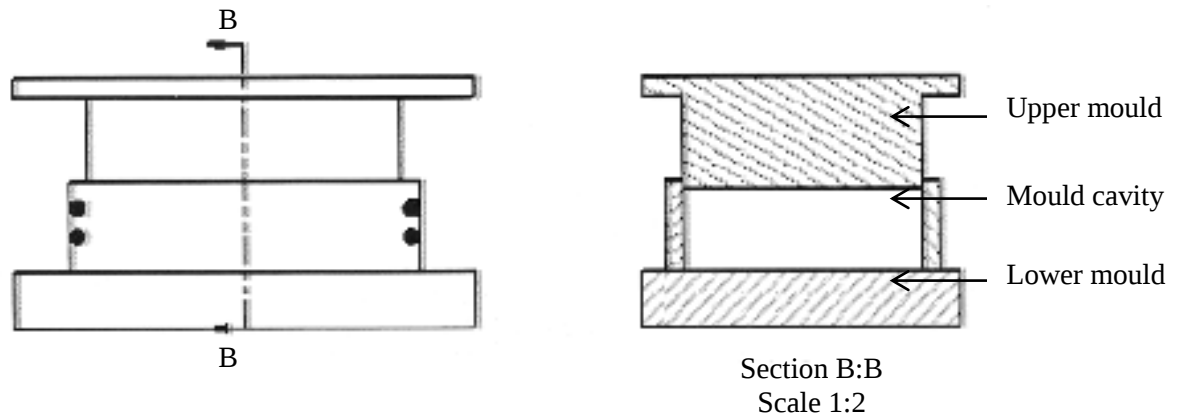


Fig. 25 Sectioned view of mould

The mould was manufactured using mild steel and was designed to have male and female parts which fit together as illustrated by the sectioned view in Fig. 25. The male part or upper mould is called the ram and is presented in Fig. 26. The female part or lower mould is assembled using a number of steel plates which create the cavity for the ram. The assembled female part is called the base and is presented in Fig.

27. Four tapped holes were placed in the corners of the ram to aid in the separation of the mould. Screws are inserted into the tapped holes and turned until they reach the base. The screws are turned further which forces the two parts apart.

The height of the ram and the thickness of the base need to be large enough to avoid large deflections caused by the forces applied. If the mould deflects more than 0.075 mm the thickness of the composite plates varies significantly. This value was calculated by analyzing the thicknesses of previous moulds manufactured in the laboratory and determining their maximum deflections for the forces used. The drawings of the mould can be found in Appendix A.



Fig. 26 Male part (ram)

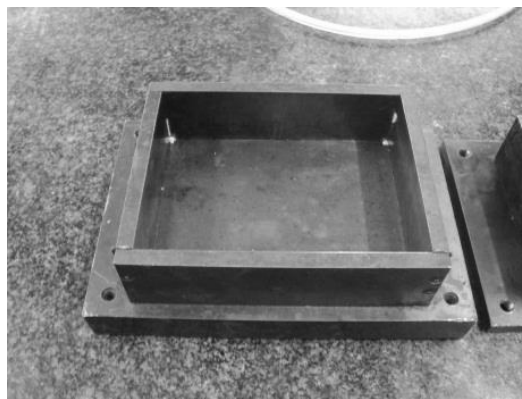


Fig. 27 Female part (base)

4.3 Testing equipment

4.3.1 Tensile testing

Tensile tests were carried out using the JJ Lloyd tensile testing machine at the University of the Witwatersrand. Specimens were mounted into rigid jaws. A 5 kN load cell was used to achieve the required resolution. An external extensometer was used to measure elongation with a gauge length of 50 mm (in accordance with ASTM D638). The test setup is presented in Fig. 28.

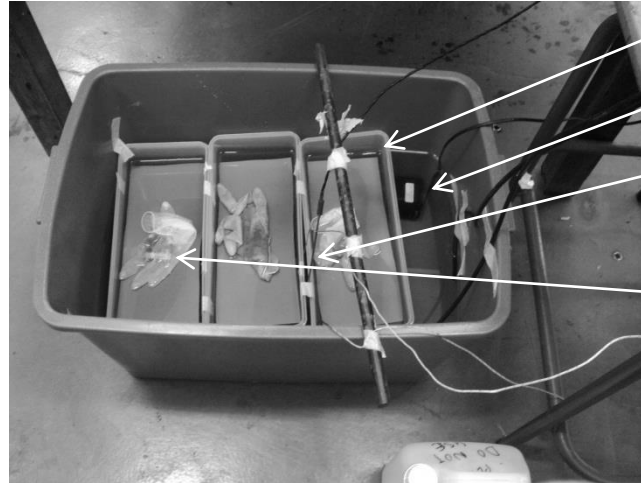


Fig. 28 Setup for tensile testing

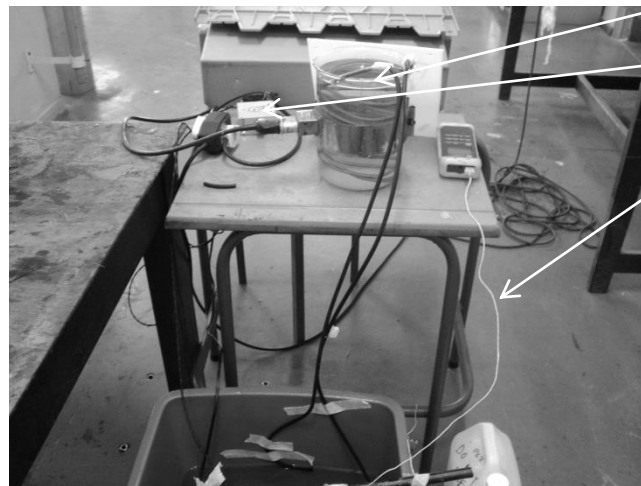
A MATLAB code was used which allowed for efficient data processing. This code was written by Rassmann [59]. The code is able to find the maximum stress prior to failure of the specimen as well as the elastic moduli of the samples, linear modulus and secant modulus. The requirement for two moduli is due to the stress-strain curves having linear and non-linear regions.

4.3.2 Water absorption testing

The equipment used for the water absorption tests is presented in Figs. 29 and 30. The test setup was designed by Rassmann [59]. The equipment consists of a pump, containers filled with distilled water, a thermocouple and controller, weights, rigid plastic sheets and a heating coil. The equipment maintains the water at a constant temperature of 23°C.



Containers filled with distilled water
Pump
Thermocouple for controller
Weights and plastic to stop samples floating



Heating Coil
Controller
Thermocouple to visually inspect temperature

Fig. 29 Water absorption test setup

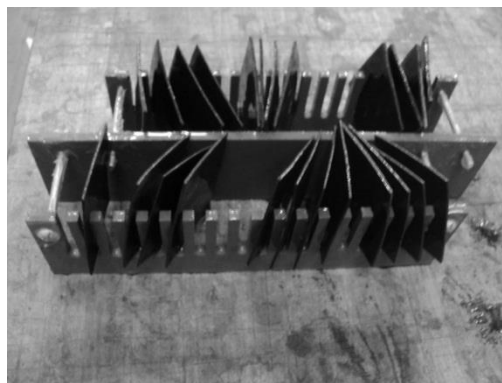


Fig. 30 Rack to hold samples

The samples were placed on racks manufactured from PVC. PVC was used since it does not oxidize in the presence of water like steel. This oxidation leads to impurities contaminating the distilled water. The

samples on the rack are presented in Fig. 30. The racks are held together using steel screws. The screws oxidize when exposed to water and they were therefore coated with silicone gel to limit this exposure.

As specified by the standard, the racks and samples were placed inside plastic containers filled with distilled water. The containers were immersed in tap water which was maintained at the required 23°C. The water temperature was controlled using a thermocouple and a controller. When the water temperature dropped below the specified limit the controller activated the pump which circulated the water through the copper heating coil located outside the tank. Once the temperature reached the upper limit the pump would turn off and the water temperature would stabilize. The water was maintained at the temperature required of $23 \pm 0.5^\circ\text{C}$. An external thermocouple was used to monitor the temperature.

The samples tended to float when immersed in the water, and therefore stiff plastic sheets and weights were placed on top of the samples to keep them submerged. The samples were in contact with the plastic sheet which is contrary to the standard, however, this was consistent across all samples being tested. The weights were kept inside latex gloves and the gloves were tied off to stop the weights from contaminating the distilled water. This is presented in Fig. 31.



Fig. 31 Weights used to keep samples submerged

4.3.3 Scanning electron microscope

Use of the SEM or scanning electron microscope was kindly offered by Professor Bhattacharyya in the Physics Department at the University of the Witwatersrand. This SEM has the capabilities of capturing images of non-conductive samples due to the low voltages used. This is essential when analyzing natural fibre composites. The SEM or field emission scanning electron microscope was manufactured by JOEL and has a model number of JSM-7001F

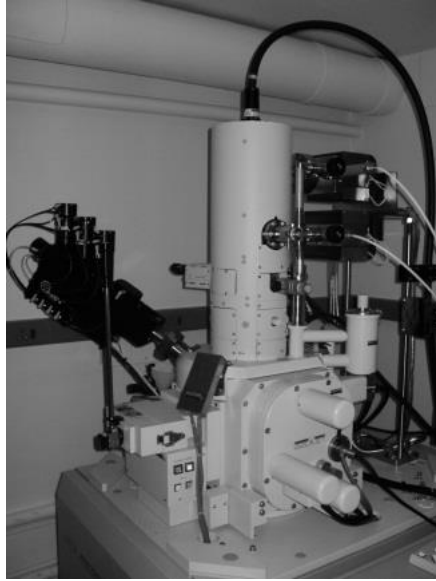


Fig. 32 SEM used in the present work

5 Experimentation

5.1 Procedure

5.1.1 Manufacture of composite plates

The process of manufacturing the composite plates involved a number of different stages:

1. Fibre treatment
2. Mat manufacture
3. Surface treatment of glass flakes
4. Mixing of filler and polypropylene
5. Compression moulding of composite plates

These stages are discussed in more detail in the following sections.

5.1.1.1 Fibre treatment

The fibre treatment was done using the method described by Asumani *et al.* [1]. Even though hemp fibres are used instead of kenaf the same technique was used. The efficiency of the process can therefore be assessed for hemp fibres. The treatment was performed as follows:

1. The hemp fibre was cut into lengths of approximately 5mm using scissors.
2. A 6% NaOH solution by mass was prepared using distilled water. This concentration was chosen since Asumani *et al.* [1] achieved the best results using this concentration.
3. The hemp fibre was immersed in the NaOH solution for 24 hours at a temperature of 45°C.
4. The fibres were then washed using running tap water. This was done by collecting the fibres in a sieve and allowing the running tap water to wash the fibres.
5. The fibres were then immersed in distilled water containing 1% by volume acetic acid to neutralize the remaining NaOH molecules.
6. 5% by mass of silane (relative to the dry mass of fibres being treated) was diluted in a 50% aqueous solution of methanol.
7. The fibres were immersed in this solution for 4 hours at 28°C.
8. The pH of the solution was maintained between 4 and 5 using acetic acid. The pH was measured using a PT-10 Sartorius pH meter.
9. Finally the fibres were washed using distilled water and then dried in an oven for 12 hours at 45°C.

5.1.1.2 Mat manufacture

1. The correct weight of fibres for the desired density is measured out. The area of the mesh used for this work was 0.038m^2 and so 13.3g of fibres give an average density of 350g/m^2 .
2. The fibres are pre-mixed in a container of water to avoid agglomeration and floating.
3. A jet of water is initiated with the pipe standing vertically.
4. The water/fibre mix is loaded into the pipe.
5. The mesh is inserted and held in place by a screw.
6. The jet of water is stopped, 3 minutes after the water begins to overflow from the top of the pipe.
7. The top end is closed off.
8. The pipe is inverted so that the originally open end is now at the bottom.
9. The end is opened and the water is allowed to flow out of the pipe.
10. The mesh with the fibre mat on its upper surface is removed.
11. The fibre mat is dried overnight in the oven at 70°C .

5.1.1.3 Surface treatment of glass flakes

The glass flakes need to be treated with silane to bond better with the polypropylene matrix. This process of treating the flakes with 3-aminopropyltriethoxysilane was performed as follows.

1. A solution of 6%, by volume, 3-aminopropyltriethoxysilane in acetone is prepared. This concentration was recommended by Glass Flake Ltd.
2. The glass flakes are sprinkled into a constantly mixed silane solution.
3. The solution is mixed for no more than 15 minutes.
4. The excess silane solution is siphoned off using a medical syringe.
5. The remaining glass flakes are dried in the oven at 80°C .
6. The final product is a cake of glass flakes which readily breaks apart.

5.1.1.4 Mixing of filler and polypropylene

Mixing of the polypropylene matrix and filler material was performed using the Haak Polylab at the Chemistry Department at the University of Johannesburg.

1. The shear mixing attachment is connected to the controller.
2. The machine is turned on and the temperature is allowed to reach the required internal temperature of 210°C and a speed of 60 rpm.

3. The filler is weighed out to achieve the weight percentage required. The shear mixer can handle a maximum of 50g of polypropylene at a time and so the quantity of filler must be determined using this value.
4. 50g of polypropylene balls are slowly introduced into the mixing chamber.
5. The filler is slowly introduced into the mixing chamber.
6. The ram is closed to stop any polypropylene or filler from leaving the mixing chamber.
7. The components are mixed for 10 minutes.
8. The machine is stopped.
9. The chamber is opened.
10. The polypropylene/filler mixture is removed from the wall and the screws.
11. The polypropylene/filler mix is cut into small pieces while hot.
12. The shear mixer is removed and the single screw extruder and die are connected to the controller.
13. Pieces of polypropylene/filler are introduced into the extruder and forced through the die onto the conveyer belt.
14. The strip of polypropylene/filler is collected.
15. The polypropylene/filler strips are cut to the required length.

5.1.1.5 Manufacture of composites plates

The manufacturing procedure is based on that explained by Asumani *et al* [1]. The development of the final manufacturing process of the composite plates is presented in Appendix C. The final process is described below which makes use of the compression moulding technique.

Preparing the mould:

1. The fibre mat is weighed.
2. The mass of matrix required to achieve the required fibre weight fraction is determined for the fibre mat being used. Since the fibre mats are similar in mass, the mass of matrix required for the composite plate having a 15% fibre weight fraction is higher than the mass required for the composite plate having a 30% fibre weight fraction. The difference in mass of matrix results in the 15% fibre weight fraction composite plate having a greater thickness than the 30% fibre weight fraction composite plate for the same area.
3. This mass of matrix strips is measured out and 10-15% is added for matrix losses.
4. “Ease release 200” is applied to the mould as specified by the manufacturer.
5. One sheet of PTFE release film is placed inside the female part of the mould.

6. Half the mass of the polypropylene matrix material is placed inside the mould. The strips of matrix material should be placed overlapped in end view in the same way that bricks are laid. Details are illustrated in Appendix C.
7. The fibre mat is placed on top of the polypropylene matrix strips.
8. The rest of the strips of matrix material are placed using the same brick laying format used in step 6.
9. The second sheet of PTFE release film is placed on top of the composite charge.
10. The mould is closed.
11. Spacers are placed as required.

Table 3 Table of spacer thickness vs. fibre weight fraction

Fibre weight %	Spacer thickness
30%	0mm
25%	2.55mm
20%	3.55mm
15%	4.05mm

Production process:

1. The mould is placed in the oven and the charge is compressed with a pressure of 200 bar as measured by the pressure in the hydraulic cylinder. This corresponds to a pressure of approximately 4.5 bar on the charge. The pressure is applied for five minutes with the oven off. This flattens the composite charge.
2. The oven is turned on and set to 275°C.
3. The pressure is dropped to 100bar in the hydraulic cylinder which corresponds to approximately 2.5 bar on the charge.
4. After an elapsed time of 30 minutes the pressure rises as high as 160 bar (3.5 bar on charge) due to the constrained thermal expansion of the mould and other steel components under compression. The pressure is reduced to 60 bar (1.3 bar on charge) over a period of 5 minutes to avoid the composite charge experiencing a rapid change in pressure.
5. After an elapsed time of 65 minutes the oven is turned off and the door is opened. The fan is turned on and the mould is allowed to cool.

6. After an elapsed time of 95 minutes the pressure has dropped on its own to between 5 bar (0.1 bar on charge) and 10 bar (0.2 bar on charge) as the mould cools down. The mould can now be released and removed from the oven.
7. The mould is cooled for a further 30 minutes outside the oven with the fan blowing on it.
8. The mould is then opened and the composite plate is removed.
9. The composite plate is weighed to determine the average fibre weight fraction.
10. The mould is then placed in cold water to cool it completely.
11. The mould is then dried and can now be used in the manufacture of another plate.

5.1.2 Tensile testing

The tensile tests were conducted according to ASTM D638 using the JJ Lloyd tensile testing machine with a 5 kN load cell. An external extensometer was used to measure elongation with a gauge length of 50 mm (in accordance with ASTM D638).

The samples used were of type I/II. The samples were cut from the composite plates into rectangles and then shaped into the required dog bone shape using a specially designed jig. The samples were placed on a mould and the mould was then passed along a diamond coated sanding head to shape the samples to the required size. Some of the polypropylene melted during the process and so the samples were cleaned up using a craft knife as well as water paper.

5.1.3 Water absorption testing

The water absorption test was conducted according to ISO R62 with slight modifications as described by Rassmann *et al.* [4].

The water absorption samples were first cut from the composite plate into a square of roughly 50x50 mm dimension. They were then sanded down using a belt sander and polished using water paper to achieve the required dimensions.

The specimens were placed in an oven at 50°C for 12 hours and then allowed to cool in the presence of silica gel for a further 12 hours. The specimens were measured and then submerged in distilled water maintained at 23°C.

After 1 hour of immersion the specimens were removed and measured. A second measurement was taken 23 hours after the first. From then on measurements were taken every 24 ± 1 hours. Once the samples began to near saturation, measurements were taken 48 ± 1 hours apart and then even 72 ± 1 hours apart.

Measurements took place in the following fashion. Samples were removed from the water and any surface water remaining was wiped away using paper towel. The samples were out of the water for 5 minutes prior to performing any measurements in order for surface water to evaporate. The samples were then measured using a scale accurate to 0.0001g. The sample dimensions were then measured namely the length, breadth, centre thickness as well as thickness 7.5mm from the edge. The length and breadth were measured using a vernier caliper accurate to 0.05mm and the thickness was measured using a micrometer accurate to 0.01mm.

5.2 Precautions

5.2.1 Fibre treatment

- Use a well-ventilated room.
- Wear a lab coat, gloves and a face mask.
- Keep the fibres in a sieve when rinsing as this helps to avoid losing fibres.
- Ensure that the fibres are immersed in the solution at all times. The fibres tend to float to the surface and so a mesh was placed over them to keep them submerged.
- Dispose of all chemicals properly.

5.2.2 Mat manufacture

- Ensure the mesh is placed inside the pipe before the water begins to overflow otherwise fibres will be lost.
- Ensure the top is sealed when rotating the pipe as any leakage could lead to fibre loss.
- Care must be taken when removing the mesh so as to avoid disturbing the fibre mat that has just been created.
- Wear clothing that can get wet as this process leads to a lot of water loss.
- Care must be taken when loading the fibre-water mix as any spillage will lead to a loss of fibres.
- Ensure the pipe is perpendicular to the ground when releasing the water from the pipe to ensure a uniform mat density.
- Once the fibres have dried, cut the required mat out of the area with the most consistent density. Even though the manufactured mat is random it is not 100% uniform and so there are areas of higher fibre concentration and areas of lower fibre concentration.

5.2.3 Glass flake treatment

- Do not exceed the decomposition temperature of 3-aminopropyltriethoxysilane (217°C) during drying.
- Do not mix for more than 15 minutes otherwise the flakes will begin to reduce in particle size.
- Ensure all the flakes are submerged in the solution.

5.2.4 Mixing of filler and polypropylene

- Ensure the compressed air and the water flow to the Haake PolyLab is connected.
- Introduce both the polypropylene and filler slowly into the shear mixing chamber as to avoid losses as well as agglomerations.
- Avoid coming into contact with the mixing chamber as the temperature is over 200°C
- Clean the machine using pure polypropylene before changing between any mixture for both extrusion and shear mixing.
- Operate the extruder using pure polypropylene prior to any sample runs to properly set the speed of the conveyor belt and the speed of the extruder. These speeds determine the dimensions of the resulting strips.
- Do not put large pieces of polypropylene/filler mix into the extruder as it may get stuck.

5.2.5 Manufacture of composites

- Dry the mould thoroughly. Any water left on its surface could lead to bubbles being formed in the composite plate being manufactured.
- Do not touch the mould after manufacture without thermally resistant gloves until it has cooled.
- Lay the strips of matrix material evenly over the mould so as to avoid uneven distribution of the matrix in the final composite plate.
- Remove the composite plates gently to avoid large distortions.
- Ensure the steel compression rods are straight and apply force through the centre of the mould as misalignment can lead to fibres being pushed to one side of the mould. Misalignment can also lead to tearing of the fibres inside the fibre mat.

5.2.6 Tensile testing

- Ensure that the test specimen is aligned straight in the grips.
- Ensure that the grips are tightly closed to avoid slippage of the specimen.

- Ensure that the external extensometer as well as the load cell is zeroed at the beginning of every test.
- Ensure the test specimen breaks in the testing area before using the measured results in any analysis.
- Ensure the extensometer is taking readings before starting a test.

5.2.7 Water absorption testing

- Ensure the containers are filled with distilled water at all times.
- Due to the logarithmic nature of the curve, a large number of readings need to be recorded at the beginning of the test.
- Keep the water level in the external container high enough for the pump to be able to operate.
- Allow the scale to settle before taking readings.
- Wear gloves so as to avoid contaminating the samples.
- Take readings as soon as possible so as to avoid excess evaporation of absorbed water.
- When using the vernier caliper take care not to cut into the specimen being tested.
- Take care to measure the thickness at a set point on the same sample due to the varying thickness of individual samples.

6 Observations

A number of different observations were made during the manufacturing process of the composite plates. These observations are relevant to the explanation of some of the results.

6.1 Glass flake treatment

The glass flakes acquired from Glass Flake Ltd did not have any surface treatment applied to them and hence needed to be treated. After the silane treatment it was apparent that the flakes changed from a white powder as presented in the left hand image of Fig. 33 to a yellow powder that tended to agglomerate together as presented in the right hand image of Fig. 33. The agglomerations are formed during the drying of the glass and tend to crumble very easily.



Fig. 33 Left: Untreated glass flakes \ Right: treated glass flakes

6.2 Fibre treatment

Two observations were made during the treatment of the fibres. Firstly a colour change between the treated and untreated fibre is clearly seen in Fig. 34. Secondly it is also noticed that the treated fibres tend to agglomerate more than untreated fibres.



Fig. 34 Left: untreated fibres \ Right: treated fibre

6.3 Extruding polypropylene/filler mix

An extruded strip of polypropylene and carbon nanotube is presented in Fig. 35. It was noticed that the sheet width and thickness varied slightly along its length. This variation does not affect manufacture of the composites since the amount of matrix material is determined by mass.



Fig. 35 Extruded polypropylene and carbon nanotube sheets

6.4 Agglomeration of carbon nanotubes and glass flakes

Some of the extruded strips of polypropylene matrix material containing carbon nanotubes had areas where the carbon nanotubes appeared to agglomerate. This indicates that the carbon nanotubes were not perfectly mixed into the polypropylene using the shear mixing process. This was not the case for every strip, however, it does have an effect on the overall results. It was also observed that some of the glass flake strips had glass flake agglomerations. This was not as prominent as in the strips containing carbon nanotubes but was still observed. Images of carbon nanotube and glass flake agglomerates are presented in Fig. 36. These images are obtained using an optical microscope.

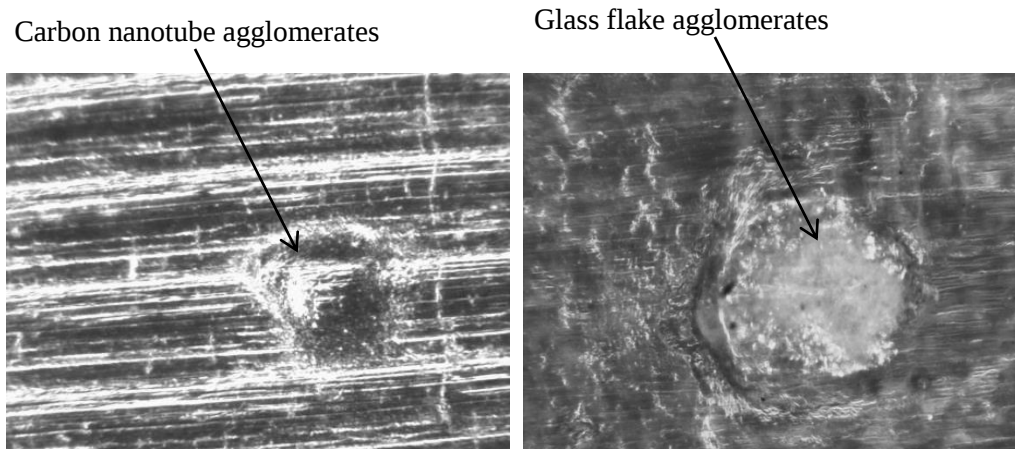


Fig. 36 Filler agglomerates in strips. Left: carbon nanotubes at 112.5x magnification \ Right: Glass flakes at 112.5x magnification

6.5 Wetting-out of fibres

Fig. 37 shows a matrix layer on either side of the fibres. This layer acts as a barrier between the fibres and the surroundings. One major obstacle when manufacturing natural fibre composites is the wetting out of the fibres. Since the matrix penetrates through the laminate from the surface, it is necessary to ensure that it flows all the way to the centre of the laminate and fully wets-out the fibres at this point. From Fig. 37 it is not apparent that the fibres have been completely wet-out and so SEM images were needed of the interior of the plate.



Fig. 37 Side view of manufactured composite

SEM images of the interiors of the plates made from pure polypropylene and treated fibres are shown in Figs. 38 to 41 for the fibre weight fractions of 15%, 20%, 25%, 30%, respectively. It can be observed from these images that there is matrix surrounding the fibres indicating that the matrix did indeed flow into the interior of the plate and fully wet-out the fibres.

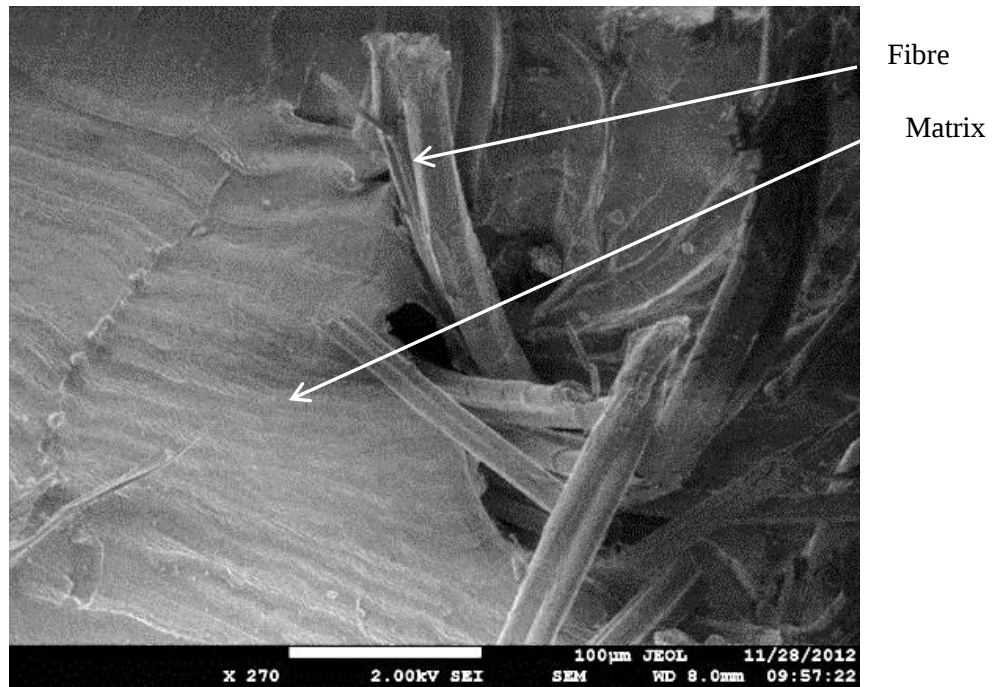


Fig. 38 SEM image of the sectioned view of the composite plate having a fibre weight fraction of 15%

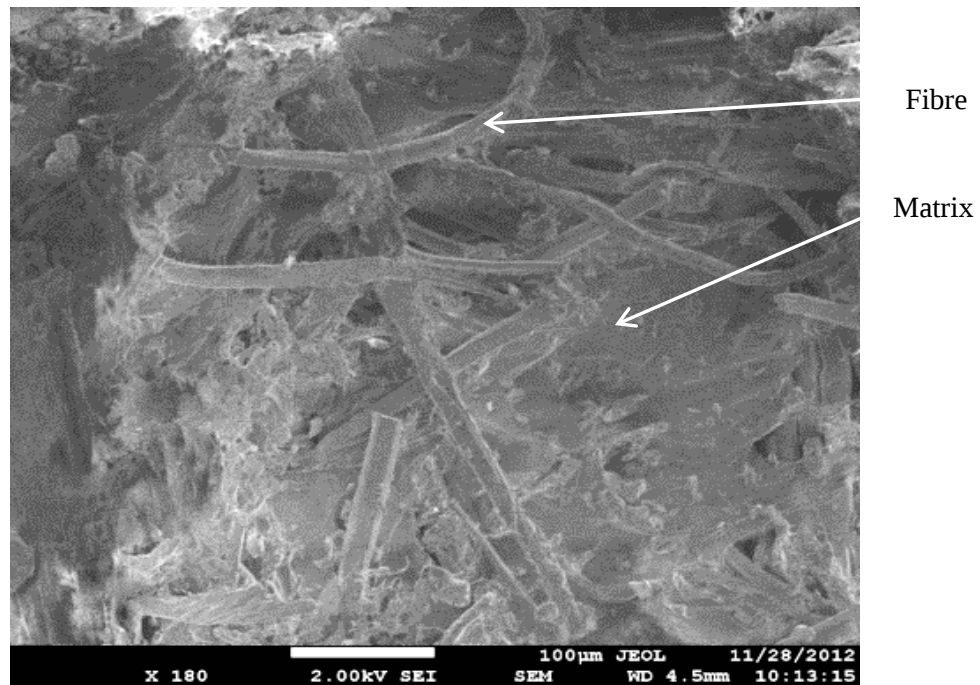


Fig. 39 SEM image of the sectioned view of the composite plate having a fibre weight fraction of 20%

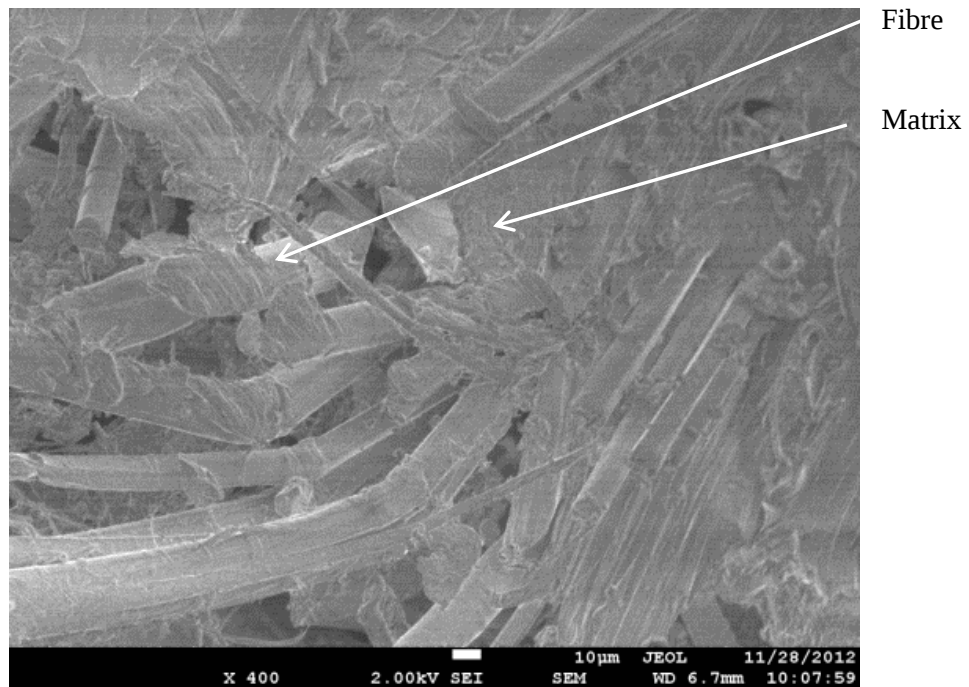


Fig. 40 SEM image of the sectioned view of the composite plate having a fibre weight fraction of 25%

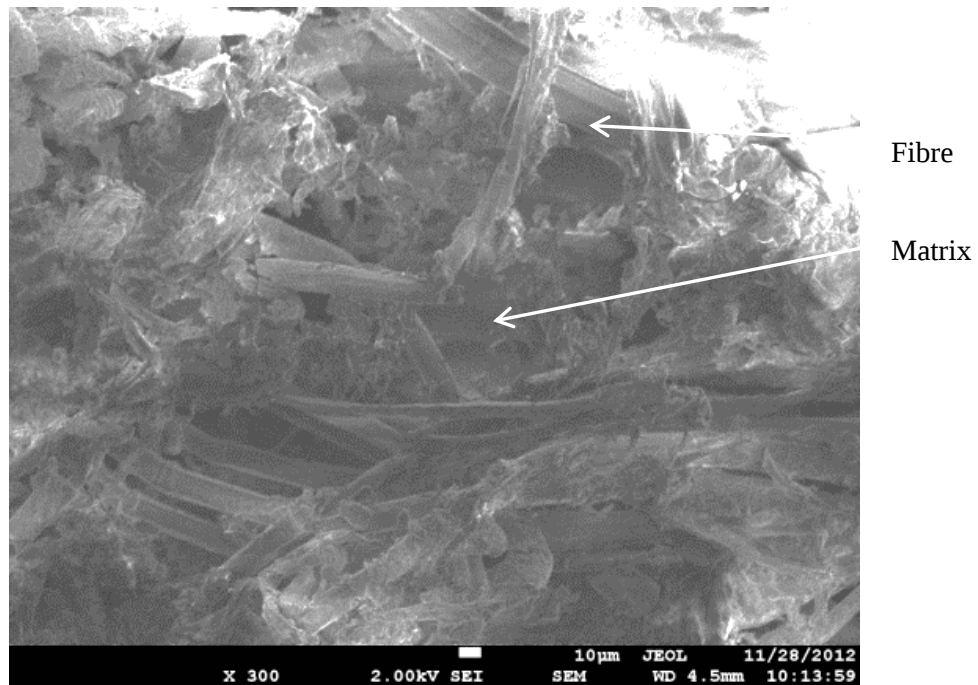


Fig. 41 SEM image of the sectioned view of the composite plate having a fibre weight fraction of 30%

Due to the limited amount of time available on the SEM, images for the composite plates containing fillers could not be obtained. An image using an optical microscope is, however, presented in Fig. 42. This image of a sectioned view of the 30% carbon nanotube and polypropylene composite clearly shows matrix present in between the fibres at the interior of the composite indicating the wetting out of the

fibres. The images for the other fibre weight fractions and for the glass flake filler are similar to that presented. It is concluded that the manufacturing process used did fully wet-out the fibres in the interior of the plate.

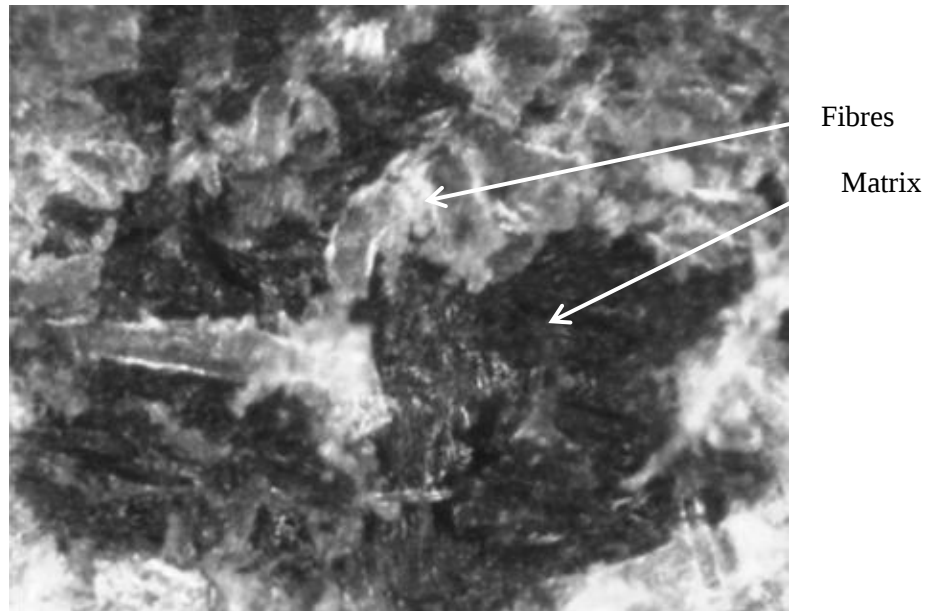


Fig. 42 Sectioned view of carbon nanotube and polypropylene composite at 112.5x magnification

6.6 Fibres on surface of plates

It was noticed that some fibres were exposed at the surface of the plate after manufacture. This issue became prominent during the water absorption test when after some time in the water the fibres began to swell. The effect was noticed to be more prevalent for composites having higher fibre weight fractions since there is less matrix present. Using the current manufacturing process this could not be avoided. Images of plates with both 15% and 30% fibre weight fraction of untreated fibres are presented in Fig. 43. It is clear that higher fibre content leads to more fibres being exposed at the surface of the composite.

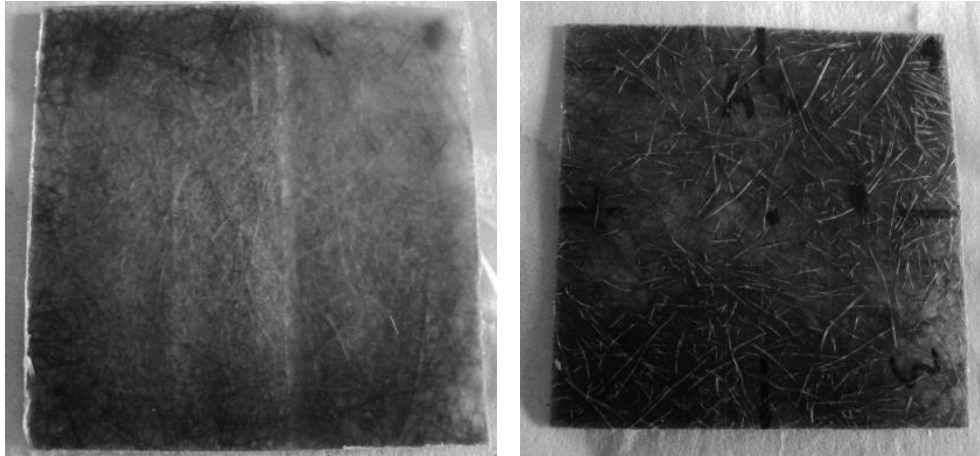


Fig. 43 Left: 15% untreated fibres \ Right: 30% untreated fibres

This phenomenon was seen for all the composites considered i.e. using treated as well as non-treated fibres and with or without fillers.

Interestingly, the treated fibres were more likely to be exposed than the untreated fibres. This effect can be observed in Fig. 44. This figure shows images corresponding to 30% fibre weight fraction for both the untreated fibres and the treated fibres.

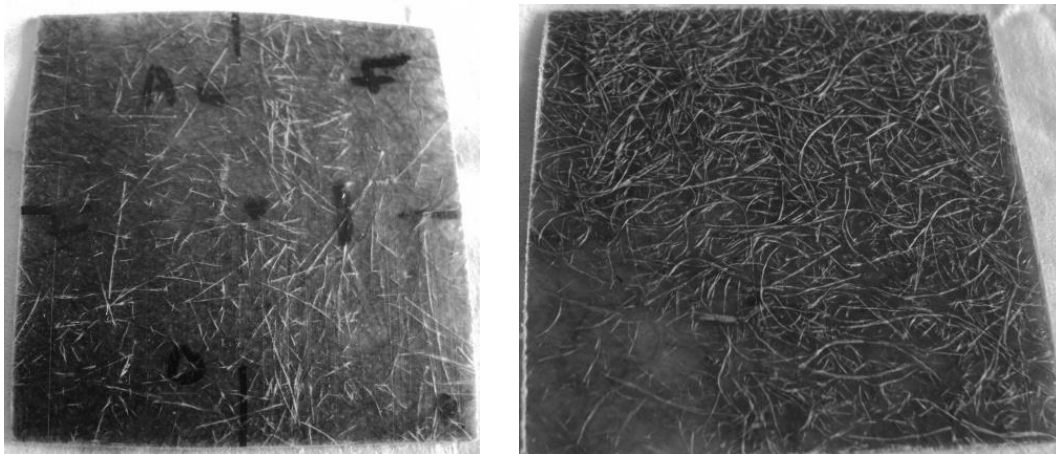


Fig. 44 Left: 30% untreated fibres Right: 30% treated fibres

Images of water absorption specimens cut from the same composite plate containing untreated fibres at a fibre weight fraction of 25% are presented in Fig. 45. These images display sample 4 and sample 3. It is evident that samples cut from the same composite plate have different amounts of fibres exposed at the surface of the plate. This is due to the uneven distribution of fibres in the fibre mat.

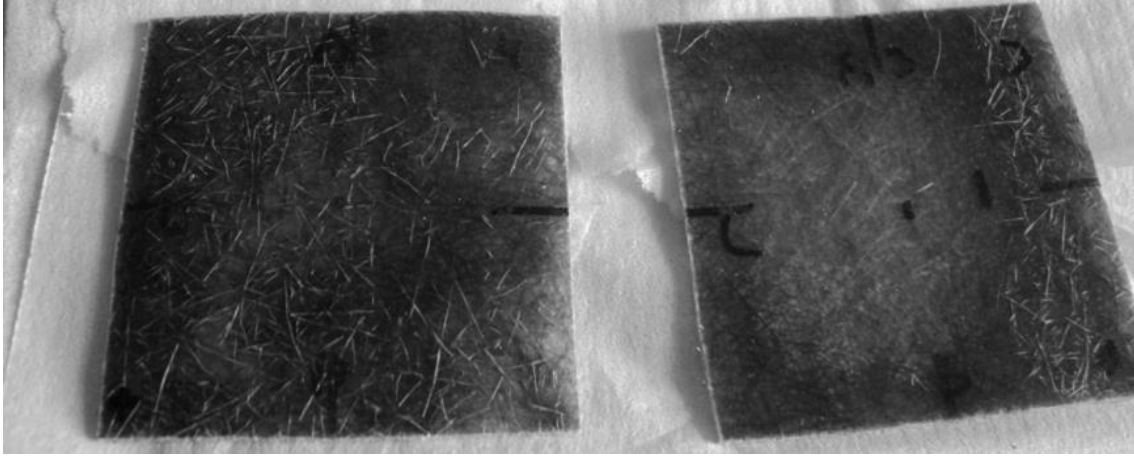


Fig. 45 Left: Sample 4 25% untreated fibres \ Right: Sample 3 25% untreated fibres

6.7 Sample distortion

Some of the manufactured plates were found to be slightly distorted. An example is presented in Fig. 46. This curvature did not seem to disappear with a longer cooling time. It is believed that the curvature results from the slight asymmetries in the plate lay-up. Variations in the fibre arrangement affect the in-plane properties of the plate and possibly also affect the through-thickness position of the fibre mat in the plate. Such asymmetries would be expected to have more impact in thinner plates due to their lower bending stiffness. This is, in fact, the case. The plates with a 15% fibre weight fraction (which are thicker than plates with a 30% fibre weight fraction) were less distorted than plates with a 30% fibre weight fraction. Some samples distorted more than others and the image presented in Fig. 46 is of a composite with a high degree of distortion.

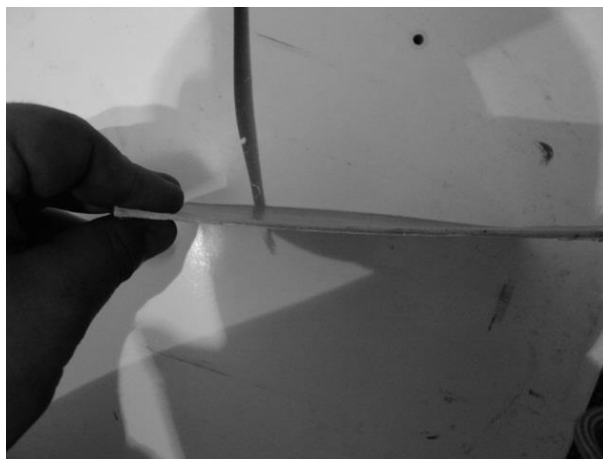


Fig. 46 Distortion of composites

6.8 Tensile testing

During the tensile tests, all the specimens broke suddenly without the phenomenon of necking occurring. All specimens failed in the test region of the dog bone shape.

6.9 Data processing

The data for the tensile testing was processed using a MATLAB code written by Rassmann [59]. This MATLAB code was designed specifically for the JJ Lloyd tensile testing machine at the University of the Witwatersrand and can be found in Appendix D. The code follows the sample calculations and outputs stress, strain, secant modulus as well as linear modulus. The processed data can be found in Appendix E.

Sample calculations for the fibre weight fraction, tensile strength, elastic modulus, change in mass and change in dimensions are presented in Appendix F.

7 Results

The following results are presented using a coded system. Each sample type has four different variations and hence the code is made up of four parts. The first variation refers to the fibres themselves, UF (untreated fibre) and TF (treated fibres). The second variation refers to the filler used; N (none), GF (Glass flakes) and CNT (Carbon nanotubes). The third variation refers to the amount of filler used and is expressed as a weight percentage of the total matrix weight. The final variation is the fibre weight fraction; 15%, 20%, 25% and 30%. These percentages are approximate values and 15%-16.5%, 20%-21.5%, 24.5%-26.5% and 29%-31% would be more accurate. This variation was caused due to a loss of matrix from the mould in the form of flash. Some samples lost more matrix than others and this led to a variation in fibre weight fraction.

An example of the code used would be as follows: UF-N-30% refers to the sample that used untreated fibres and contains no filler at a fibre weight fraction of 30%. A second example would be TF-GF350nm-8%-30% refers to the sample that used treated fibres containing 350 nm thick glass flakes at a concentration of 8% relative to the matrix and a fibre weight fraction of 30%.

7.1 Tensile strength

In the following data, error bars represent one standard deviation. A minimum of five samples were tested for each configuration of the composite. Fig. 47 compares the tensile strength for composites manufactured using treated and untreated fibres. These composites did not contain any filler. Fig. 48 compares the tensile strength for treated fibre composites containing 2% carbon nanotubes against treated fibre composites containing no fillers. A 2% concentration of carbon nanotubes corresponds to the middle of the three concentrations tested. Fig. 49 compares the tensile strength of the treated fibre composites containing 8% glass flakes with a thickness of 350 nm with that of treated fibre composites containing no filler. This concentration of glass flakes was recommended by the supplier as the optimum for polypropylene matrix. Fig. 51 shows the tensile strength of treated and untreated kenaf and hemp fibre composites at a fibre weight fraction of 30%.

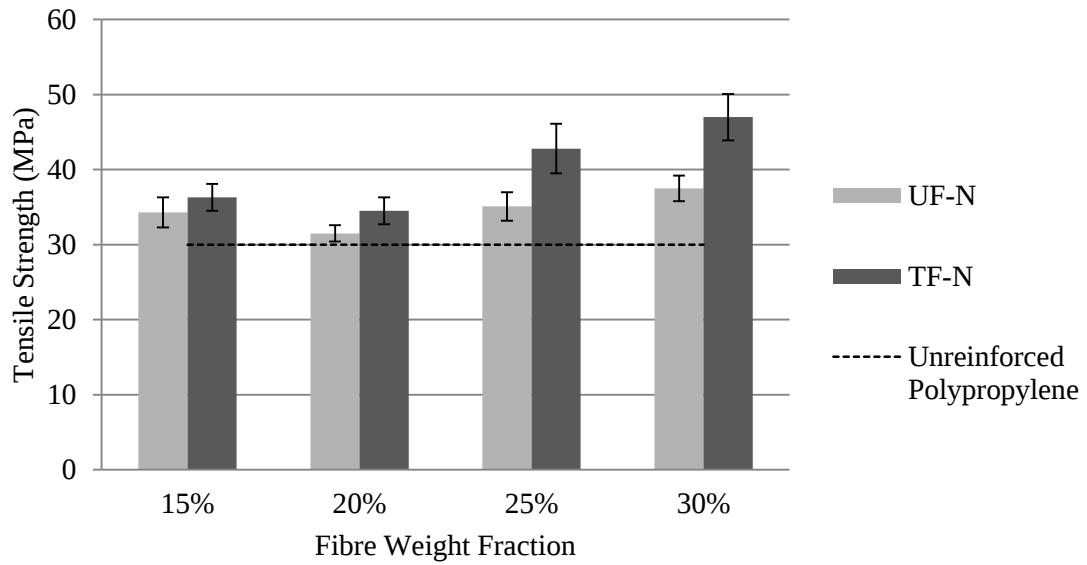


Fig. 47 Effects of fibre treatment on tensile strength

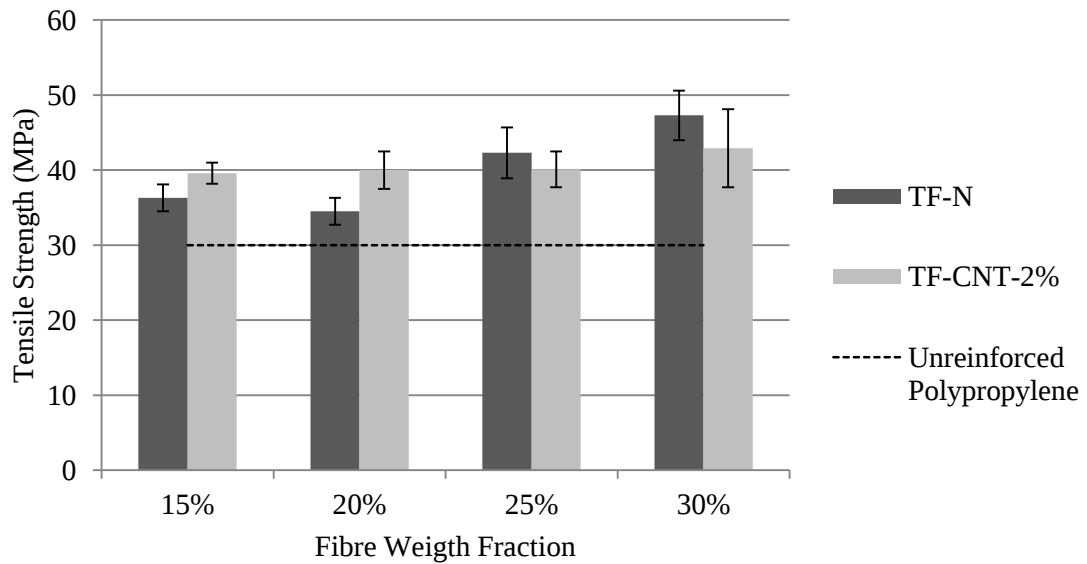


Fig. 48 Effect of 2% carbon nanotubes on tensile strength

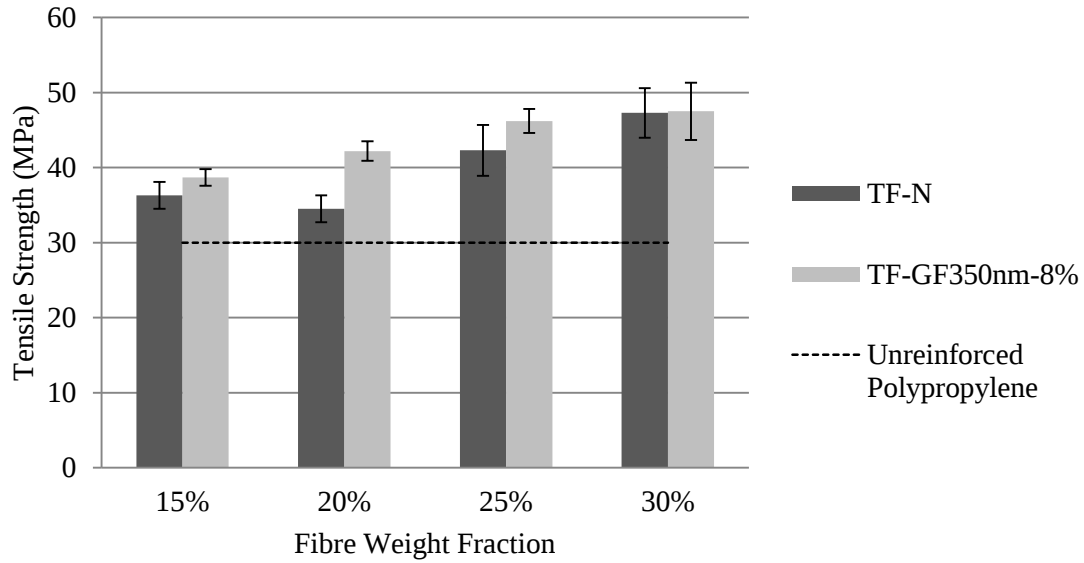


Fig. 49 Effect of 8% 350nm glass flakes on tensile strength

The composite containing 30% fibre weight fraction seems to achieve the best results and so the variations in the filler content are compared for this fibre weight fraction. Fig. 50 compares the tensile strength of the composites having a fibre weight fraction of 30%. The horizontal dotted line refers to the strength of the TF-N composite at a fibre weight fraction of 30%. Fig. 51 shows the tensile strength of treated and untreated kenaf and hemp fibre composites at a fibre weight fraction of 30%.

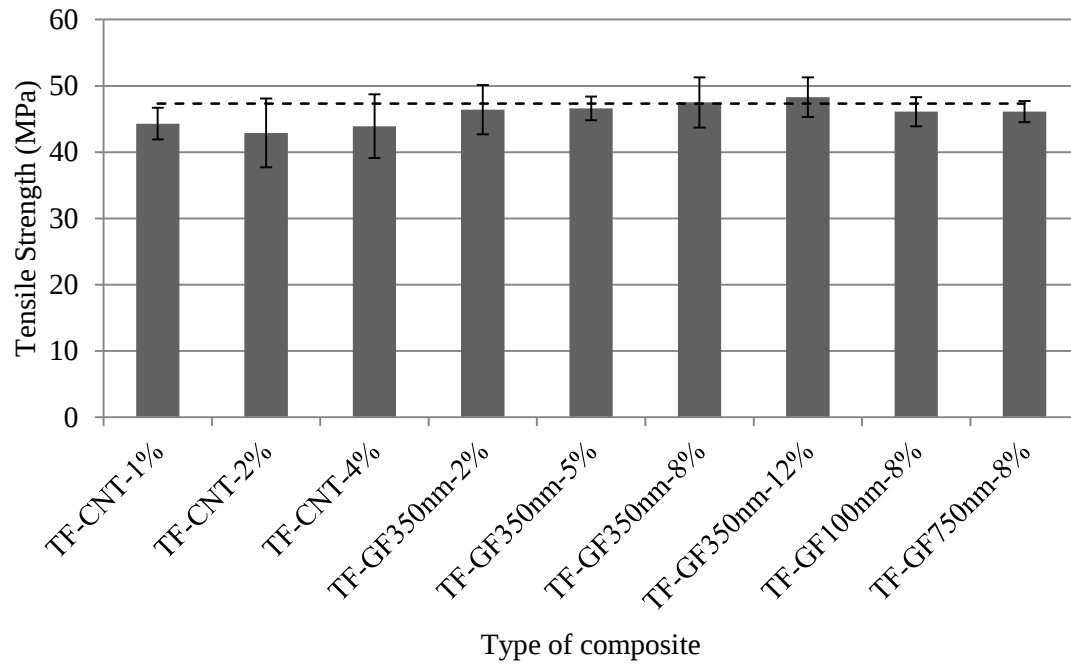


Fig. 50 Effect of variation in filler content on tensile strength for a fibre weight fraction of 30%

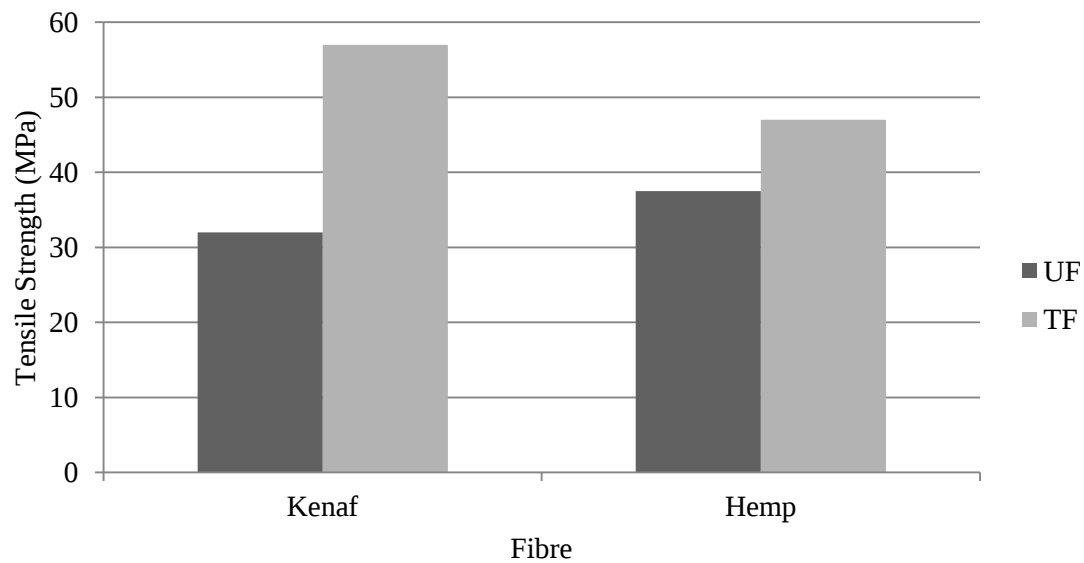


Fig. 51 Comparison of the tensile strength for kenaf fibre and hemp fibre composites at a fibre weight fraction of 30%

7.2 Tensile modulus

The linear and secant moduli were also investigated. The following graphs show the stress–strain curve for all composites configurations at a fibre weight fraction of 30%. All other fibre weight fractions followed a similar trend.

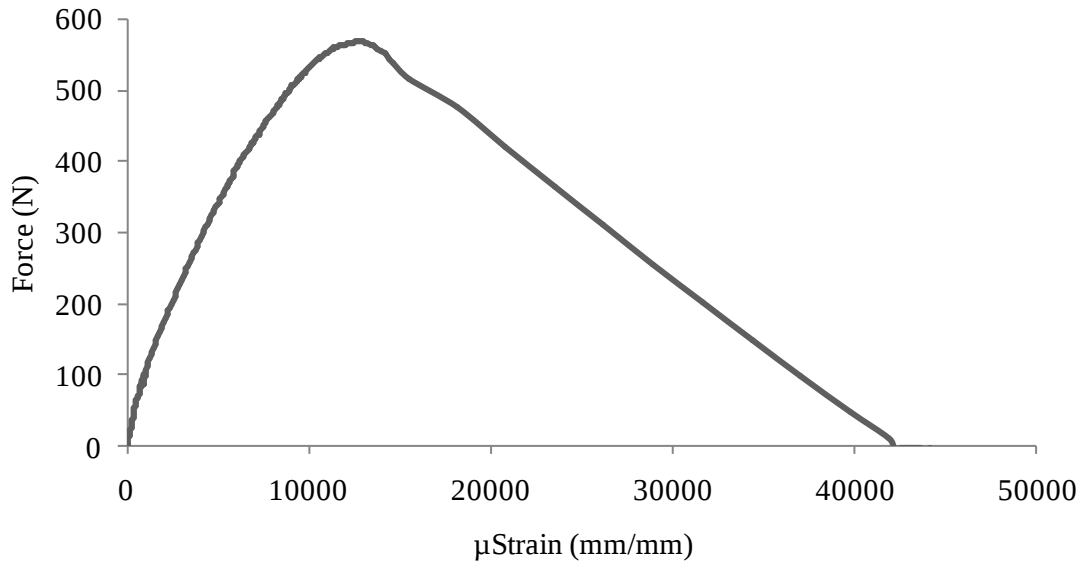


Fig. 52 Stress vs Strain for untreated fibres at a fibre weight fraction of 30%

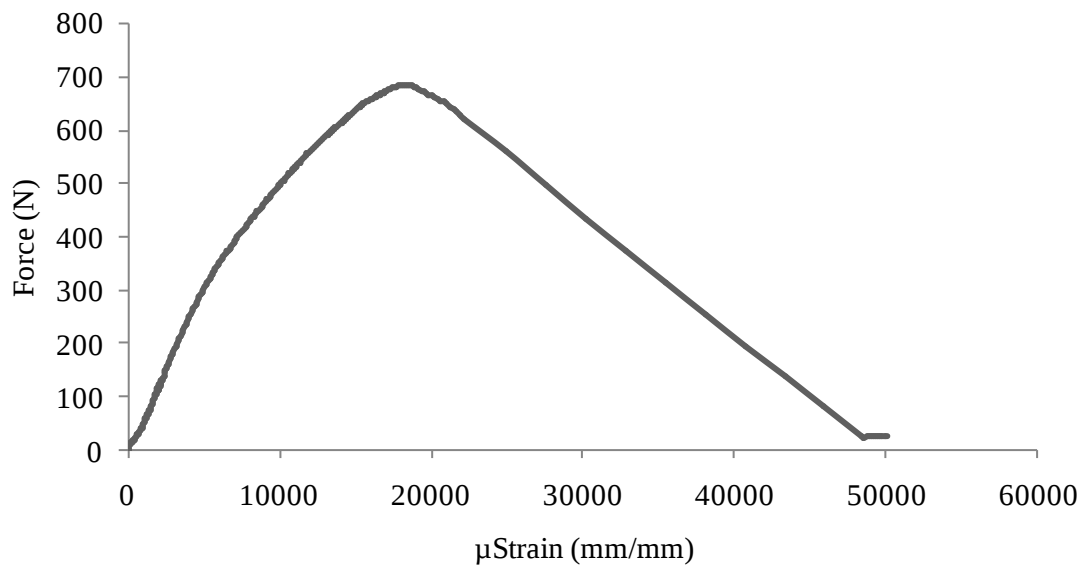


Fig. 53 Stress vs strain for treated fibre at a fibre weight fraction of 30%

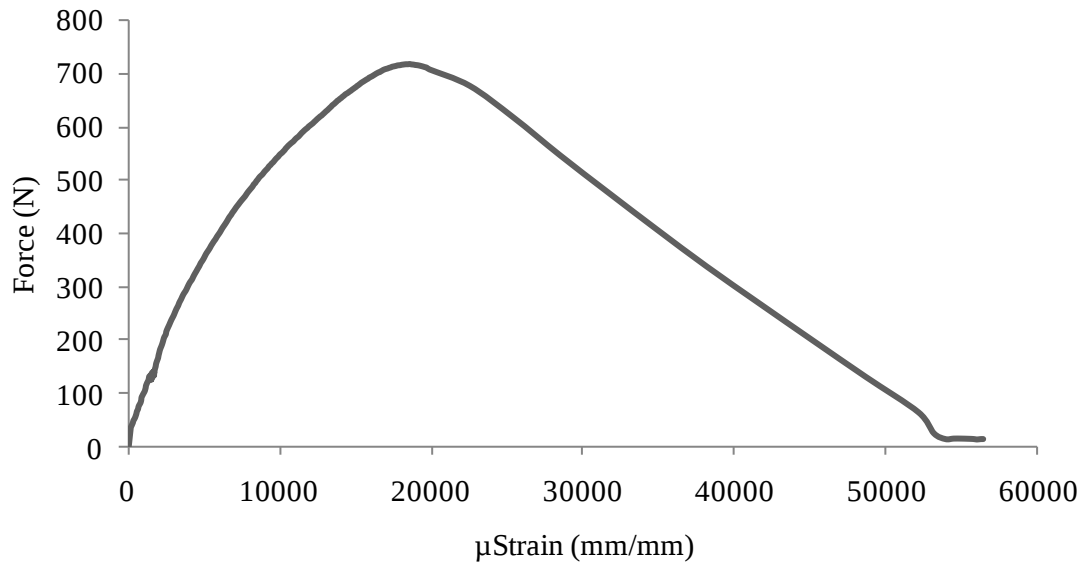


Fig. 54 Stress vs strain for composite containing 2% carbon nanotubes at a fibre weight fraction of 30%

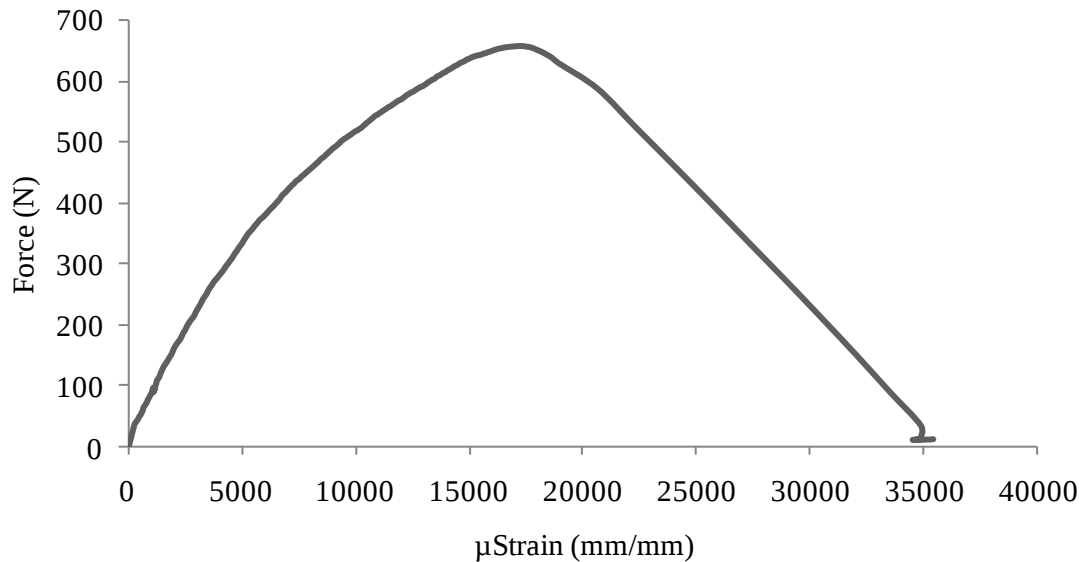


Fig. 55 Stress vs strain for a composite containing 8% glass flakes at 350nm at a fibre weight fraction of 30%

The linear modulus is the gradient of the linear region of the stress strain curve. It is evident from the stress strain curves that the linear region is small and so the secant modulus is calculated using a strain of 10 000 $\mu\epsilon$. The following graphs compare the moduli at different configurations of the composite for different fibre weight fractions. Figs. 51 and 52 compare the linear and secant moduli, respectively, for

composites manufactured using treated and untreated fibres. These composites did not contain any filler. Figs. 53 and 54 compare the linear and secant moduli, respectively, for treated fibre composites containing 2% carbon nanotubes with treated fibre composites containing no fillers. Figs. 55 and 56 compare the linear and secant moduli, respectively, for the treated fibre composites containing 8% glass flakes with a thickness of 350 nm with that of treated fibre composites containing no filler.

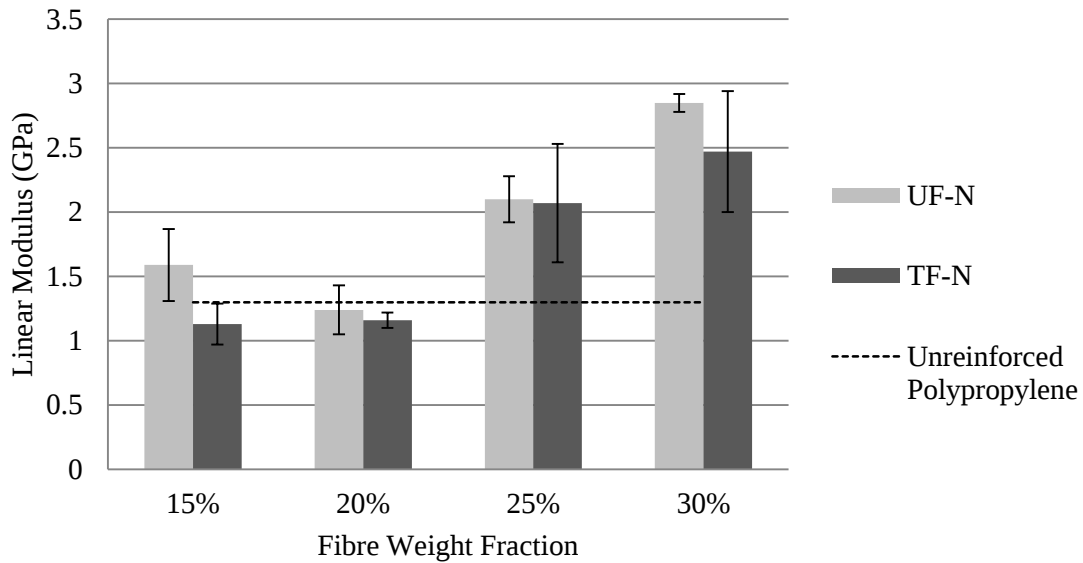


Fig. 56 Effect of fibre treatment on linear modulus

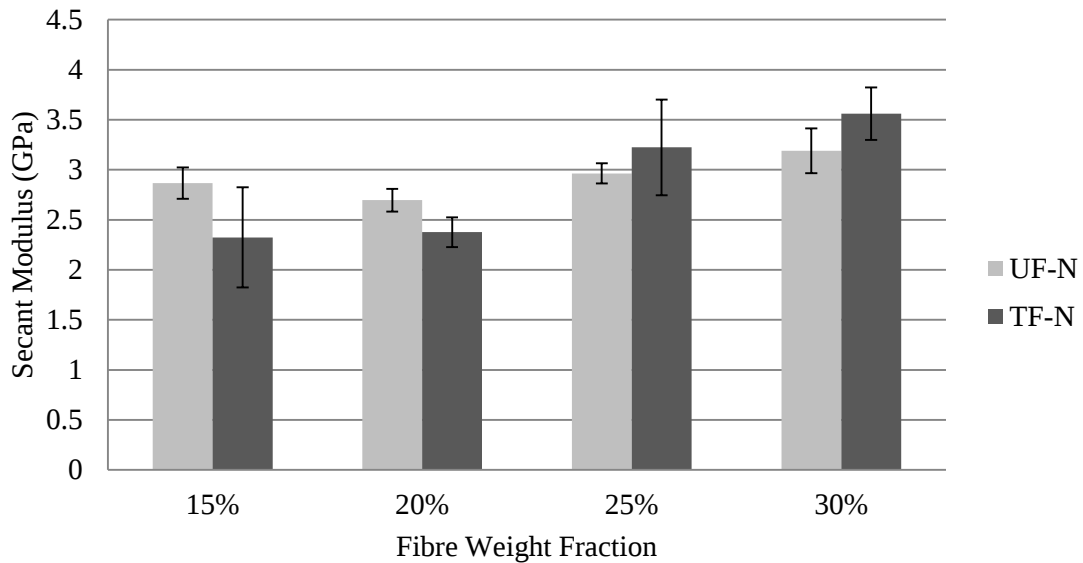


Fig. 57 Effect of fibre treatment on secant modulus

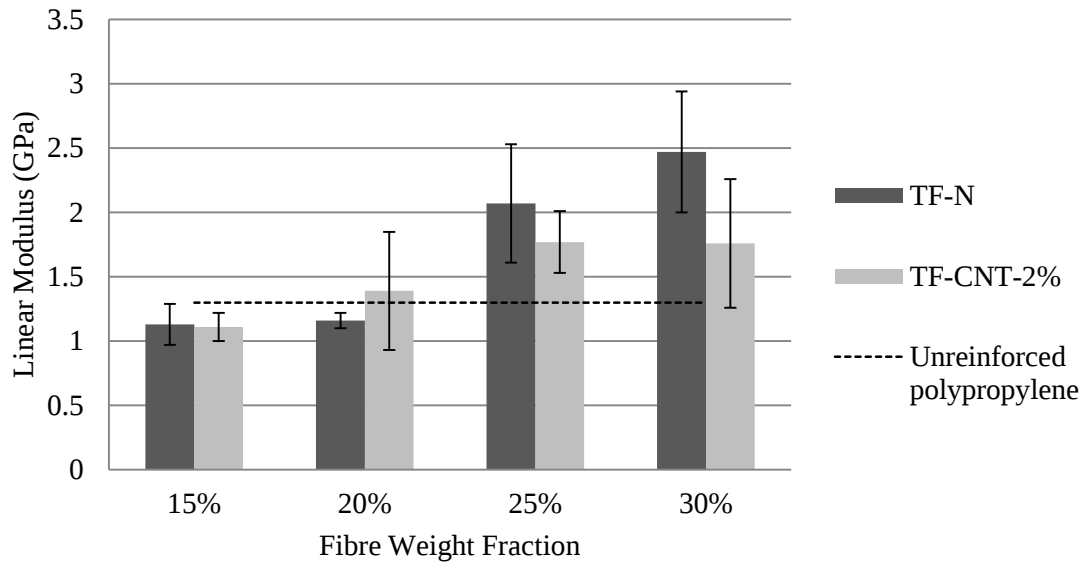


Fig. 58 Effect of carbon nanotubes with a concentration of 2% on linear modulus

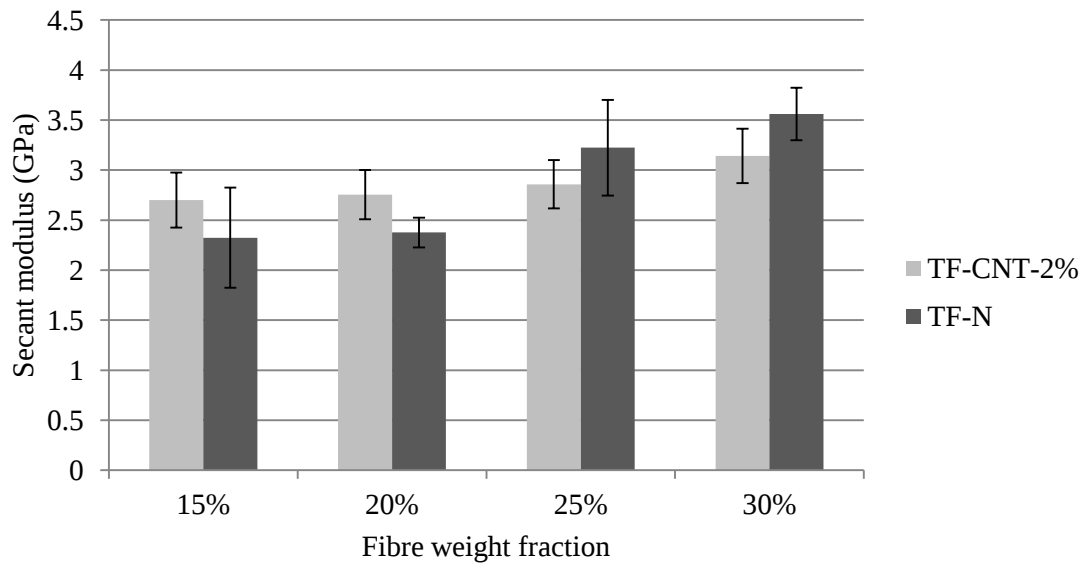


Fig. 59 Effect of carbon nanotubes with a concentration of 2% on secant modulus

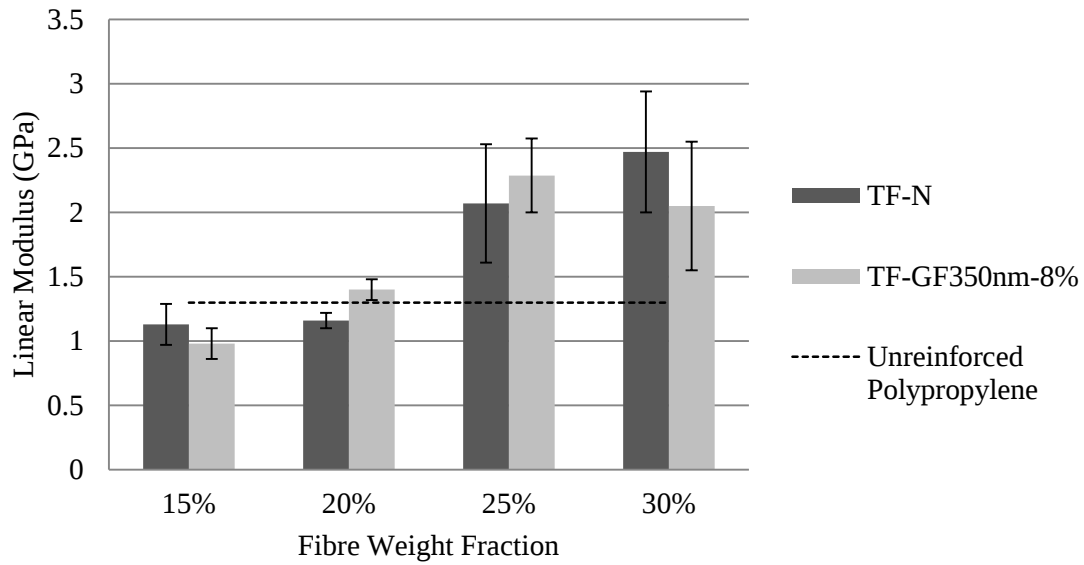


Fig. 60 Effect of 350 nm glass flakes with a concentration of 8% on linear modulus

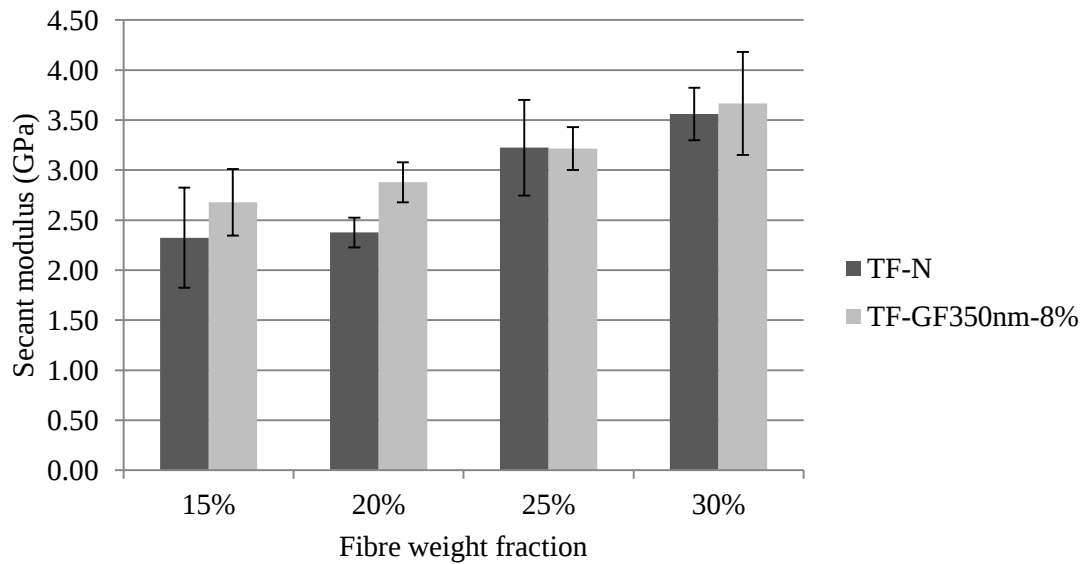


Fig. 61 Effect of 350 nm glass flakes with a concentration of 8% on secant modulus

Figs. 57 and 58 illustrate the effect that the variation in filler content has on the linear and secant moduli, respectively, for a fibre weight fraction of 30%. The horizontal dotted line refers to the modulus of the TF-N composite at a fibre weight fraction of 30%.

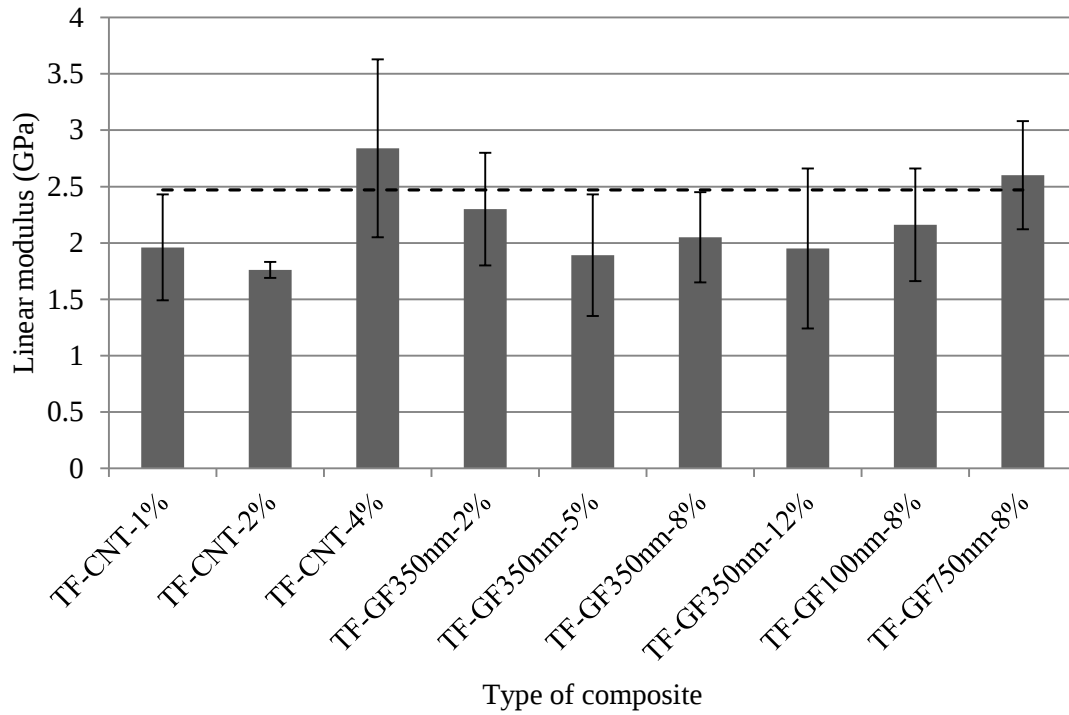


Fig. 62 Effect of variation in filler content on linear modulus for a fibre weight fraction of 30%

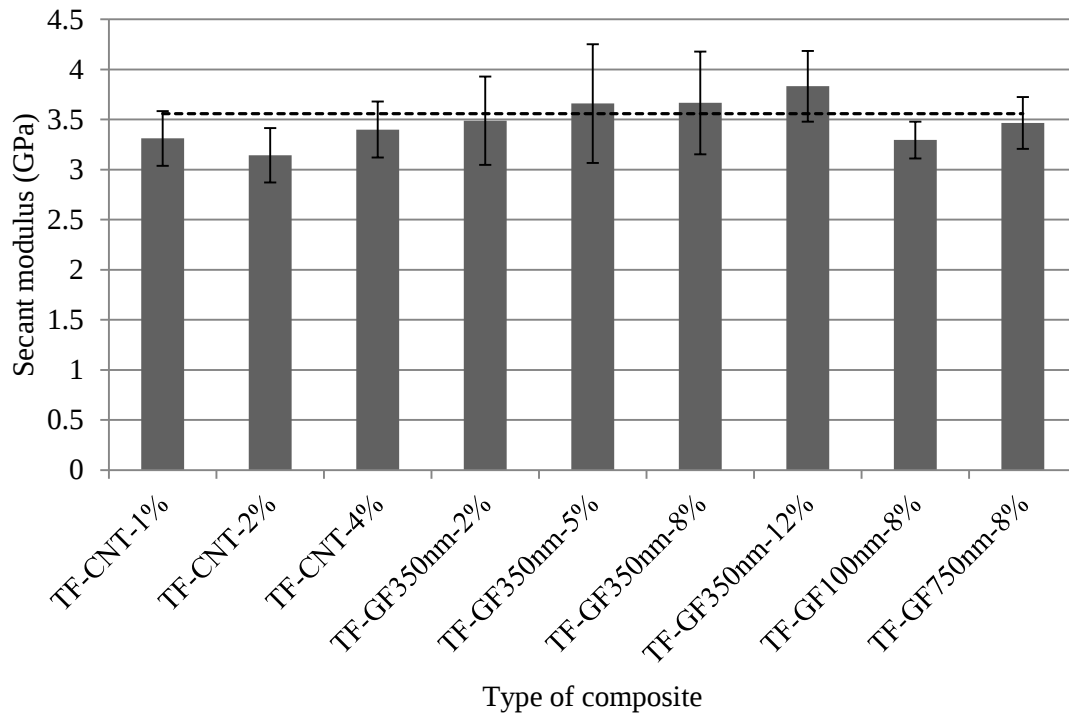


Fig. 63 Effect of variation in filler content on secant modulus for a fibre weight fraction of 30%

7.3 Water absorption

The samples were immersed in water over a period of time and their change in mass, length, breadth, centre thickness and edge thickness were measured periodically. The dimensional change was measured for the first 648 hours, while the mass change was measured for a period of 1080 hours. Due to the time consuming measuring process the samples were not all measured after exactly the same period of time. The variation in the time that each specimen was tested at are, however, not significant in comparison to the total immersion time.

7.3.1 Mass change

The change in mass over time is considered. This measurement has the least error inherent in the measurement and was found to be the most consistent. Different samples are consequently compared to one another using this data.

A least squared exponential curve is fitted to the data using the formula:

$$y = a \times (1 - e^{-bt})$$

where 'a' refers to the maximum height of the curve, 'b' refers to the rate at which the curve approaches 'a' and 't' refers to the time in hours that the samples have been exposed to water. These values correspond to the two aspects of water absorption that need to be compared. Rate of water absorption which will be referred to using the symbol (β) corresponds to 'b' and the saturation value corresponds to 'a'. This saturation value refers to the change in mass as the time tends to infinity. Fig. 64 shows a change in mass vs. time for the TF-N composites at all fibre weight fractions. The error bars on the graphs represent one standard deviation from the four samples measured for each composite. The remaining water absorption curves are presented in Appendix G.

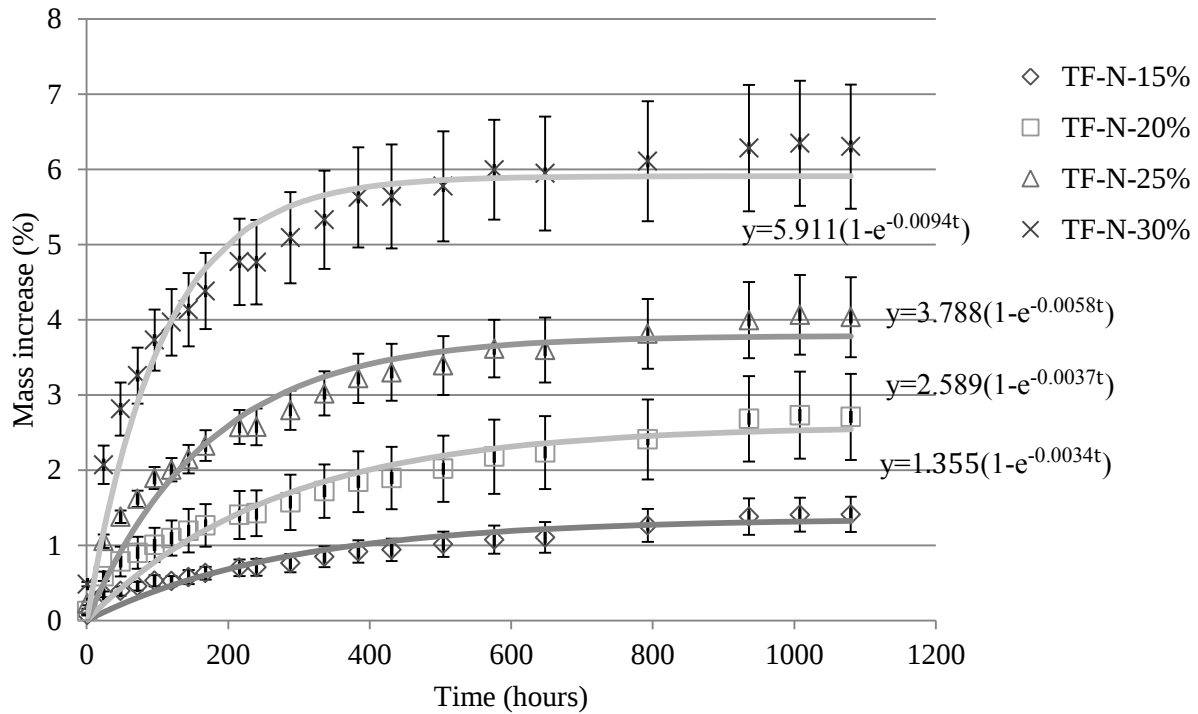


Fig. 64 Effect of fibre weight fraction on mass increase for treated fibres after 1080 hours

There are two separate parameters that need to be looked at to compare the water absorption of the composites, the rate of water absorption (β) and the saturation value. Figs. 60 and 61, respectively, compare the saturation value and rate of water absorption (β) for composites manufactured using treated and untreated fibres. These composites did not contain any filler. Figs. 62 and 63 compare the saturation value and rate of water absorption (β), respectively, for treated fibre composites containing 2% carbon nanotubes against those of treated fibre composites containing no fillers. Figs. 64 and 65 compare the saturation value and rate of water absorption (β), respectively, for the treated fibre composites containing 8% glass flakes with a thickness of 350 nm against those of treated fibre composites containing no filler. Figs. 66 and 67 illustrate the effect the variation in filler content has on the saturation value and rate of water absorption (β), respectively, for a fibre weight fraction of 30%. A full set of the values can be found in Appendix G. Fig. 73 illustrates the effect the fibre weight fraction has on the total water absorbed after 1080 hours.

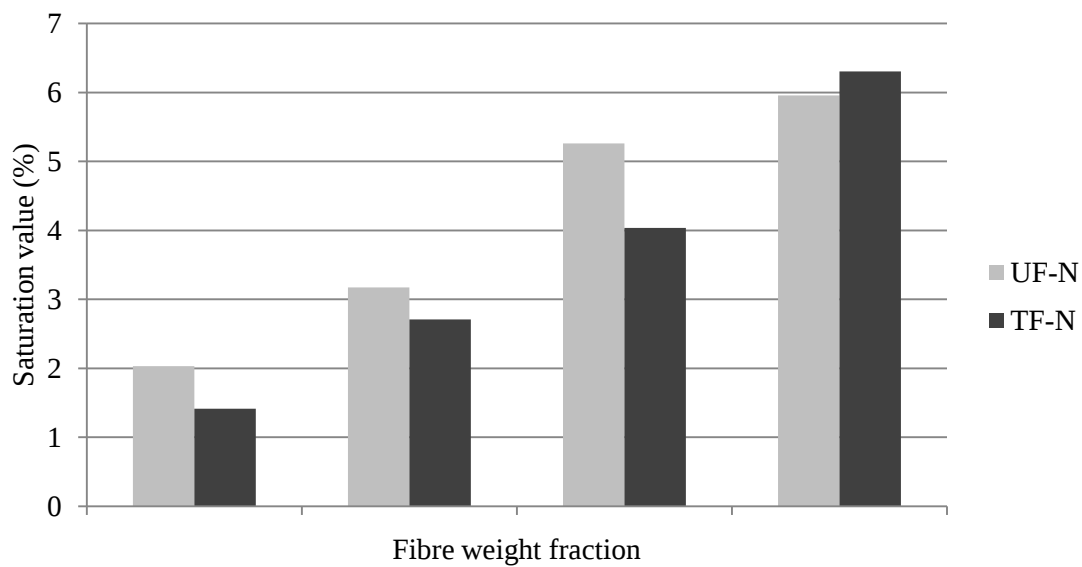


Fig. 65 Effects of fibre treatment on saturation value

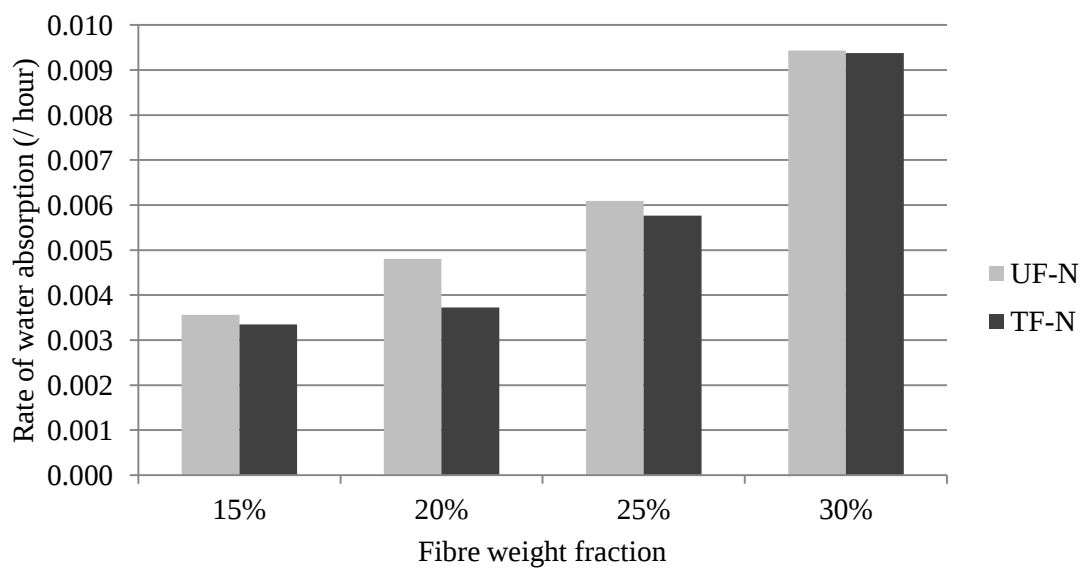


Fig. 66 Effects of fibre treatment on the rate of water absorption

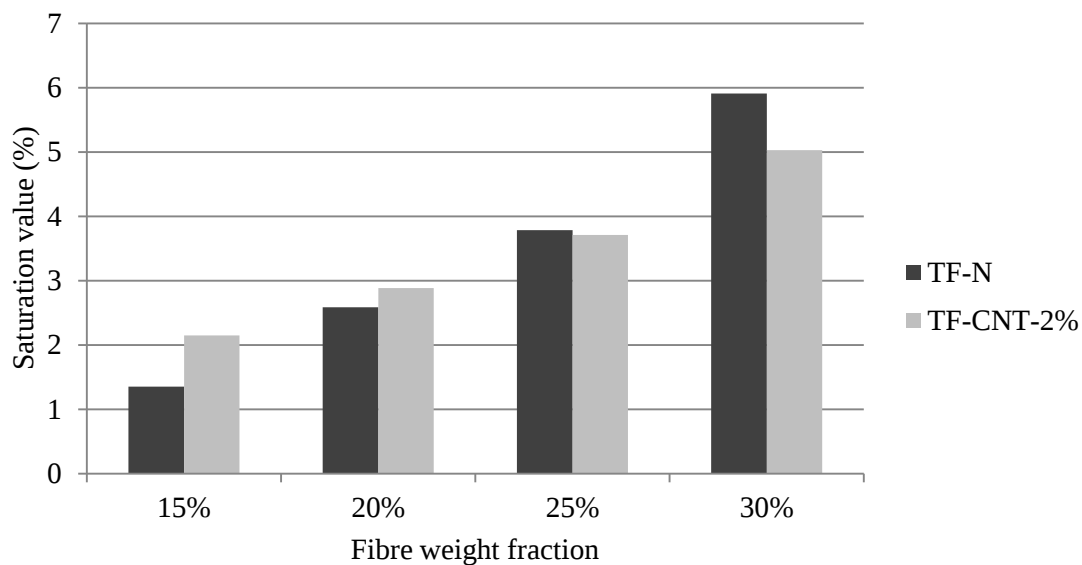


Fig. 67 Effect of carbon nanotubes with a concentration of 2% on saturation value

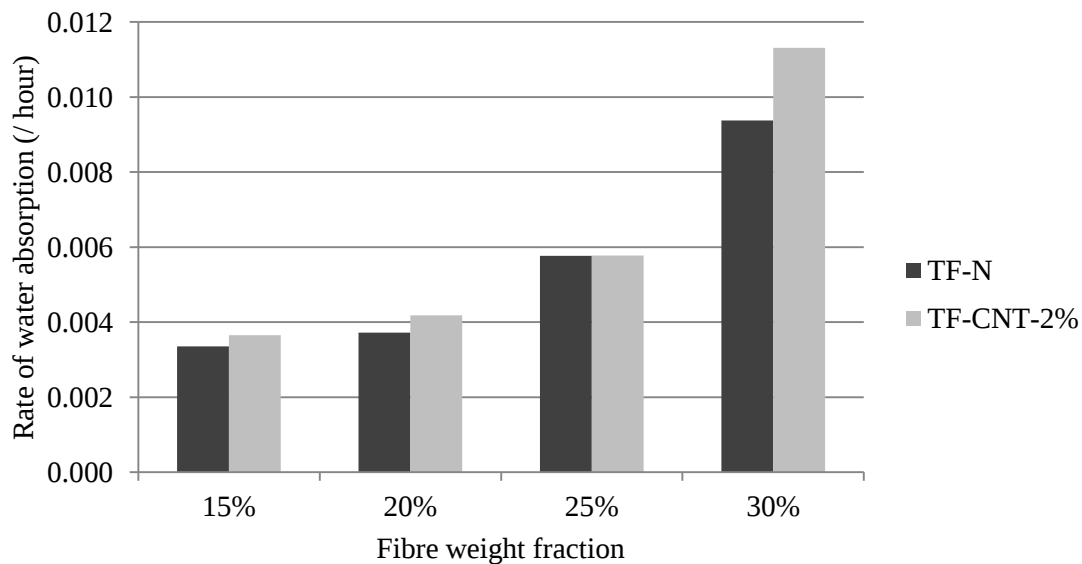


Fig. 68 Effect of carbon nanotubes with a concentration of 2% on rate of water absorption

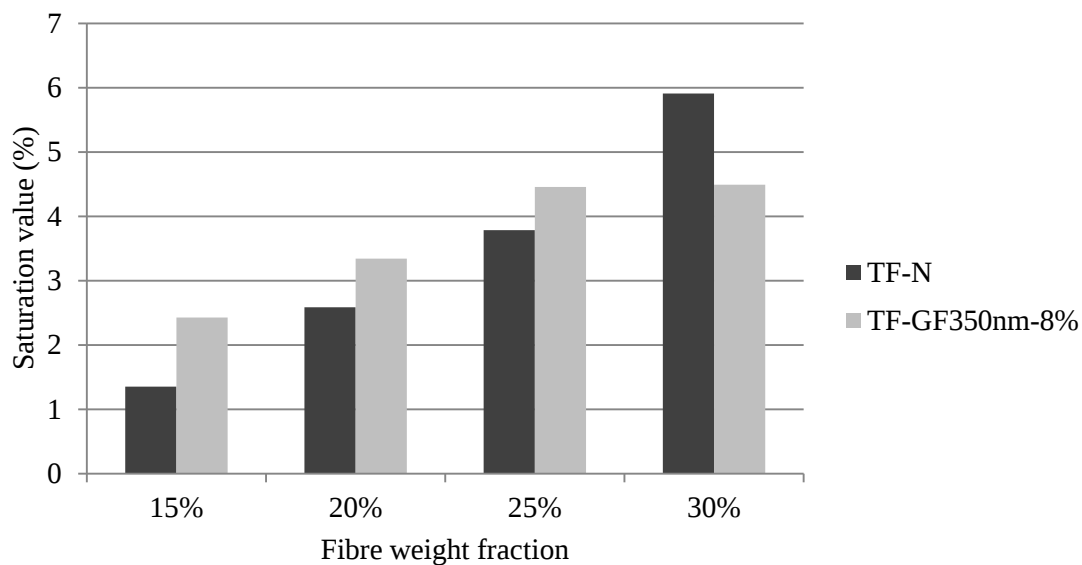


Fig. 69 Effect of 350 nm glass flakes with a concentration of 8% on saturation value

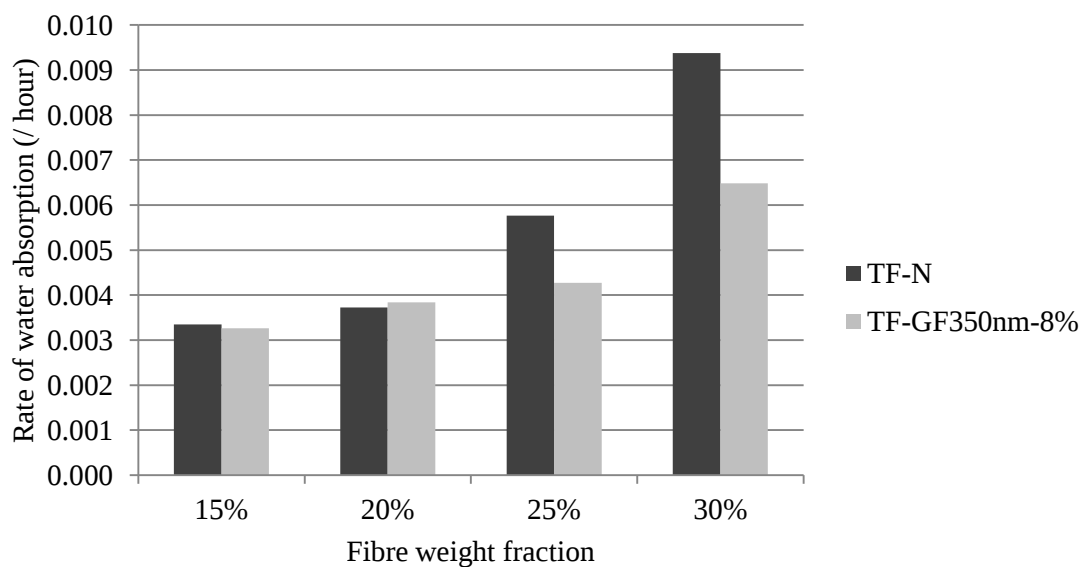


Fig. 70 Effect of 350 nm glass flakes with a concentration of 8% on rate of water absorption

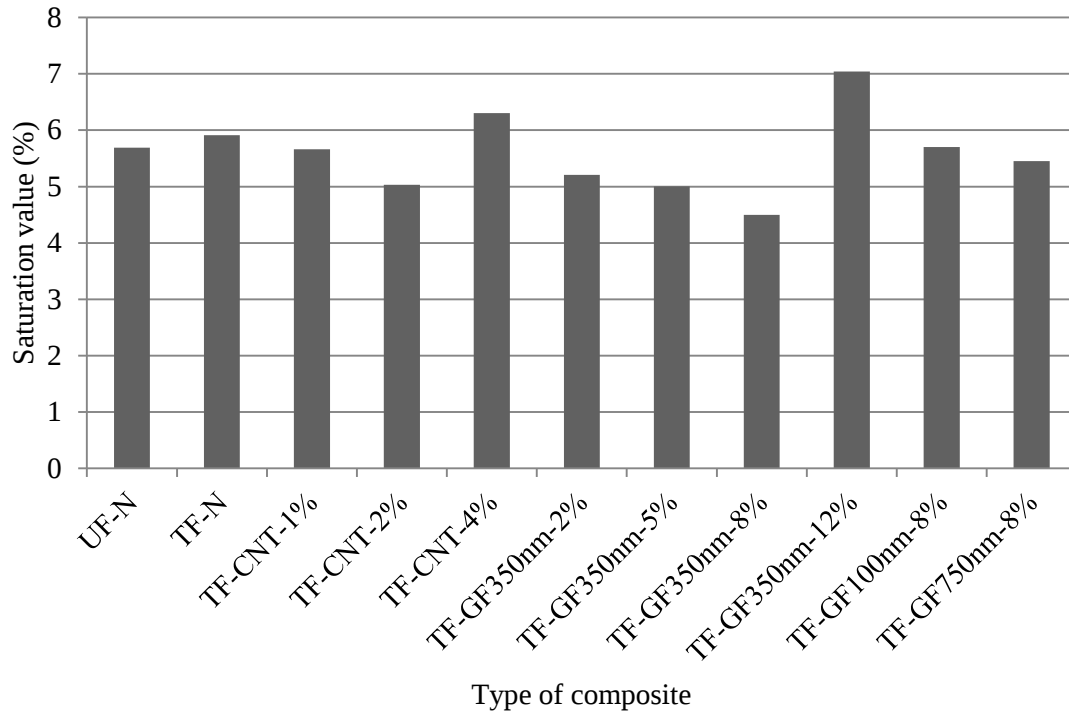


Fig. 71 Effect of variation in filler content on saturation value for 30% fibre weight fraction

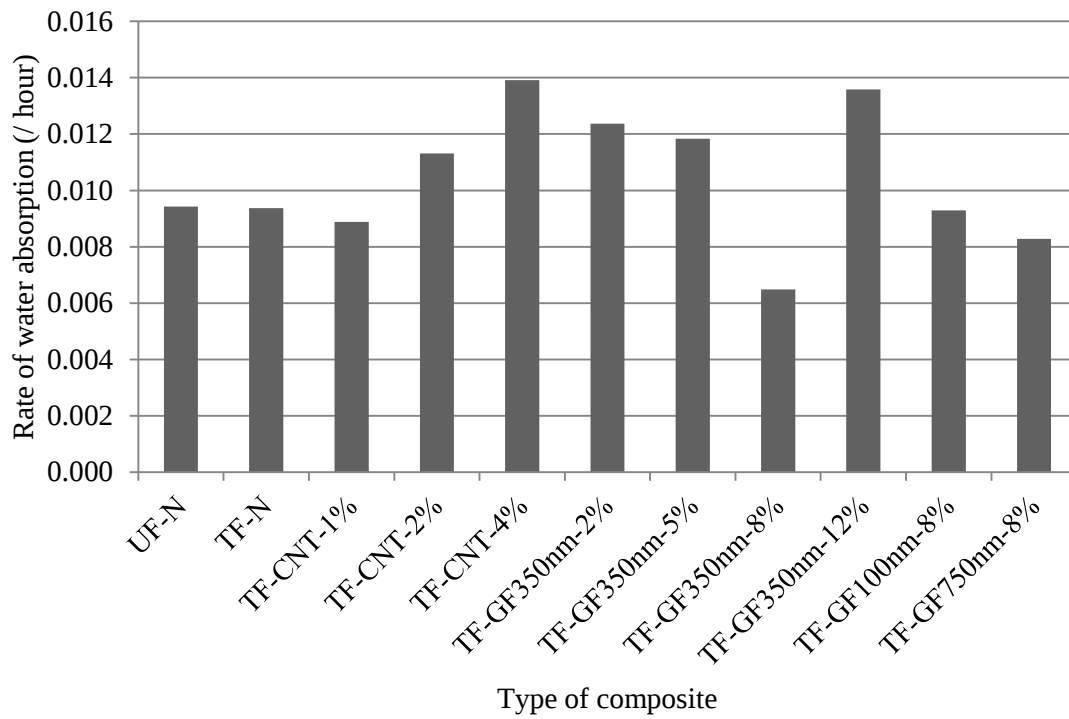


Fig. 72 Effect of variation in filler content on rate of water absorption for 30% fibre weight fraction

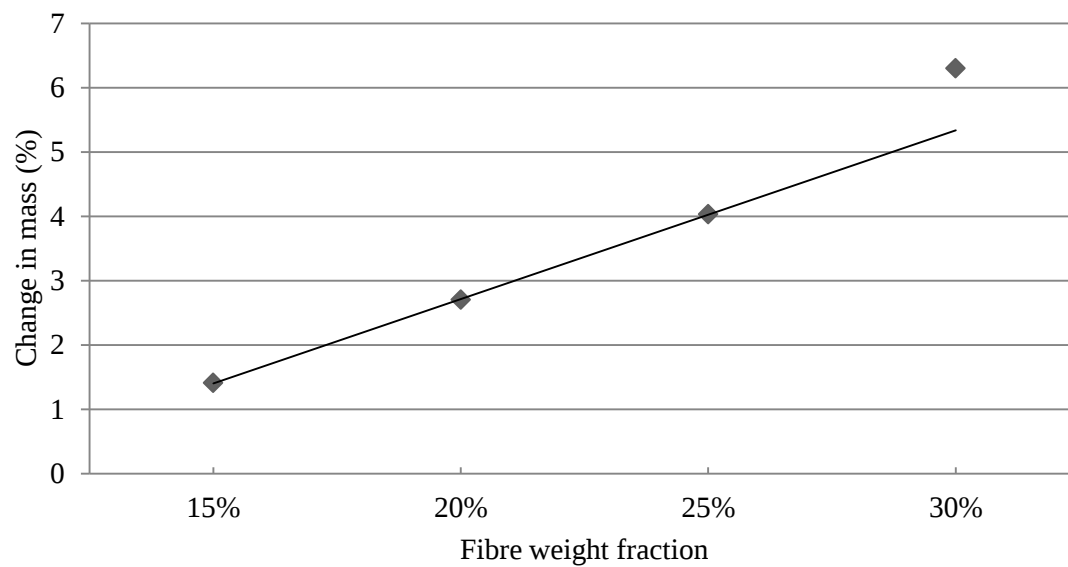


Fig. 73 Change in mass vs. fibre weight fraction of the TF-N composite after 1080 hours

7.3.2 Dimensional change (Area, Thickness)

The thickness, length and breadth were measured every 24 hours. The measurements were taken over a total time period of 648 hours. The reason for the shorter time as compared to the mass change is that the dimension measurements were very time consuming and enough data had been recorded after 648 hours to determine trends. Change in individual dimensions is not presented. What are presented instead are changes in area. The fibre mat is randomly aligned and cut from a circular disk. “Length” and “breadth” are therefore arbitrary constructs. The changes in the “length” and “breadth” directions should therefore be similar, but it was found that changes in area showed less scatter and so are presented instead.

All curves presented in this section comprise data averaged from the four samples used for each composite configuration. All error bars represent one standard deviation across the four samples. Fig. 74 shows the change in area for the TF-N composite at a fibre weight fraction of 15%. There is a definite trend visible and can be expressed using the equation:

$$y = a \times (1 - e^{-bt})$$

The change in area for the various composite configurations followed the same trend as for the TF-N-15%. The fibre weight fraction had no apparent effect on the change in area and so all fibre weight fractions are not plotted.

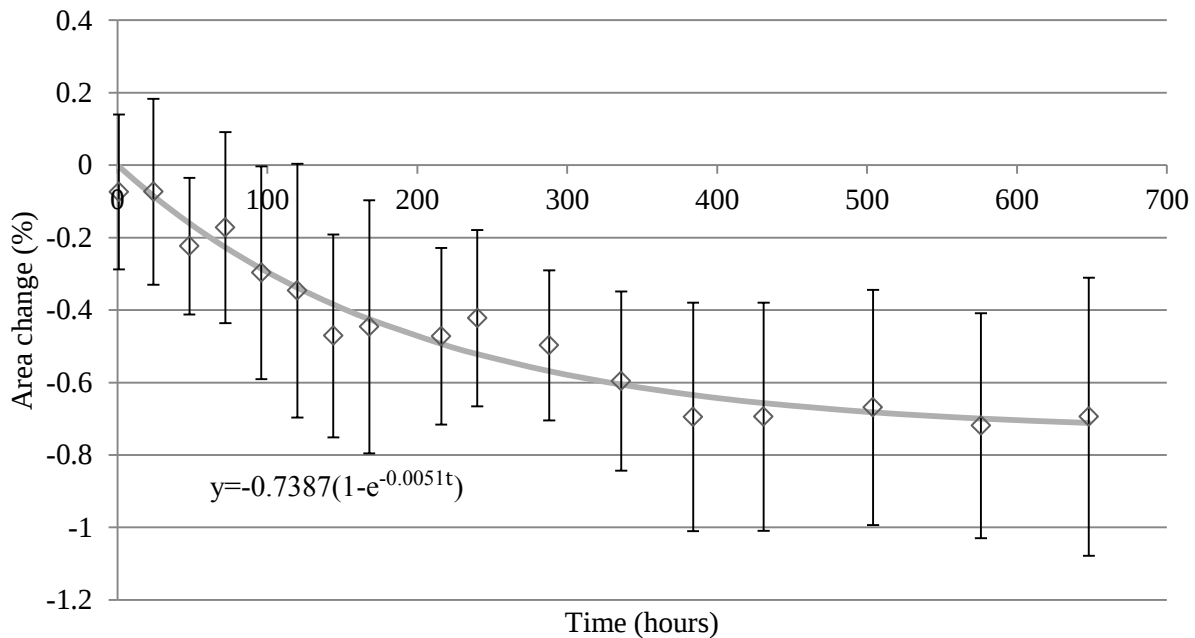


Fig. 74 Change in area for TF-N composite at a fibre weight fraction of 15%

Fig. 75 compares the change in area with time for composites manufactured using treated and untreated fibres. These composites did not contain any filler. Fig. 76 compares the change in area with time for treated fibre composites containing 2% carbon nanotubes with treated fibre composites containing no fillers. Fig. 77 compares the change in area with time for the treated fibre composites containing 8% glass flakes with a thickness of 350 nm with that of treated fibre composites containing no filler.

Fig. 78 illustrates the effect the variation in filler content has on the change in area with time for a fibre weight fraction of 30%. The remaining data for a change in area is presented in Appendix G.

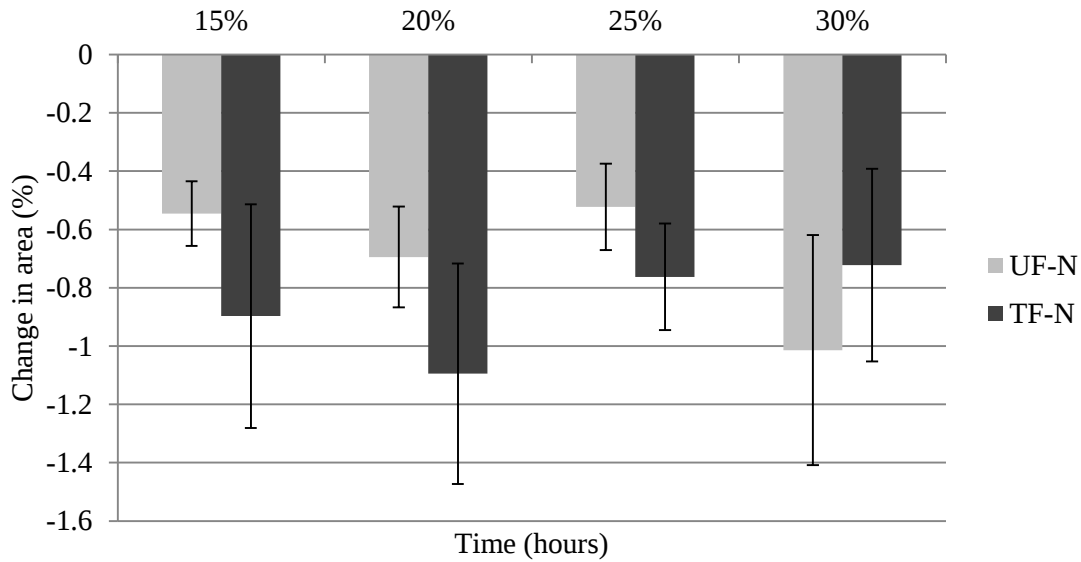


Fig. 75 Effect of fibre treatment on the change in area for various fibre weight fractions after 648 hours

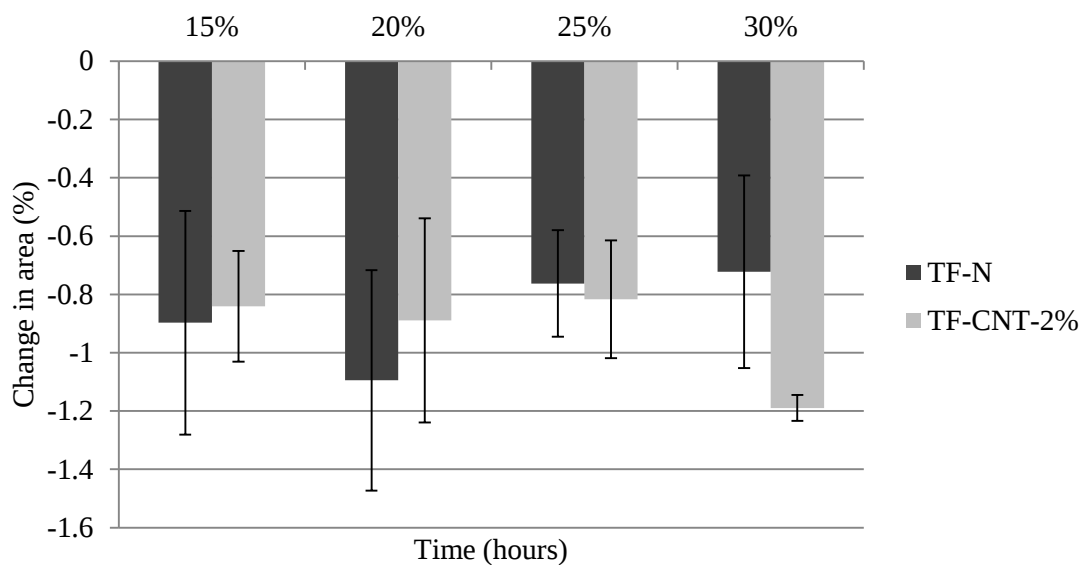


Fig. 76 Effect of carbon nanotubes with a concentration of 2% on the change in area for various fibre weight fractions after 648 hours

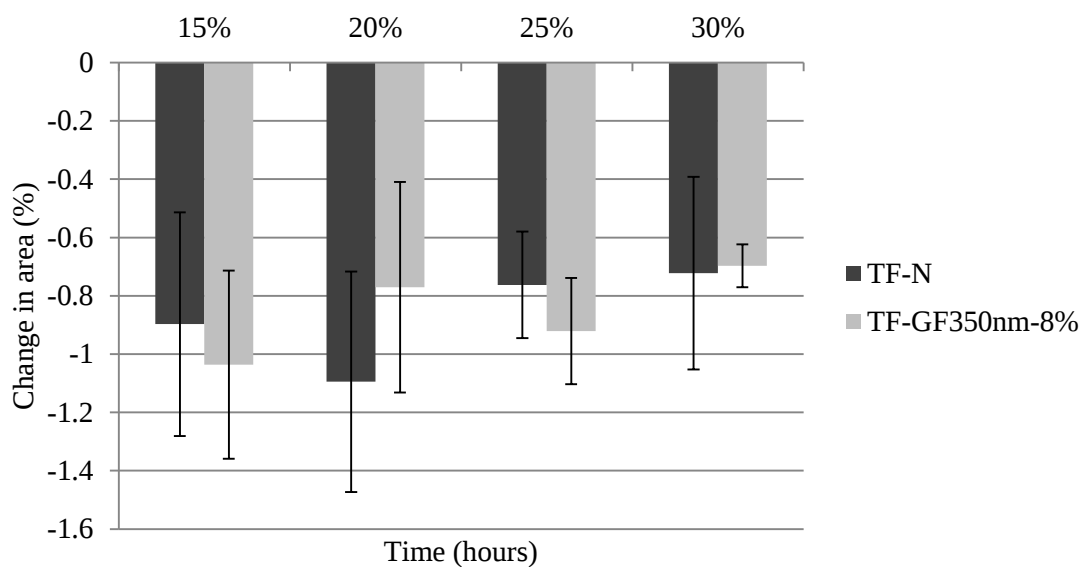


Fig. 77 Effect of 350 nm glass flakes with a concentration of 8% on the change in area for various fibre weight fractions after 648 hours

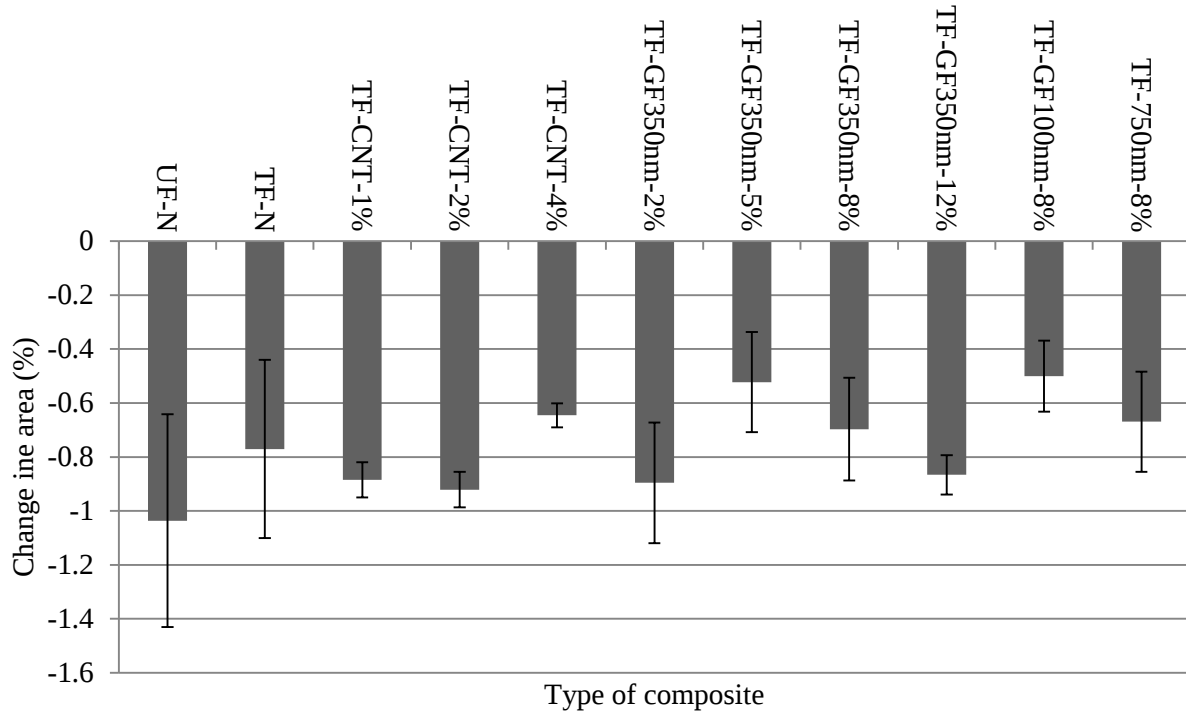


Fig. 78 Effect of variation in filler content on change in area for a fibre weight fraction of 30%

As with the area measurements, the thickness measurements were taken over a period of 648 hours. The measurements were taken at the centre as well as 7.5mm from each edge of the specimen. The edge measurements were averaged over all four edges of each specimen.

Figs. 74 and 75 show the change in centre and edge thicknesses, respectively, with time for the TF-N composite at various fibre weight fractions. The trend followed by the data can be expressed using the equation:

$$y = a \times (1 - e^{-bt})$$

The change in centre thickness at 15% and 20% and the change in edge thickness at 15% do not follow any trend since the changes are small and so susceptible to scatter. Figs. 76 and 77 compare the change in centre and edge thicknesses, respectively, with time for composites manufactured using treated and untreated fibres. These composites did not contain any filler. Figs. 78 and 79 compare the change in centre and edge thicknesses, respectively, with time for treated fibre composites containing 2% carbon nanotubes against those of treated fibre composites containing no fillers. Figs. 80 and 81 compare the change in centre and edge thicknesses, respectively, with time for the treated fibre composites containing 8% glass flakes with a thickness of 350 nm against those of treated fibre composites containing no filler.

Figs. 82 and 83 illustrate the effect that the variation in filler content has on the change in centre and edge thicknesses, respectively, with time for a fibre weight fraction of 30%. Fig. 89 compares the change in centre thickness and change in edge thickness for the TF-N composite at various fibre weight fractions. The rest of the data can be found in Appendix G.

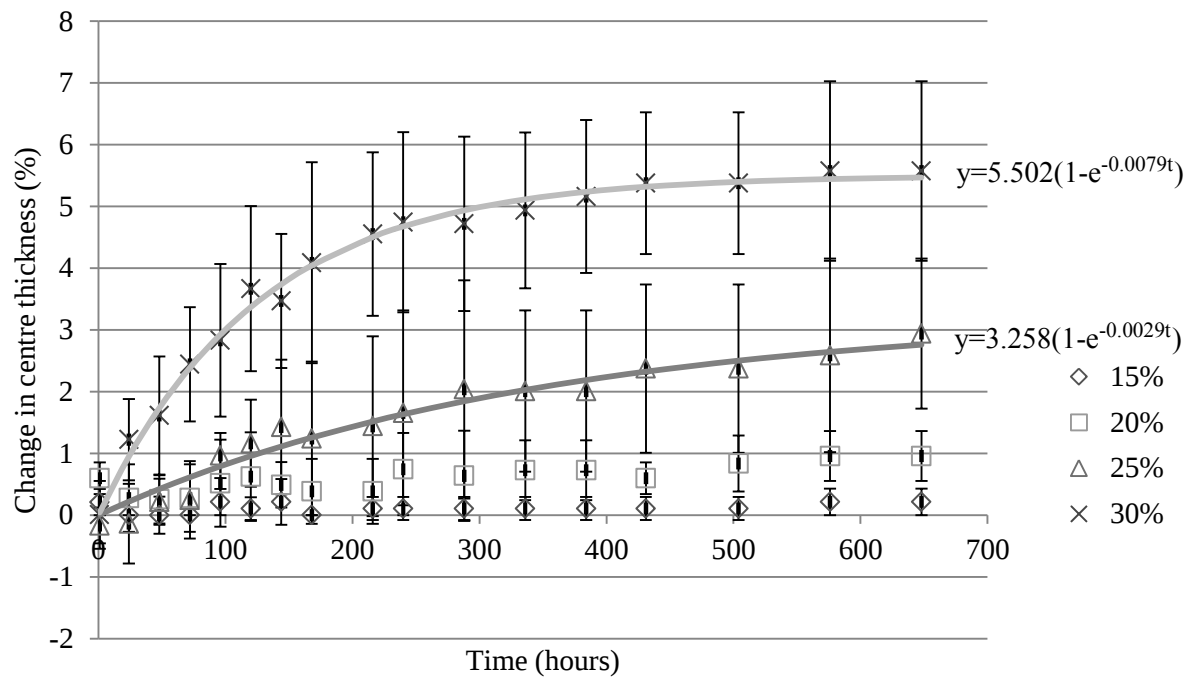


Fig. 79 Effect of fibre weight fraction on change in centre thickness for TF-N composite after 648 hours

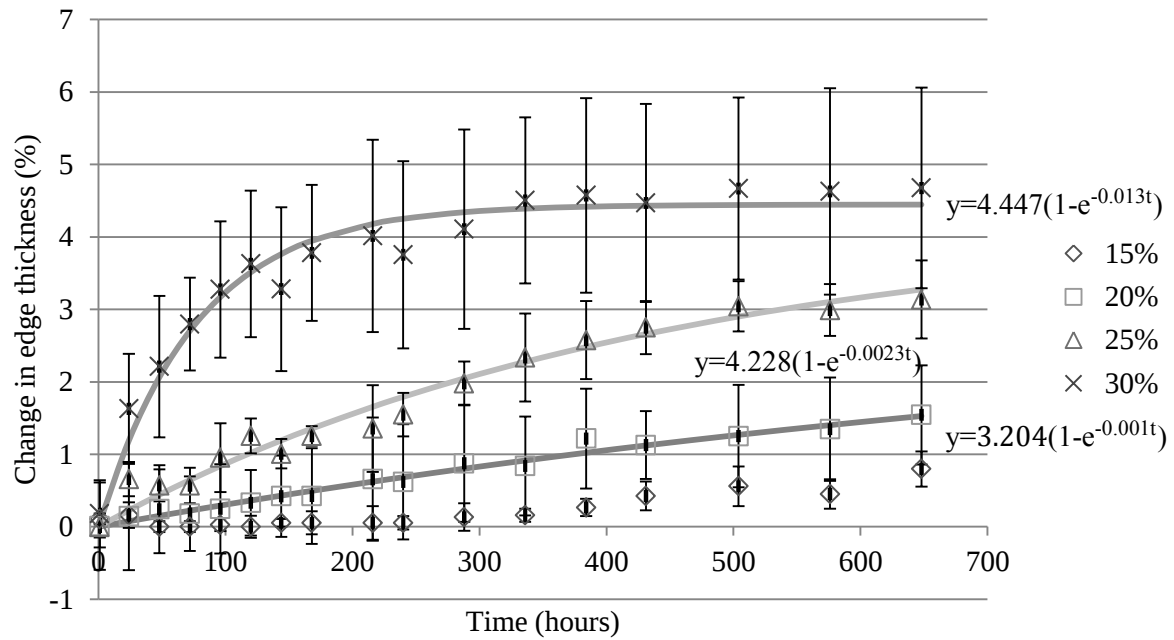


Fig. 80 Effect of fibre weight fraction on change in edge thickness for TF-N composite after 648 hours

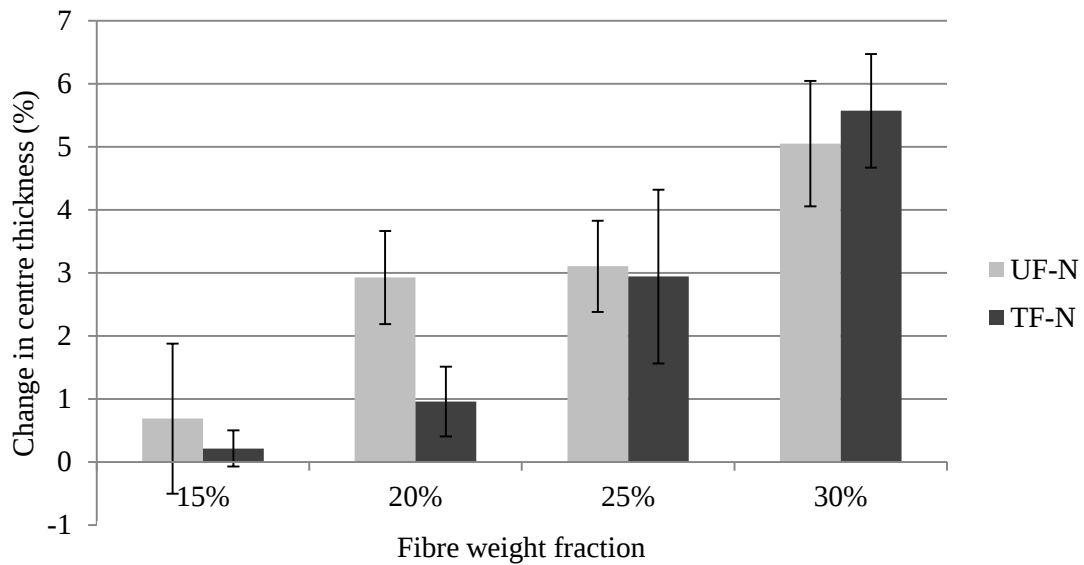


Fig. 81 Effect of fibre treatment of change in centre thickness for various fibre weight fractions after 648 hours

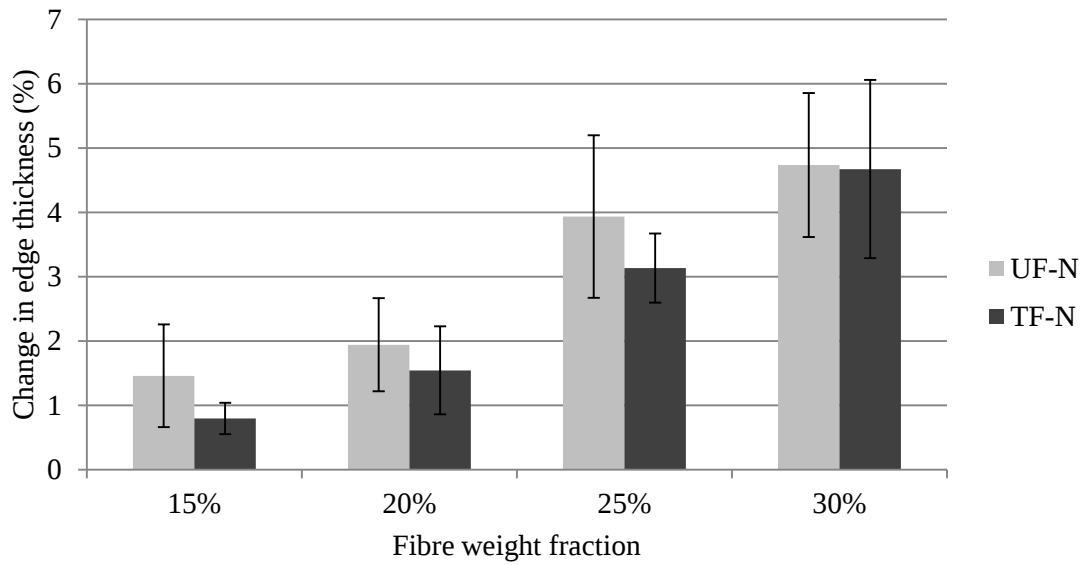


Fig. 82 Effect of fibre treatment of change in edge thickness for various fibre weight fractions after 648 hours

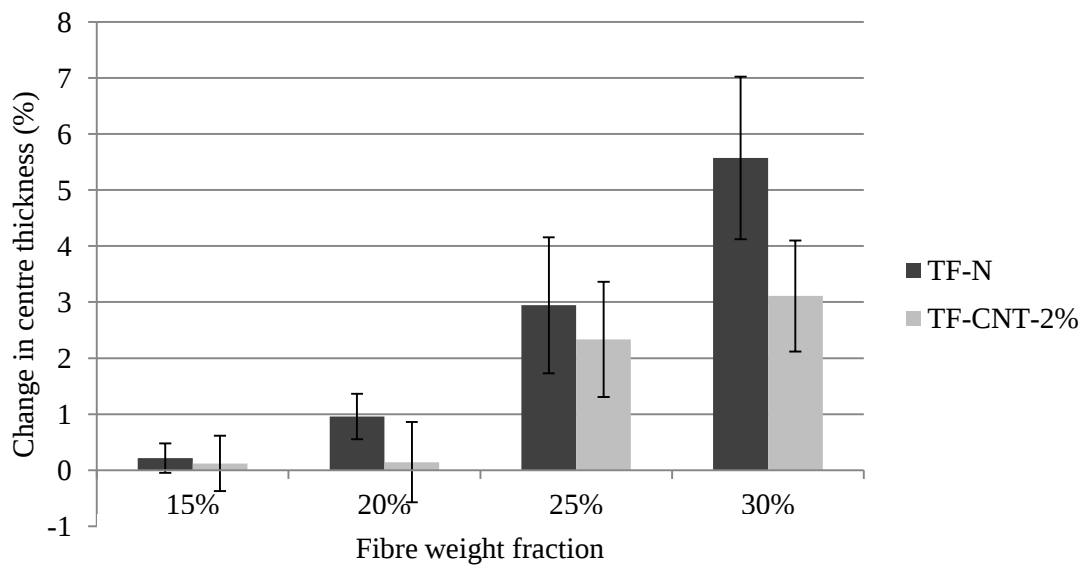


Fig. 83 Effect of carbon nanotubes with a concentration of 2% on the change in centre thickness for various fibre weight fractions after 648 hours

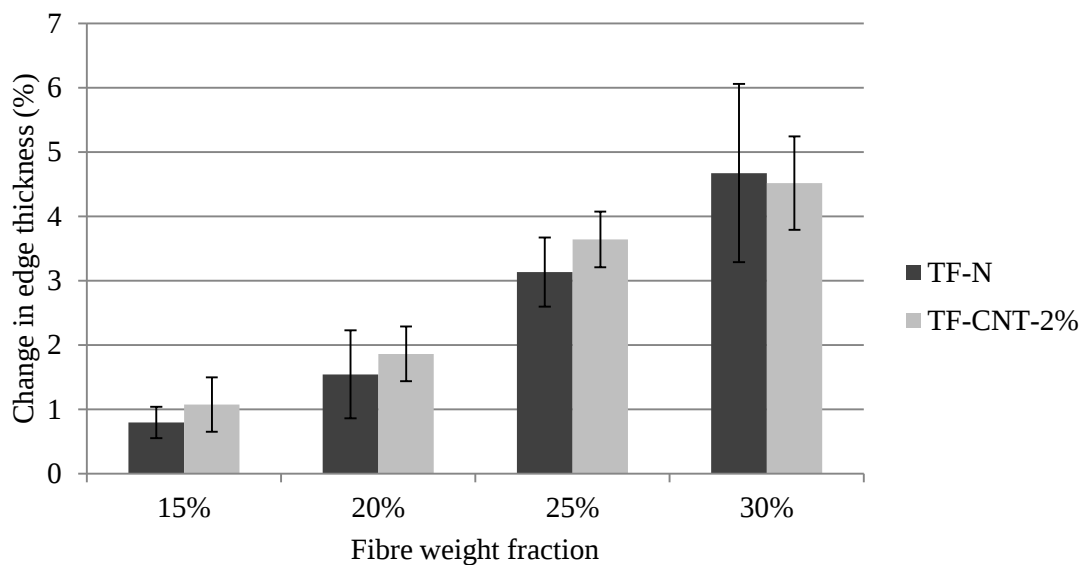


Fig. 84 Effect of carbon nanotubes with a concentration of 2% on the change in edge thickness for various fibre weight fractions after 648 hours

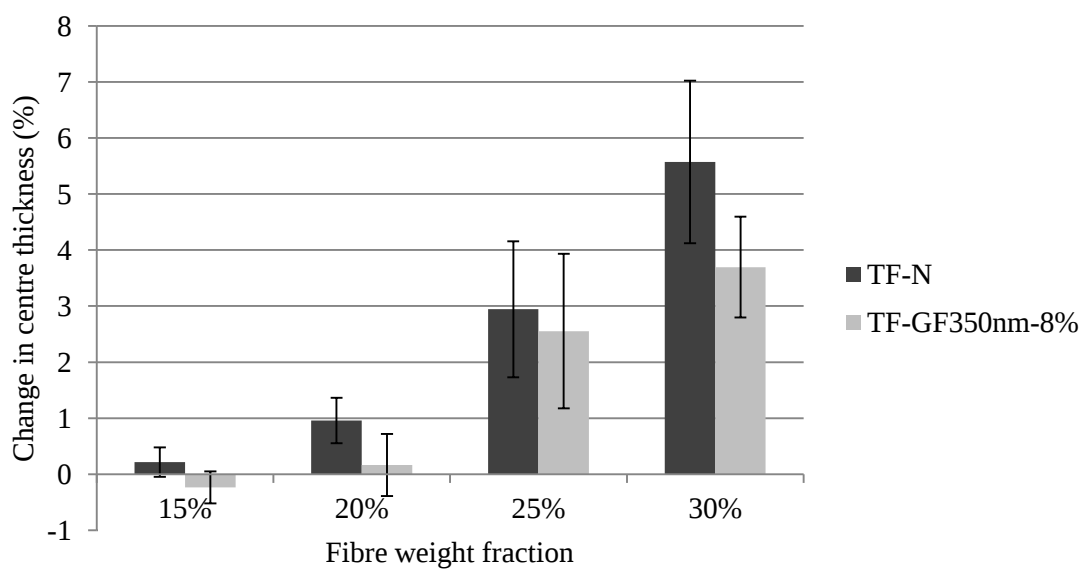


Fig. 85 Effect of 350 nm glass flakes with a concentration of 8% on the change in centre thickness for various fibre weight fractions after 648 hours

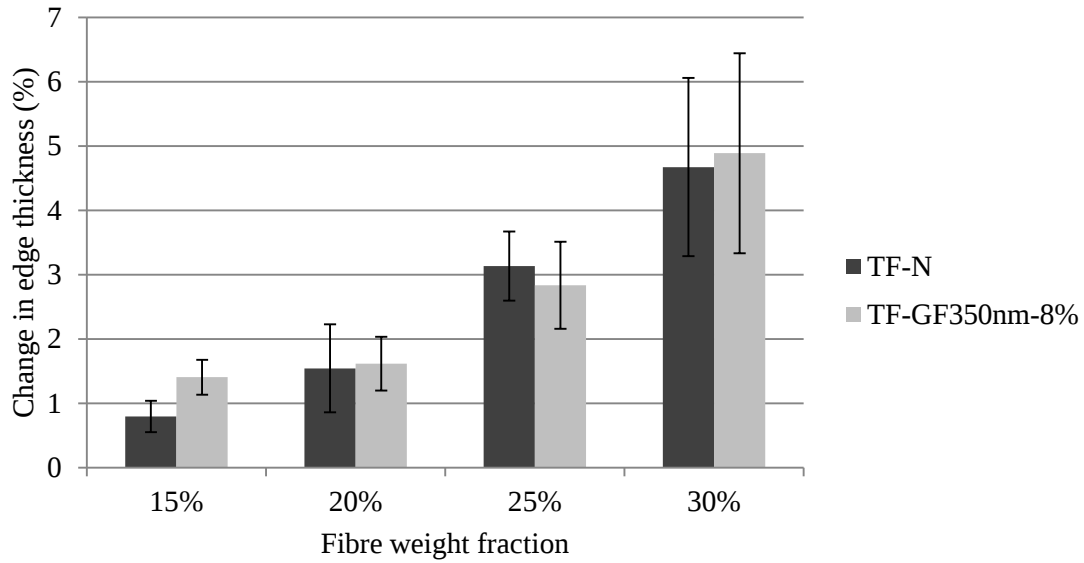


Fig. 86 Effect of 350 nm glass flakes with a concentration of 8% on the change in edge thickness for various fibre weight fractions after 648 hours

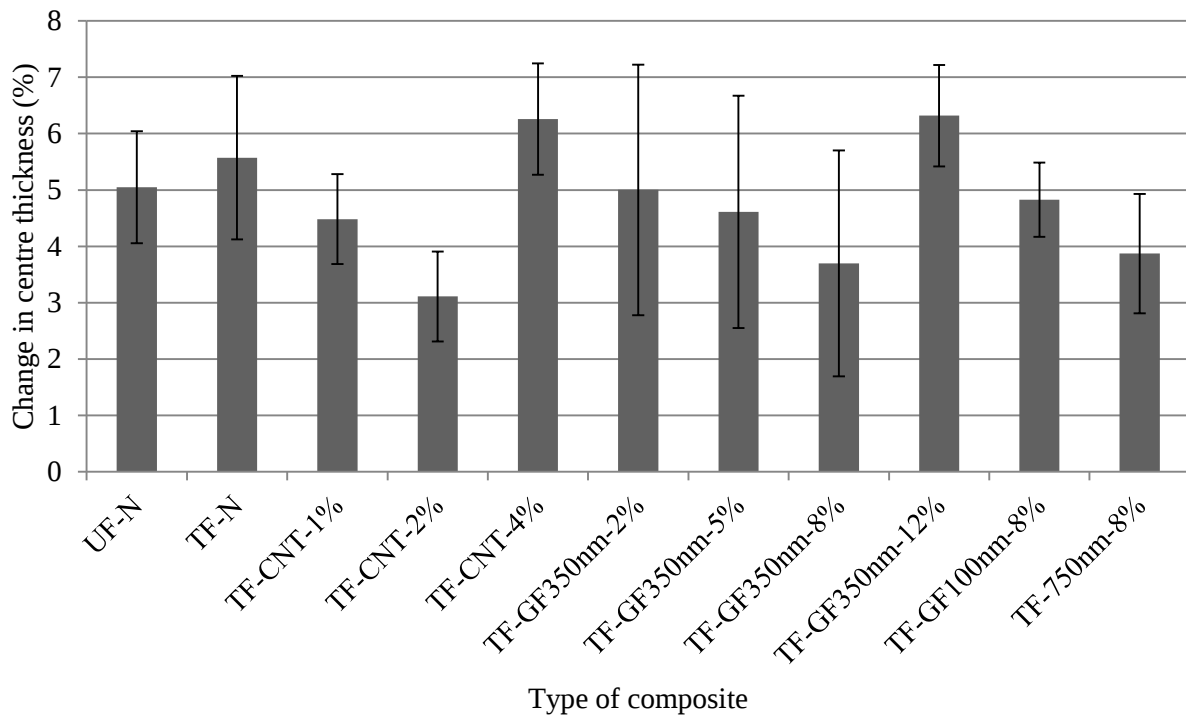


Fig. 87 Effect of variation in filler content on change in centre thickness for a fibre weight fraction of 30%

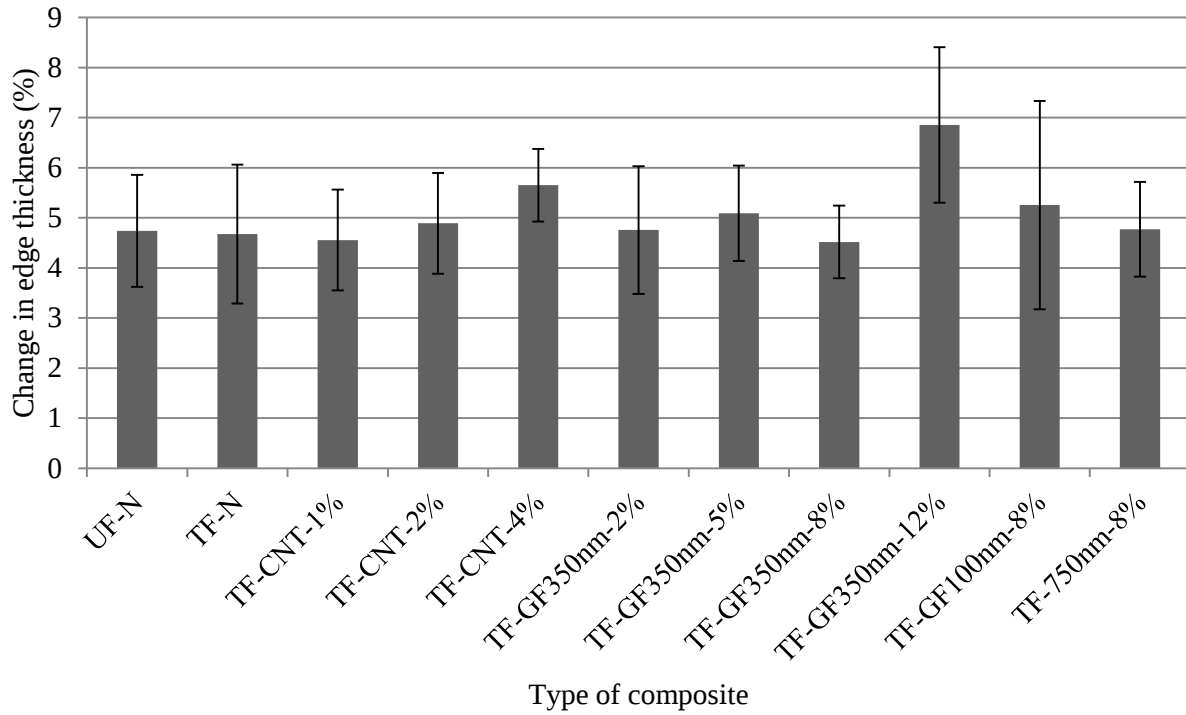


Fig. 88 Effect of variation in filler content on change in edge thickness for a fibre weight fraction of 30%

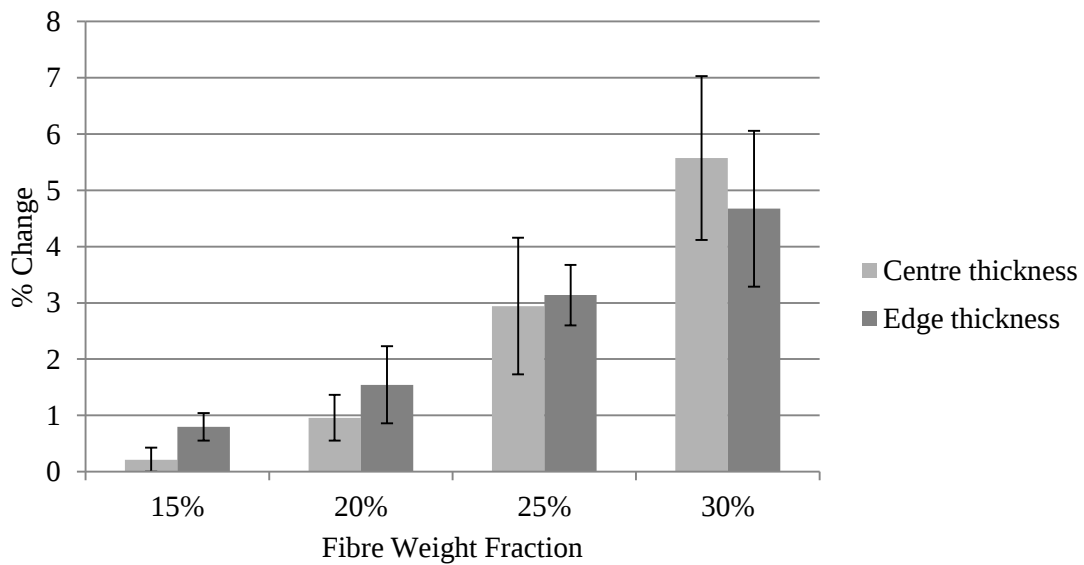


Fig. 89 Comparison between the centre and edge thickness change for TF-N composite at various fibre weight fractions

7.4 SEM images

Untreated as well as alkali treated hemp fibres were investigated using a scanning electron microscope. In addition to this, the fracture surfaces of various composites were also investigated. Some of the images taken are presented in the following figures.

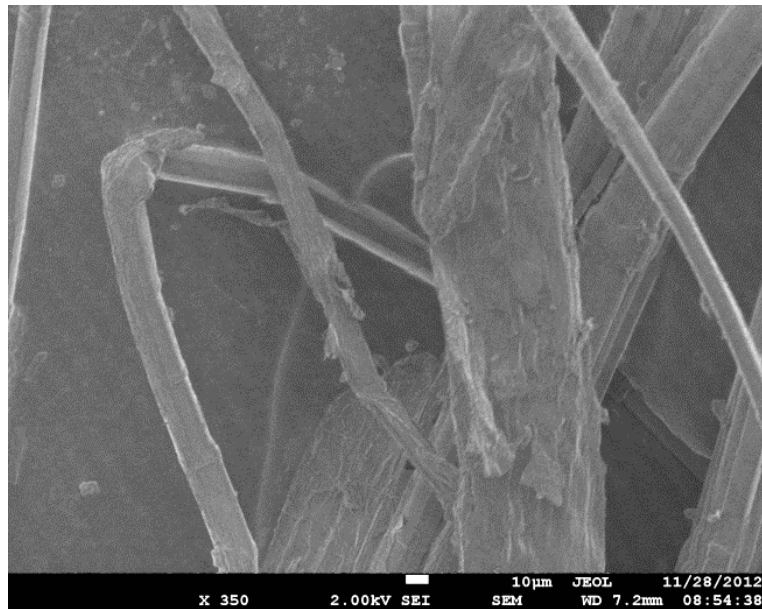


Fig. 90 Untreated fibres showing surface debris

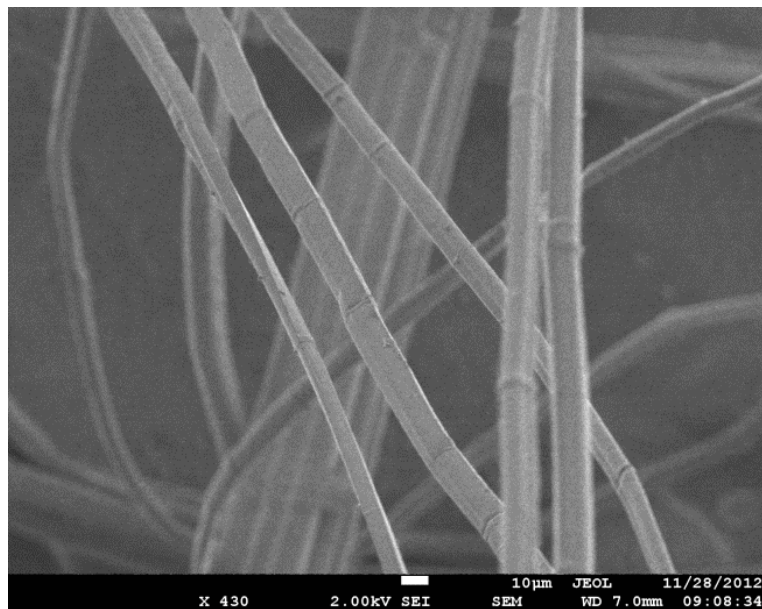


Fig. 91 Fibres treated with 6% NaOH

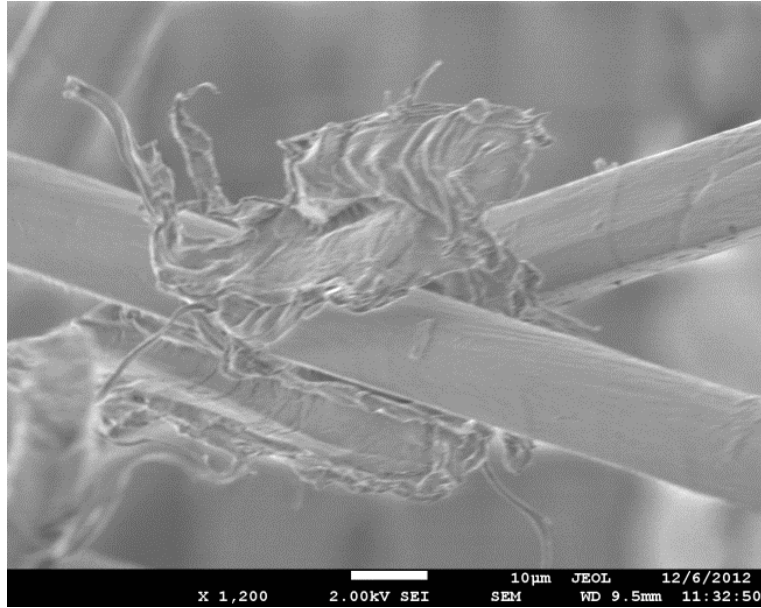


Fig. 92 SEM image showing matrix bonded to the fibres in the TF-N-30% composite

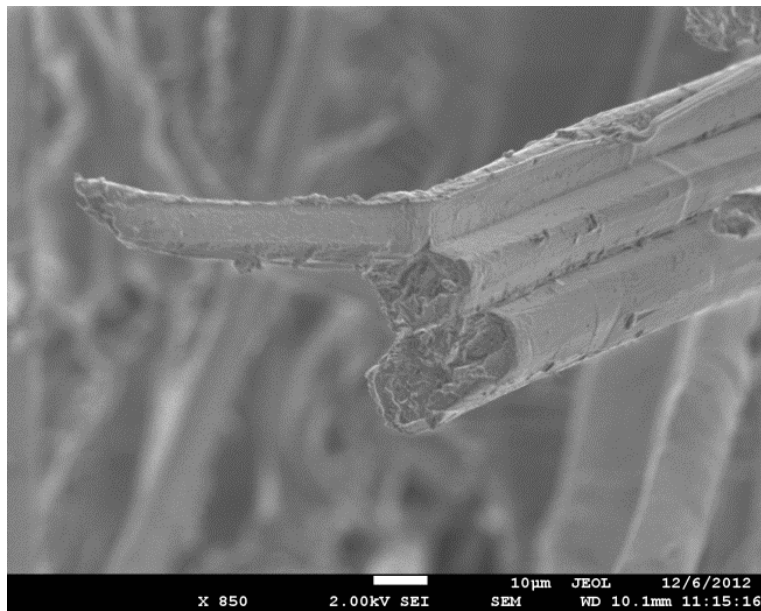


Fig. 93 SEM image of fibre breakage in the TF-N-30% composite

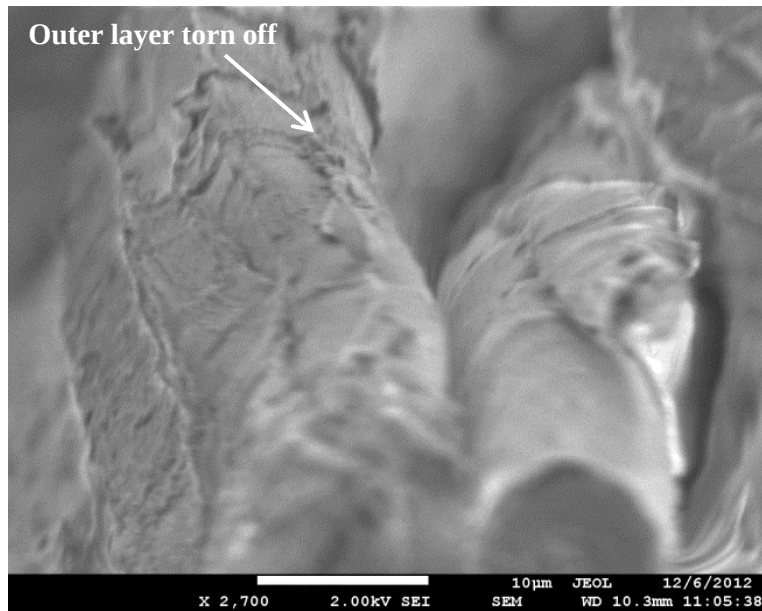


Fig. 94 SEM image showing evidence of the outer layer of the fibre being torn from the interior in the TF-N-30% composite

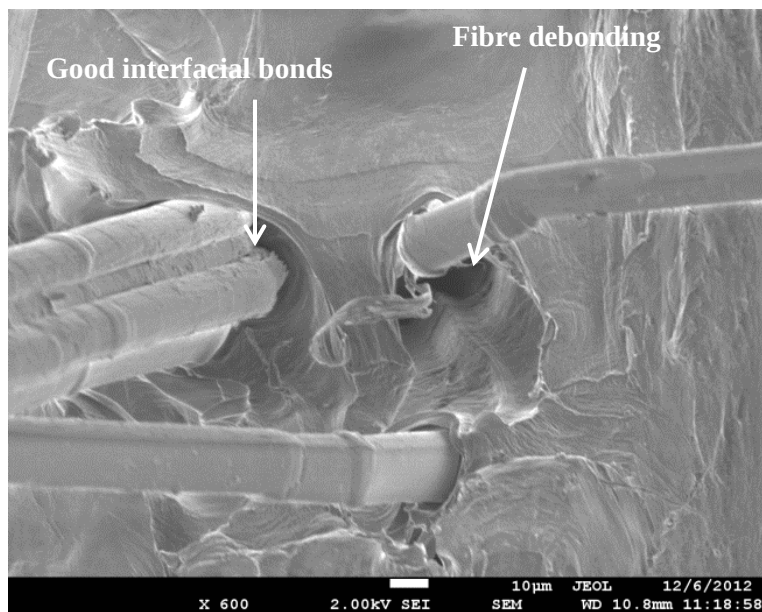


Fig. 95 SEM image showing evidence of both fibre debonding and good interfacial bonds in the TF-N-30% composite

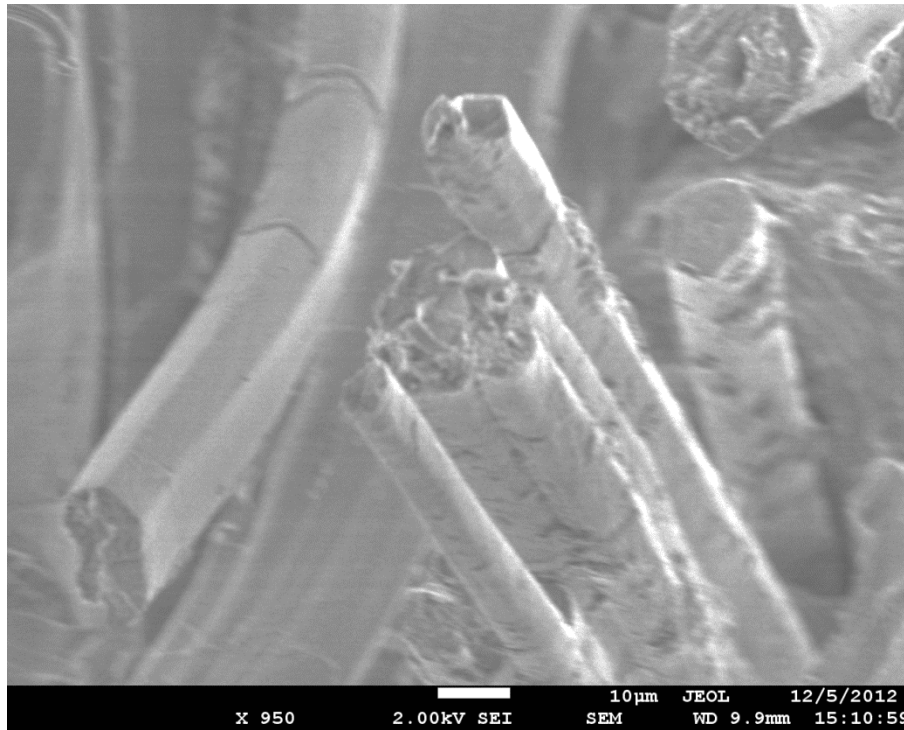


Fig. 96 SEM image showing fracture surface of the UF-N-30% composite

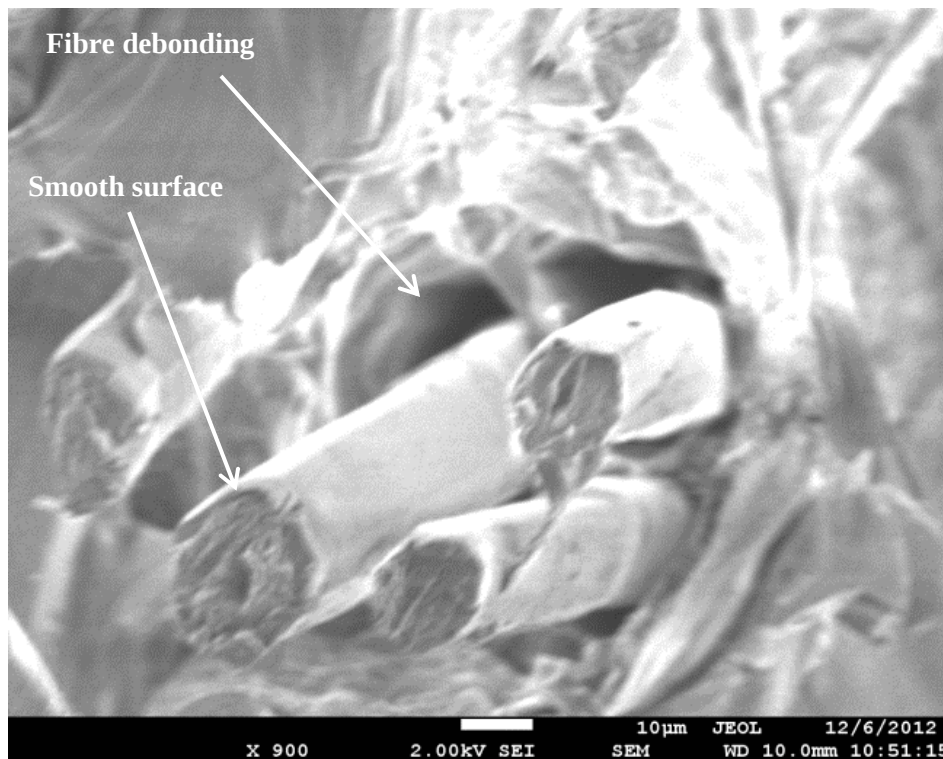


Fig. 97 SEM image showing fibre debonding in UF-N-30% composite

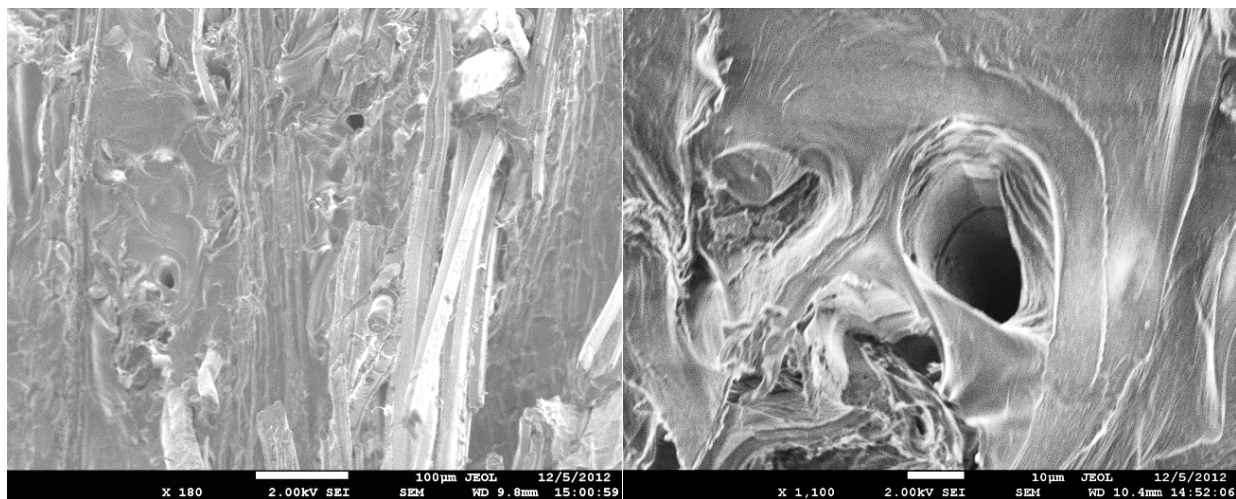


Fig. 98 SEM image of holes on fracture surface indicating fibre pull out in UF-N-30% composite. Left: Holes at 180x magnification / Right: One hole at 1100x magnification

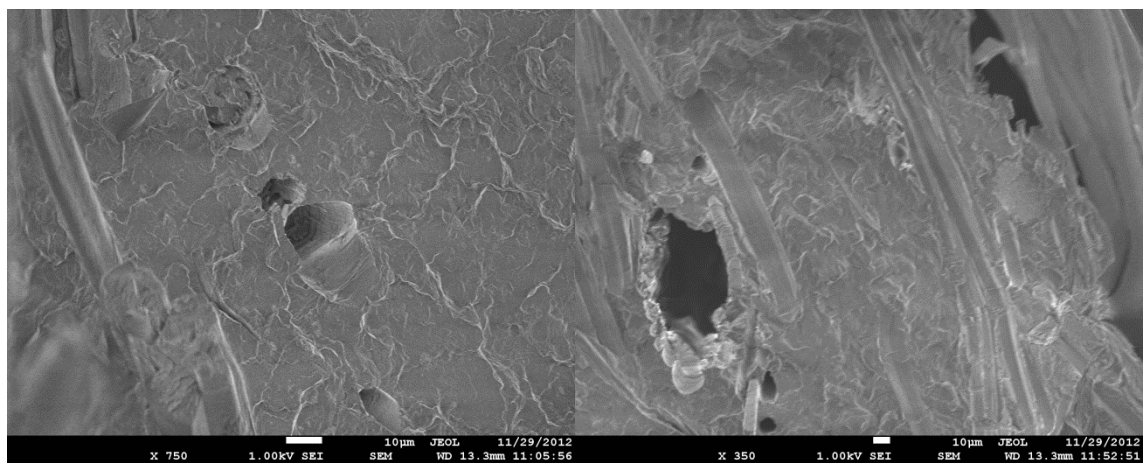


Fig. 99 SEM images of holes indicating fibre pull out in the TF-CNT-2%-30% composite

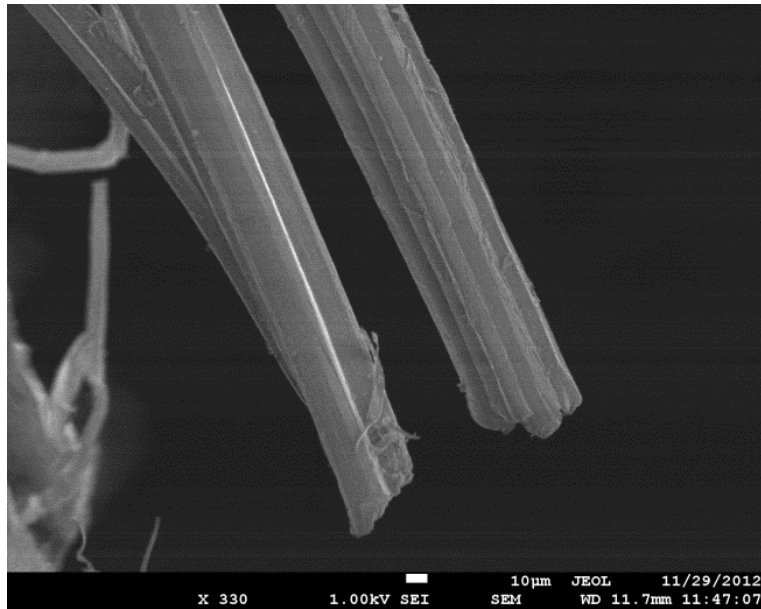


Fig. 100 SEM image showing lack of matrix bonded to fibres in the TF-CNT-2%-30% composite

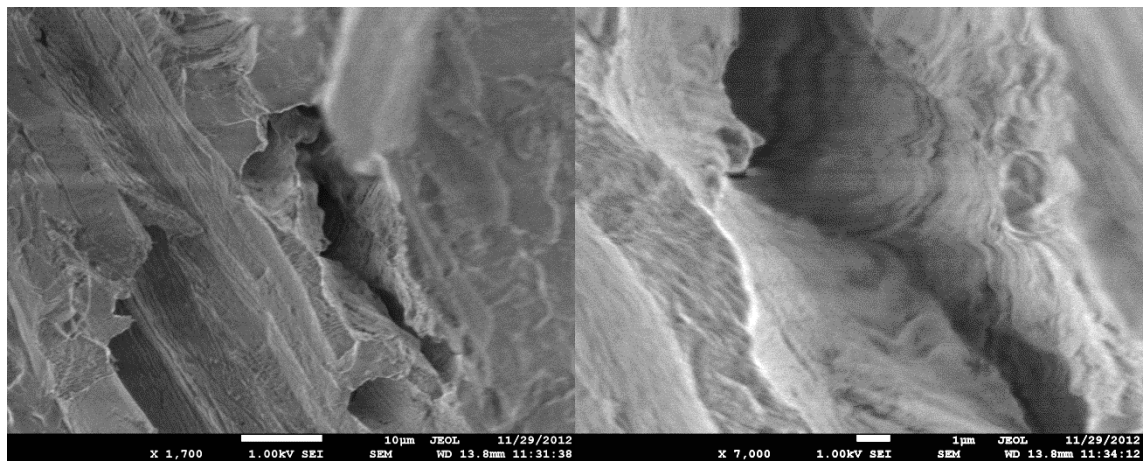


Fig. 101 SEM image of fracture surface of TF-CNT-2%-30% showing holes Left: Image at 1700x magnification / Right Image at 7000x magnification

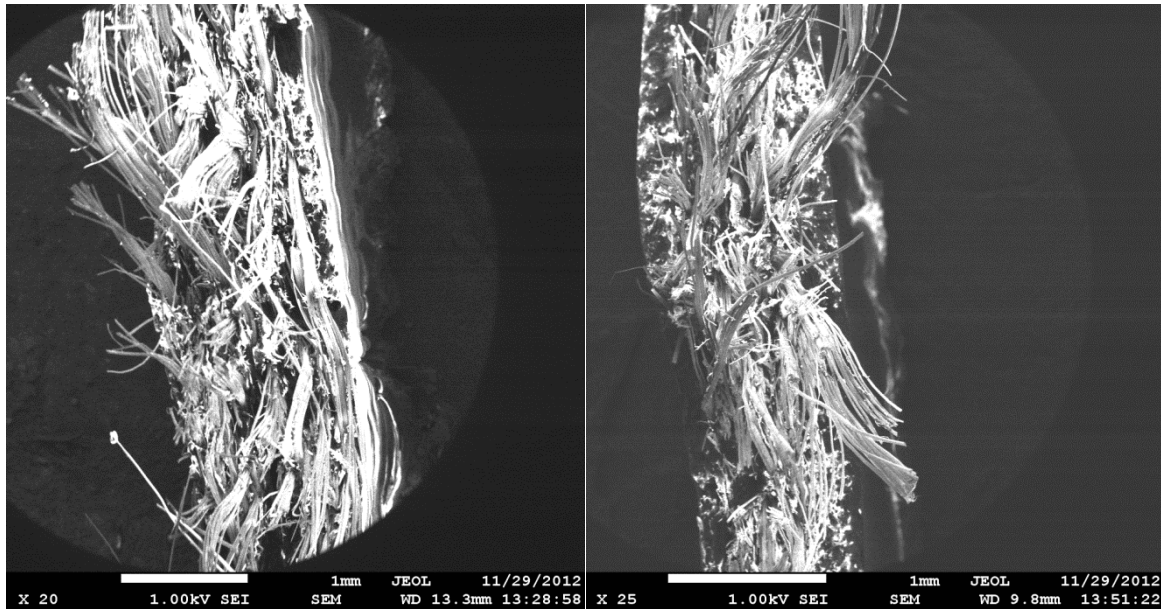


Fig. 102 SEM images showing the difference in fibre density for different samples in the TF-350nmGF-8%-30% composite: Left: Sample 4 / Right: Sample 5

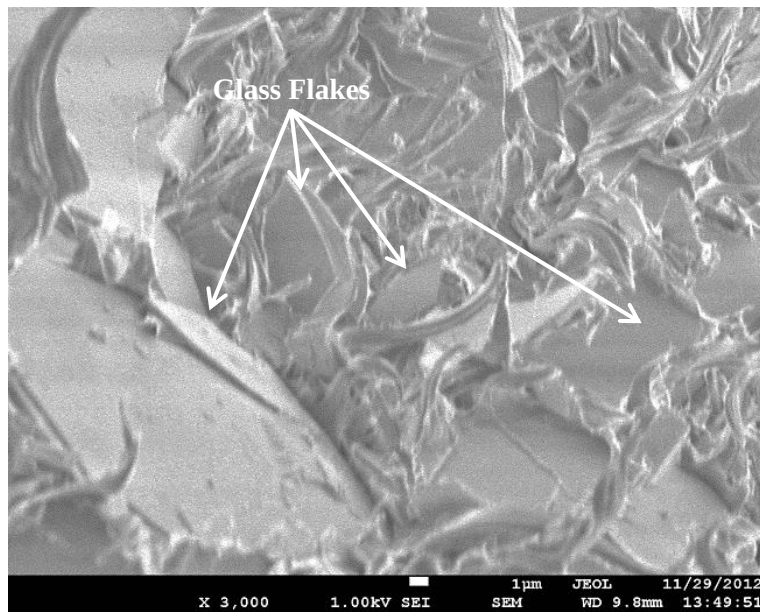


Fig. 103 SEM image showing evidence of glass flakes inside the TF-GF350nm-8%-30% composite

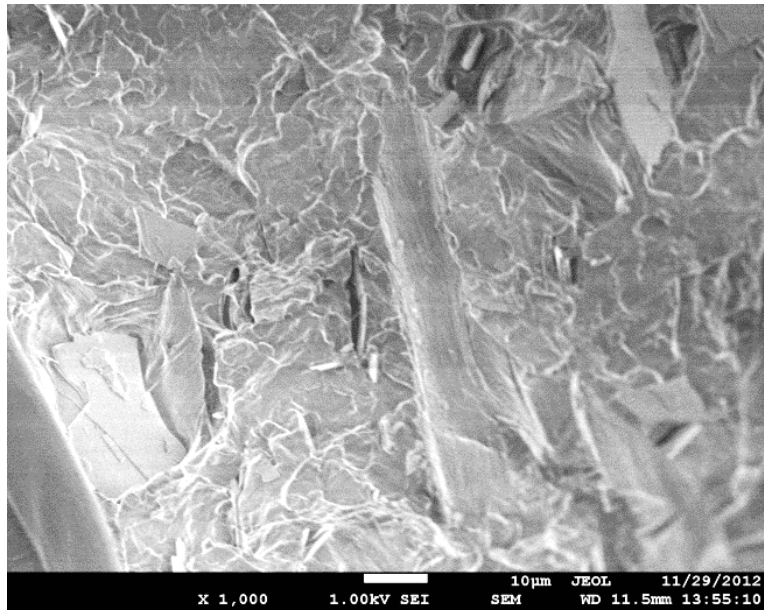


Fig. 104 SEM image of thin holes indicating glass flake pull out in the TF-GF350nm-8%-30% composite.

8 Discussion

The following discussion is split into two sections. The first section discusses the effect the fibre weight fraction and fibre treatment has on the tensile and water absorption properties of the composite plates. The effect the water absorbed has on the dimensions of the composites is also discussed in this section. The second section discusses the effect the fillers have on the tensile and water absorption properties of the composite plates.

8.1 Fibre treatment

8.1.1 Tensile properties

The effect of the fibre treatment on the tensile strength of the hemp fibre composites is illustrated in Fig. 47. For all fibre weight fractions, treated and untreated, the composites achieved a higher tensile strength when compared to unreinforced polypropylene. From this it is evident that the natural fibres have improved the strength of the polypropylene matrix. The treated fibre composites achieved higher strengths when compared with the untreated fibre composites. This trend is observed for all fibre weight fractions. The relative increase in strength is higher as the fibre weight fraction increases, 5% and 25% when comparing the 15% and 30% fibre weight fractions, respectively. This increase in strength can be explained by looking at the interfacial bonding between the fibres and the matrix. According to Mohanty *et al.* [35] a suitable coupling agent, such as silane, can be used to overcome the chemical incompatibility between the polar nature of lignocellulosic fibres and the non-polar nature of thermoplastics so as to improve the interfacial bonds. The improved interfacial bonds can explain the improved strength. The more fibres present, the more significant the increase. Fig. 92 shows polypropylene matrix bonded to the hemp fibres inside the TF-N-30% composite. This is in contrast to the absence of matrix on the surface of the untreated hemp fibres in Fig. 96. This comparison supports the theory of improved interfacial bonds as the matrix remains present on the surface of the treated fibres and not the untreated fibres after material failure.

It can be seen from Fig. 90 that the untreated hemp fibres have some surface debris. This is similar to the untreated kenaf fibres used by Asumani *et al.* [1]. Mohd Edeerozy *et al.* [34] stated that the alkali treatment cleans and chemically modifies the fibre surface, increases the surface roughness and improves significantly the mechanical properties of the fibres. Fig. 91 shows the fibres treated with 6% NaOH. It is evident that the alkali treatment has cleaned the fibres of this debris. This contributes to the improved strength of the TF-N composites as there is more exposed cellulose increasing the number reaction sites for the silane to bond to the fibres [36].

Figs. 88 to 90 show SEM images of the fracture surface for the TF-N-30% composite. These images reveal that the predominant mode of failure is due to fibre breakage. Figs. 88 and 89 show fibre breakage and evidence of the outer layer being torn off the interior of the fibre, respectively. These are fibre dominated failure modes. This indicates strong interfacial bonds between the matrix and the fibres. Fig. 95, however, shows evidence of both fibre debonding and good interfacial bonds. This indicates that the bonding could still be improved. Similar images to Fig. 94 were seen by I'Ons *et al.* [60]. These authors investigated a fibre treatment of polypropylene and found similar tearing of the fibre's outer layer. According to I'Ons *et al.* [60] this is evidence of a change in failure mode indicating improved interfacial bonding. These improved interfacial bonds lead to the improved strength seen in Fig. 47.

Figs. 92 and 93 show SEM images of the fracture surface of the UF-N-30% composite. These images reveal that contrary to the TF-N composites the predominant modes of failure are fibre debonding and fibre pull-out. Evidence of fibre debonding is illustrated in Fig. 97. There is a lack of fibre breakage as seen by the smooth surface, resulting from scissor cuts during manufacture of the mats, in Fig. 97 indicating more matrix dominated failure. The holes seen in Fig. 98 are evidence of fibre pull-out and not voids. These failure modes are clear evidence of poor interfacial bonds. These poor interfacial bonds lead to ineffective load transfer between the matrix and fibre. This is the cause of the poor strengths achieved by the UF-N composites.

From Fig. 47 it appears that the tensile strength increases with increasing fibre weight fraction. This trend is expected since the fibres have a higher tensile strength than the polypropylene matrix and so the higher the fibre content the stronger the resulting composite. The 20% fibre weight fraction deviated from this trend. The tensile strength dropped below the 15% fibre weight fraction for both treated and untreated fibres. This is an unexpected phenomenon which needs to be investigated. The moduli of the fibres were thus looked at to try and help understand this. Figs. 51 and 52 show the linear and secant moduli for the TF-N and UF-N composites. As with the tensile strength, the 20% fibre weight fraction does not follow the expected trend for both moduli. It is possible that the fibres were damaged at the compression stage of manufacture. As fibres are compressed together, the point of contact between two fibres may become damaged. This would result in failure at lower loads. The modulus, however, should not be affected as the damaged caused is localized at a specific point. Thus, the stiffness of the entire fibre should remain the same. The 30% fibre weight fraction was the only fibre weight fraction that was manufactured without stops (or spacer). Thus the fibre mat acts as the stop for the ram and so the fibres experience the full compressive force. Fundamentally this is the only difference in the manufacturing process between any fibre weight fraction. It would thus be expected that if the fibres were damaged due to the compressive load, there would be a drop in properties between the 25% and 30% fibre weight fractions and not at a

fibre weight fraction of 20%. It must be concluded that something else is causing this unexpected behavior. The composites were viewed using the SEM to specifically try and understand the cause of the unexpected phenomenon. One possible explanation for this trend is that at 15% the fibres are properly wet-out and at the higher fibre percentages they are not. If the composites were dry in the centre there would still be an increase in strength with increasing fibre weight fraction and so results may not show this. The SEM was used to look for dry fibres at the centre of the composite. No dry fibres were evident as illustrated by the SEM images in Figs. 38 to 41. These SEM images show that at all fibre weight fractions there is no evidence that the fibres are not fully wet-out. Matrix is clearly visible between all the fibres. The reason for the unexpected behavior at a fibre weight fraction of 20% remains unknown.

The 30% fibre weight fraction achieved the highest tensile strength. This correlates well with Asumani *et al* [1] even though the reinforcement used was different. Since the higher fibre weight fractions were not tested, it is not possible to determine if a drop off in strength occurs beyond this weight fraction or at some higher value. Dhakal *et al.* [61] and Keller [62] saw an increase in tensile strength up until 26% and 27% fibre volume fraction (approximately 32% and 33% fibre weight fraction)¹ of hemp, respectively. Mutje *et al.* [63] saw an increase in strength up until a fibre weight fraction of 30%. Keller [62] and Mutje *et al.* [63] saw a drop off in strength above 27% fibre volume fraction and 30% fibre weight fraction, respectively. These used different manufacturing techniques and resin systems, however the trend is consistent. This indicates that large improvements will most likely not be seen above a fibre weight fraction of 30%.

A 25% increase in strength is achieved using a 30% fibre weight fraction. This is much less than the 100% increase seen by Asumani *et al.* [1]. The difference in results obtained may be due to an inherent property of the fibres themselves. The alkali treatment removes a certain amount of hemicellulose, lignin, wax and oils covering the external surface of the fiber cell wall [36]. This increases the number of reaction sites for the silane to bond [1]. The weight percentage of lignin and hemicellulose present in hemp fibre are 6.06% and 10.7%, respectively [37]. In contrast the weight percentage of lignin and hemicellulose present in kenaf are 14.7% and 19.21%, respectively [38]. There is higher percentage of lignin and hemicellulose that can be removed from the kenaf fibre and so the effect of the treatment is more significant. From Fig.51 it is evident that the strength of the untreated kenaf fibre composite is slightly lower (approximately 5 MPa less) than that of the untreated hemp fibre composite. After the fibre treatment, however, the strength of the treated kenaf fibre composite is much larger (approximately 10

¹ Volume fraction is not equal to weight fraction. The density of hemp is 1480kg/m³ [28] and the density of polypropylene is 910 kg/m³ [2]. Therefore, the fibre volume fraction can be converted to a fibre weight fraction using these densities.

MPa) than that of the treated hemp fibre composite. It is plausible that the difference in lignin and hemicellulose percentage is the reason for this trend. A 50% improvement in strength was seen by Mehta *et al.* [10] using silane treated hemp. This is double the improvement in strength achieved during this work using the same natural fibre. Perhaps the hemp fibres underwent different treatments during cultivation prior to the alkali-silane treatments. This may alter the properties of the fibres and so the effects of the treatment may vary. Mehta *et al.* [10] also used a different silane compound to treat the fibres and manufactured the composites using a different matrix system. These differences may be the reason for the lower level of improvement experienced during this work.

Figs. 51 and 52 show the effect the fibre treatment has on the linear and secant moduli, respectively. Both the linear and secant moduli follow the same trends. The following discussion will not be specific as to which modulus is being referred to since the discussion applies to both moduli equally.

Figs. 51 and 52 show that the moduli increase with an increase in fibre weight fraction. This trend was expected as similar trends were experienced by Asumani *et al.* [1], Rassmann *et al.* [4] and Mutje *et al.* [63]. The fibres themselves have a modulus of 25 GPa [5, 11] and the matrix has a modulus of 1.3 GPa [2]. According to Mutje *et al.* [63] it is accepted that the modulus increases with the percentage of reinforcement, and that this depends on the degree of dispersion of the reinforcement in the matrix. If more fibres are added to the matrix the resulting composite will consequently become stiffer.

The TF-N composites achieved lower moduli than the UF-N composites at 15% fibre weight fraction. Taking into account the scatter there is no significant difference in moduli, when comparing the TF-N and UF-N composites, for fibre weight fractions ranging from 20% through to 30%. These results are contrary to results obtained by Asumani *et al.* [1]. It is possible that the decrease in modulus is a result of fibre degradation due to the alkali treatment. This would occur if the concentration of NaOH was too high for the hemp fibres. Asumani *et al.* [1] noticed that NaOH concentrations above 6% lead to the kenaf fibre degradation. Suardana *et al.* [13] noticed a drop off in strength at NaOH concentrations above 4% using hemp fibres, indicating fibre degradation. The strength of the composites will still increase due to the coupling agent and so the improved tensile strength results do not negate the possibility of fibre degradation. A range of NaOH concentrations were not tested and so SEM analysis of the fibre was done to determine if any fibre degradation was visible. Fig. 91 shows an SEM image of hemp fibre treated with 6% NaOH. There is no evidence of fibre degradation seen in this image.

Beckerman and Pickering [5] and Mutje *et al.* [63] investigated the effect of maleated polypropylene (MAPP) as a coupling agent to improve the mechanical properties of hemp fibre composites. They found that the MAPP improved the interfacial bonding of the hemp and the matrix. This was seen by the

increased tensile strengths achieved. Contrary to Asumani *et al.* [1] both works found no correlation between this improved interfacial bonding and the modulus. In some cases the modulus seemed to deteriorate as the interfacial bonds increased. Similar trends were experienced by Maldas and Kokta [64]. They investigated the effect of MAPP on woodflour-filled polypropylene composites. This is similar to trends experienced within this work. Improvements in tensile strength and SEM images indicate better interfacial bonds which were discussed previously. There is, however, not necessarily any improvement in tensile modulus as a result of this increased interfacial bonding.

8.1.2 Water absorption

Fig. 64 shows the change in mass over time for the TF-N samples exposed to water. This figure shows two important results of the water absorption tests. Firstly the shape of the data can be described using the equation:

$$y = a \times (1 - e^{-bt})$$

After long time periods, the mass stops increasing since it is fully saturated. The saturation value is represented by 'a'. The rate at which the water is absorbed is represented by 'b'. These two values can be used to compare different data sets. From the trend of the data it is evident that the samples absorb water more rapidly during the early stages of the test. Once the samples near saturation, the rate of water absorption slows down and the curve begins to flatten out. A similar trend was seen, when exposing natural fibres to water, by Rassmann *et al.* [4], Kymalainen *et al.* [65], Doan *et al.* [66] and Law and Ishak [67].

It is evident from Fig. 65 that the amount of water absorbed (saturation value) increases with an increase in fibre weight fraction. It is also evident from Fig. 66 that the rate of water absorption increases with an increase in fibre weight fraction. This indicates that the composites containing more fibres will reach their higher saturation value quicker than composites containing fewer fibres. A similar trend was noted by Rassmann *et al.* [4] and Law and Ishak [67]. It is important to note that the edges of the samples were not sealed off. This allows two separate paths for water diffusion (through the surface and along the fibres at the edge of the composite). The fibres are hydrophilic and the matrix is hydrophobic and so the water can diffuse along the fibres much easier than through the matrix. Therefore the majority of the water will diffuse through the edge and not through the surface. The composites tend to have a layer of polypropylene on either side of the fibres. This layer is thicker at lower fibre weight fractions and acts as a barrier for the water absorption. An image of this matrix barrier is presented in Fig. 37. The matrix barrier should limit the water absorbed by the composites as polypropylene is hydrophobic. This improvement is minimized since the samples were not sealed off at the edges. The fibres are short and

only penetrate a short distance into the composite. Further movement of water requires these fibres to be in contact with other fibres creating a pathway to the centre of the composite. The samples with the higher fibre content will thus have more fibres and so a higher chance of fibres coming into contact with one another. This will result in more water being absorbed and at a higher rate. This trend applied for all composites tested i.e. UF-N, TF-N, TF-CNT-2% and TF-350nmGF-8%.

The change in mass over time has the least error inherent in the measurement and was found to be the most consistent. The different composites are consequently compared to one another using this data. The effect of fibre treatment on water absorption is presented in Figs. 60 and 61. It can be seen that at fibre weight fractions of 15% to 25% the rate of water absorption and the saturation value of the composites with treated fibres have decreased relative to the composites with untreated fibres. At a fibre weight fraction of 30% both the rate of water absorption and the saturation value have increased when compared to the untreated fibre composite. This trend is contrary to that at lower fibre weight fractions. This phenomenon can be explained by considering Fig. 44. There appears to be more fibres exposed on the surface of the treated fibre composites. This creates a second pathway through the matrix for the water to diffuse to the centre of the composite. This leads to a higher rate of water absorption and a higher saturation value. The effect the fibres, at the surface, have on the water absorption of the TF-N-30% composite is illustrated in Fig. 73. There appears to be a linear trend within the data when comparing fibre weight fraction. The 30% fibre weight fraction, however, deviates from this trend. There appears to be more water absorbed than expected. The fibres exposed at the surface of the composites were only observed once the samples have been in the water for a short period of time. This is due to the fact that only then do they begin to swell and become more prominent and visible.

The increase in fibre numbers exposed on the surface of the composite may be a result of fibre agglomeration. It was observed that the treated fibres tended to agglomerate more than the untreated fibres during mat manufacture. These treated fibres tend to stick together and so may not be able to slide over one another as easily as the untreated fibres. The fibres will remain stiffer in the through-thickness direction. It is possible that when compressed the stiffer fibres will exert an opposing force stopping them from becoming flat. These fibres will remain at the surface and may penetrate through the matrix.

The change in area vs. exposure time for TF-N-15% is presented in Fig. 74. Contrary to results obtained by Rassmann *et al.* [4], the area decreased with increased exposure time. It is believed, though, that this effect is an artificial result of increasing curvature of the plate. The edges of the samples tended to curve inwards after manufacture. This is illustrated by Fig. 46. It is believed that the curvature results from the slight asymmetries in the plate lay-up. Variations in the fibre arrangement affect the in-plane properties of

the plate and possibly also affect the through-thickness position of the fibre mat. The asymmetry and variation in fibre mat position cause the coefficient of hygroexpansion to vary through the thickness of the composite. This difference in hygroexpansion causes the length of the composite to increase at different rates through the thickness. This leads to the curvature increasing as the fibres absorb more water. The increased curvature results in decreased measurements of length and breadth of the composites when taken from edge to edge. The decrease in dimensions leads to a decrease in area. No correlation between fibre weight fraction and area is seen in Fig. 75. Since these results are not believed to accurately reflect the true change in area, they will not be discussed any further.

Figs. 74 and 75 show the change in centre and edge thicknesses, respectively, for the TF-N composite at the various fibre weight fractions. It is evident that both the centre thickness and the edge thickness tend to increase with increasing exposure time. This effect was also documented by Almgren *et al.* [68] and Law and Ishak [66]. The fibres tend to swell more than they stretch [4] when immersed in water. The fibre swelling was also experienced by Rassmann *et al.* [4] and Law and Ishak [66]. They explain that the cell wall of the fibres contain hydroxyl and other oxygen-containing groups that attract moisture through hydrogen bonding. When the composites are immersed in water the cell wall will begin to swell, and the fibres expand until the cell wall is saturated with water [67]. The cell walls absorb most of the water and so greater swelling of the fibres is experienced in the radial direction than along their length [68]. This results in the change in through-thickness being larger than the change in in-plane dimension. The change in centre thicknesses for the composites with 15% and 20% fibre weight fractions were very low, less than 1% over 648 hours. Therefore, the data is highly susceptible to scatter and so a trend could not be accurately plotted. The same phenomenon was experienced for the edge thickness data for the 15% fibre weight fraction. The reason for the scatter is that some samples of the same fibre weight fraction have more fibres exposed on the surface than others. This is illustrated in Fig. 45. These samples consistently absorbed more water than the others of the same nominal type. The scatter appeared to be larger at higher fibre weight fractions since these composites have more fibres exposed on the surface.

From Figs. 74 and 75 it appears that the change in thickness tends to increase as the fibre weight fraction increases. This effect was experienced by all the samples tested and correlates well with data obtained by Rassmann *et al.* [4] and Law and Ishak [67]. In general, the edge thickness change was greater than the centre thickness change as seen from Fig. 89. This is because the fibres at the edges were exposed directly to the water and so these fibres absorbed more water than the fibres at the centre which were not exposed directly to the water. The water needed to travel along the fibres or diffuse through the matrix barrier to reach the fibres at the centre. The samples that did not follow this trend were those samples with more fibres exposed at the centre of the composite. Fig. 44 shows the TF-N-30% composite with a large

number of fibres exposed at the centre. Water is able to flow through these fibres and into the centre of the composite. From Figs. 74 and 75 it is evident that the saturation values for the change in centre thickness differs from the saturation values for the change in edge thickness at the same fibre weight fraction. This is contrary to what one would expect. At saturation the thickness change should be constant across the composite as no more water can be absorbed. The scatter present within the results prevents the thickness data from correlating as expected.

Assuming that polypropylene is inert, it would be expected that the change in thickness of the 30% fibre weight fraction would be approximately double the change in thickness of the 15% fibre weight fraction. However, the change in thickness experienced by the 30% fibre weight fraction was over ten times more than for the 15% fibre weight fraction. A similar phenomenon was documented by Law and Ishak [67]. They claim that the change in thickness of a composite having a 40% fibre weight fraction was 12 times more than for a composite having a 20% fibre weight fraction. The explanation for this is that a higher concentration of fibres within the composite increases the likelihood of fibres contacting one other. The rate of water diffusion along the fibres towards the centre of the composite will be compounded by this effect.

8.2 Fillers (carbon nanotubes and glass flakes)

8.2.1 Tensile properties

Fig. 48 shows the effect of adding 2% by weight of multi-walled carbon nanotubes to the polypropylene matrix reinforced with treated fibres. It can be seen that the strength does not increase with increasing fibre weight fraction as for the composites with no carbon nanotube filler. Instead the strength of all composites from 15% through to 25% is essentially identical. The strength of the composite with 30% fibre weight fraction is only slightly higher, however, the large amount of scatter within those results indicates that there is no clear improvement over the other fibre weight fractions. Therefore at the low fibre weight fractions, 15% and 20%, there is an improvement in strength relative to composites without fillers. Since the strength remains constant, the higher fibre weight fractions, 25% and 30%, are weaker than those without fillers.

Prashanta *et al.* [42] used a range of multi walled carbon nanotube concentrations to reinforce polypropylene. Improvements in tensile strength were seen from 19% using a concentration of 1% by weight to 35% using a concentration of 5% by weight. From results and work done by Prashanta *et al.* [42] it would appear that the strength of the polypropylene has been improved by the carbon nanotubes. From Fig. 48 it seems as if the amount of fibres present within the composite does not affect the overall strength as the strength seems to remain constant with increasing fibre weight fraction. It is possible that the improved strength of the matrix has been achieved at the expense of the interfacial bonds between the matrix and the fibres. SEM images showing matrix dominated failure modes support the idea of poor interfacial bonding between the fibre and the matrix. Fig. 99 shows holes present inside the matrix which is evidence of fibre pull-out. These holes were more frequent within the composites containing carbon nanotubes than the treated fibre composites without any filler. Fig. 100 shows an absence of matrix present on the surface of the fibres. The absence of matrix indicates that the interfacial adhesion is not strong enough to retain the matrix on to the surface of the fibre after material failure. The treated fibre composites, however, displayed evidence of good interfacial bonding seen by the fibre dominated failure modes evident in Figs. 87 to 90. Thus the addition of carbon nanotubes resulted in a reduction in interfacial bonding.

The poor tensile results may also be caused by holes within the composite. These holes are displayed in Fig. 101. The holes are not areas where fibre has been pulled out as their dimensions are not circular as seen in Fig. 99. These holes result from the carbon nanotube agglomerates created during the mixing process. Polypropylene cannot penetrate through these carbon nanotube agglomerates resulting in empty

space. The agglomerates are illustrated in Fig. 36. These holes create areas of high stress concentration and so lower the failure strength of the composite.

As discussed Prashanta *et al.* [42] saw improvements in strength using multi walled carbon nanotubes with concentrations ranging from 1%-5%. This indicates that some improvements should be seen over the range of carbon nanotube concentrations investigated. Fig. 50 shows the effect the concentration of carbon nanotubes have on the tensile strength of the composites at a fibre weight fraction of 30%. From Fig. 50 no improvement is evident with an increase in carbon nanotube concentration. The carbon nanotube concentration of 1% achieved the highest strength, however, when the scatter and outliers are taken into account there is no significant trend experienced within the results. The lack of trend seems to be a result of the opposing effects the carbon nanotube concentration has on the strength of the matrix and strength of the interfacial bonds. The increase in concentration of carbon nanotubes should improve the strength but simultaneously also decrease the strength of the interfacial bonds. These changes may occur at different rates and therefore a single trend is not visible. The strength for all carbon nanotube concentrations is below that of composites containing no filler. At even the lowest carbon nanotube concentration there is still a reduction in strength.

Fig. 49 shows the effect of adding 350 nm thick glass flakes to polypropylene at a concentration of 8% by weight. This concentration and size was chosen on the advice given by the suppliers. In contrast to the composites containing carbon nanotubes, the strength increases with increasing fibre weight fraction. This indicates that the glass flakes do not negatively affect the interfacial bonds between the matrix and the fibres. Glass flakes presumably increase the viscosity of polypropylene and so the matrix would have more difficulty penetrating through the fibres than for composites containing no filler. A drop in strength at the 20% fibre weight fraction, similar to the TF-N and UF-N composites, was not experienced and so using a more viscous matrix resulted in fully wet-out composites. The hypothesis of dry fibres for the TF-N and UF-N composites is again negated.

Similar images of the fracture surface of the glass flake composites were seen when compared to the treated fibre composites. These images showed evidence of matrix bonded to the fibre and fibre breakage as seen for the TF-N composites in Figs. 87 and 88, respectively. This indicates good interfacial bonds between the matrix and the fibres.

The glass flakes improve the tensile strength of the composites by 7%, 22%, 8% and 1% for 15%, 20%, 25% and 30% fibre weight fractions respectively. The improvement at a fibre weight fraction of 20% is larger than the others since the strength using no filler at this fibre weight fraction is lower than expected as discussed previously. At 30% fibre weight fraction, however, there is only a 1% improvement relative

to the composites containing no filler. Scatter in the tensile strength results was caused by the uneven fibre distribution of the hemp fibre mats. This uneven fibre distribution led to some areas having a higher fibre density than other areas. This is evident from Fig. 102. This figure shows a comparison between SEM images of two samples containing 350 nm glass flakes at a concentration of 8% and fibre weight fraction of 30%. Sample 4 had a higher fibre density and achieved a 16% higher tensile strength than sample five.

Fig. 103 shows an SEM image of the glass flakes inside the composite. The glass flakes appear to be bonded well to the matrix as there was no evidence found showing areas of debonded polypropylene. The adequate bonding resulting from the silane treatment improves the overall strength of the composite. Fig. 104 shows holes which might seem similar to the holes seen inside the carbon nanotubes composites. The holes would result from glass flakes agglomerates. Polypropylene cannot penetrate through the agglomerates resulting in empty space. From Fig. 36 it appears that there are glass flake agglomerates present inside the matrix after the mixing process, similar to those encountered for the carbon nanotubes, however, their frequency were much less. On closer inspection, it is evident that these are not holes resulting from the glass flakes agglomerates. Due to the shape of the holes it appears that the glass flakes have been pulled out of the matrix. The flake pull-out is evidence of poor bonding between the matrix and the glass flakes. Even though there appeared to be good bonding, there is evidence showing it can still be improved in some areas.

Fig. 50 shows the effect the concentration and size of the glass flakes have on the tensile strength of the composites at a fibre weight fraction of 30%. It can be seen from this figure that the tensile strength increases slightly with an increase in glass flake concentration up until 12%. Up until a concentration of 8% the strength of the composites is lower than the strength without the filler indicating that the mechanical properties have decreased slightly. It can also be seen that the 350 nm glass flake achieved the highest strength. Both the 100 nm and 750 nm glass flake composites achieved results slightly lower than the composites containing no filler. Changes in strength resulting from the glass flakes are very small for all glass flake size and concentration. The improved strength at the high fibre weight fraction is most likely dominated by the fibres and so the glass flakes have little effect irrespective of size and concentration.

Figs. 53 to 56 show the effect the fillers have on the linear and secant moduli. Figs. 57 and 58 show the effect the filler concentration and size have on the linear and secant moduli. As with the UF-N and TF-N composites both the linear and secant moduli follow the same trend. As a result of the scatter present

within the results there is no direct correlation between the filler, the filler percentage or the filler size on the tensile moduli.

8.2.2 Water absorption

One interesting observation needs to be discussed prior to the discussion on the effect the filler has on water absorption properties. The change in edge thickness for treated fibre composites with and without fillers seems to follow an approximately linear trend with increasing fibre weight fraction. This trend is illustrated by looking at Figs 79 and 81. This constant trend is expected as fibres at the edge of the samples are directly exposed to the water and therefore not affected by the fillers.

Figs. 62 and 63 show the effect of adding 2% by weight of multi-walled carbon nanotubes to the polypropylene matrix on the saturation value and rate of water absorption, respectively. Both the saturation value and the rate of water absorption increase with increasing fibre weight fraction. This is similar to the trend experienced by the TF-N composite. The effect of the carbon nanotubes will be discussed with respect to the change in mass and compared to that of the TF-N composites. This comparison is done since all composites containing fillers use treated fibres as the reinforcement.

It is important to note that at the 30% fibre weight fraction the treated fibre composites performed very poorly when compared to the lower fibre weight fractions and so it is difficult to compare results. It is thus important to compare results at the lower fibre weight fraction to understand the effect of the carbon nanotubes. At the lower fibre weight fractions the composites containing carbon nanotubes performed poorly, however, at the 25% fibre weight fraction results were similar. This decline in results is most likely due to the holes created in the matrix by the carbon nanotube agglomerates. It was explained previously, however, that these holes create pathways for the water to diffuse through the thickness of the composite. This allows the water to penetrate through to the centre of the composite easier. This may lead to the higher rate of water absorption experienced. It is known that water molecules can diffuse down single walled carbon nanotubes of diameter 1.08 nm [69]. In the present work we are unsure of the diameter of the innermost wall of the multi walled carbon nanotubes in use, but since they have an outer diameter of around 15 nm there is plenty of room to accommodate an inner most wall of 1.08 nm. It would therefore be expected that the rate of diffusion would increase above that were diffusion along the natural fibres is the only route into the interior of the specimens. This mechanism would not affect glass flake composites as the glass flakes are flat and have no holes through their centre. The water will find it difficult to diffuse through the glass flakes and so will have to diffuse around them. The results using glass

flakes should thus be better than using carbon nanotubes. This is not evident from Figs. 62 to 65 and so the hypothesis is not valid.

Figs. 66 and 67 show the effect the concentration of carbon nanotubes have on the saturation value and rate of water absorption, respectively, for composites at a fibre weight fraction of 30%. The 2% concentration of carbon nanotube absorbs the least amount of water once it reaches saturation. The rate of water absorption increases with increasing carbon nanotube concentration. This may be a result of the increased number of holes at higher carbon nanotube concentrations. Having said this, there seems to be no carbon nanotube concentration that improves the water absorption properties of the composites.

Figs. 64 and 65 show the effect of adding 8% by weight of 350 nm thick glass flakes to the polypropylene matrix on the rate of water absorption and the saturation value, respectively. Similar trends were experienced as compared to the carbon nanotubes. No improvements were seen at low fibre weight fractions. A similar phenomenon as for the carbon nanotubes can explain this result. The rate of water absorption using the 8% concentration is significantly lower at higher fibre weight fractions. The glass flakes are packed randomly on top of one another through the thickness of the composite. This should force the water to diffuse around the layers of glass flakes. This would not stop the water from penetrating through to the centre of the composite and so saturation values should not be as effected. The time taken, however, to reach this saturation value should be increased.

Figs. 66 and 67 show the effect the concentration and size of glass flakes have on the saturation value and rate of water absorption, respectively, for composites at a fibre weight fraction of 30%. Both the saturation value and the rate of water absorption seem to decrease with an increase in glass flake concentration up to a concentration of 8%. Above this concentration the properties appear to drastically deteriorate. At a glass flake concentration of 12%, there appeared to be more filler agglomerates evident than at the lower concentrations. These agglomerates presumably create holes and thereby pathways for the water to diffuse through the thickness to the centre of the composite. This may be the reason for the poor performance at the higher concentration. The 350 nm thick glass flakes achieved the best results when compared to the other glass flake sizes. It is interesting to note that the best performing glass flake size and concentration corresponds well with the information given by the supplier.

9 Conclusions

Based on the results of the investigation, the following conclusions can be drawn:

1. An increase in fibre weight fraction increased both the tensile strength and the elastic moduli.
2. A fibre weight fraction of 30% achieved the highest tensile strength.
3. Treatment of the hemp fibre using 6% NaOH and 3-aminopropyltriethoxysilane improves the tensile strength of the composites due to the improved interfacial bonding between the fibres and the matrix.
4. Treatment of the hemp fibre using 6% NaOH and 3-aminopropyltriethoxysilane reduced the tensile moduli.
5. Mass increase resulting from water absorption can be described using the following formula:
$$y = a \times (1 - e^{-bt}).$$
6. The saturated water content of the composites increase with an increasing fibre weight fraction. There are more fibres present at high fibre weight fractions. This increases the amount of water absorbed by the composite. In addition to the increase in fibre content, more fibres became exposed at the surface. This allowed water to diffuse along these fibres and so the composites absorbed more water.
7. Since the composite edges were not sealed, the composites swelled more around the edges than at the centre.
8. Fibre treatment improves the water absorption properties of the composites below a fibre weight fraction of 30%. A drop off in properties at 30% fibre weight fraction results from the fibres being exposed on the surface of the composite.

9. The carbon nanotubes improve the strength of the polypropylene matrix. This improved strength improves the tensile strength of the composite at low fibre weight fractions. The carbon nanotubes, however, may reduce the strength of the interfacial bonds between the matrix and the fibres. This results in a reduction in tensile strength at high fibre weight fractions.
10. Glass flakes slightly improve the tensile strength. The TF-GF350nm-12% composite achieved the highest tensile strength.
11. As a result of the scatter present within the results there is no direct correlation between the filler, the filler percentage or the filler size on the tensile moduli.
12. No significant improvement on the water absorption properties resulted from the carbon nanotubes.
13. At high fibre weight fractions, there appears to be an improvement in water absorption properties using 350 nm glass flakes at a concentration of 8%. No significant improvement on the water absorption properties resulted from the other glass flake sizes or concentrations.

10 Recommendations

1. Varying the parameters used for the rheomixer may be able to limit the number of agglomerates created during mixing of the matrix and filler. Reduction of agglomerates may result in improved water absorption properties and less variation in the mechanical properties.
2. An investigation into varying the concentrations of NaOH needs to be done to determine the best process for hemp fibre composites.
3. Experimental investigation into why fibres are exposed on the surface of the composite at high fibre weight fractions should be done.
4. Ways in which to improve the interfacial bonding of composites containing carbon nanotubes should be investigated.

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12 Appendix A (Development of mat manufacture process)

Non-woven hemp fibre mat could not be purchased at the required density and so methods of creating them in-house needed to be determined. A number of different methods were investigated and these were as follows:

- Water Trough
- Compressed Air Pipe
- Water Pipe

12.1 Water trough

The first method investigated was the water trough method. This method involved stirring fibres, roughly 5 mm in length, in a container filled with water. This allowed the fibres to disperse randomly inside the water. This was followed by lifting a wire mesh such that it would collect the randomly distributed fibres and create a uniform non-woven mat. The apparatus used for this method can be seen in Fig. 105. The idea behind this method is that the fibres would randomly disperse themselves inside the water due to the vigorous mixing process and once the mesh has been raised there would be a random distribution of fibres on top of the mesh. The fibres would then dry and a non-woven mat would result.

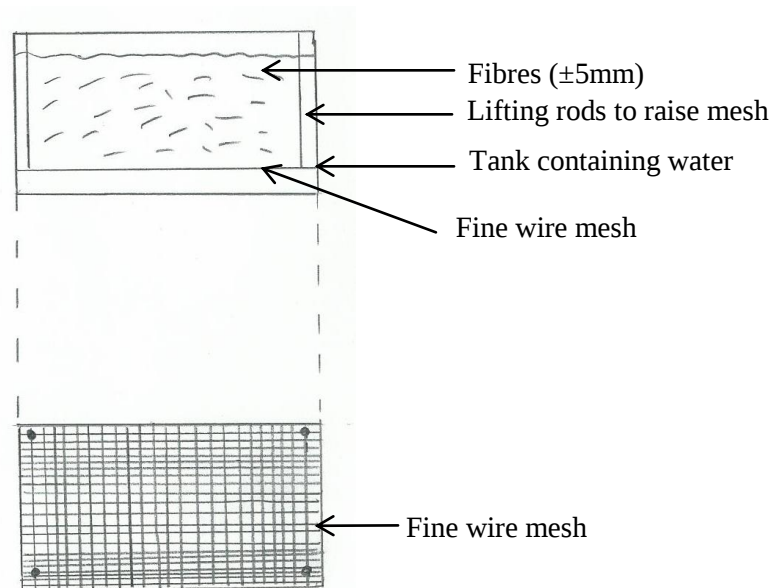


Fig. 105 Water trough design

A number of issues arose during testing of this process. Firstly it was very difficult to pull the mesh up without tilting it, which led to more fibres being forced one way or another. This problem was solved by

joining the lifting rods to a frame above the mesh. Secondly, the fibres hooked onto one another when they come into contact in the water which large highly dense areas and areas where there are very little to no fibres present. Vigorous mixing was used to separate the agglomerates but the fibres begin to agglomerate very quickly once the mixing process stopped. One method of solving this problem is to create a very high trough with a very low fibre volume fraction. This approach would limit contact between individual fibres and thereby limit agglomeration allowing for a more uniform fibre distribution. This idea this led to the second method, the use of compressed air inside a long pipe.

12.2 Compressed air pipe

This method uses the fact that when a fluid flows through a pipe there is turbulence at the inlet. This would allow the short fibres to disperse evenly and randomly in the turbulence at the entrance to the pipe. A mesh at the exit of the pipe would catch the fibres. This set up is illustrated in Fig. 106. Instead of water, compressed air was investigated to try to avoid high water losses. The fibres could either be hand fed into the air stream as illustrated in Fig. 106 or can be loaded into a chamber which as illustrated in Fig. 107. The chamber operates in the following manner. Compressed air flows into the chamber through the inlet tube. This creates a pressure difference between the inside and outside of the chamber. A second tube (exit tube) is connected between the chamber and the entrance of the pipe. Since air flows from high pressure to low pressure, the air inside the chamber flows up the pipe towards the mesh. The fibres inside the chamber follow the air stream and collect on the mesh.

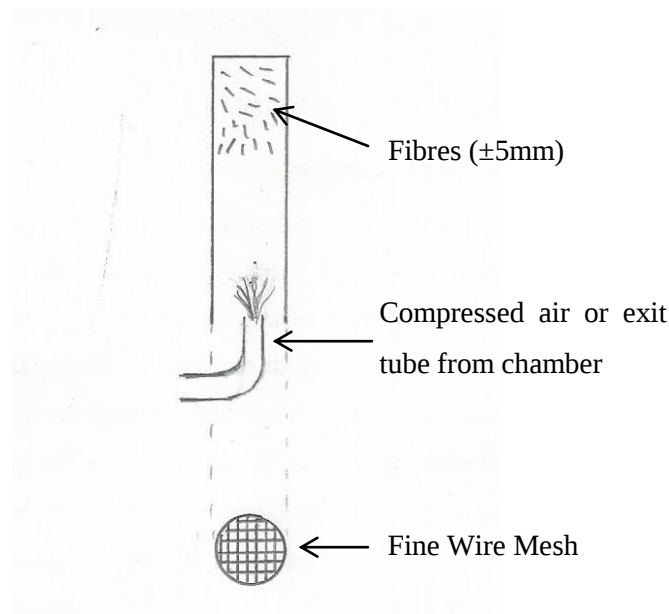


Fig. 106 Compressed air pipe

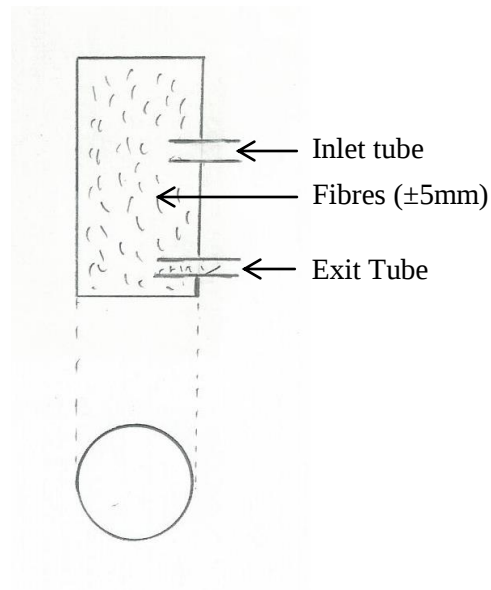


Fig. 107 Chamber

Again a number of issues arose during testing of this method. Firstly, if the fibres blocked the exit pipe the chamber burst. This happened frequently due to the size of the tubes used. The second issue was that the fibres seemed to collect around the edge of the mesh as opposed to being randomly distributed. The final issue is that not all the fibres were collected on the mesh. This was due to two problems; some of the fibres passed through the mesh and some simply fell off the mesh once air flow ceased. This issue made manufacture of mats with specific aerial densities more difficult.

It was subsequently decided to abandon the use of compressed air and to revert back to the use of water as the suspension medium.

12.3 Water pipe

The water pipe method uses the concept of turbulence inside a pipe as well as at the inlet to randomly disperse the fibres to achieve a uniformly distributed non-woven mat. At the inlet, the water flows into the pipe similar to a fountain and creates vortices in the corners which mix the fibres. Once water is pumped into the pipe, the fibres are forced up to the top of the pipe and collected on the mesh. Small scale tests were done and good results were obtained and so this was the final method chosen. The method is discussed in greater detail in section 4.2

13 Appendix B (Drawings)

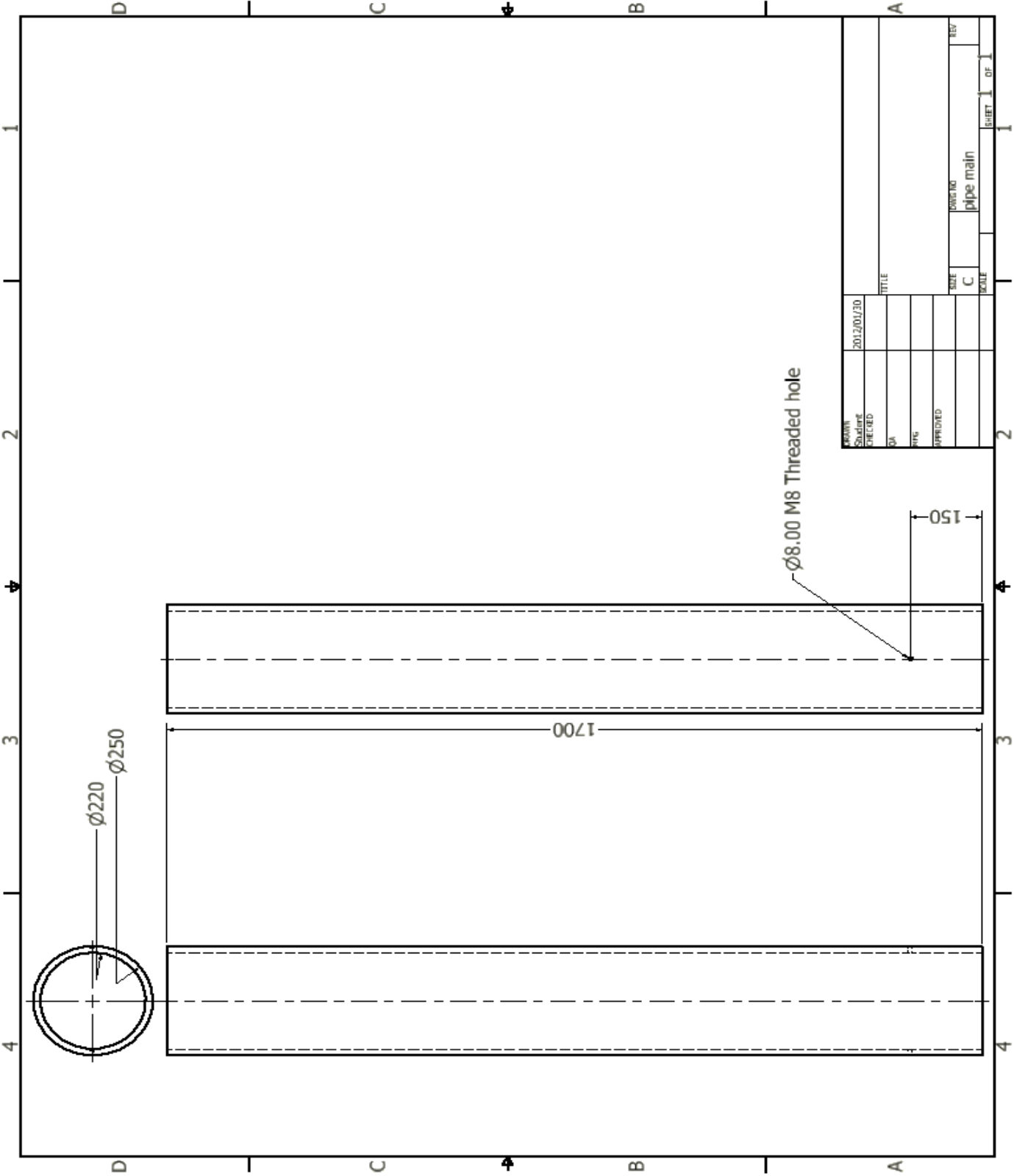
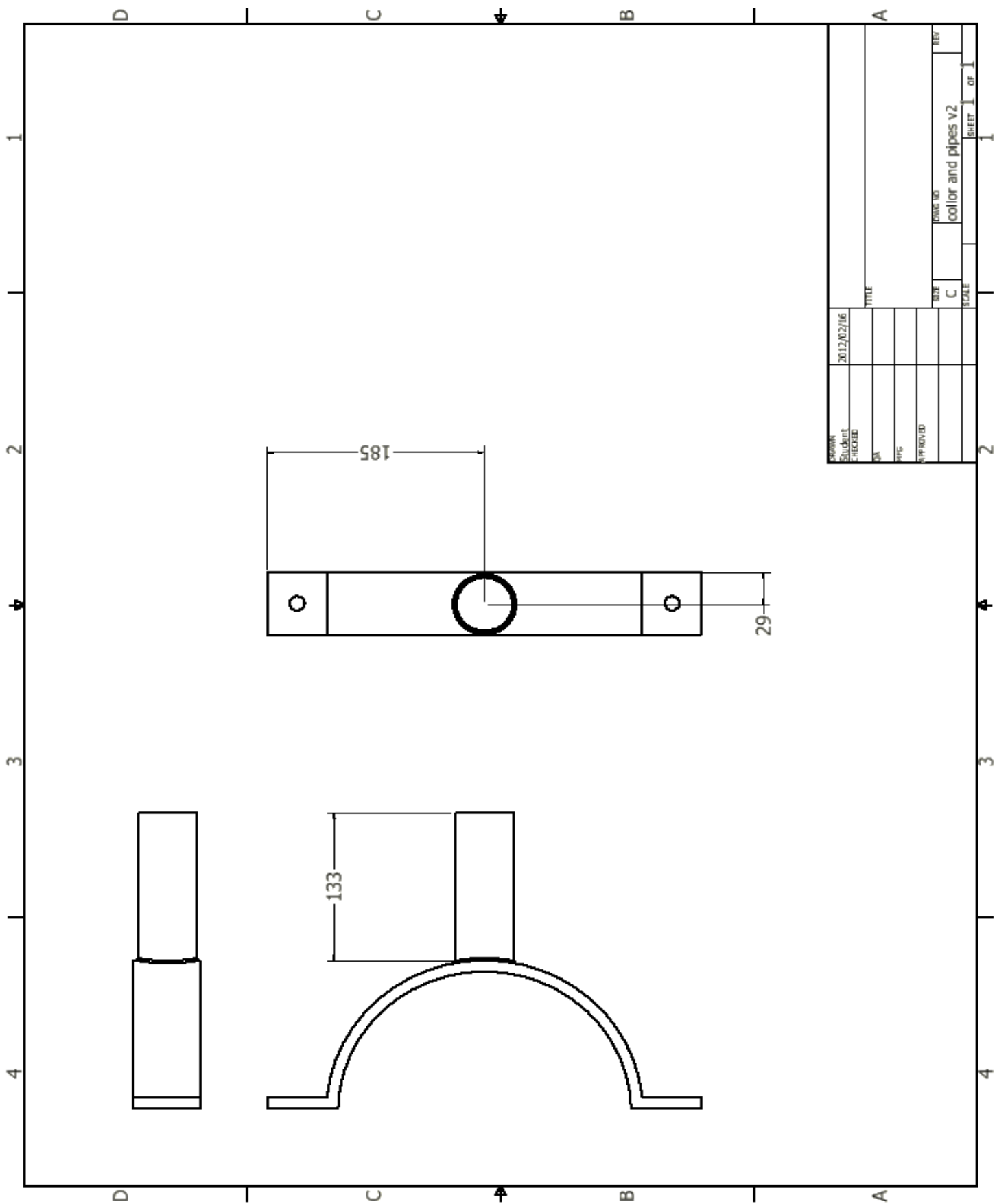


Fig. 108 Pipe used for water pipe method



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Fig. 109 Clamp and bar assembly

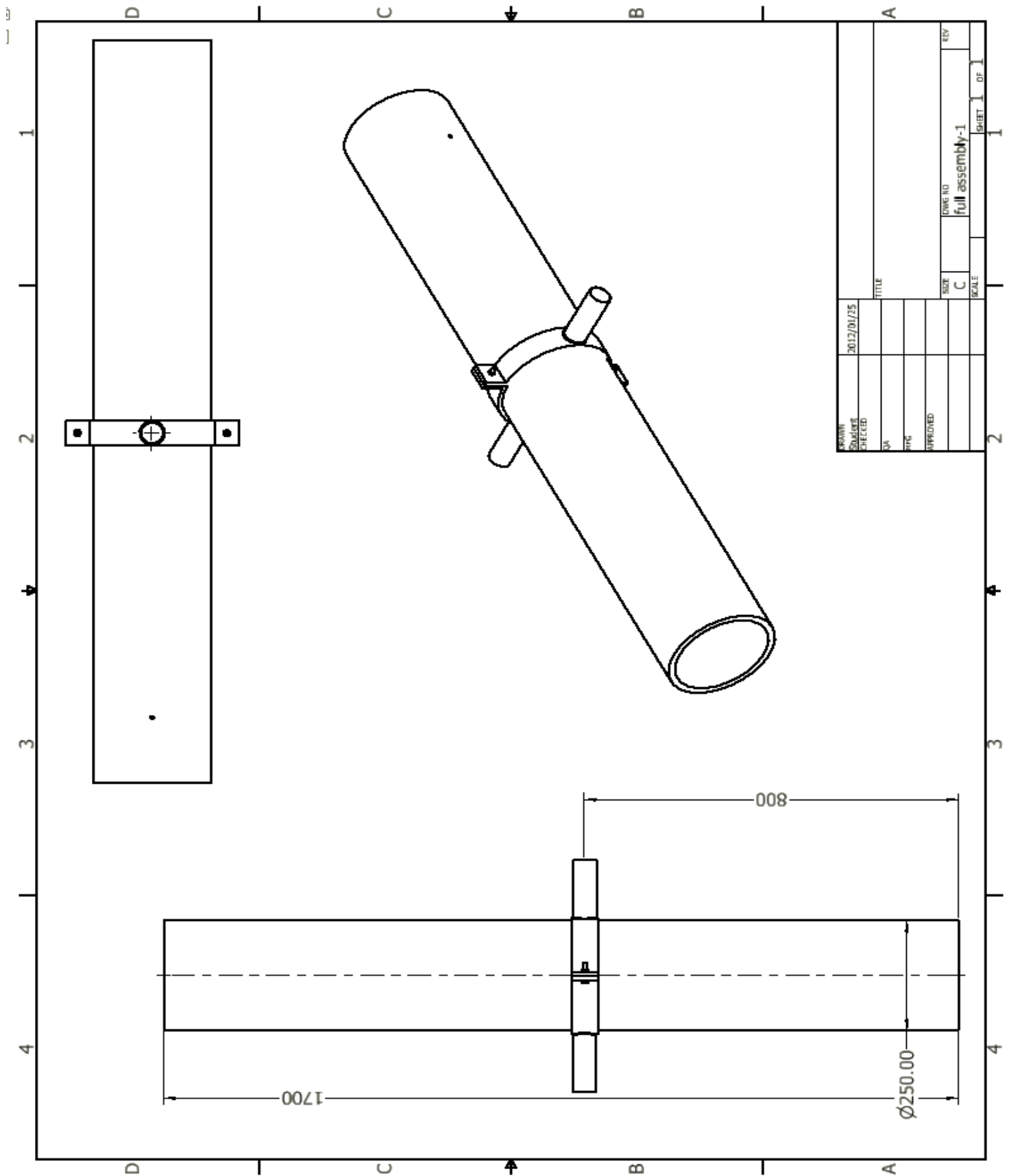
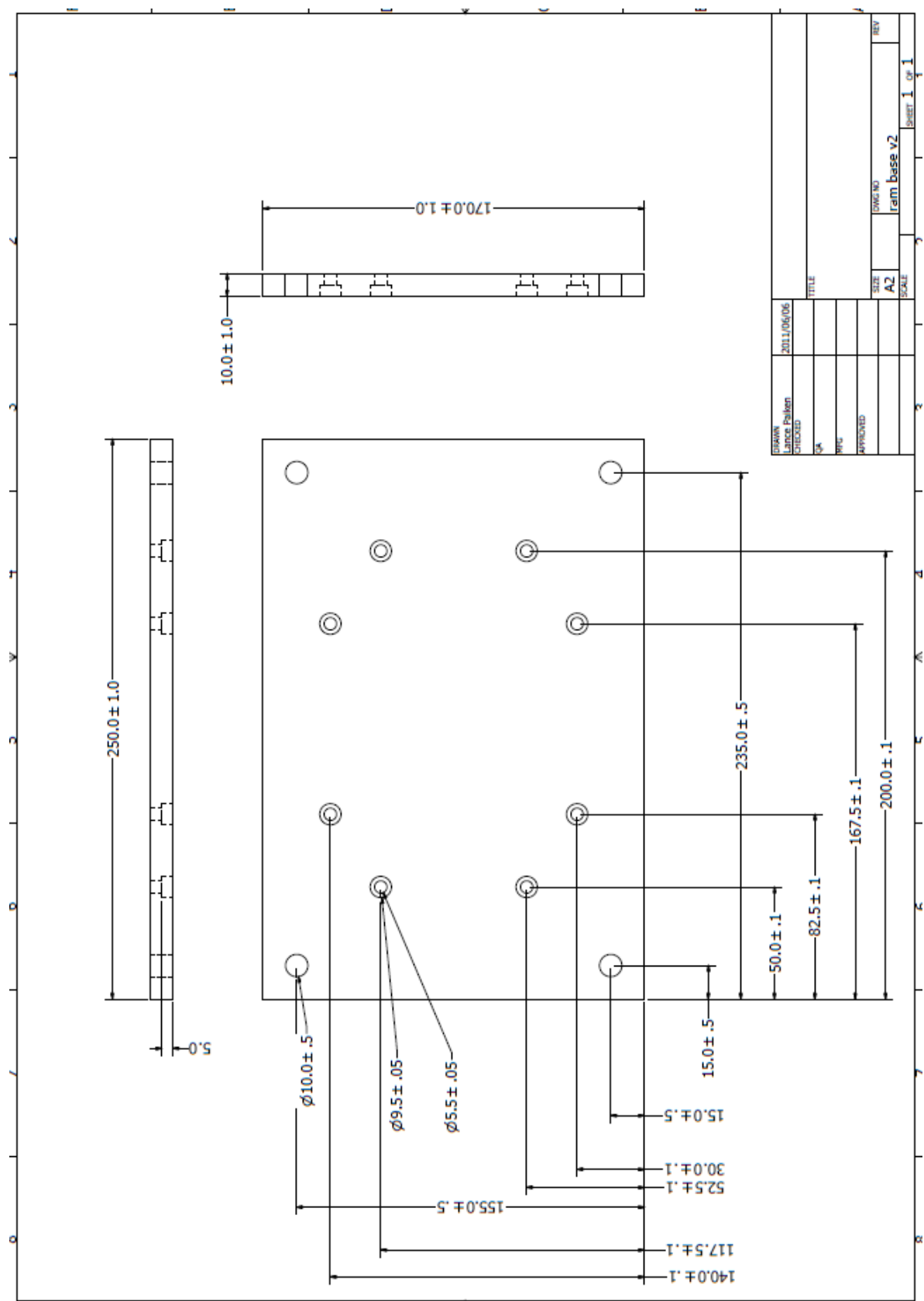


Fig. 110 Full assembly for water pipe method



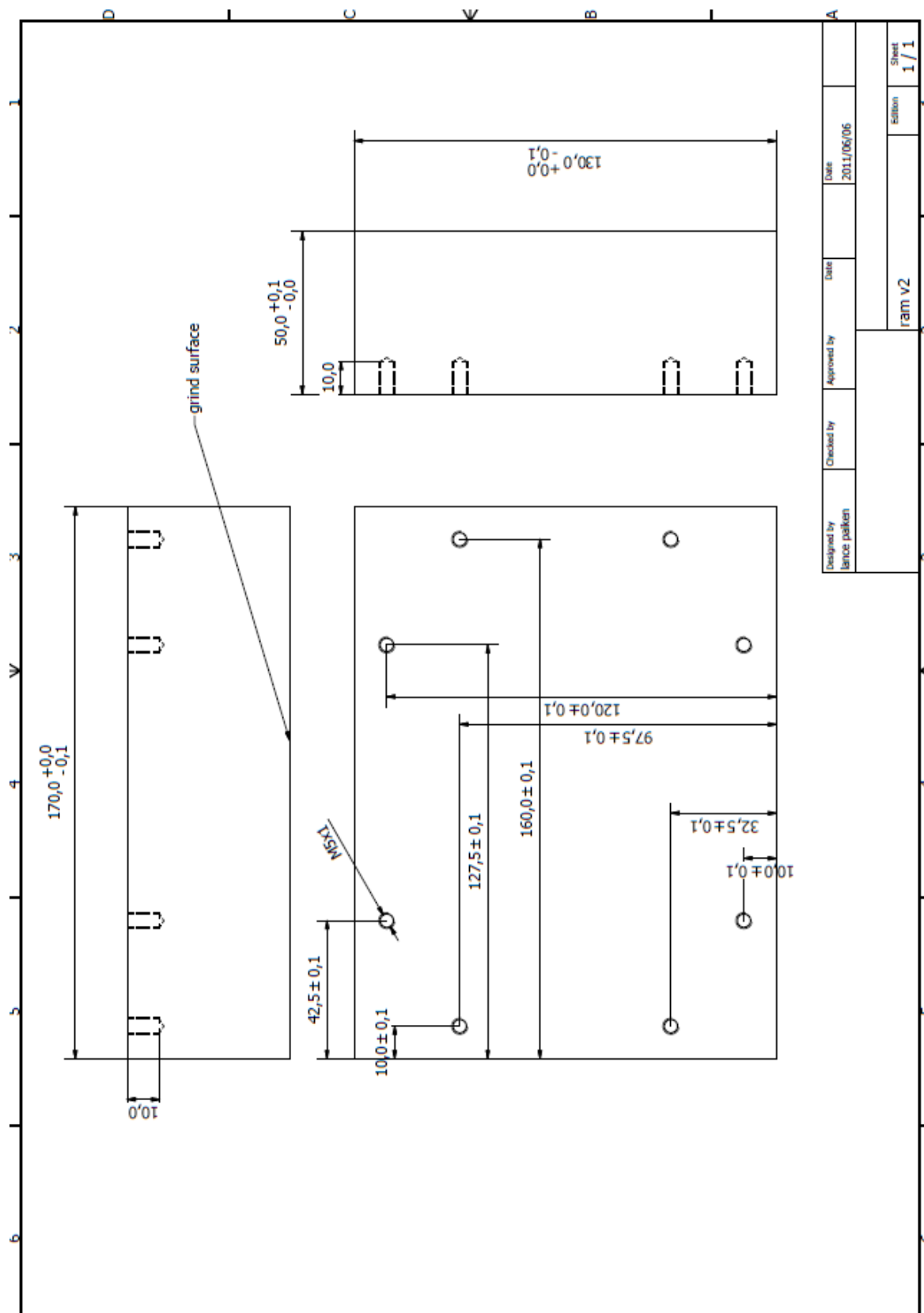


Fig. 112 Drawing of solid piece for male part

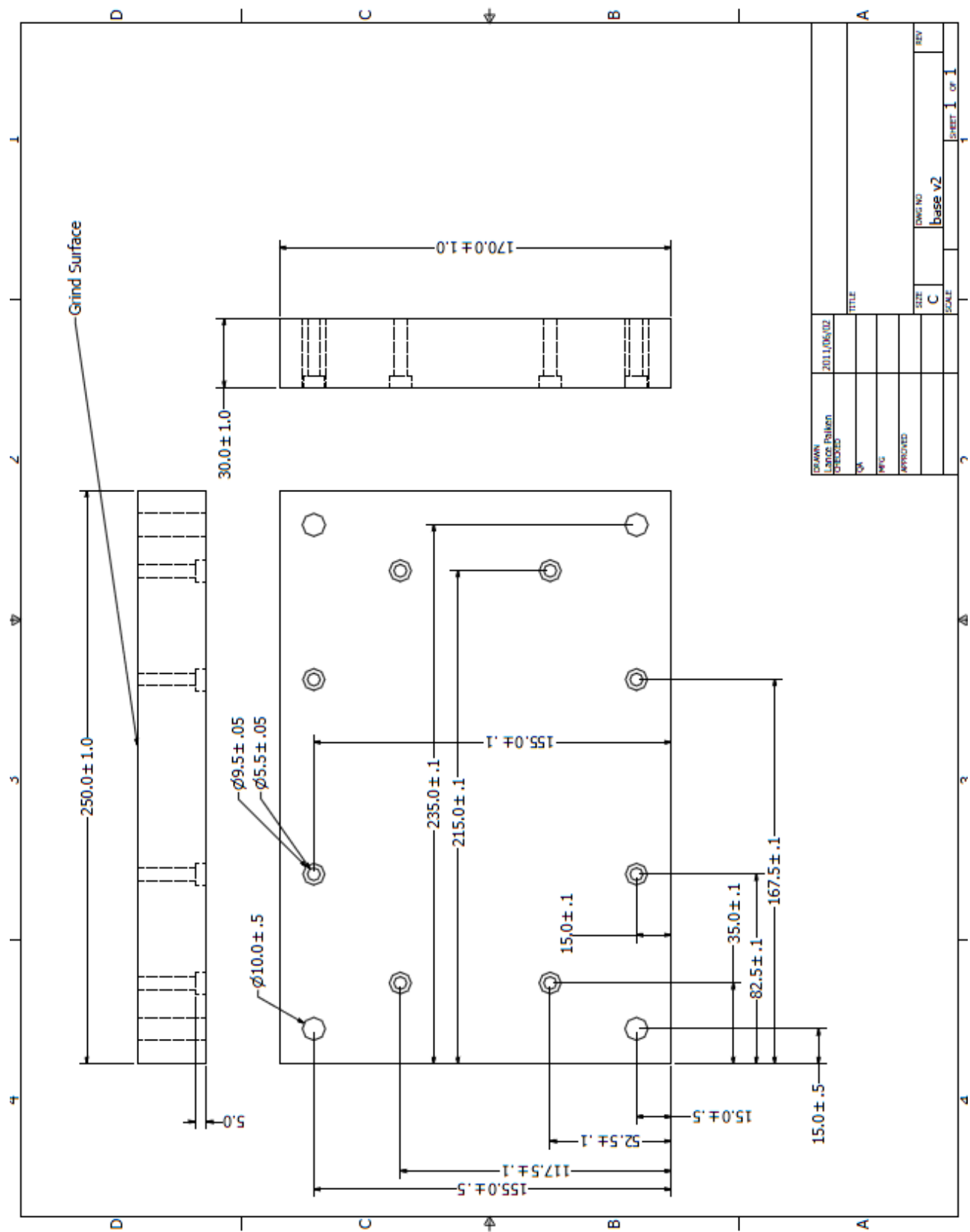


Fig. 113 Drawing of base for female part

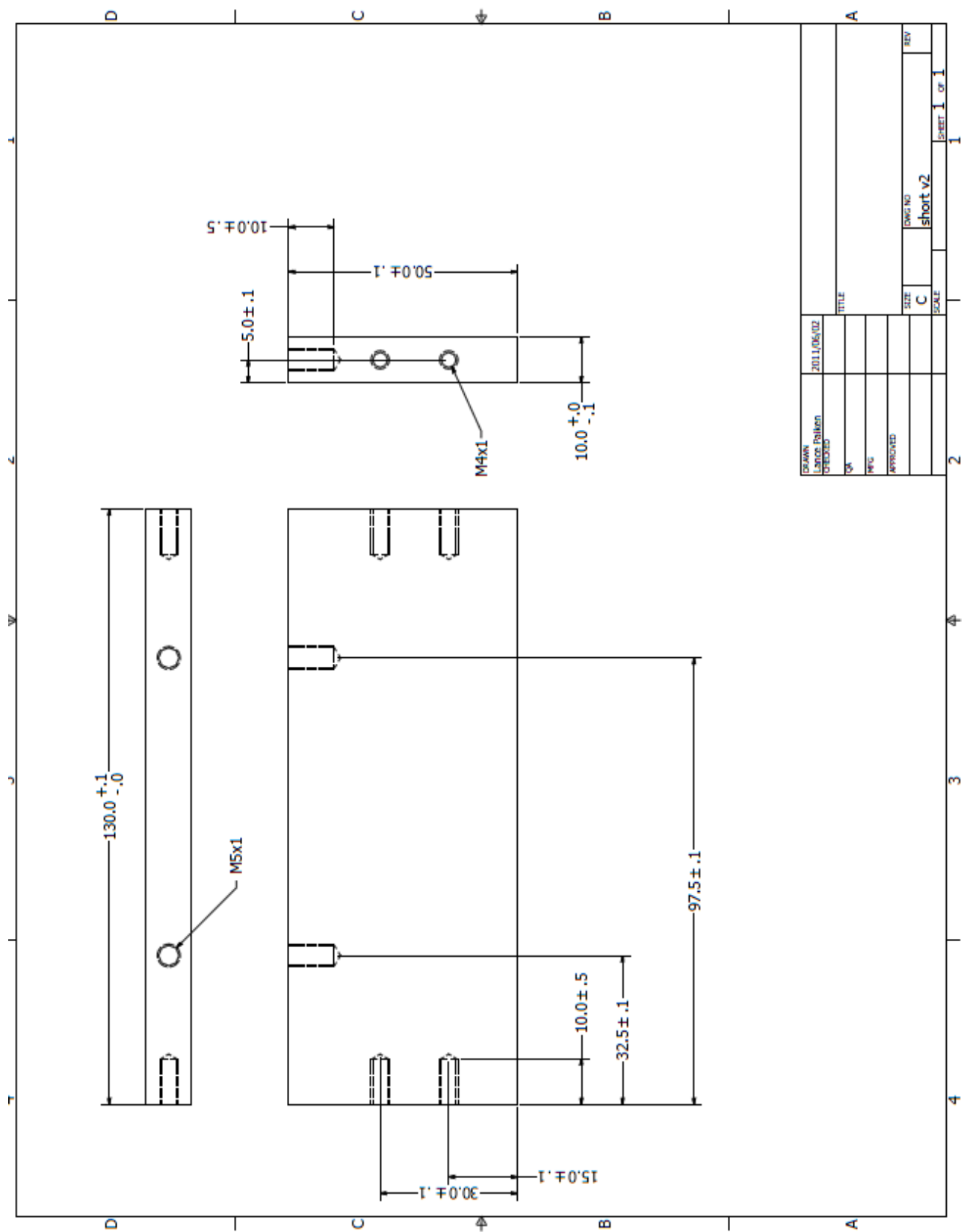


Fig. 114 Drawing of short side piece for female part

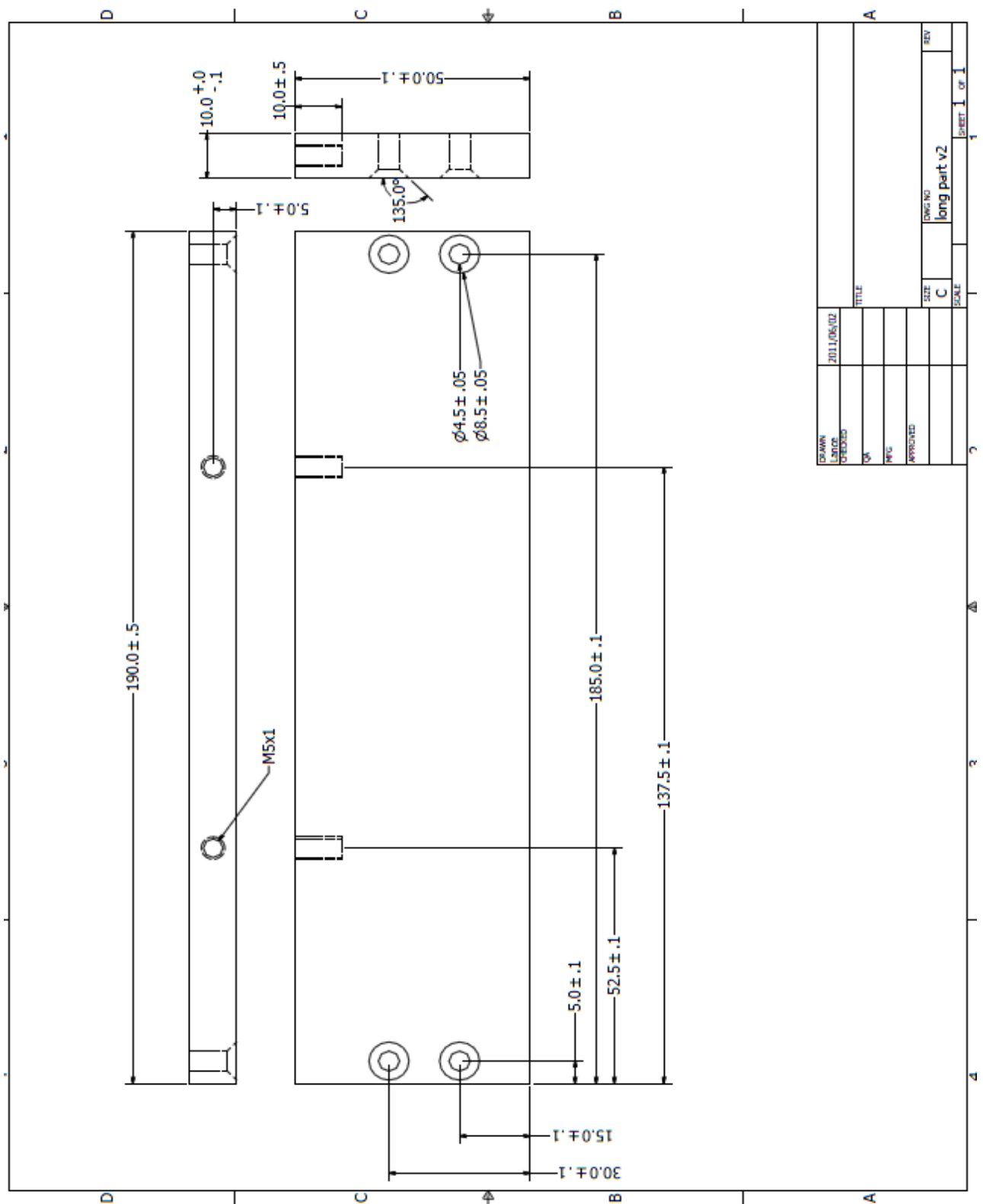


Fig. 115 Drawing of long side piece for female part

14 Appendix C (Development of composite manufacturing process)

The development of the manufacturing process began with the parameters set out by Asumani *et al.* [1]. These parameters produced good results. This needed to be altered since the mould used is different, the fibres used are different and the matrix used is not in a single sheet. The process set out by Asumani *et al.* [1] is as follows:

The material charge is made by sandwiching polypropylene sheets and layers of kenaf fibre mats impregnated with polypropylene powder. The material charge is loaded into the mould cavity. Thin PFA sheets of 0.2 mm thickness were used as a release agent to allow smooth finishing of the surface of the composite plates. The closed mould is loaded in to the oven and a compressive load of 75 bar is applied to the mould for 15 minutes to compact the material charge. The temperature of the oven is increased and stabilized at 250°C for a period of 35 minutes. The compressive load is then reduced and maintained at 50 bar for a period of 15 minutes. The temperature of the oven is reduced to ambient temperature and the compressive load is gradually decreased from 50 bar to approximately 5 bar within 5 minutes. The closed mould is allowed to cool down under compressive load of 5 bar for 20 minutes. The fabricated laminate is released from the mould at ambient temperature.

The parameters were tested and changed according to the results achieved

Table 4 Parameters set out by Asumani *et al.* [1]

Time (Minutes)	Temperature of Oven (°C)	Pressure (Bar) [pressure after adding springs and new mould]
0-15	250	75 [100]
15-20	250	Reduced to 50 [65]
20-35	250	50 [65]
35-40	0	Reduced to 5 [6.5]
40-60	0	5 [6.5]

The manufacturing process differed in a number of ways. Firstly the matrix used by Asumani *et al.* [1] was thin sheets of polypropylene created by melting polypropylene balls and compressing them in the oven. The polypropylene used for this research was manufactured using the extruder at the University of Johannesburg to minimize the differences between different composites i.e. with and without fillers.

The polypropylene sheets collected from the extruder were in one continuous strip therefore the first step was to cut the extruded sheet into strips 170mm in length to place them into the mould. The strips however would not remain flat since they were looped when collected therefore needed to be ironed flat and allowed to cool. This led to the strips displayed in Fig. 116. The black strip is the polypropylene and carbon nanotube mix, the white strip is pure polypropylene and the yellow strip is polypropylene/glass flakes mix.

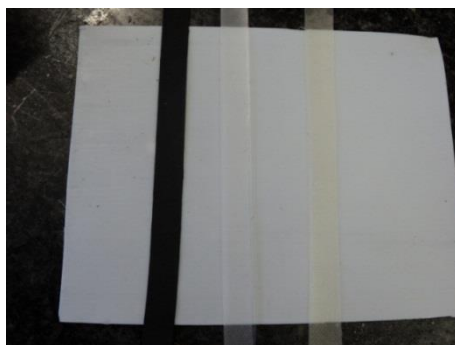


Fig. 116 Strips of matrix Left: carbon nanotube and polypropylene Middle: polypropylene Right: glass flakes and polypropylene

Prior to placing the mould in the oven the composite charge needs to be created. This is simply the fibre and matrix layup. This was done by placing a sheet of PTFE inside the female part of the mould followed by half the required number of matrix strips. The matrix strips should be overlapping in end view the same way that bricks are laid. This is displayed in Fig. 117. A sheet of fibre mat is placed on top of the matrix followed by the rest of the matrix strips placed using the same brick laying format. Finally a second PTFE sheet is placed and the mould is closed.

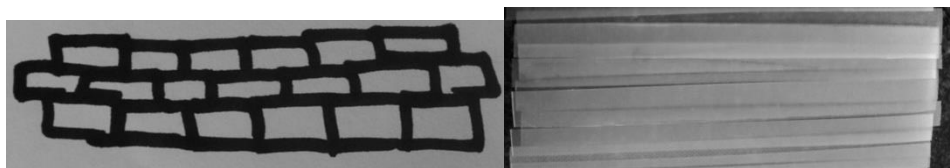


Fig. 117 Brick laying format of matrix strips

After the first test run i.e. using the parameters set up by Asumani *et al.* [1] altered slightly due to the springs being placed and the new mould it was found that the polypropylene did not melt at all. This was due to the interior of the mould did not reaching the required melting temperature of the polypropylene. It was found that the mould needed to be in the oven for a longer period due to the increase in amount of steel used for the new mould. The temperatures inside the new mould were investigated at each point in

time to determine what the required processing time needed to be to melt the polypropylene. Fig. 118 illustrates the temperature vs. time for the mould at 300°C.

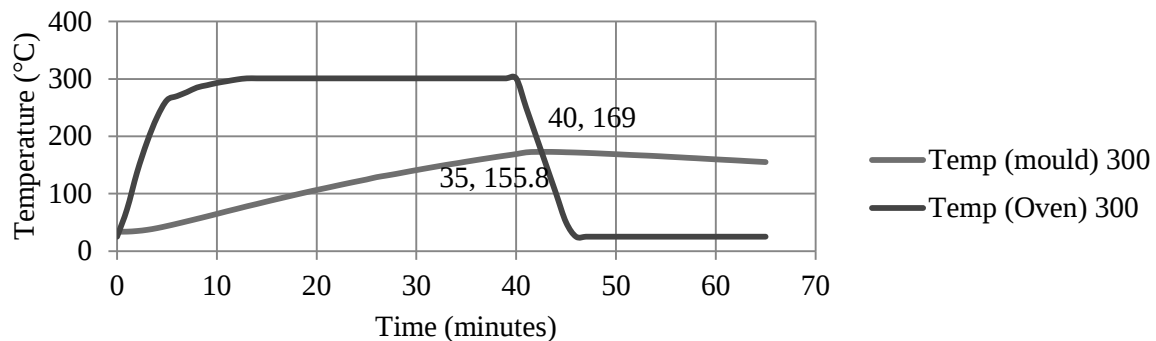


Fig. 118 Temperature vs time using an oven temperature of 300°C

It was discovered that the temperature at the top surface differed from that at the bottom surface of the mould resulting in only one surface melting sufficiently. The non-melted surface is illustrated in Fig. 119. This test was done using crisscross matrix strips which were not used for any plates further on in testing.

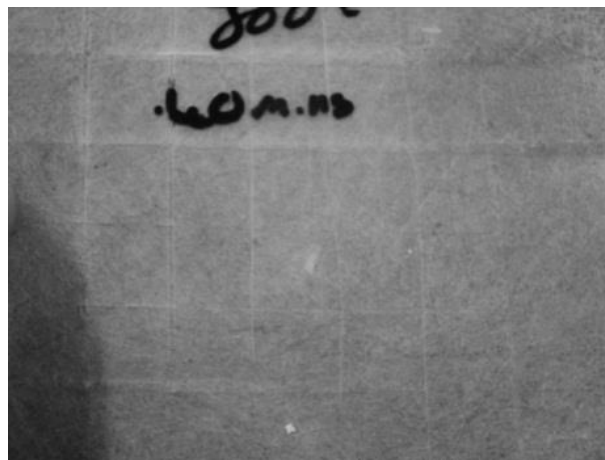


Fig. 119 Non-melted surface

Graphs of interior mould temperature vs. time for both the top and bottom surfaces is illustrated in Fig. 120 and Fig. 121 for an oven temperature of 275°C and 250°C, respectively. There is a 10°C and 9°C difference at the maximum temperature, respectively, which is a significant difference. The top refers to the male part of the mould and the bottom refers to the female part of the mould. One can see that the male part gets hot quicker since it is more exposed to the hot air than the female part of the mould.

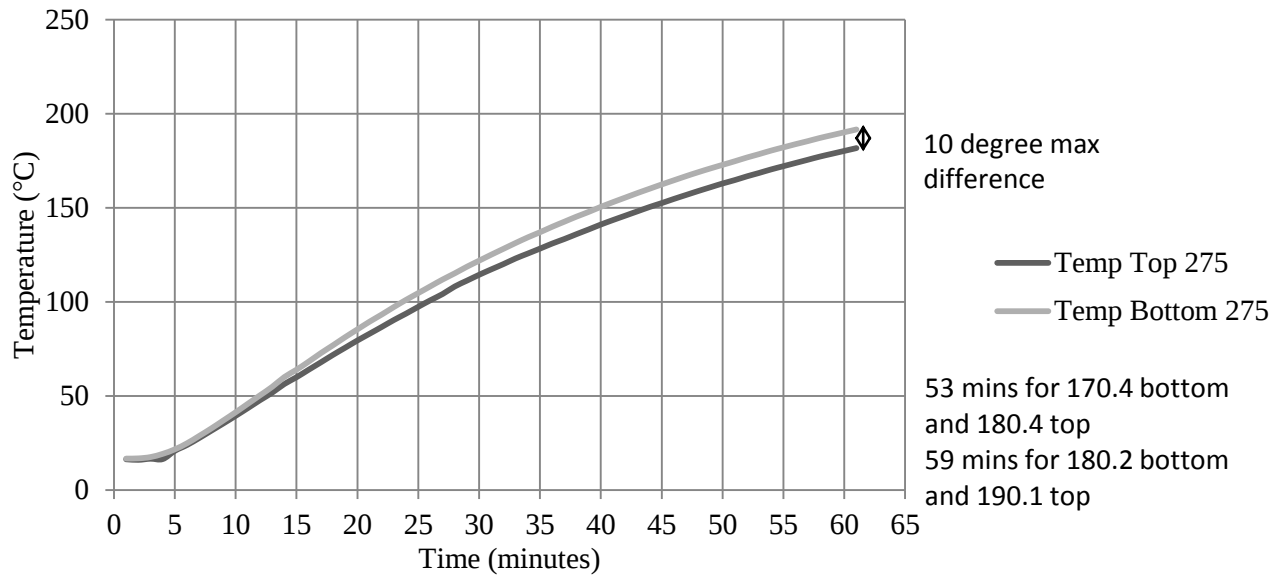


Fig. 120 Graph of temperature vs. time at 275°C

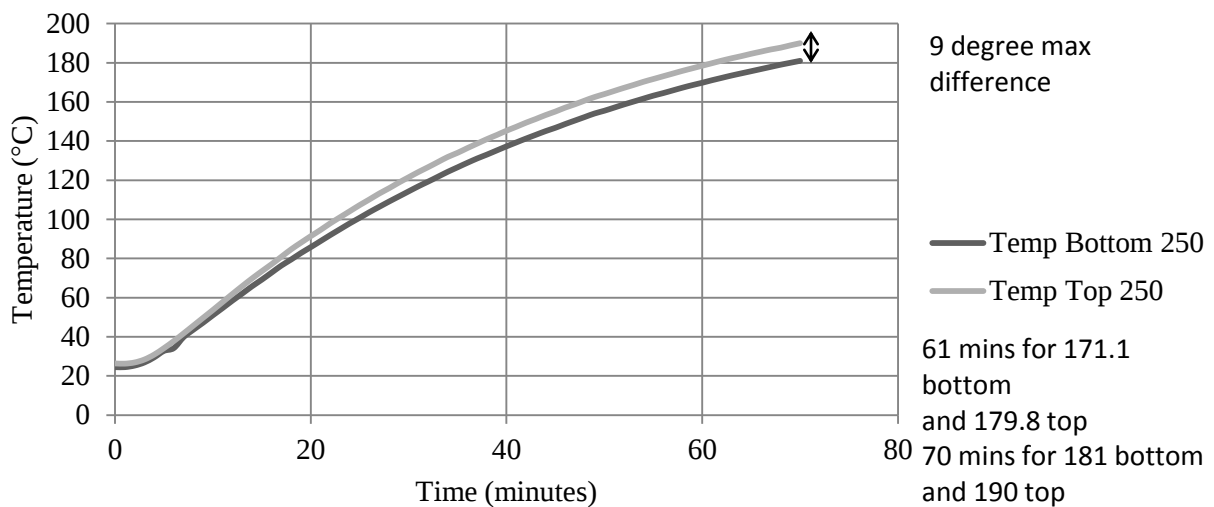


Fig. 121 Graph of temperature vs. time at 250°C

The final time chosen for manufacture is 60 minutes at a temperature of 275 °C. This allows the temperature to rise slowly yet still reaches the required temperature of 180°C for both surfaces within a reasonable period of time. This temperature is above the melting temperature of polypropylene yet not too high that it damages the fibres.

A problem arose during manufacture which was a significant issue that needed to be resolved. There were large bubbles forming inside the composite which is displayed in Fig. 122. A number of theories were

investigated to determine the cause of the bubbles. Those included lack of matrix for the required spacer size, lack of pressure forcing out the water molecules and excess water absorbed by the fibres.



Fig. 122 Bubbles formed inside the composite

The final theory determined to be the cause of the bubbles was that the fibres have a high affinity to moisture and so absorb a large amount of water. This water evaporates during manufacture and bubbles get trapped inside the matrix. This was proven by tests done to evaluate the moisture composition after drying. This was compared to the kenaf fibre used by Asumani *et al.* [1]. It is evident that the hemp has a large amount of absorbed as well as adsorbed water inside.

Table 5 Moisture Properties of Kenaf and Hemp Fibre

	kenaf	Hemp
Moisture		
Absorbed water	5%	9%
Adsorbed water	2%	4%
Cellulosic compounds/Organics	86%	85%

The difference between absorbed and adsorbed water is that the absorbed water permeates or dissolves into a liquid or solid whereas adsorbed water is water molecules that bond to the surface of the material. The fibre mats were kept with silica gel at all times to absorb the water from the fibres however the adsorbed water needs to be removed by compressing the fibres and breaking the bonds prior to evaporation. This was achieved by first applying a pressure of 200 Bar to the composite charge and then heating the mould. The pressure was then dropped over the period of the manufacturing process.

The mould was cooled using a fan for half an hour under pressure and 15 minutes at atmospheric pressure. The pressure dropped on its own during the first half an hour due to the steel shrinking after being cooled. This creates the next problem faced during the manufacture of the composites. Occasionally

after cooling the polypropylene tended to crystallize and lead to white forming on the composite which is displayed in Fig. 123. This was found to be due to the rapid cooling of the polypropylene changing the microstructure of the polypropylene. This was overcome by allowing the mould to cool for an extra 15 minutes under the fan.



Fig. 123 Crystallized polypropylene

One precaution that needs to be discussed and explained is that precaution which states that the rams of the compression moulding machine need to be aligned and that the rams need to apply force at the centre of the mould. If this is not done a moment is created and the fibres are forced to one side. This leads to fibres being pulled apart, ripped and squished to the side of the composite plate. This is displayed in Fig. 124. Another aspect that can cause the squish and fibre ripping is the mould cavity size. If the cavity is too small it leads to excess matrix flash pulling the fibres to the edge. This excessive matrix flash also leads to the fibre weight fraction increasing excessively and so spacers were used to create a mould cavity large enough for the different fibre weight fractions. A table of spacer vs fibre weight fraction is presented in Table 3.

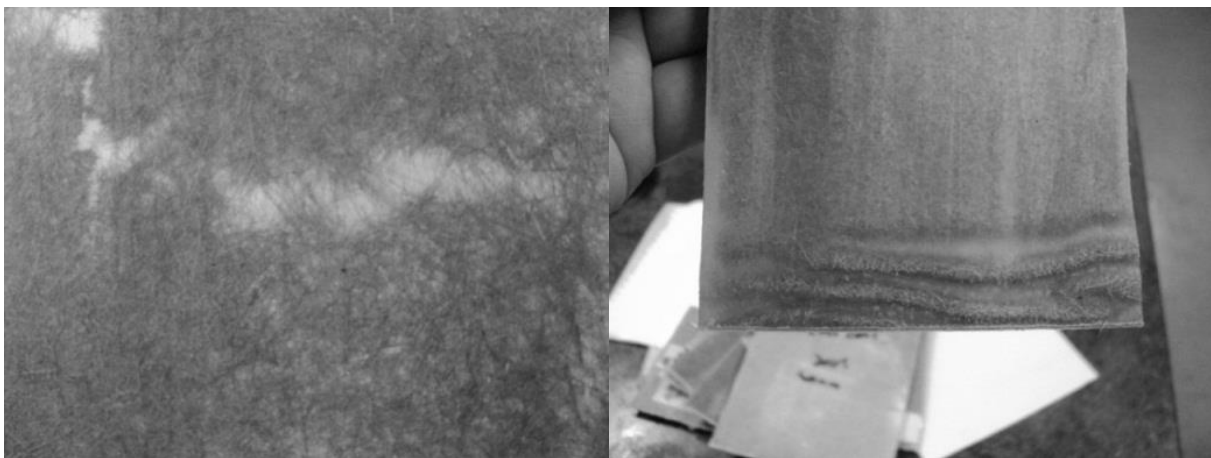


Fig. 124 Left: Fibre ripping Right: Fibre squeeze out

The final manufacturing process is discussed in the procedure section. A table of parameters, however, is presented in Table 6.

Table 6 Table of Final Process vs. Time for Composite Manufacture

Time (Minutes)	Temperature of Oven (°C)	Pressure (Bar)
0-5	0	200
5-30	275	100
30-35	275	Dropped to 60 over the 5 Minutes
35-65	275	60
65-95	0	Pressure dropped
95-125	0	Atmospheric

15 Appendix D (MATLAB code)

```
%CODE FOR ANALYSING TENSILE TEST DATA
% - Lance Paiken
%=====
clear;
prompt = {'type the directory containing tensile test files'};
default = {'C:\Users\Lance\Desktop\Research\tensile data\CSV'};
title = 'directory';
lineNo = 1;
directory = inputdlg(prompt, title, lineNo, default);
directory = char(directory);
prompt = {'Enter the Number of Specimens'};
number_of_specimens = inputdlg(prompt);
M = str2num(number_of_specimens{1});
%cd(directory);
%[filename1, pathname1] = uigetfile('*.txt', ['Select File Containing
Dimensions ']);
for z=1:M
specimen_number = num2str(z);
% CONSTANTS
guage_length = 50;
%=====
% 1. TENSILE DATA FILE GETS OPENED
% The data is sorted into FORCE and EXTENSION
cd(directory);
[filename2, pathname2] = uigetfile('*.txt', ['Select Specimen
',specimen_number]);
data = csvread(filename2);
%=====
% 2. DIMENSIONS CAN BE OBTAINED EITHER FROM A *.txt FILE OR ENTERED
% MANUALLY
% OPTION 1 - USE A *.txt FILE
% dimensions = dlmread(filename1);
% area = dimensions(z,1);
% OPTION 2 - ENTER DIMENSIONS MANUALLY
prompt = {'Enter specimen width (mm):', 'Enter specimen thickness (mm):', 'or
Enter specimen area (mm^2):'};
default={'0', '0', '0'};
title = 'Specimen Dimensions';
lineNo=1;
dimensions = inputdlg(prompt,title,lineNo,default);
width = str2num(dimensions{1});
thickness = str2num(dimensions{2});
area = str2num(dimensions{3});
if area <= 0
area = width*thickness;
end
%=====
% 3. STRESS AND STRAIN ARE CALCULATED FROM THE INPUT DATA &
% DIMENSIONS
stress = (1/area)*data(:,1);
strain = (1/guage_length)*data(:,2);
%=====
% 4. USER IS REQUESTED TO SELECT LINEAR REGION
scrsz = get(0,'ScreenSize');
```

```

figure('Position',[0.5*(scrsz(3)-scrsz(3)*0.75)    0.5*(scrsz(4)-scrsz(4)*0.75)
scrsz(3)*0.75 scrsz(4)*0.75]);
figure(1)
plot(strain, stress);
xlim([0 1.1*max(strain)]);
ylim([0 1.1*max(stress)]);
xlabel('Uncorrected Strain');
ylabel('Stress [MPa]');
grid on
% Introduce crosshairs to select origin
x = 0.15*max(xlim);
y = 1.05*max(ylim);
text(x,y, ['Select Start and End of Linear Region of ',filename2]);
[start_linear_strain, start_linear_stress]=ginput(1);
[end_linear_strain, end_linear_stress]=ginput(1);
% Now we want to disregard anything before the start and end of the
% linear region
N=length(stress);
a = 1;
while a <= N
if strain(a) <= start_linear_strain
lowerlim = a;
elseif strain(a) <= end_linear_strain
upperlim = a;
else
break
end
a = a + 1;
end
lowerlim = lowerlim + 1;
linear_strain = strain(lowerlim:upperlim);
linear_stress = stress(lowerlim:upperlim);
p = polyfit(linear_strain, linear_stress, 1);
f = polyval(p,linear_strain);
%=====
% 5. THE FITTED LINEAR CURVE IS USED TO DETERMINE THE POINT OFZERO
% STRAIN
grad = p(1);
y_int = p(2);
zero_strain = (-y_int)/grad;
for b=1:N
strain(b)=strain(b)-zero_strain;
end
%=====
% 6. THE MAXIMUM STRESS, MAXIMUM STRAIN AND ELASTIC MODULUS ARE CALCULATED
c = 1;
max_stress = 0;
while c <= N
if stress(c) >= max_stress;
max_stress = stress(c);
failure_point = c;
end
c = c + 1;
end
maximum_stress(z)=stress(failure_point);
maximum_strain(z) = strain(failure_point);
secant_modulus(z) = (maximum_stress(z)/maximum_strain(z))/1000;

```

```

linear_modulus(z) = grad/1000;
maximum_stress_squared(z)=(stress(failure_point))^2;
maximum_strain_squared(z)=(strain(failure_point))^2;
secant_modulus_squared(z)=(((stress(failure_point))^2)/((strain(failure_point))^2))/(1000^2);
linear_modulus_squared(z) = (grad^2)/(1000^2);
closereq;
end
average_maximum_stress = sum(maximum_stress)/M;
average_maximum_strain = sum(maximum_strain)/M;
average_secant_modulus = sum(secant_modulus)/M;
average_linear_modulus = sum(linear_modulus)/M;
stress_std_deviation=((sum(maximum_stress_squared)-(average_maximum_stress^2)*M)/(M-1))^0.5;
strain_std_deviation=((sum(maximum_strain_squared)-(average_maximum_strain^2)*M)/(M-1))^0.5;
secant_modulus_std_deviation=((sum(secant_modulus_squared)-(average_secant_modulus^2)*M)/(M-1))^0.5;
linear_modulus_std_deviation=((sum(linear_modulus_squared)-(average_linear_modulus^2)*M)/(M-1))^0.5;
%=====
% 7. THE RESULTING VALUES ARE WRITTEN INTO text FORMAT
results = [maximum_stress; maximum_strain; secant_modulus;
linear_modulus];
average_results = [average_maximum_stress; average_maximum_strain;
average_secant_modulus; average_linear_modulus];
std_deviation = [stress_std_deviation; strain_std_deviation;
secant_modulus_std_deviation; linear_modulus_std_deviation];
cd C:\Tensile_Results_masters_15_tf;
[filename3, pathname3] = uinputfile('*.txt', 'Save Results As:');
fid = fopen(filename3,'w');
fprintf(fid,'\t Stress \t Strain \t Secant_Modulus \tLinear_Modulus\n');
fprintf(fid,'\t MPa \t mm/mm \t GPa \t GPa\n');
for y=1:M
y_str=num2str(y);
fprintf(fid,'Specimen %c', y_str);
fprintf(fid,'\t %3.1f \t %7.5f \t %3.2f \t %3.2f\n',results(:,y));
end
fprintf(fid,'\n');
fprintf(fid,'Average');
fprintf(fid,'\t %3.1f \t %7.5f \t %3.2f \t %3.2f\n',average_results);
fprintf(fid,'Std. Dev. %c');
fprintf(fid,'\t %3.1f \t %7.5f \t %3.2f \t %3.2f\n',std_deviation);
fclose(fid);

```

16 Appendix E (Tensile results)

The following Tables represent the tensile results achieved during testing.

16.1 Untreated fibre composites

Table 7 Tensile results UF-N-15.8%

	Tensile Strength	Strain to Failure	Secant_Modulus	Linear_Modulus
	MPa	mm/mm	GPa	GPa
Specimen 1	38.1	0.025	2.68	1.97
Specimen 2	33.2	0.032	2.97	1.75
Specimen 3	32.8	0.035	2.63	1.22
Specimen 4	35.0	0.035	2.89	1.37
Specimen 5	33.3	0.030	2.97	1.77
Specimen 6	33.1	0.027	3.06	1.46
Average	34.3	0.030	2.87	1.59
Std. Dev.	2.0	0.0039	0.16	0.28

Table 8 Tensile results UF-N-20%

	Tensile Strength	Strain to Failure	Secant_Modulus	Linear_Modulus
	MPa	mm/mm	GPa	GPa
Specimen 1	32.6	0.025	2.52	1.50
Specimen 2	31.6	0.033	2.69	1.09
Specimen 3	32.5	0.033	2.78	1.15
Specimen 4	30.3	0.032	2.64	1.08
Specimen 5	30.5	0.025	2.85	1.40
Average	31.5	0.030	2.70	1.24
Std. Dev.	1.1	0.0045	0.11	0.19

Table 9 Tensile results UF-N-25.6%

	Tensile Strength	Strain to Failure	Secant_Modulus	Linear_Modulus
	MPa	mm/mm	GPa	GPa
Specimen 1	34.0	0.015	2.80	2.29
Specimen 2	37.2	0.022	3.06	2.20
Specimen 3	36.4	0.020	3.00	2.14
Specimen 4	32.5	0.018	3.06	2.07
Specimen 5	35.5	0.022	2.90	1.82
Average	35.1	0.019	2.96	2.10
Std. Dev.	1.9	0.0029	0.10	0.18

Table 10 Tensile results UF-N-30.5%

	Tensile Strength	Strain to Failure	Secant_Modulus	Linear_Modulus
	MPa	mm/mm	GPa	GPa
Specimen 1	34.7	0.014	3.20	2.82
Specimen 2	37.4	0.016	2.96	2.80
Specimen 3	39.2	0.017	2.99	2.91
Specimen 4	38.3	0.016	3.59	2.93
Specimen 5	38.0	0.018	3.21	2.77
Average	37.5	0.016	3.19	2.85
Std. Dev.	1.7	0.0014	0.23	0.07

16.2 Treated fibre composites

Table 11 Tensile results TF-N-15.6%

	Tensile Strength	Strain to Failure	Secant_Modulus	Linear_Modulus
	MPa	mm/mm	GPa	GPa
Specimen 1	35.0	0.044	2.16	0.96
Specimen 2	36.5	0.043	2.12	1.02
Specimen 3	36.0	0.043	2.13	1.17
Specimen 4	39.3	0.033	3.31	1.38
Specimen 5	34.7	0.038	1.9	1.13
Average	36.3	0.040	2.32	1.13
Std. Dev.	1.8	0.0045	0.50	0.16

Table 12 Tensile results TF-N-20.3%

	Tensile Strength	Strain to Failure	Secant_Modulus	Linear_Modulus
	MPa	mm/mm	GPa	GPa
Specimen 1	33.4	0.033	2.29	1.15
Specimen 2	36.4	0.036	2.55	1.16
Specimen 3	32.7	0.033	2.22	1.15
Specimen 4	33.3	0.035	2.26	1.09
Specimen 5	36.5	0.033	2.56	1.27
Average	34.5	0.033	2.38	1.16
Std. Dev.	1.8	0.0014	0.15	0.06

Table 13 Tensile results TF-N-24.8%

	Tensile Strength	Strain to Failure	Secant_Modulus	Linear_Modulus
	MPa	mm/mm	GPa	GPa
Specimen 1	36.6	0.031	2.20	1.38
Specimen 2	42.5	0.024	3.21	1.97
Specimen 3	43.1	0.021	3.52	2.38
Specimen 4	45.4	0.022	3.53	2.65
Specimen 5	45.7	0.024	3.60	2.26
Specimen 6	43.5	0.027	3.28	1.77
Average	42.8	0.025	3.22	2.07
Std. Dev.	3.3	0.0038	0.48	0.46

Table 14 Tensile results TF-N-30.8%

	Tensile Strength	Strain to Failure	Secant_Modulus	Linear_Modulus
	MPa	mm/mm	GPa	GPa
Specimen 1	50.5	0.020	3.87	2.83
Specimen 2	42.2	0.026	3.10	1.87
Specimen 3	49.7	0.023	3.69	2.54
Specimen 4	48.0	0.020	3.73	3.00
Specimen 5	45.4	0.020	3.64	2.67
Specimen 6	45.9	0.026	3.33	1.93
Average	47.0	0.022	3.56	2.47
Std. Dev.	3.1	0.0030	0.26	0.47

16.3 Carbon nanotube composites

Table 15 Tensile results TF-CNT-2%-16%

	Tensile Strength	Strain to Failure	Secant_Modulus	Linear_Modulus
	MPa	mm/mm	GPa	GPa
Specimen 1	40.3	0.039	2.72	1.17
Specimen 2	40.7	0.040	3.09	1.18
Specimen 3	40.8	0.039	2.88	1.20
Specimen 4	37.8	0.048	2.31	0.94
Specimen 5	38.2	0.043	2.50	1.06
Average	39.6	0.042	2.70	1.11
Std. Dev.	1.4	0.0037	0.27	0.11

Table 16 Tensile results TF-CNT-2%-20.85%

	Tensile Strength	Strain to Failure	Secant_Modulus	Linear_Modulus
	MPa	mm/mm	GPa	GPa
Specimen 1	40.8	0.035	2.90	1.33
Specimen 2	39.2	0.025	2.84	2.16
Specimen 3	39.1	0.040	2.56	1.13
Specimen 4	37.3	0.043	2.39	0.98
Specimen 5	43.8	0.035	3.08	1.36
Average	40.0	0.036	2.75	1.39
Std. Dev.	2.5	0.0066	0.25	0.46

Table 17 Tensile results TF-CNT-2%-26.1%

	Tensile Strength	Strain to Failure	Secant_Modulus	Linear_Modulus
	MPa	mm/mm	GPa	GPa
Specimen 1	43.5	0.029	3.26	1.60
Specimen 2	40.1	0.025	2.83	1.75
Specimen 3	36.7	0.027	2.82	1.48
Specimen 4	40.1	0.022	2.50	2.02
Specimen 5	40.2	0.02416	2.88	2.02
Average	40.1	0.025	2.86	1.77
Std. Dev.	2.4	0.0028	0.24	0.24

Table 18 Tensile results TF-CNT-1%-29.9%

	Tensile Strength	Strain to Failure	Secant_Modulus	Linear_Modulus
	MPa	mm/mm	GPa	GPa
Specimen 1	44.9	0.029	3.38	1.67
Specimen 2	41.3	0.025	3.38	1.74
Specimen 3	42.8	0.030	3.20	1.50
Specimen 4	45.0	0.019	3.72	3.36
Specimen 5	47.6	0.034	2.88	1.52
Average	44.3	0.028	3.31	1.96
Std. Dev.	2.4	0.057	0.27	0.79

Table 19 Tensile results TF-CNT-2%-29.55%

	Tensile Strength	Strain to Failure	Secant_Modulus	Linear_Modulus
	MPa	mm/mm	GPa	GPa
Specimen 1	41.3	0.022	3.64	1.94
Specimen 2	38.2	0.036	2.89	1.12
Specimen 3	38.3	0.021	2.93	2.46
Specimen 4	47.1	0.033	3.05	1.55
Specimen 5	49.5	0.032	3.20	1.69
Average	42.9	0.029	3.14	1.76
Std. Dev.	5.2	0.0068	0.27	0.50

Table 20 Tensile results TF-CNT-4%-30.45%

	Tensile Strength	Strain to Failure	Secant_Modulus	Linear_Modulus
	MPa	mm/mm	GPa	GPa
Specimen 1	45.2	0.024	3.37	2.00
Specimen 2	43.4	0.015	3.30	3.42
Specimen 3	50.3	0.021	3.62	3.01
Specimen 4	37.1	0.015	2.95	3.10
Specimen 5	43.4	0.018	3.76	2.65
Average	43.9	0.019	3.40	2.84
Std. Dev.	4.8	0.0040	0.28	0.54

16.4 Glass flake composites

Table 21 Tensile results TF-GF350nm-8%-15.6%

	Tensile Strength	Strain to Failure	Secant_Modulus	Linear_Modulus
	MPa	mm/mm	GPa	GPa
Specimen 1	39.3	0.044	2.55	1.01
Specimen 2	39.2	0.042	2.61	1.09
Specimen 3	37.1	0.050	2.40	0.86
Specimen 4	39.7	0.041	3.33	1.08
Specimen 5	38.0	0.052	2.50	0.84
Average	38.7	0.046	2.68	0.98
Std. Dev.	1.1	0.0048	0.33	0.12

Table 22 Tensile results TF-GF350nm-8%-20.8%

	Tensile Strength	Strain to Failure	Secant_Modulus	Linear_Modulus
	MPa	mm/mm	GPa	GPa
Specimen 1	42.9	0.033	3.15	1.51
Specimen 2	40.8	0.035	2.81	1.31
Specimen 3	42.3	0.037	2.55	1.36
Specimen 4	41.0	0.033	2.88	1.39
Specimen 5	43.9	0.033	3.00	1.44
Average	42.2	0.034	2.88	1.40
Std. Dev.	1.3	0.0018	0.20	0.08

Table 23 Tensile results TF-GF350nm-8%-25.6%

	Tensile Strength	Strain to Failure	Secant_Modulus	Linear_Modulus
	MPa	mm/mm	GPa	GPa
Specimen 1	45.6	0.021	3.52	2.69
Specimen 2	48.5	0.028	3.24	2.01
Specimen 3	44.0	0.025	2.92	2.24
Specimen 4	46.2			
Specimen 5	46.4	0.025	3.18	2.21
Average	46.2	0.025	3.22	2.29
Std. Dev.	1.6	0.0030	0.21	0.29

Table 24 Tensile results TF-GF350nm-8%-31%

	Tensile Strength	Strain to Failure	Secant_Modulus	Linear_Modulus
	MPa	mm/mm	GPa	GPa
Specimen 1	48.3	0.024	4.48	2.09
Specimen 2	51.7	0.022	4.03	2.81
Specimen 3	43.5	0.029	3.08	1.60
Specimen 4	50.3	0.028	3.31	2.12
Specimen 5	43.4	0.029	3.43	1.61
Average	47.5	0.026	3.67	2.05
Std. Dev.	3.8	0.0032	0.51	0.50

Table 25 Tensile results TF-GF350nm-2%-29.8%

	Tensile Strength	Strain to Failure	Secant_Modulus	Linear_Modulus
	MPa	mm/mm	GPa	GPa
Specimen 1	40.2	0.016	3.18	2.83
Specimen 2	48.7	0.027	2.97	1.93
Specimen 3	48.6	0.030	3.27	1.86
Specimen 4	48.6	0.021	4.10	2.50
Specimen 5	45.9	0.021	3.92	2.37
Average	46.4	0.023	3.49	2.30
Std. Dev.	3.7	0.0054	0.44	0.40

Table 26 Tensile results TF-GF350nm-5%-29.8%

	Tensile Strength	Strain to Failure	Secant_Modulus	Linear_Modulus
	MPa	mm/mm	GPa	GPa
Specimen 1	47.2	0.033	4.72	1.51
Specimen 2	48.3	0.020	3.74	3.17
Specimen 3	43.7	0.030	3.16	1.59
Specimen 4	46.6	0.033	3.05	1.61
Specimen 5	47.4	0.034	3.63	1.59
Average	46.6	0.030	3.66	1.89
Std. Dev.	1.8	0.0059	0.59	0.71

Table 27 Tensile results TF-GF350nm-12%-29.85%

	Tensile Strength	Strain to Failure	Secant_Modulus	Linear_Modulus
	MPa	mm/mm	GPa	GPa
Specimen 1	50.2	0.023	3.86	2.78
Specimen 2	51.3	0.032	4.07	1.75
Specimen 3	50.0	0.028	4.02	1.90
Specimen 4	45.3	0.031	3.14	1.64
Specimen 5	44.7	0.030	4.07	1.68
Average	48.3	0.029	3.83	1.95
Std. Dev.	3.0	0.0036	0.35	0.48

Table 28 Tensile results TF-GF100nm-8%-30.6%

	Tensile Strength	Strain to Failure	Secant_Modulus	Linear_Modulus
	MPa	mm/mm	GPa	GPa
Specimen 1	43.1	0.020	3.53	2.76
Specimen 2	47.4	0.025	3.13	2.11
Specimen 3	46.6	0.029	3.05	1.74
Specimen 4	48.6	0.033	3.46	1.55
Specimen 5	44.6	0.022	3.31	2.62
Average	46.1	0.026	3.30	2.16
Std. Dev.	2.2	0.0053	0.18	0.53

Table 29 Tensile results TF-GF750nm-8%-29.8%

	Tensile Strength	Strain to Failure	Secant_Modulus	Linear_Modulus
	MPa	mm/mm	GPa	GPa
Specimen 1	44.3	0.015	3.56	2.73
Specimen 2	48.5	0.019	3.91	2.91
Specimen 3	46.5	0.021	3.43	2.89
Specimen 4	45.2	0.025	3.24	2.60
Specimen 5	45.9	0.028	3.19	1.87
Average	46.1	0.022	3.47	2.60
Std. Dev.	1.6	0.0050	0.26	0.43

17 Appendix F (Sample calculations)

17.1 Fibre weight fraction

An exact fibre weight fraction was not easily obtained due to matrix flash. This varied from composite to composite. The fibre weight fraction was determined after manufacture for each composite as discussed above. This was done as follows.

$$W_f = \frac{\text{mass of fibre}}{\text{total composite mass}}$$

Where the mass of fibre was weighed prior to manufacture and the total composite mass was weighed after manufacture. This leads to a rough average fibre weight fraction for the entire composite plate. Note that some areas may have a higher weight fraction and some areas may have a lower fibre weight fraction than this average.

17.2 Tensile data

The following calculations shall be done using the 30% TF fibres sample 1. The variables needed for the tensile test calculations can be seen below.

$$F (\text{force at failure}) = 775.3N$$

$$\Delta L (\text{extension at failure}) = 0.958mm$$

$$L (\text{original length or gauge length}) = 50 \text{ mm}$$

$$W (\text{width}) = 13 \text{ mm}$$

$$t (\text{thickness}) = 1.18 \text{ mm}$$

The ultimate tensile strength can be calculated using the following formula. The corresponding sample calculation can be confirmed using the MATLAB code. For 30% TF sample 1 the ultimate tensile strength was calculated to be 50.5Mpa using the MATLAB code which correlates to that calculated using the formula below.

$$\sigma = \frac{F}{w \times t}$$

$$\sigma = \frac{775.3}{13 \times 1.18}$$

$$\sigma = 50.5MPa$$

The modulus of elasticity of the composites can be calculated using two different methods as discussed above, the linear modulus as well as the secant modulus. The secant modulus is simply the modulus at maximum stress hence can be calculated by dividing the max stress by the corresponding strain. The linear modulus however is calculated by determining the slope of the straight section of the stress strain curve.

In order to determine the strain at failure, the MATLAB code extends a line (from the linear region) to determine a zero-stress point and the corresponding value of strain. This point of zero-stress is then used as a new origin for the curve, thus offsetting the “bedding-in” region. The secant modulus is then calculated by dividing the stress at failure by the corresponding strain [59].

$$E_{Secant} = \frac{\sigma_{max}}{\varepsilon} = \frac{\sigma_{max}}{\frac{\Delta L}{L}}$$

$$E_{Secant} = \frac{50.5}{\left(\frac{0.958}{50}\right)} = 2.64 \text{ Gpa}$$

The linear modulus can be calculated from the MATLAB code. The user is prompt to choose the start and end of the linear region. The code fits a best fit curve to this data and then calculates the gradient of this line and this is the linear modulus.

The water absorption specimen used was specimen 2 having 22.5% fibre volume fraction. The data was observed after 312 hours of being in the water.

17.3 Water absorption data

The specimen used for the water absorption sample calculations is the 30% TF composite sample 1. The data that follows occurred after 648 hours.

Initial mass (m_0) = 2.9877g

Initial breadth (b_0) = 49.65mm

Initial length (L_0) = 50.85mm

Initial centre thickness (tc_0) = 1.15mm

Initial edge thickness (te_0) = 1.1475mm

The edge thickness is an average over all four edges.

Mass at 648(m_{648}) = 3.1923g

Breadth at 648 (b_{648}) = 49.55mm

Length at 648 (L_{648}) = 50.7mm

Centre thickness at 648 (tc_{648}) = 1.21mm

Edge thickness at 648 (te_{648}) = 1.225mm

The percentage change can be calculated from the following formula.

$$\%X = \frac{X_2 - X_1}{X_1} \times 100$$

Therefore we can calculate the change in dimensions as follow.

$$\text{mass change} = \frac{m_{648} - m_0}{m_0} \times 100 = \frac{3.1923 - 2.9877}{2.9877} \times 100 = 6.85\%$$

$$\text{breadth change} = \frac{b_{648} - b_0}{b_0} \times 100 = \frac{49.55 - 49.65}{49.65} \times 100 = -0.2\%$$

$$\text{length change} = \frac{L_{648} - L_0}{L_0} \times 100 = \frac{50.7 - 50.85}{50.85} \times 100 = -0.3\%$$

$$\begin{aligned} \text{area change} &= \frac{(L_{648} \times b_{648}) - (L_0 \times b_0)}{(L_0 \times b_0)} \times 100 = \frac{(50.7 \times 49.55) - (50.85 \times 49.65)}{(50.85 \times 49.65)} \times 100 \\ &= -0.496\% \end{aligned}$$

$$\text{centre thickness change} = \frac{tc_{648} - tc_0}{tc} \times 100 = \frac{1.21 - 1.15}{1.15} \times 100 = 5.2\%$$

$$\text{edge thickness change} = \frac{te_{648} - te_0}{te} \times 100 = \frac{1.225 - 1.1475}{1.1475} \times 100 = 6.75\%$$

18 Appendix G (Water absorption)

Table 30 Rate of water absorption and saturation value for varying fibre weight fractions.

	UF-N		TF-N		TF-CNT-2%		TF-GF-8%	
(%)	β	Saturation value	β	Saturation value	β	Saturation value	β	Saturation value
15	0.30	2.03	0.21	1.41	0.34	2.25	0.39	2.54
20	0.48	3.17	0.42	2.71	0.48	3.09	0.55	3.55
25	0.82	5.26	0.63	4.03	0.63	3.98	0.75	4.73
30	0.94	5.96	0.95	6.30	0.80	5.45	0.83	5.44

Table 31 Rate of water absorption and saturation value for varying carbon nanotube concentrations.

	TF-CNT-1%	TF-CNT-2%	TF-CNT-4%
Saturation value	5.663	5.03	6.303
β	0.008879	0.01131	0.01391

Table 32 Rate of water absorption and saturation value for varying glass flake size and concentrations

	TF-GF350nm-2%	TF-GF350nm-5%	TF-GF350nm-8%	TF-GF350nm-12%
Saturation value	5.21	5.003	4.4955	7.04
β	0.01237	0.01183	0.006481	0.01358
	TF-GF350nm-8%	TF-GF100nm-8%	TF-GF750nm-8%	
Saturation value	4.4955	5.701	5.453	
β	0.006481	0.009293	0.008277	

18.1 Mass increase vs. time

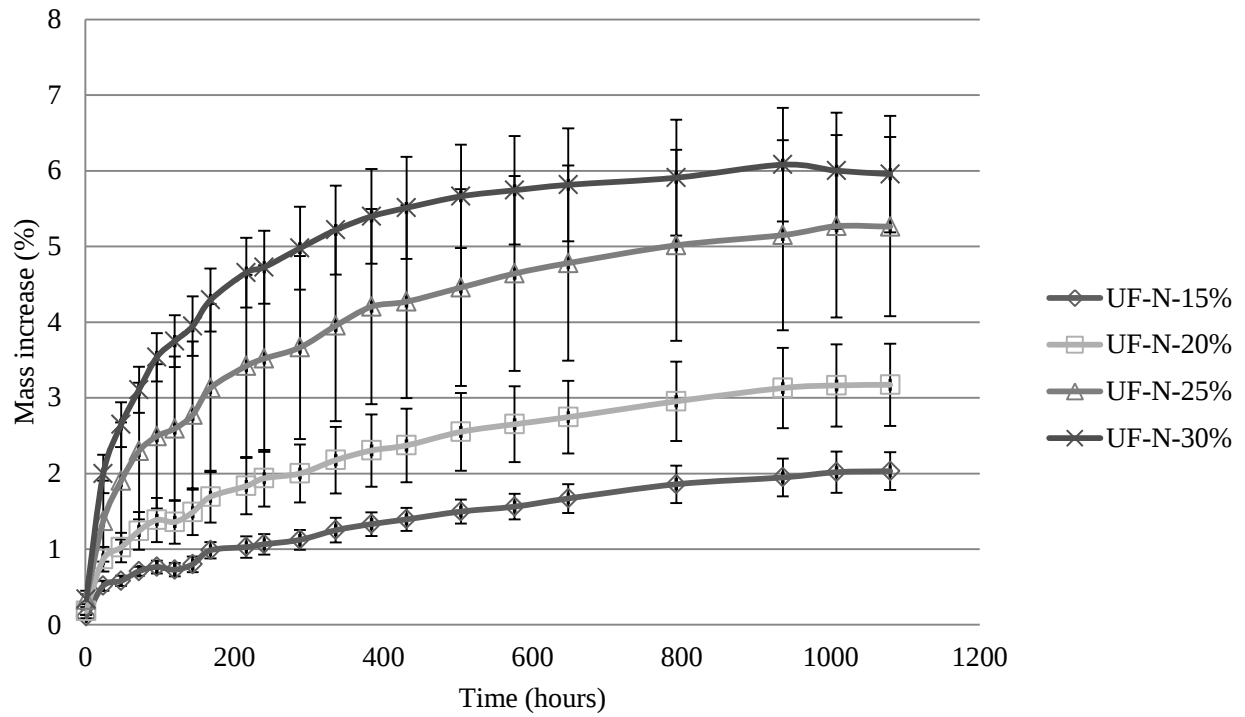


Fig. 125 Effect of fibre weight fraction on increase in mass for untreated fibres after 1080 hours.

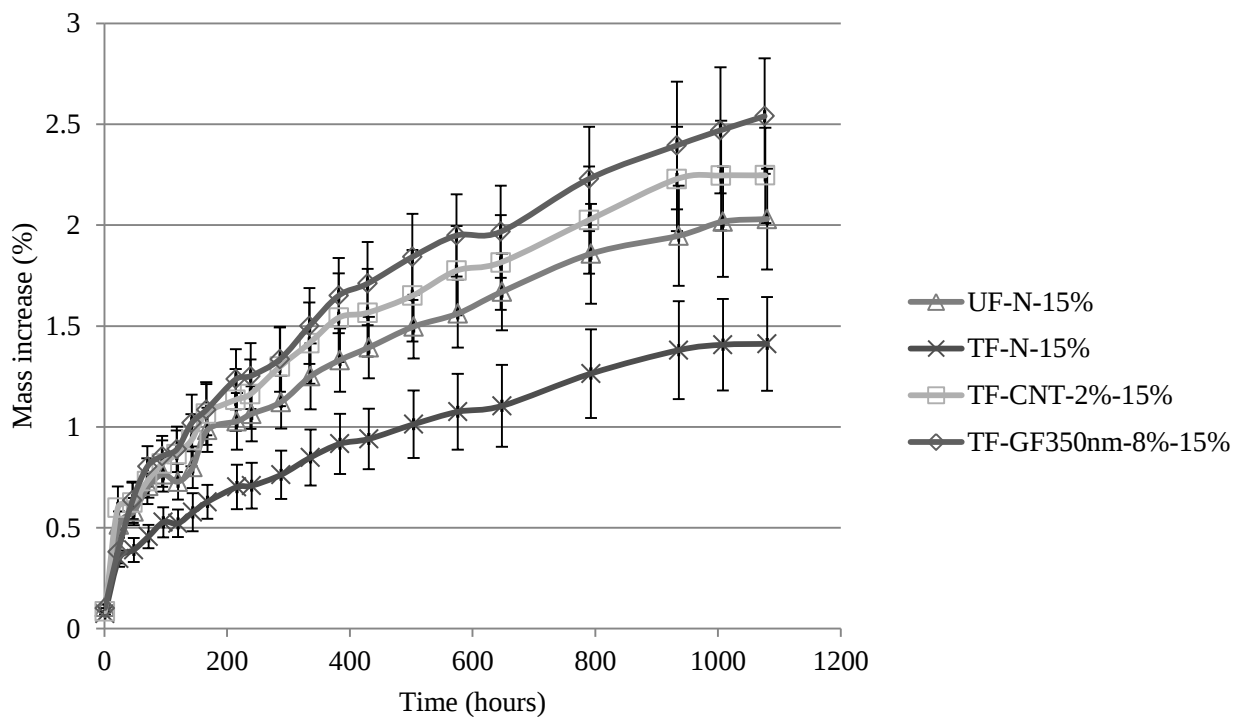


Fig. 126 Effect of filler on mass increase for fibre weight fraction of 15% after 1080 hours.

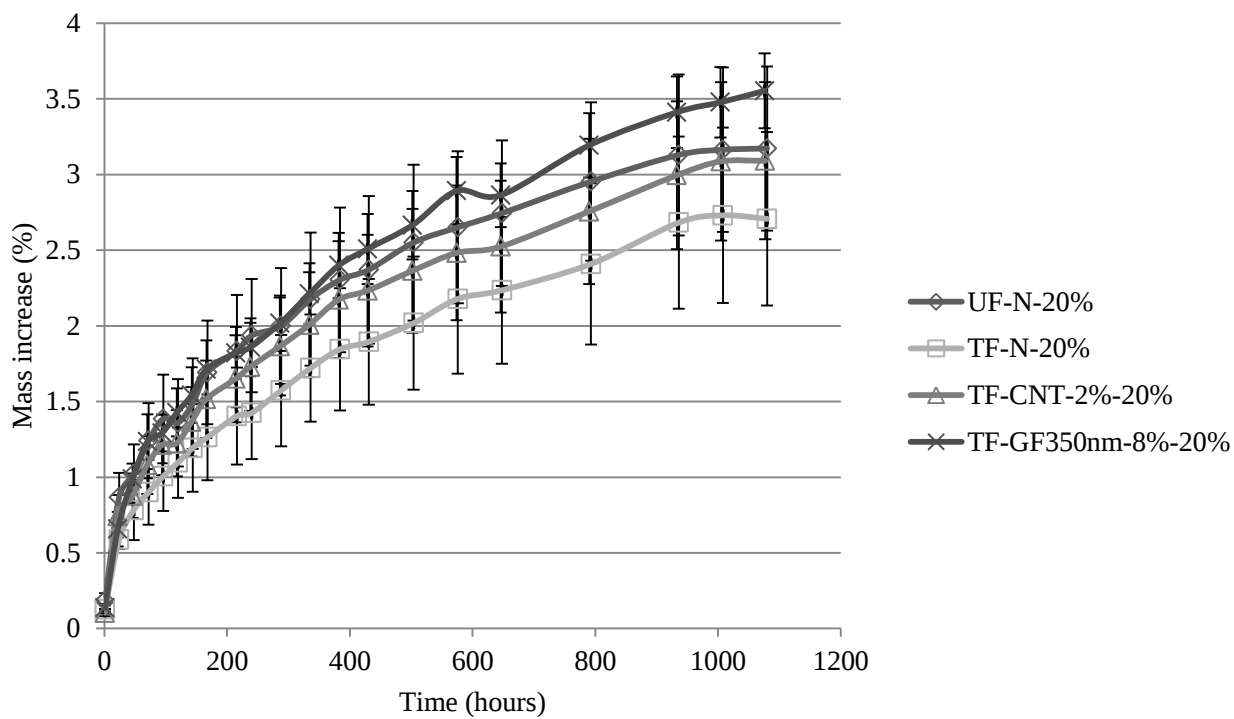


Fig. 127 Effect of filler on mass increase for fibre weight fraction of 20% after 1080 hours.

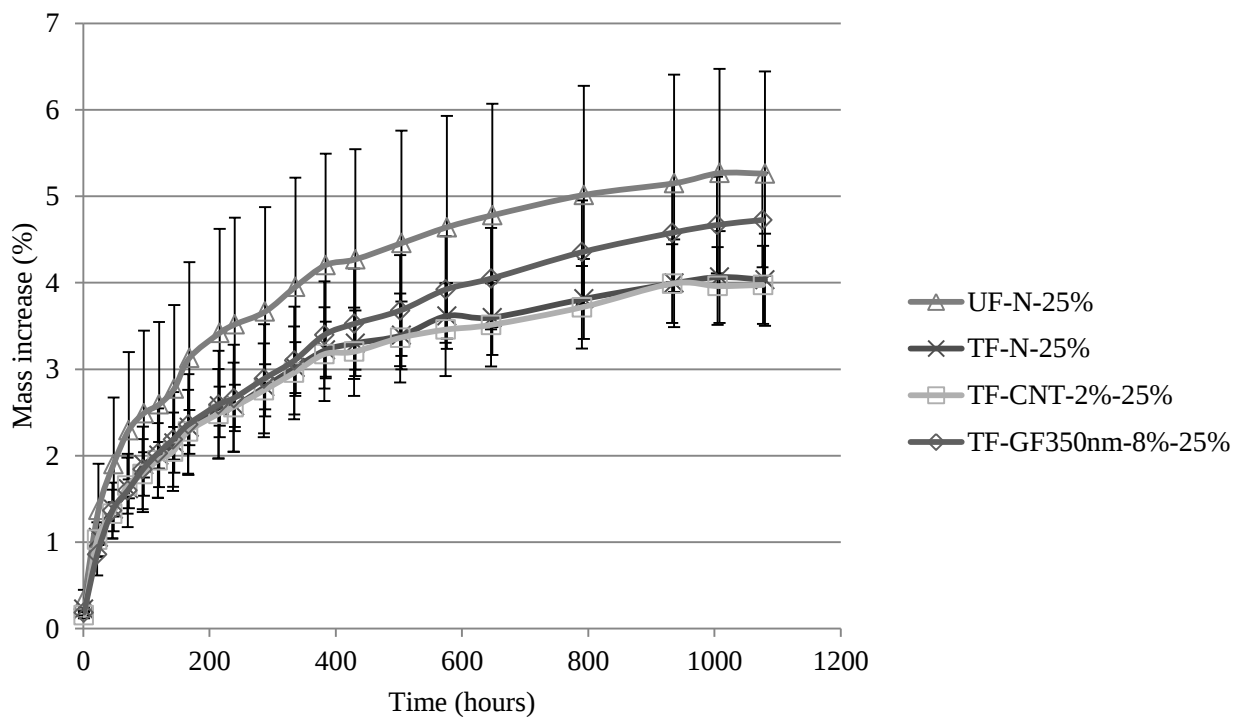


Fig. 128 Effect of filler on mass increase for fibre weight faction of 25% after 1080 hours.

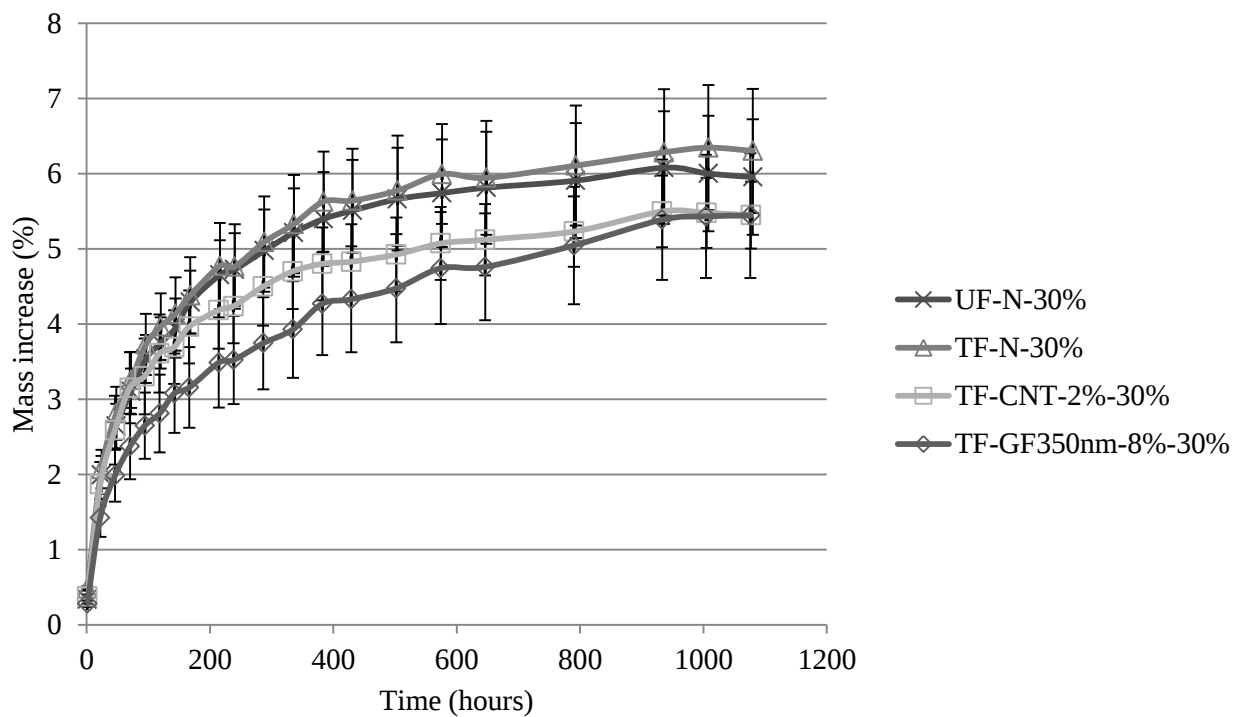


Fig. 129 Effect of filler on mass increase for fibre wight faction of 30% after 1080 hours.

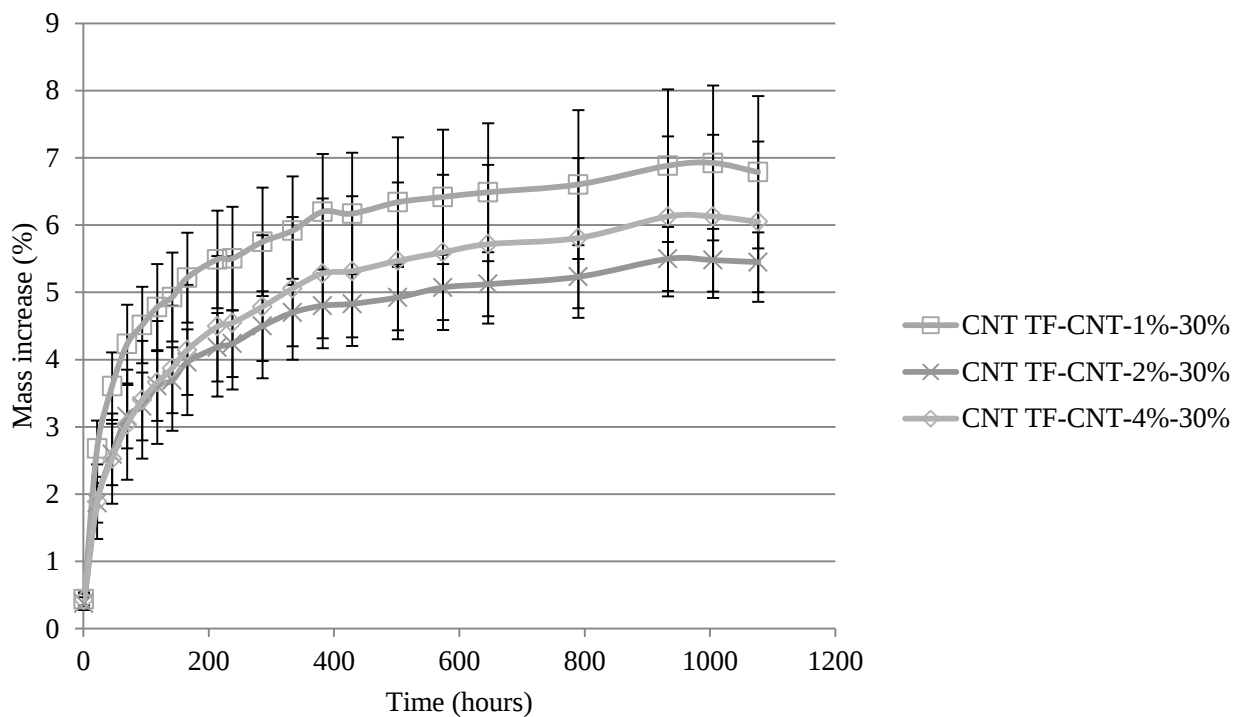


Fig. 130 Effect of carbon nanotube percentage on mass increase after 1080 hours.

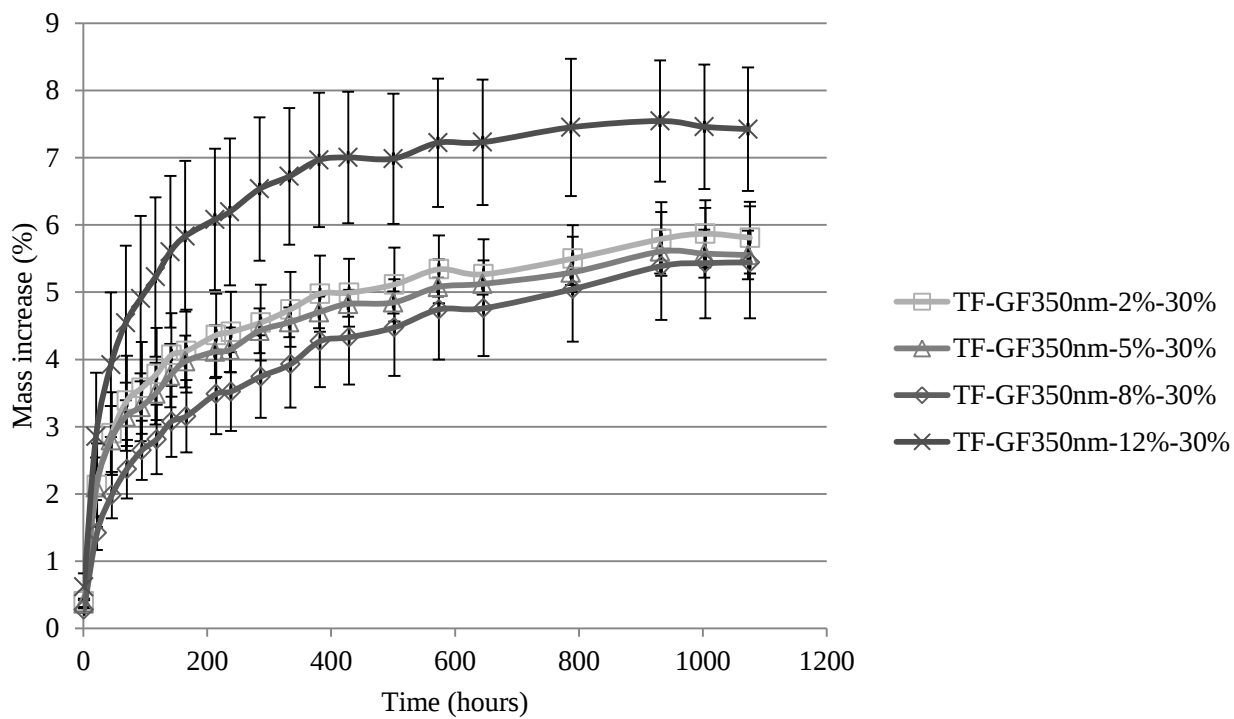


Fig. 131 Effect of glass flake percentage on mass increase after 1080 hours

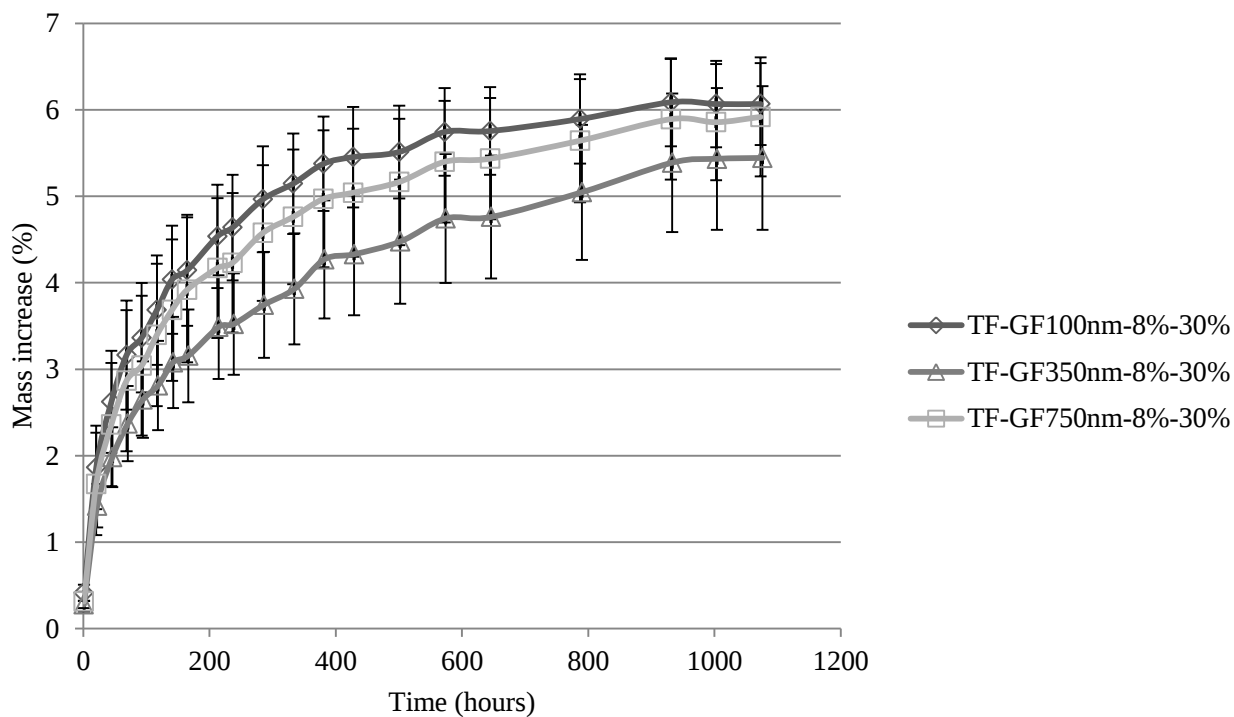


Fig. 132 Effect of glass flake size on water absorption after 1080 hours.

18.2 Area Change vs. Time

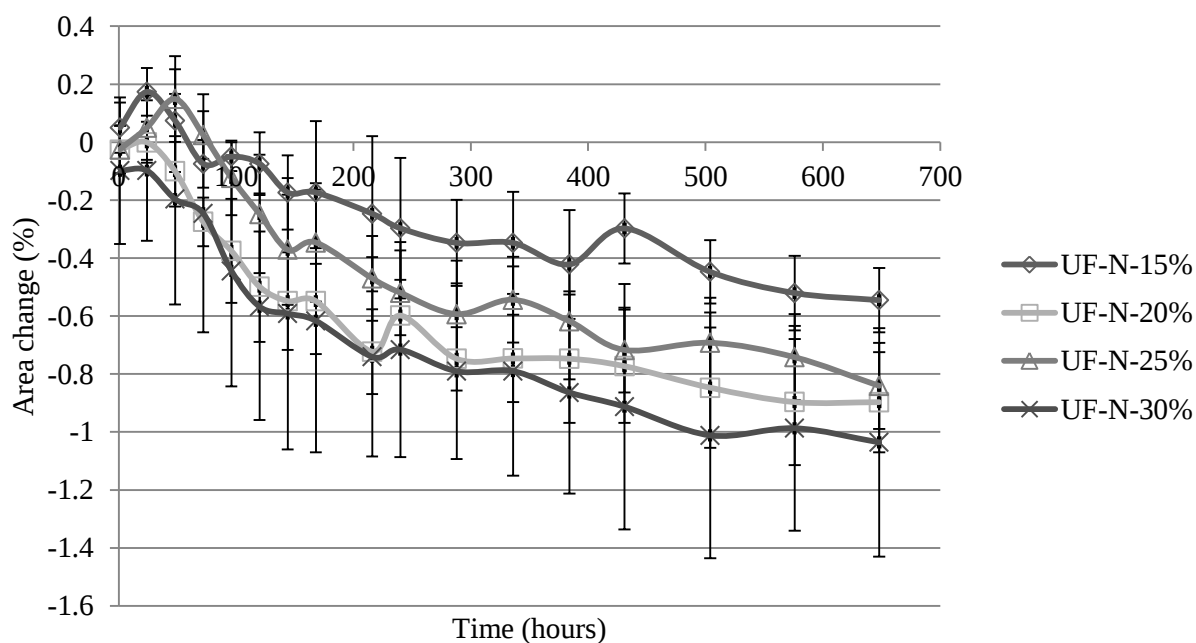


Fig. 133 96 Effect of fibre weight fraction on change in area for UF after 648 hours

18.3 Centre Thickness Change vs. Time

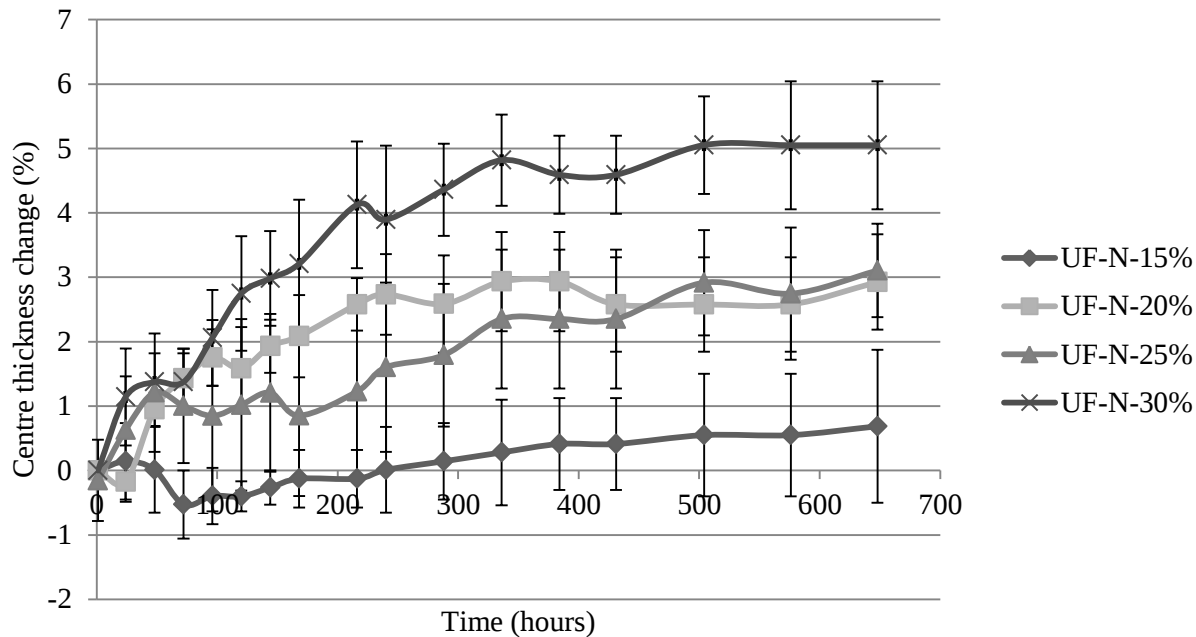


Fig. 134 Effect of Fibre weight fraction on change in centre thickness for UF after 648 hours.

18.4 Edge Thickness Change vs. Time

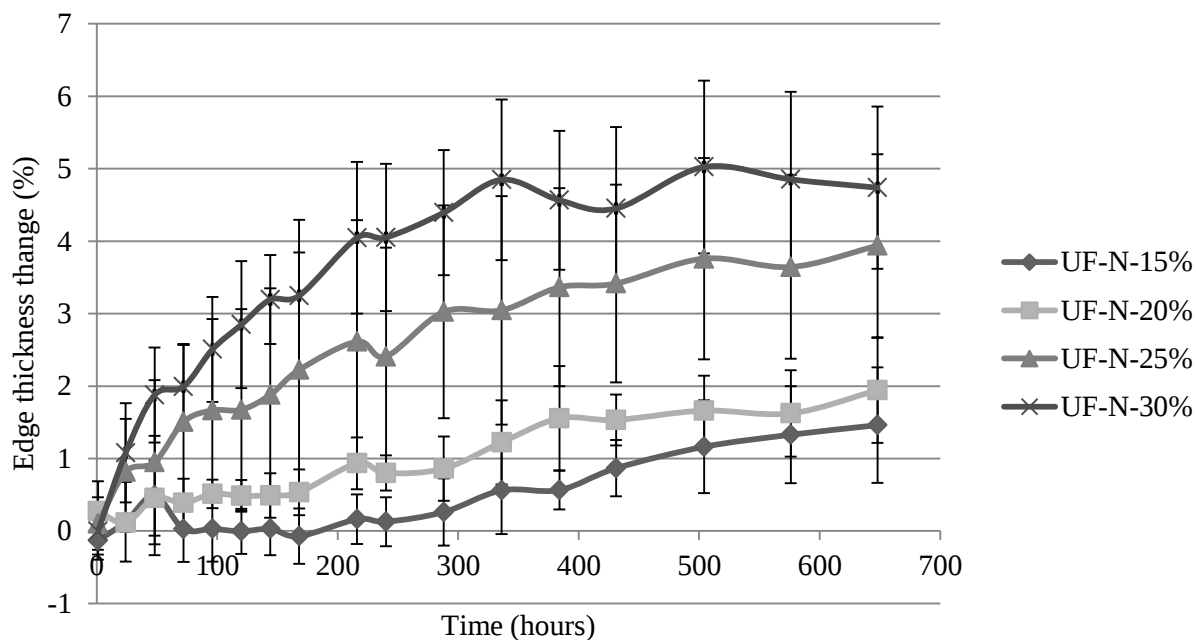


Fig. 135 Effect of Fibre weight fraction on change in edge thickness for UF after 648 hours.