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Effect of hemp fibre length on the properties of polypropylene composites

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Abstract. Hemp fibre (HF) is a natural fibre that has gained increased application in interior material for automobile industries (Sanjay, et al., 2016). However, good interfacial bonding between fibre/matrix is necessary to enhance the mechanical properties of the composite (Pickering, et al., 2007). This study focuses on the effect of fibre length, alkali and silane treatments on the mechanical and physical properties of hemp fibre reinforced polypropylene composites. Compression moulding technique was used to produce the composite, fibre lengths of 50, 100 and 150 mm were selected and combined with polypropylene powder at a fibre/PP ratio of 60/40%, a pressure of 1.67 MPa and temperature between 160–200 °C. The results obtained show that longer fibres enhanced mechanical strength. The tensile test result, for instance, shows a 21% increase in flexural strength at 150 mm compared to the fibre length of 50 mm. The modification resulted in a 46% decrease in strength, especially for 150 mm long fibres. This may have been as a result of fibre damage, inadequate modification, less quality fibre or higher initial moisture content in the modified fibres as observed from FTIR spectroscopy. Further investigation of these factors is required to be able to conclusively determine if they may have affected the mechanical performance (Alao, 2018).

Key words: Hemp fibre, interfacial bonding, polymer composite, modification, polypropylene, fibre length, moisture content, mechanical properties.

INTRODUCTION

Increasing research are ongoing to develop environmentally friendly, sustainable and reusable composite materials such as hemp, flax and sisal as replacement to glass fibres and other carbon-based materials used as reinforcements for plastic polymers. (Placet, 2009). The drawback comes from the fact that these materials are mainly obtained from hydrocarbon fuel which are considerably highly air polluting, not sustainable and hardly recyclable, especially when combusted (Masuelli, 2013).

According to (Alao, 2018), researchers such as (Wambua et al., 2000) identified that the suitability of these natural fibres as reinforcement for thermoplastic composites requires analysis and comparison of the mechanical properties with that of their glass fibre counterpart. Comparable results of the strength properties have been obtained between these fibres and that of glass. Other factors that must be determined are the temperature and humidity that ensures the sustainability of fibre integrity (Davies &

Bruce, 1998). The polymer viscousity is the main deciding factor for the selection of the temperature (Gassan & Bledzki, 2001).

Alao (2018) in this project focused on researching hemp fibre, a variety of cannabis sativa plant species. Islam et al. (2010) identified the constituent of the fibre as crystalline cellulose (55–72 wt.%), hemicellulose (8–19wt.%) and lignin (2–5 wt.%) and comparison between other natural fibres shows that industrial hemp is a strong and stiff material with the ability to reinforce polymer.

Suardana et al. (2011) defined this hemp fibre (*Cannabis Sativa L)* as a cheap, high-quality natural fibre that has been increasingly applied as a suitable interior material in the automobile industry because it possesses outstanding mechanical properties. While Sisti et al., (2017) corroborated the fibre content as shown in Table 1 below to be cellulose, hemicelluloses and lignin. They are generally extracted by retting before use as a composite reinforcement (Alao, 2018)

Table 1. The chemical composition of hemp fibres. Source: (Suardana et al., 2011)

Hemp Fibre	Cellulose	Pectin	Hemicelluloses	Lignin	Waxes & oils
wt.%	70.2–76.12	0.9–1.55	12.28–22.4	3.7–5.7	0.8–1.59

It was asserted by (Alao, 2018) that the polymer matrix is an essential material in fibre reinforced polymer (FRP) composite. These polymers are classified into thermosetting and thermoplastics, but the latter according to (Malkapuram et al., 2011) are the most widely used. These includes, Polypropylene (PP), Polyethylene (PE) and Poly (vinyl) chloride (PVC). The origin of these polymers can further be used to categorize them into synthetic or Bio based. Those obtained from petroleum-based products are called synthetic polymers while carbohydrate-rich substances like corn and sugar cane are termed Bio based, or biodegradable polymers (Mohanty et al., 2005).

Factors such as commercial availability, low density (0.92 g cm^{-3}), good heat stability, impact resistance, ease of processing and low investment input have made polypropylene (PP) the most commonly used polymer matrix. This polymer also has the ability to improve the chemical and stain resistance of the final composite material. (Denis, et al., 2016). But, (Harutun, 2003) noted that the mechanical properties of the resulting composite are most likely dependent on the reinforcing material and production parameters.

This was further corroborated by (Ho, et al., 2012) in which factors such as effective fibre/polymer matrix interface adhesion, fibre content, processing parameters and conditions were emphasized as the important factors influencing the mechanical performance of FRP composites produced from natural fibres like hemp fibre.

Alao (2018) stressed the set-back of using natural fibres with polymer matrix as pinpointed by Wambua et al. (2000). Natural fibres in FRP composites combined with these matrices causes poor fibre/matrix adhesion which is because of the heterogenous nature and high-water sorption rate of the fibre. This issue leads to poor stress transfer between the fibre and matrix, causing weak mechanical performance of the final composite material. Following recent research, surface treatment was identified as a means to improve the adhesive fibre-matrix interface bonding.

The presence of cellulose in natural fibres causes hydrophilic properties while polymer matrices are hydrophobic leading to incompatibility when combined. There is

low wetting of the fibres by the molten polymer resulting in low dispersion, inadequate reinforcement and bad mechanical properties (Harutun, 2003). Better adhesion between the fibre and polymer can be enhanced by chemically modifying the fibre surface to increase the hydrophobicity (Denis, et al., 2016) & (Malkapuram et al., 2011). Currently, modification with a solution of alkaline and silane are being researched (Alao, 2018). To extract cellulose fibres, mild treatments with alkaline have been used. This has led to improved fibre packing and orientation of the chain molecules (Gassan & Bledzki, 1999).

Although combined treatment of hemp fibres with alkaline and coupling agent such as maleic anhydride (MA) grafted PP (MAPP) produced composite with better tensile properties, modification with 25% of alkaline alone was found to increase the young modulus of the composite by almost 50% (Pickering et al., 2007). Ho, et al. (2012) thus, defined a coupling agent as any substance that adheres two materials together and serves to improve the reaction between the fibre and the matrix. These chemicals can modify the mechanical properties of the thermoplastic matrix making them more polar. They react with both the polymer and fibre surface to improve adhesion (Harutun, 2003).

In this research, the aim was to determine how fibre length affects hemp fibre reinforced polypropylene composites, the effective treatment method and hemp fibre amount required to produce a functional FRP composite. The properties of the produced hemp fibre reinforced polypropylene (HFRP) composites were determined.

MATERIALS AND METHODS

Materials

The hemp bale was supplied by Hempson OÜ. ICORENE supplied Polypropylene powder with trade name ICORENE® PP CO14RM having a density of 0.9 g cm^{-3} and melt flow rate (MFR) of 13 g per 10 min. NaOH granules (98% concentration), ethanol (96.7% concentration), acetic acid (Lachner: 99.8% concentration and molar mass of 60.05 g mol^{-1}), silane (3 – Aminopropyl-triethoxy silane: 98% concentration), tap water and distilled water were used to modify the fibre while, litmus paper was used to confirm the removal of NaOH after modification.

Modification of the hemp fibres

Hemp fibres were separated from the hurd by hand and cut into lengths of 50, 100, and 150 mm. For modification, 150 g of the fibres were first soaked at room temperature 23 °C in a solution of 1,000 mL tap water and NaOH granules 5 wt.% HF for 30 min, thoroughly washed with tap water, checked with litmus paper to ensure there was no residual alkaline, before drying in the oven at 80 °C for 24 h. The modification was completed by washing these fibres with a solution of Silane 3 wt.% HF in 9/1 ethanol and distilled water before oven drying at 80 °C for 24 h. The solution was first neutralized with 20 mL acetic acid and steered for 30 mins to activate the silane.

Production of test specimen

A mixture of HF and PP powder 60/40 was used. 135 g of modified/unmodified hemp fibres and 54. of polypropylene powder were weighed using a Mettler Toledo PL202-s and then combined in a hot press. To ease processing of the unmodified hemp

fibres, they were first immersed in water for 10 min, drained, cold-pressed at 1.65 MPa for an additional 10 min before drying at 80 °C for 24 h.

The fibres were then combined with PP before placing the mixture in the press at a temperature of 190 °C for 15 min without pressure and then a pressure of 1.65 MPa was introduced for 10 min at temperatures between 190 °C and 210 °C.

Tensile test

The tensile test was performed according to EN-ISO 527-4 (1997) using an Instron 5688 tensile testing machine and test specimens with a dimension of 150 mm x 25 mm. Test condition was at a temperature of 23 °C, relative humidity of 20% and a test rate of 5 mm min^{-1}. In carrying out the test, the load was applied to a test specimen placed between two grips until failure.

Compressive test

The test was done according to EVS-EN-ISO 14126 (2000) test standard for composites. Instron 5688 and test specimen with a length of 110 ± 1 mm and width of 10 ± 0.5 mm was used to conduct the test. Prior to testing, all specimens were covered with veneer sheets of 50 x 10 x 2 mm at 50 mm from each end using a polyvinyl acetate (PVA) glue. Test speed was 1 mm min^{-1} ± 0.5 mm min^{-1}.

Flexural test

The flexural test was based on EN-ISO 14125 (1998) test standard. The dimension of specimens was (80 x 10) mm. The test was carried out using Instron 5688 machine at standard laboratory atmosphere of 23 °C and relative humidity of 20%.

Water absorption and swelling test

The water absorption and thickness swelling test was performed according to international test standard EVS-EN 317 (2000). Test specimens had a dimension of (50 x 50 mm) while the thickness was measured using the veneer calliper before the test. Specimen edges were first dipped in wax to prevent water from directly absorbing through them. To calculate the percentage change in mass (C) and thickness (T) of the specimen the initial mass and thickness were determined using the following equations:

$$C = \frac{m_2 - m_1}{m_1} \cdot 100\%, \quad (1)$$

Where m_1 is the mass (g), after initial drying and before immersion; m_2 is mass (g), after immersion.

$$T = \frac{t_2 - t_1}{t_1} \cdot 100\%, \quad (2)$$

Where t_1 is the average thickness (mm), after initial drying and before immersion; t_2 is the average thickness (mm) after immersion.

Air permeability

International standard EN 12114 (2000) was used to perform the test. The influence of fibre modification on the insulation properties of the composite was determined using airflow resistivity. Specimens dimension was (100 x 100) mm with special air permeability tape called seal flex from tesa used to seal the edges to prevent air leakage. Fig. 1. shows the schematics of the test apparatus.

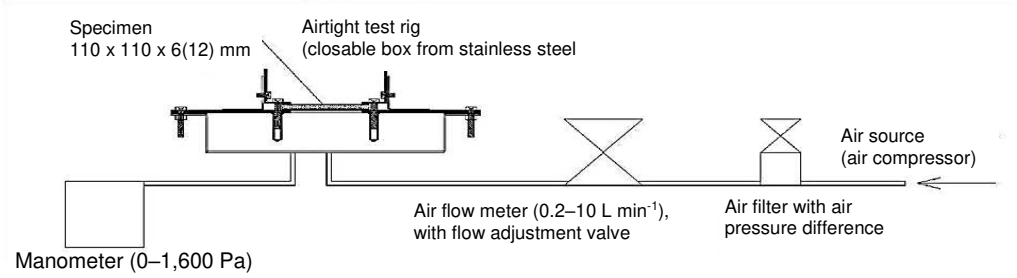

Figure 1. Equipment's complex scheme for carrying out the air permeability test. Source: (Kukk, 2016).

The pressure was introduced through a small pipe from the bottom of the apparatus. The maximum and minimum pressure difference (Δpmax & Δpmin) were 1,000 Pa and 50 Pa. Three pulses of pressure were administered to the specimens and maintained for at least 2 mins. Each pulse produced a pressure difference of 1,100 Pa. While some specimens were airtight at this pressure. Further testing was done at pressures of 1,000, 652, 425, 277, 181, 118, 77 and 50 Pa for specimens with air flow until there was no airflow recorded. Specimens that are airtight at 1,100 Pa (Stage 1) required no further testing at these pressures (Stage 2). Second phase test pressures were calculated based on the following equation.

$$\Delta p_i = 10^i \frac{\log \Delta p_{max} - \log \Delta p_{min}}{N} + \log \Delta p_{min} \quad (3)$$

Where Δp – pressure difference (Pa), N – total number of pressure steps, i – number of pressure steps.

For this test, Δpmax = 1,000 Pa, Δpmin = 50 Pa

Fourier transform infrared spectroscopy (FTIR)

The spectroscopy was performed using fibre strands and thin sheet specimen of modified and unmodified HFRP composites cut with a scalpel. Each was separately placed under the clamp and measured with peak points marked. The spectra range and resolution were 4000–500 cm^{-1} and 4 cm^{-1} respectively.

RESULTS AND DISCUSSION

The data presented in results shows modified samples as S(length)M and where numbers represent fibre lengths. i.e. S50M (composites of modified hemp fibres of 50 mm) S50 (composites of unmodified hemp fibres).

Tensile properties

The maximum tensile strength of all specimen is illustrated in Fig. 2. The result shows an increased tensile strength as fibre length increased but not with modification. S150 produced the best result (25 MPa) compared to a 47% decrease for S150M. The most significant decrease was 59% for 50 mm HF after modification. This overall poor result may suggest that it was not sufficiently modified or that these fibres were weaker compared to those of unmodified HFRP as they were produced about 60 days later. According to (Pickering et al., 2007), the time of harvest of HF can affect the strength

properties of the composites. The tensile results obtained for the unmodified composites especially S150 conform slightly with (Puech et al., 2018) were tensile strength of 24.5 ± 0.1 MPa and modulus of 2.6 GPa were reported for untreated HF, but these fibres were only 2 mm long and the composites were produced using co-rotating twin screw extruder. Hence, with appropriate modification long HF may perform better than shorter ones since 40% more strength is obtained at 150 mm.

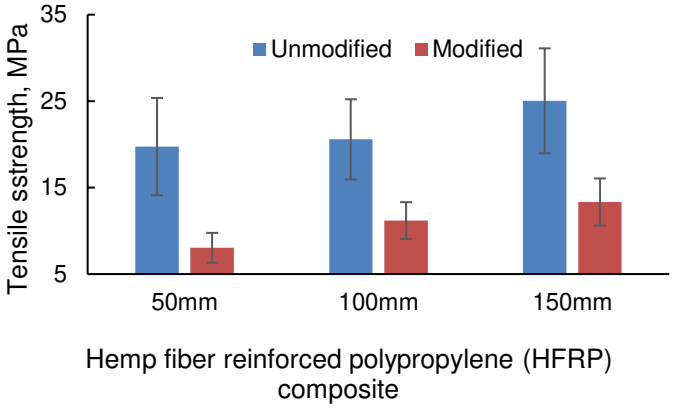

Figure 2. Tensile strength of modified and unmodified HFRP composites with varying fibre lengths (Alao, 2018).

The Young's modulus of the HFRP composite is shown in Fig. 3. The result is similar to that of tensile properties where 150 mm unmodified fibre composites performed better. S150 had the highest elastic modulus at approximately 4.5 GPa and S50M was the lowest at 1.8 GPa. Overall modulus increased by 56.8% and 20% for modified and unmodified HFRP composites as fibre length increased from 50 to 150 mm.

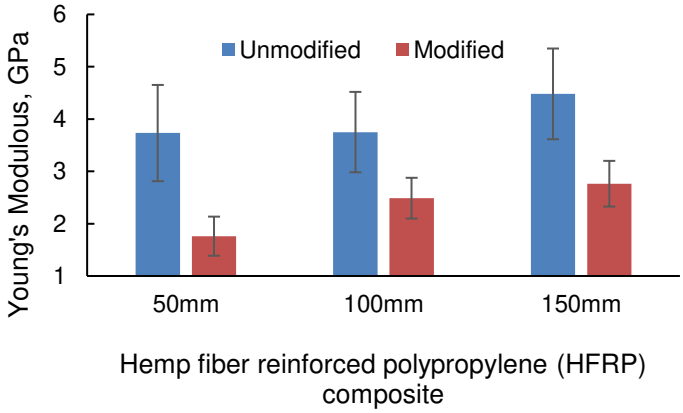

Figure Tõrge! Dokumendis pole määratud laadis teksti.**.** Young's modulus of unmodified & modified HFRP composites with varying fibre lengths (Alao, 2018).

These results are not exactly unexpected as recent research by (Sepe et al., 2018) on hemp fibre reinforced epoxy composites produced by vacuum infusion process showed a 25% decrease in tensile strength and 7% lower tensile modulus after treatment with 5% alkaline solution. The research also presented a 10 and 15% increase in the tensile modulus when silane was used to treat the fibres as compared to results from untreated and alkali-treated fibre composites. The decrease in tensile strength after alkali modification is purely attributed to excessive removal of lignin and hemicellulose content of the fibre, while treatment with silane is shown to improve bonding between the matrix and fibre. It may thus be inferred that for this research, the bond between the modified HF and PP powder was poor.

Compressive properties

The compressive strength result is shown in Fig. 4. Here, modification did not enhance compressive properties. The best result was obtained for S150, 21 MPa and S150M 18 MPa. A decrease of 35% was recorded for 50 mm fibres after modification.

Strength increased with fibre length from 50 to 100 mm to 16% and 27% from 100 to 150 mm. The modified fibres showed a slightly different trend with compressive strength decreasing by 12% from 50 to 100 mm but an increase of 16% from 50 to 150 mm conclusively shows that an increase in the fibre length, enhances the compressive strength of the composite.

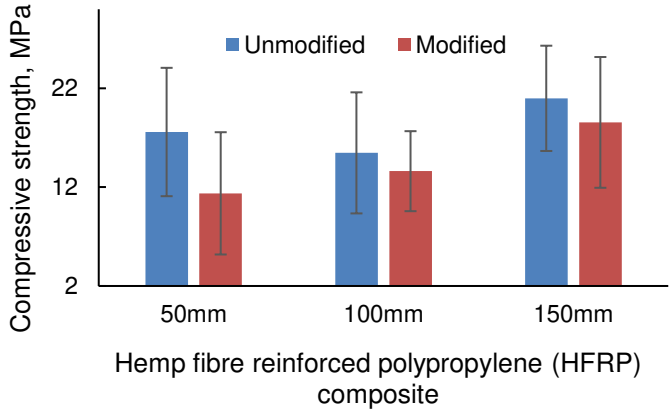

Figure 4. The maximum compressive strength of unmodified & Modified HFRP composites with varying fiber lengths (Alao, 2018).

In Fig. 5 where the compressive modulus is depicted, a similar result is also seen where higher modulus was obtained for the unmodified HFRP composites compared to those of the same length. The compressive modulus for S50, S100 and S150 were 39%, 28% and 29% higher than S50M, S100M and S150M accordingly. Showing that 50 mm fibres had the most significant decrease in strength after modification and also produced 27 and 7% lower performance than S150M (1.26 GPa) and S100M (0.98 GPa).

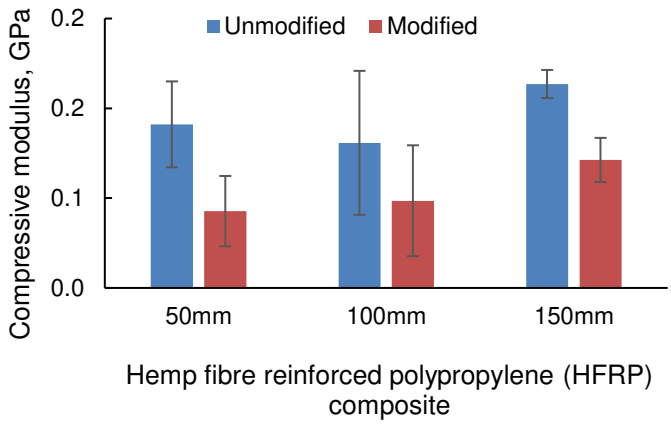

Figure5. Compressive modulus of HFRP composite from modified and unmodified hemp fibres (Alao, 2018).

Flexural properties

Flexural test results shown in Fig. 6, presents poor outcome for the modified compared to unmodified HFRP composites. It decreased on average by 56%. 150 mm HF showed the most significant decrease of 63%. An increase in fibre length favoured higher flexural performance in particular for the unmodified fibre composites where S150 produced 32.67 MPa. It was 11 and 38% more than S100 and S50. There was no significant difference in the flexural strength for 100- and 150-mm fibres after modification but both were 12% more than 50 mm long hemp fibres.

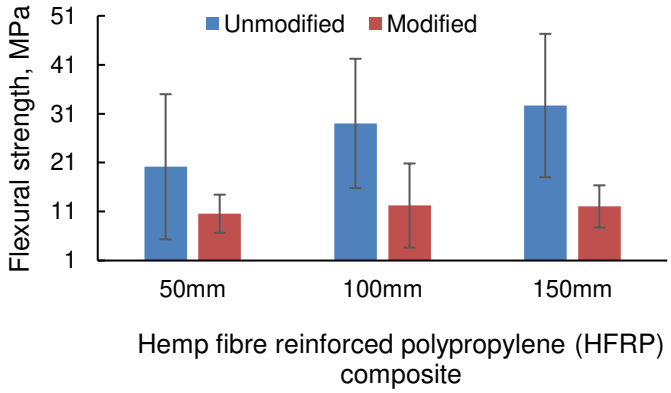

Figure 6. Flexural strength of HFRP composites with varying fibre lengths (Alao, 2018).

The flexural modulus from the modified and unmodified hemp fibre composite is shown in Fig. 7. The result is also identical to that of flexural strength. The best modulus, 3 GPa by S150 decreased 84% after fibre modification (S150M). Commonly, flexural modulus increased with fibre length except for modified fibre where it is unclear why 150 mm fibre yielded 13% less than 50 mm.

These results affirm some past research that flexural strength increases with fibre length. Thomason et al. (1996), Joseph et al. (2002) and Sathishkumar et al. (2012) have shown that using fibres with higher initial lengths enables the composite material to carry higher bending loads. The poor results for the modified HFRP composite, on the other hand, may be attributed to reduced load sharing ability caused by low interaction between polymer and matrix as a result of the ineffective modification. Combined treatment of hemp fibre with NaOH and silane from previous research have shown improvement of bonding between fibre surface and matrix leading to improved flexural properties even when compared to treatments with only NaOH (Sood & Dwivedi, 2017).

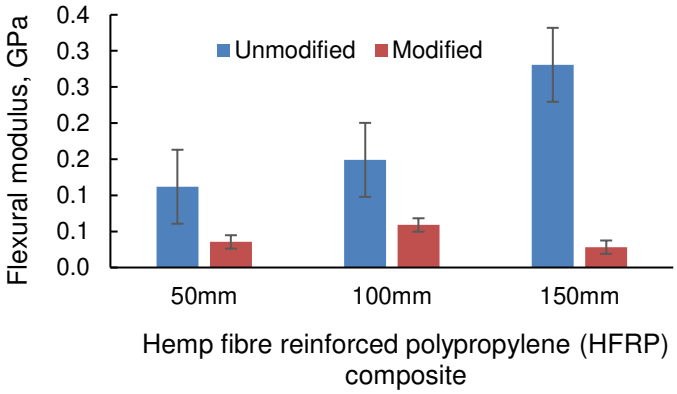

Figure 7. Flexural modulus of HFRP composites with varying fibre lengths (Alao, 2018).

Water absorption and swelling properties

The water absorption results and image of S150M are as shown in Fig. 8. Test standard allows 672 hr. (28 days) of immersion, however, an additional 672 hr. was used for this research. During the first period of soaking (24 hr.), modified fibre composites had higher water uptake than unmodified ones. S150M absorbed 65% while S150 increased by only 13%. At the end of the additional 672 hr of immersion, S150M had gained 85%, showing massive water sorption. Although, this may have been partly due to the wax coming off the edges during the first hours of immersion as shown in the image.

From the result, there appears to be no logical correlation between the lengths of the fibres and water uptake, especially for modified fibre composites. However, if wax removal in S150M is regarded and the fact that S100M absorbed 8% less moisture than S50M is highlighted, it could be concluded, that the water absorption of all the composite examined decreases with increase in fibre length. Higher water uptake recorded in this research could be inferred from (Pickering et al., 2015) which is shown to be influenced by large fibre volume fraction. Hargitai et al. (2008) in their research with a nonwoven fleece of PP fibres using fibre combinations of 30, 40, 50, and 70%, discovered that water sorption characteristics of a composite were affected by the fibre content with a composite of 70% hemp fibre showing 42% water absorption after about 19 days of immersion.

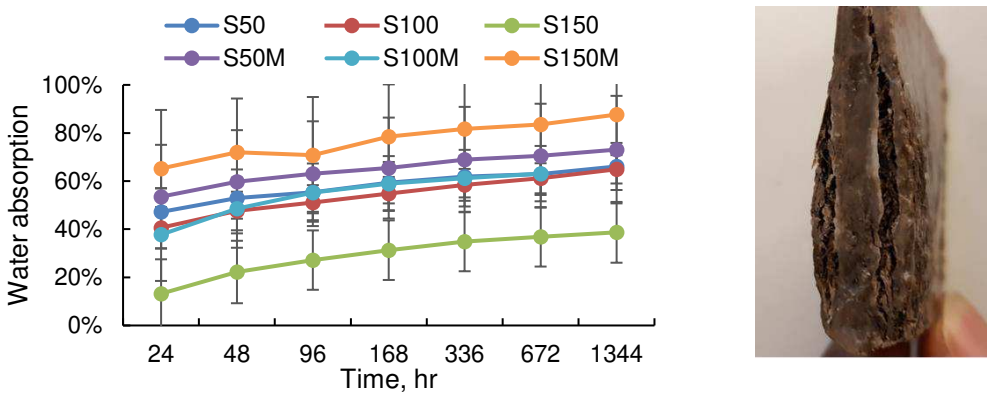

Figure 8. Water absorption of unmodified/modified HFRP composites with varying length of hemp fibres (Alao, 2018).

In Fig. 9, the swelling was constant at 14% for S150 during the first 48hr. 29%-dimensional change shown by S50 was the most significant of all the samples, but this was only 4% higher than that of the composite of the same length. There was no significant difference in thickness swelling results for S100 and S100M, though S150M increased by 2 % compared to S150.

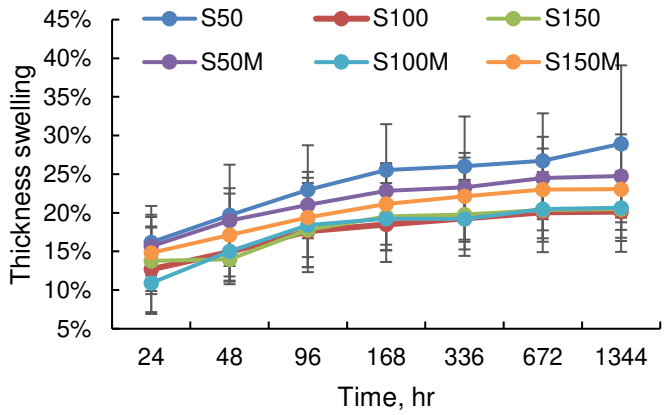

Figure 9. Thickness swelling of HFRP composite with varying length of hemp fibres (Alao, 2018).

Air permeability properties

The mean values of air flow in the first and second stage pressure test are shown in Table 2 below. Specimens from unmodified HFRP composite performed better. This confirms (Nazire et al., 2012) research where it was stated that alkaline treatment causes a drop-in basis weight and a decrease in airflow resistivity.

Table 2. Mean values of air flow in first and second pressure test stages (Alao, 2018)

Pressure stage	Test pressure (Pa)	Mean values of air flow of specimen S50 (L min^{-1})	Mean values of air flow of specimen S50M (L min^{-1})	Standard deviation S50M	Mean values of air flow of specimen S100 (L min^{-1})	Standard deviation S100	Mean values of air flow of specimen S100M (L min^{-1})	Standard deviation S100M	Mean values of air flow of specimen S150 (L min^{-1})	Standard deviation S150	Mean values of air flow of specimen S150M (L min^{-1})	Standard deviation S150M
1	1,100	0	0.3	0.09	0.12	0.20	0.25	0.27	0.09	0.15	0.36	0.12
	1,100	0	0.3	0.09	0.12	0.20	0.25	0.27	0.09	0.15	0.36	0.12
	1,100	0	0.3	0.09	0.12	0.20	0.25	0.27	0.09	0.15	0.36	0.12
2	50	0	0.00	0.00	0.00	0,00	0,00	0.00	0.00	0.00	0.00	0.00
	77	0	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
	118	0	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
	181	0	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
	277	0	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
	425	0	0.00	0.00	0.00	0.00	0.07	0.13	0.00	0.00	0.07	0.12
	652	0	0.08	0.14	0.07	0.12	0.11	0.19	0.00	0.00	0.10	0.17
	1,000	0	0.27	0.09	0.11	0.19	0.17	0.29	0.08	0.14	0.32	0.11

Only unmodified HFRP composites of 50 mm long HF were totally airtight at the 1st stage of the pressure test. There seems to be no direct relationship between the fibre lengths and the air permeability and at 425 Pa, S100M and S150M were still not airtight. The growth rate at 425 Pa for S150M/S100M was constant with 652 Pa, 0.9, but then increased by more than 100% to 1.94 at 1,000 Pa. Compared to S100M, the airflow rate of S150M was twice much. This may be attributed to the longer fibre length. Further analysis may be required to assume this because the flow at 1,000 Pa is 41% less for S100M compared to S50M.

The correlation between fibre volume fraction and porosity was emphasized by (Pickering et al., 2015) to be maxed at fibre contents of 50–60 m%, with higher causing increased porosity. Hence, the general lack of airtightness in this research may have been because of the hemp fibre content used (Alao, 2018).

FTIR properties

The FTIR spectrum of the treated and untreated hemp fibres, as well as their composites, are shown in Fig. 10. The spectrums are all similar. The region between 3,500 to 3,000 cm^{-1}, shows wide stretched peak of hydrogen bond for water which is particularly obvious for the modified fibre because of the high absorbance. This shows it contains higher moisture than the unmodified fibre.

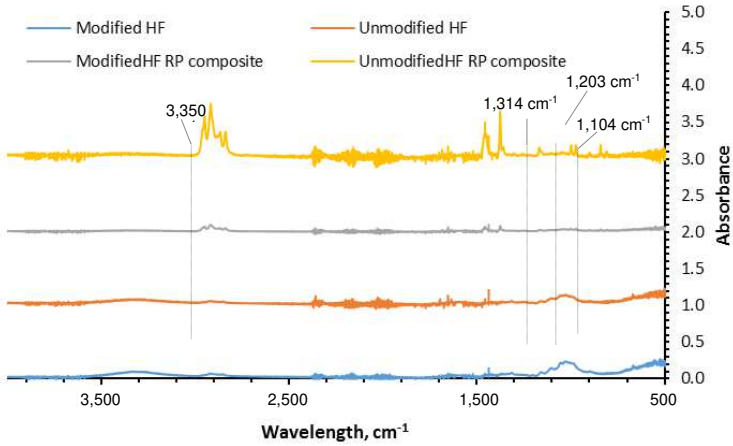

Figure10. FTIR spectra of modified and unmodified hemp fibres (Alao, 2018).

The spectra show that all specimen have similar features within the wavelengths of 1500–2,500 cm^{-1}. Although, the peak seems to intensify in the wavelengths below 1,500 cm^{-1} for the unmodified fibres compared to modified ones which indicate chemical treatment induced the C-O-C stretching causing a reduction in peak intensity. Theresa et al., 2017, published that NaOH modification is especially responsible for this. Another study by Sepe et al., 2018 on the influence of chemical treatments on mechanical properties of hemp fibre reinforced composites showed a decrease in weak and strong peaks of 1,734 cm^{-1} and 1,373 cm^{-1} respectively for hemp fibre composites modified with different concentrations of alkali (1% wt., 5% wt. and 20% wt.) which increases with the concentration of NaOH. This treatment caused the removal of a part of the

hemicellulose from the surface of the fibre. However, as seen, no new peaks were observed after silane modification which may indicate ineffective treatment.

The dislocation of natural fibres was analysed using Fourier-transform infrared spectroscopy by Dasong & Mizi, 2011. It was deduced that significant differences in the spectra are obtained between bands below 1,500 cm^{-1}, although this spectra show CH2 rocking vibration (1,314 cm^{-1}), C-O-C symmetrical stretching (1,203 cm^{-1}), and C-C, C-OH, C-H ring and side group vibrations (1,104 cm^{-1}), no evidence of silane attachment was observed (Alao, 2018).

CONCLUSIONS

It could be concluded that 60% fibre content may have led to the overall poor mechanical performance and increased porosity of the final composite material due to poor impregnation and inadequate compaction in the composite material. This is because previous research have determined that fibre mechanical properties and porosity is maxed at a fibre content of 50% and starts to decrease. Based on air permeability result, alkalinization treatment may have caused further low outcome for modified composite fibres because of the low rigidity arising from lignin and hemicellulose removal.

The FTIR analysis showed some evidence of ineffective modification with silane and possible higher moisture content in the treated fibres.

Although properties improved with fibre length, further investigation is required to ascertain the main reason for the general unsatisfactory performance.

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