An investigation on the use of sodium hydroxide treated *Pandanus utilis* fibres in compression moulded polyester composite

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Abstract: In this study, the use of *Pandanus utilis* natural fibre as a reinforcing agent in polyester was investigated with the aim of evaluating the tensile strength of the fibre, and its wettability based on two different NaOH treatments. Furthermore, the effect of the NaOH treated chopped fibre length on the tensile strength of the fibre composite was evaluated. The results have shown that a better tensile strength was obtained for the composites with the 2.5% NaOH treated fibres (11.10 ± 2.53MPa) as compared to the composite made with 0.5% NaOH treated fibres (8.32 ± 1.30 MPa), primarily due to a better wettability of the 2.5% alkaline treated fibre (87.37° internal contact angle) with the polyester resin. Within the tested range of short chopped fibre length (6 mm to 15 mm), it was shown that there was a general decrease in the tensile strength of the fibre composite.

Keywords: tensile strength; contact angle; NaOH treatment; *Pandanus utilis*; fibre length; wettability.

Reference to this paper should be made as follows: L'Entêté, L. and Ramasawmy, H. (2022) 'An investigation on the use of sodium hydroxide treated *Pandanus utilis* fibres in compression moulded polyester composite', *Int. J. Student Project Reporting*, Vol. 1, No. 1, pp.68–82.

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1 Introduction

In this era where the world is facing a major environmental problem of plastic pollution, people are focusing their ideas towards the use of natural fibres in different applications, particularly for the development of green functional products. For the past three decades there has been an increasing use of natural plant fibres in the manufacture of composites, particularly in the automotive and construction sectors. This is due to the relatively high specific strength, bio-degradability property, relative ease of extraction, lower cost of production, and low abrasive properties of natural fibres.

For Small Island Developing States (SIDS), the demand for glass-reinforced products (GRP) such as roof slates, interior wall cladding materials, soft partitioning for low walls, water tanks, septic tanks, utility boxes, and wind turbine blades are increasing year by year showing the significant use of glass fibres. However most SIDS do not have local raw material resources and have to import glass fibres, which involves high transportation cost and leading to a high carbon footprint. Glass fibre tends to cause additional machining cost of 2.65 €/cm^3 since the tools required in the manufacturing process of GRP need to be replaced frequently given the abrasive nature of glass fibre (Kolar et al., 2014)

In order to meet the sustainable development goals (SDGs), governmental authorities in SIDS have to provide their citizens with new opportunities for business development in the economic sectors such as agriculture, fishing and tourism. Thus, the creation of a micro industry for the production and extraction of fibres from agro wastes (such as banana, pineapple, coir) and other local biomass (such as Pandanus and palm species) will not only prevent the farmers from losing their jobs but also allows the development on the agricultural sector, and building better resilience against the adverse effects of climate change. This would help to alleviate poverty and minimise social inequalities.

Natural fibres have proved to gain more strength under alkaline treatment depending on the proper concentration and soaking time. Treated date palms fibre (DPF) with 5% wt NaOH yielded an optimum tensile strength of 460 MPa (Oushabi et al., 2017). On the other hand the optimum treatment for banana fibre was found to be 11 g/L NaOH for 150 min at 90°C (Vishnu Vardhini and Murugan, 2017). After being soaked for 10 min at 55°C at 5% wt NaOH, treated flax fibre has an optimum tensile strength of 611 MPa (Aly et al., 2012). These findings clearly show that the optimum conditions of an alkaline treatment depend largely on the nature of the fibre, basically on its physical structure and chemical constituents.

However, the main objective of treating natural fibre is to remove surface impurities, improve its tensile strength and improve the fibre's adherence to a matrix within a composite. But the optimum alkaline treatment conditions for producing the strongest fibre may well differ from the alkaline conditions which will produce the stronger fibre composite. This is because there are other factors, particularly the fibre to matrix adhesion which tends to play an important role in influencing the composite mechanical strength. For example, the optimum treatment for producing a strong single Kenaf fibre was found to be 4% wt NaOH for a period of 30 min at 60 °C but the author did not mention the specific alkaline treatment which leads to an increase in strength of 11.84% of the composites (Othman et al., 2018). Taha et al. (2007) did optimise the strength of date palm fibre (DPF) for its application in polymeric composites, mentioning that alkaline treatment also modifies the surface of the fibre. Furthermore, Rizal et al. (2018) noted an improvement in the tensile strength of fibre composites when increasing the soaking time of the alkaline treatment of the Typha fibre. Benyahia et al. (2013) reported that the strength of Alfa reinforced polyester was increased with an increased in the concentration of NaOH for the same soaking time.

Few authors did consider the impact of optimising the alkaline treatment in terms of the adherence of the treated fibre with the base matrix. Their main concern was to improve factors like the length or weight ratio of the fibre. According to Pickering et al. (2007) hemp fibre propylene composites proved to be stronger (47.2 MPa) with a weight fraction of 40% and a length of 1–3 cm compared to fibre having length of 10 cm. Bassyouni (2018) reported that at a weight fraction of 30% compared to 0%, 10% and 20%, sisal reinforced polypropylene has the highest tensile strength. According to Santhiarsa (2016), ijuk fibre at a length of 50 mm had a relatively higher tensile strength compared to a fibre length of 10 mm.

Research has also been conducted on the *Pandanaceae* species and their application in biocomposite. An optimum tensile strength of 17 MPa was obtained using '*Pandanus Fascicularis*' in polyester composites while the potential application of *Pandanus utilis* in epoxy composites as a substitution to glass fibre was discussed by Deesoruth et al (2014), with a *Pandanus utilis* fibre composite having a compressive strength of 97.9 MPa for a weight fraction of 10% of the optimum treated fibre (5% wt NaOH for 45 min at 75°C) (Vigneshwaran et al., 2014).

In the present study, an investigation was performed in order to evaluate the effect of the NaOH concentration on the wettability property of the *Pandanus utilis* fibre, and of the resulting effect on the tensile strength of the treated fibre polyester composite during compression moulding. Furthermore, the effect of the fibre chopped length on the tensile strength of the reinforced (compression moulded) composite was studied and compared to the fibreglass composite being produced industrially by a local company.

2 Methodology

2.1 Extraction

The extraction of the fibre was done using a Phoenix Decorticator, and all the *Pandanus utilis* leaves [Figure 1(a)] were taken from the same tree to minimise the effect of environmental factors (temperature and relative humidity), geographical location and soil quality. The fibre were then cleaned and dried in an oven for 24 hours at 60ºC [Figure 1(b)].

2.2 Alkaline treatment

The fibres were treated under two set of conditions; a first set with 0.5% wt NaOH for 14h (SFOT) as being the optimum alkaline condition to obtain the maximum fibre tensile strength of 160 MPa as per the published work (Rafidison et al., 2018). Given that the objective is to improve the wettability of the fibre to the matrix, an exposure to a higher concentration of alkaline treatment would lead to the defibrillation of the fibre and increased roughness. Thus, a second set of conditions, 2.5% wt NaOH for 2h (SFST) was conducted, which, according to Rafidison et al. (2018), should result in a decrease of 40% in the tensile strength (120 MPa). A fibre to solution ratio of 1 to 30 was used and the fibres were completely immersed in the respective solution. After the appropriate soaking time, the fibres were neutralised using distilled water and dried in an oven for 24h at 60ºC.

Figure 1 (a) The *Pandanus utilis*' leaves obtained from the tree (b) The untreated fibre extracted from the leaves after decortication (see online version for colours)

(a) (b)

2.3 Manufacturing of the composites

The treated fibre composite was fabricated based on the method used by one local manufacturer of composite. The local manufacturer uses 6 mm chopped glass fibres to produce GRP for different commodity products by compression moulding. Thus the baseline for *Pandanus utilis* fibre length was set at 6mm, and the effect of the two alkaline treatments on the 6 mm chopped fibres were evaluated by testing the composite tensile strength.

Figure 2 The samples prepared from industrial compression for tensile test (see online version for colours)

As a second part of the composite study, SFST (2.5% wt NaOH for 2h) treated chopped fibres of length 6 mm, 9 mm, 12 mm and 15 mm, were used to manufacture composites by compression moulding as shown in Figure 2, and the tensile strength of the resulting composites was investigated.

In each case, a fibre volume fraction of 15% was used, as per the local manufacturer's practice. Since a total of 250g of mixture is required to produce one plate, a digital weighing scale with a resolution of 0.1g was used to measure the respective raw materials of the composite. The fibres were added slowly to the polyester resin during the mixing process and the resulting dough was mixed using a motorised vertical blender for achieving homogeneity. The dough was then placed in a compression moulding machine (available at the local company used for producing GRP products) and a load of 1,000 kg/cm^2 at a temperature of 150ºC for 1 min to produce rectangular slate of 315 mm × 250mm × 4mm. The composite slates were then allowed to cool at room temperature.

2.4 Tensile testing

The tensile test of the *Pandanus Utilis* fibre was done according to the ASTM C-1557 with a load cell of 10kgf at a speed of 3.5mm/min (for failure of fibre to occur within 30 seconds, as per the ASTM standard) on a Testometric M500–50 AT machine. The gauge length used was 25.4 mm and the overall fibre length was 30mm. 20 single fibre specimens were tested as shown in Figure 3 and their individual cross-sectional area was measured at the broken point (during the tensile test). Several fibre mark prints of a singular fibre were done by gently pressing the fibre in soft plasticine at several places. Then an image of the marks was captured by the USB microscope as shown in Figure 4. Using ImageJ software, the appropriate area was measured to determine the cross-section of the tested fibre.

Figure 3 The fibre samples used during tensile testing (see online version for colours)

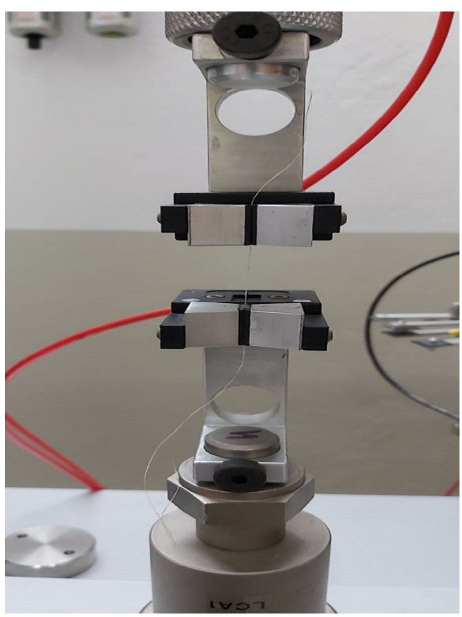
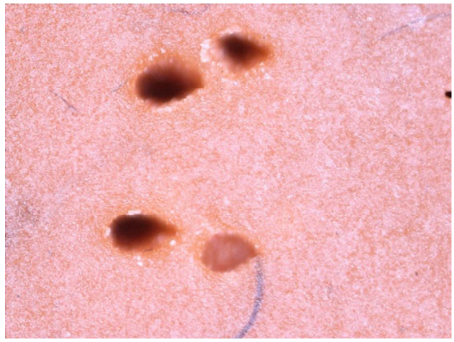
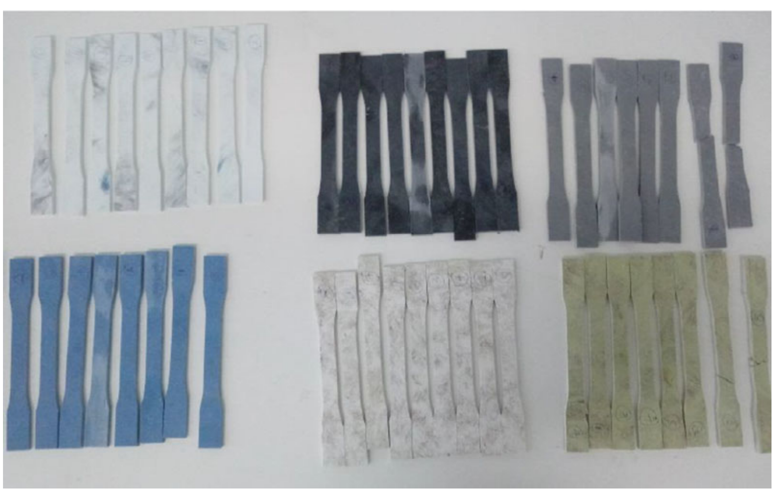


Figure 4 The image of the cross-sectional area of the broken fibre during tensile test (see online version for colours)



The samples for the tensile test of the natural fibre composites were prepared based on the shape and dimensions of specimen Type 1 of the ASTM D-638 standard as shown in Figure 5. Thus the dumbbell shape of each sample was cut from the compression moulding composite plate by using a CNC milling machine. The tensile test was conducted on the Testometric M500–50 AT machine with a load cell of 5,000 kgf and a speed of 0.35 mm/min with a sample size of 9 specimens. The cross-sectional dimensions of each of the 9 composite samples for each condition were measured using a Vernier caliper and an average value was computed.

Figure 5 The different samples made for the respective tensile test of the fibre composites (see online version for colours)



2.5 FTIR

FTIR tests were performed on the untreated and NaOH treated fibres using a Bruker single bounce ATR-FTIR spectrometer, equipped with the OPUS software. The instrument was calibrated before each measurement, and all the spectra were recorded in the range from 4000 to 500cm^{-1} and normalised before analysis on the OriginPro 8 software.

2.6 Wettability test

Each specimen from SFST and SFOT conditions were prepared on separate glass slide and the inter-fibre gaps were minimised thus increasing the accuracy of the testing method. The tests were carried out on Kruss Drop Shape Analyzer-DSA 100 using a water droplet of 2 μL at 25ºC with 5 measurements being taken on each specimen and Young Laplace Law Fitting method was used. The mean value of the five measurements for each sample was reported.

3 Results and discussion

3.1 Tensile test of the single fibre

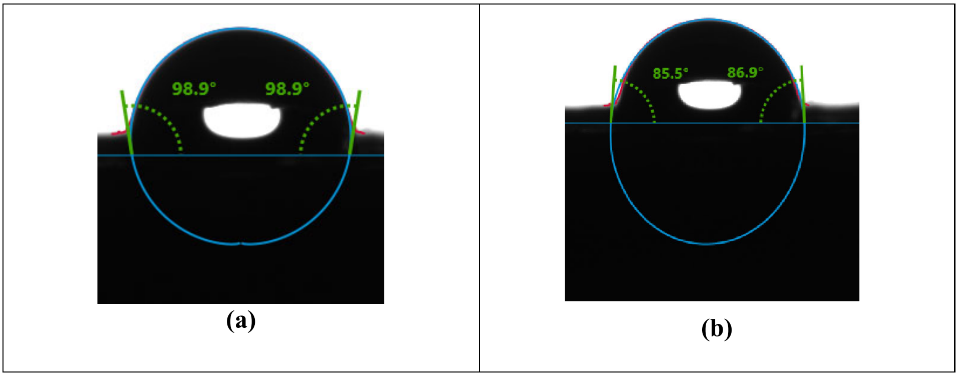
The tensile strength of the single fibre obtained for the optimum treatment (SFOT: 0.5% wt NaOH for 14h) is 168.2 ± 12.6 MPa while the tensile strength of the untreated fibre is 152.5 ± 84.1 MPa. An increase of 22% in the tensile strength of the fibre is recorded similar to the findings of Rafidison et al. (2018). This increase in the tensile strength after an optimum alkaline treatment is lower as compared to findings for other fibres. For example, Oushabi et al. (2017) have recorded an increase of 76% in the tensile strength of the DPF after the latter was treated with its optimum alkaline treatment.

The second alkaline treatment (SFST: 2.5% wt NaOH for 2 h) has yielded a fibre tensile strength of 117.9 ± 12.3 MPa, about 30% lower as compared to the highest tensile strength obtained with the SFOT NaOH treatment. This shows that a higher concentration of the alkaline treatment as compared to the optimum condition produced a treated fibre with a lower tensile strength. The same observation was made by Pickering et al. (2007), where an increase of 5% wt in its NaOH concentration led to a decrease of 45 MPa in the treated hemp fibre, although the soaking time was reduced by 30 min.

3.2 Wettability test

The mean contact angle (internal angle being measured) obtained for the SFOT sample was 95.23 ± 3.49° and for the SFST specimen was 87.37 ± 4.97°, showing that the more aggressive alkaline treatment does affect the hydrophobicity of the fibre. Figures 6(a) and 6(b) show the results of the contact angle for one measurement for the SFOT and SFST treated fibres respectively. The SFST condition allows the fibre to reach the hydrophilic state as compared to a hydrophobic state obtained with the SFOT condition. The same observation was made with Typha fibre by Rizal et al. (2018) where the internal contact angle of the water droplet decreases with an increase in the soaking time (from 1 hour to 8 hours) at a constant alkaline concentration of 5% w.t NaOH.

Figure 6 (a) and (b) The internal angle measurements of the water droplet on the SFOT and SFST fibre respectively (see online version for colours)



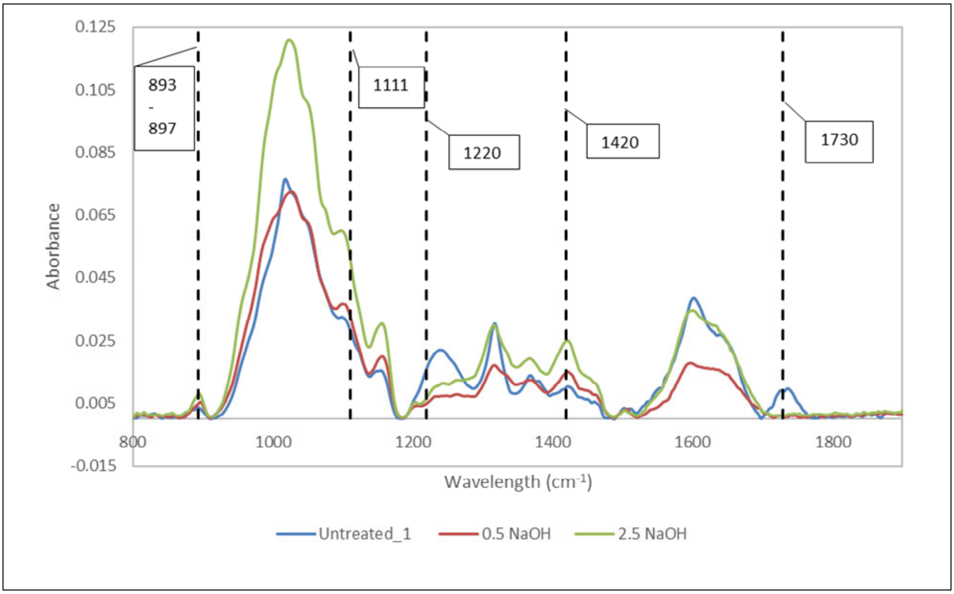
The aggressive SFST alkaline treatment does provide more OH ions to penetrate the primary wall and to cause more swelling of the fibre. The average cross-sectional area of the 2.5% NaOH (SFST) treated fibre is 0.0566 mm^2 whereas that for the 0.5% NaOH (SFOT) is 0.0265 mm^2. These experimental results show that there is a rather significant increase in the cross-sectional area for the 2.5% NaOH treated fibres as compared to the 0.5% NaOH treated, which indeed support the idea of higher amount of swelling for the 2.5% NaOH treatment. This leads to a partial defibrillation of the fibre, which explains the lower fibre tensile strength. At the same time the removal of the wax layer and other surface impurities lead to a roughening of the fibre surface. So it is believed that the swelling and partial defibrillation of the fibre together with the surface roughening provides a larger surface area for the fibre to better wet the polyester matrix. This could subsequently lead to a higher load transmission from the polyester matrix to the internal fibres, thereby improving the tensile strength of the resulting composite.

In order to further understand the difference in the effect of the chemical treatments on the properties of the fibres the detection of specific chemical functional groups by the FTIR technique is presented in the next section.

3.3 FTIR results

The significant decrease in the peak at a wavelength of 1,730 cm^{-1} for both the 0.5% and 2.5% NaOH treatment shows that hemicellulose has been removed almost completely (Figure 7). Thus a small concentration of 0.5% NaOH is sufficient to remove hemicellulose. This change has also been observed in Alfa fibre when treated with 5% NaOH (Benyahia et al., 2013). Roy et al. (2012) observed the same phenomenon as the concentration of the alkaline solution is increased when treating jute fibre. Rizal et al. (2018) reported that the absorbance peak at 1,735 cm^{-1} is reduced in Typha fibre after being treated with sodium hydroxide showing the successful removal of the carbonyl group present in hemicellulose.

Figure 7 FTIR spectrum of the untreated, 0.5% NaOH and 2.5% NaOH fibre conditions for a wavelength region of 1,850 cm^{-1} to 1,650 cm^{-1} (see online version for colours)

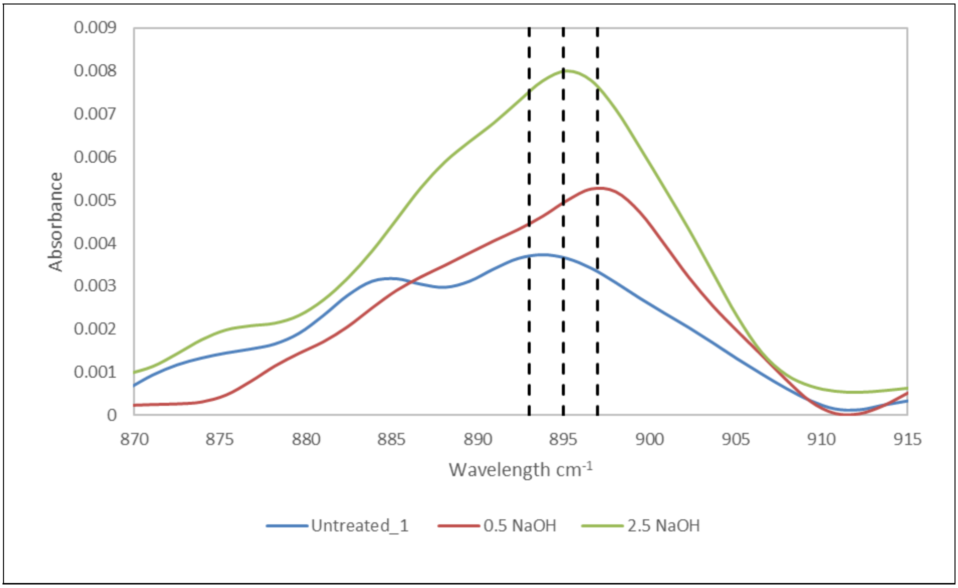


In the present study the peak around 1,220 cm^{-1} is also lower for the alkaline treated fibres, showing that lignin component has also been partially removed. Since hemicellulose and lignin (partially) have been removed from the natural fibre after the alkaline treatment, the treated fibre has a higher ratio of cellulose present in the fibre.

SFST treated fibre showed a higher peak around 1,420 cm^{-1} which would mean that there is relatively higher transformation of cellulose I into cellulose II for the 2.5% NaOH treated fibres. The higher alkaline concentration would cause a better penetration of OH ions into the primary wall of the fibres, leading to the removal of hemicellulose, and fibre swelling leading to higher defibrillation. A higher defibrillation would then lead to an increased in the surface roughness of the fibres, which would yield a better wettability property with a polymer.

The peak in the narrow range of 893–897 cm^{-1} is higher for the SFST (Figure 8), and occurs at 895 cm^{-1} whereas that for SFOT is at 897 cm^{-1}. According to Nelson and O'Connor (1964), the presence of cellulose I is revealed by a peak at 897 cm^{-1} whereas for cellulose II, a peak is obtained at 893 cm^{-1}. In the presence study, the 2.5% wt NaOH solution is not sufficiently strong to lead to a fully mercerised condition, where cellulose I would have been transformed into cellulose II. In the present case, it is believed that for the SFST fibres, there has been a partial conversion of cellulose I to cellulose II, which explains the peak at 895 cm^{-1}. Indeed Nelson and O'Connor reported that a peak at 895 cm^{-1} was obtained with a 1:1 ratio of a mixture of cellulose I and II. The cellulose II are normally smaller crystallites, with a close packed crystal structure, and this configuration would normally lead to a higher tensile strength. But given that the 2.5% NaOH (SFST) treatment has also induced a higher fibre swelling, with an increased in the cross-sectional area (as discussed in Section 3.2), then this can explain the decrease in tensile strength at the 2.5% NaOH treatment.

Figure 8 FTIR spectra in the narrow range of 893-897 cm^{-1} for the 3 conditions (see online version for colours)



Thus in the present study, it can be expected that the composite manufactured with SFST treated fibres would have a higher tensile strength as compared to composite with SFOT fibres. This will be discussed in the next section.

3.4 Tensile strength of the fibre based composite

A first comparison of the tensile strength of the natural fibre composite and the glass reinforced plastic (GRP) produced using the industrial procedure (fibre chopped at 6 mm length) in force at the local company was made. Since the density of the *Pandanus utilis* fibre is half the glass fibre, industrial volume ratio was used instead of the mass ratio for adequate mixing of the natural fibre and the polyester resin (Ramasawmy et al., 2017). The tensile strength of the SFOT fibre polyester composite was 8.32 ± 1.30 MPa, and 11.10 ± 2.53 MPa for SFST fibre polyester composite. Based on a t-test, the calculated t value is 2.953 whereas the critical t value is 2.306, which confirms that there is a statistical difference between the two mean values. These results of tensile strength confirm the results of the contact angle measurements where SFST fibres had a smaller internal contact angle as compared to the SFOT fibres. Thus the SFST fibres have a better wettability property with the polymer resin leading to better mechanical strength.

On the other hand the 6 mm chopped fibre glass polyester composite fibre has a tensile strength of 33.10 ± 2.48 MPa, which is 198% higher than that of the SFST. However, the SFST composite does not represent an optimised condition for highest interfacial shear strength between the fibre and the polyester matrix. Furthermore the effective impact of the fibre length and fibre ratio in the matrix has also not yet been optimised in this study.

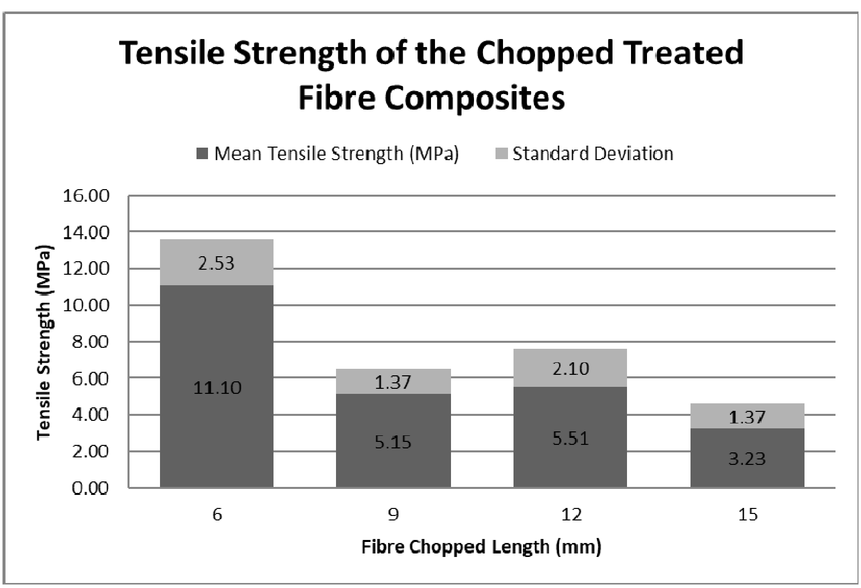
It is observed that although the SFST fibres have 40% lower tensile strength (118 MPa) as compared to the SFOT fibres (168 MPa), the SFST fibre composite has a tensile strength which is 33.7% higher than SFOT based fibre composite. This implies that the adhesion of the treated fibre with the base polyester matrix plays a critical role in transmitting the load from the matrix to the fibres. The higher adhesion of the SFST fibre to the matrix is explained by the lower internal contact angle as compared to the SFOT fibre. The same observation was made by Rizal et al. (2018) with Typha fibre, where the maximum tensile strength of the Typha fibre reinforced composite was recorded for an alkaline treatment of 5% wt NaOH for 4 hours, which had a lower contact angle than for the NaOH treatment for 1 hour soaking time. This clearly showed that the hydrophilicity state of the treated fibre do affect the final strength of the biocomposite.

3.5 Effect of the fibre length on the tensile strength of the composite

The tensile strength of the respective fibre composite produced using the different fibre length is shown in Figure 9. As the length of the fibre is increased, the tensile strength of the fibre composite decreases to a minimum value of 3.23 ± 1.37 MPa at 15 mm chopped length. Deesoruth et al. (2014) have determined that the critical length of *Pandanus utilis* fibre is L_c = 2.64mm. The present findings show that the tested length L between 2Lc (6 mm) and 6Lc (15 mm) of the treated fibre lead to a reduction in the tensile strength of the composite. The same observation was made by Yang et al. (2019).

Takagi and Ichihara (2004) observed fibre pull-out phenomenon when NaOH treated bamboo fibres of 8 mm length or less was used in a resin, whereas fibre fracture occurred when the fibre length was 15 mm or more. These authors concluded that the fibre critical length was about 12 mm whereas theoretically they calculated the critical length as being 30 mm. However, these authors did not perform any NaOH process optimisation to determine the best fibre to resin adhesion.

Figure 9 Tensile strength of the different composites produced using different SFST fibre length (see online version for colours)



Iqbal and Mousumi (2017) had also investigated the effect of jute fibre length (10-30 mm) on the tensile strength of a polystyrene untreated fibre composite. According to the authors calculation the critical length (L_c) was 0.24 mm, and the minimum fibre length to be considered as a continuous fibre ($15L_c$) was 3.6 mm. They have reported no significant improvement in the tensile strength with fibre length of 10 mm but an increase of about 18% at fibre length of 30 mm as compared to the pure unreinforced polymer. It can be observed that these authors have used fibre lengths much greater than the minimum continuous untreated fibre, and that a significant improvement in the TS of the composite was only achieved at very long length, typically $125L_c$ (Iqbal and Mousumi, 2017).

Bisaria et al. (2015) investigated the effect of untreated jute fibres (5–20 mm) on the mechanical properties of the fibre epoxy composite. These authors have reported a general decrease in the tensile strength of the resulting composite for fibre length between 5 and 20 mm as compared to that of the unreinforced polymer. These results are to some extent along the same line as those of Iqbal and Mousumi (2017) particularly for fibre length of 20 mm or less. Both Bisaria et al. (2015) and Iqbal and Mousumi (2017) have used the hand lay-up technique to fabricate the composite, but each with a different procedure, and also with a different polymer.

From the findings of Bisaria et al. (2015), Iqbal and Mousumi (2017), Takagi and Ichihara (2004), and the results of the present study, it is noted that there are several factors which would impact on the resulting tensile strength of the composite, namely, the optimised interfacial shear strength of the fibre to polymer, the fibre to resin ratio, the distribution of the fibres in the polymer (which would affect the void spaces in between the fibres network), presence of air bubbles, and the method of fabrication of the composite (which relates to the effective pressure applied to bind the fibre to the polymer).

Alkaline treatment with NaOH will tend to modify the surface roughness of the fibre thereby increasing the area of contact with the polymer. But the NaOH has to be optimised for maximum fibre to matrix interfacial shear strength rather than maximum tensile strength of the fibre. Furthermore the shape and size of the fibres also play an important role in ensuring a proper adhesion to the matrix.

4 Conclusions

This study has shown that the adhesion of the natural fibre to the polymer is a key factor in attempting to develop a high strength natural fibre based composite. In order to achieve this goal, several factors need to be considered and optimised such as the optimum fibre wettability property, optimum chopped fibre length, fibre to resin ratio and the composite fabrication method. In this study it has been shown that the 0.5% NaOH treated fibre (with the highest tensile strength) does not produce the composite with the highest mechanical strength and with 2.5%NaOH treated fibre, a stronger composite is obtained. Using SFST chopped fibre of 6mm, same as the length being used for producing GRP, a biocomposites having a mean tensile strength (11.10 MPa) of 33% of GRP (33.10 MPa) can be manufactured. Thus it is better to use a partially damaged or defibrillated fibre than the fibre with the maximum tensile strength for the manufacturing of the fibre composite. Furthermore, a fibre length L between 2Lc and 6 Lc has shown to produce a weaker fibre composite. It should be noted that in this study, the range of fibre lengths and the method for the dough preparation were based on the current practice at a leading local composite manufacturing company.

Based on the results, it is being recommended that a study be performed to identify the optimum NaOH treatment which would yield the best adhesion with the polyester matrix, and to subsequently evaluate the tensile strength of the resulting NaOH treated fibre composite. Once the optimum NaOH treatment (to yield the best fibre wettability) is obtained, a second study would need to be done in order to evaluate the optimum treated fibre length that would provide better load transmission, thereby, yielding the highest composite tensile strength.

4.1 Lessons learnt

Lesson 1 It would have been relevant to undertake a second (different) test to validate the results of the contact angle test for evaluating the wettability property of the fibre. For example, pull out test could have been used to determine the interfacial shear strength of the fibre and help to validate the result of the contact angle test.

Lesson 2 In order to use fewer chemicals for the treatment, the raw fibres were first packed as small rolls and dipped in the NaOH solution. However, it was then observed that such a decision caused the fibre to become very much entangled with each other, with significant coalescence. Thus after the treatment, it became difficult to separate the fibres, and there was also a higher risk of fibre damage. To avoid this problem, the fibres should be kept straight throughout the treatment, cleaning, drying, up until the chopping stage.

Lesson 3 For the preparation of the dough (for compression moulding), a small quantity of the fibres should be added to the resin and mixed thoroughly. This process should be repeated until all necessary fibres have been properly added and mixed with the polymer. If all the fibres are added at one go to the polymer and mixed, the fibres tend to blow away during the mixing stage, owing to the fibre low density.

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Abbreviation

SFOT: Single fibre optimum treatment

SFST: Single fibre second treatment