

**Effects of Alkali-Silane Treatment on Tensile Strength of Woven Kenaf
Reinforced Polypropylene Composites**

by

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Dissertation submitted in partial fulfilment of
the requirement for the
Bachelor of Engineering (Hons)
(Mechanical Engineering)

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CERTIFICATION OF APPROVAL

Of Research Project

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A project dissertation submitted to the
Mechanical Engineering Programme
Universiti Teknologi PETRONAS
in partial fulfilment of the requirement for the
BACHELOR OF ENGINEERING (Hons)
(MECHANICAL ENGINEERING)

Approved by,

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SEPTEMBER 2013

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

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ABSTRACT

Natural fibre reinforced plastics (NFRP) had been developed by researchers all over the world nowadays because natural fibre offers a lot of advantages such as low cost and does not threaten the environment over glass and carbon fibres. However, limitations occur in order to produce the NFRP in mass due to the limited adhesion or bonding between the matrix and fibre which caused the reduction of their mechanical properties. The main reason behind the lack of bonding is because of their difference in nature, matrix being hydrophobic and fibre is hydrophilic in nature. Therefore, in this paper surface treatment had been done on the woven kenaf fibre. The fibre had been treated with 5% concentration of NaOH solution for 24 hours at room temperature. Then, the woven kenaf is being washed with tap water and 1% concentration of acetic acid in order to neutralise the remaining NaOH on the fibre. Then the kenaf is dried in the oven at 45°C for 12 hours. Next, the kenaf is immersed in a mixture of 5% of the fibre weight of three-aminopropyltriethoxysilane solution, 50% aqueous solution of methanol, and 1% concentration of acetic acid for 4 hours at 28°C. The fibre is then washed and dried in the oven for another 12 hours at 45°C. The processing method used in order to produce the composite is compression moulding technique. The composites are being processed at 185°C under 10 tons of compression force. The tensile strength of the woven kenaf increased about 62% when compared to neat PP alone. However, slight decrement in tensile strength of the treated composite compared to untreated composite. Based on the FESEM analysis done, it could be seen that the decrement was due to the deterioration of the woven kenaf fibre maybe because being damp for too long.

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Table of Contents

CERTIFICATION OF APPROVAL	ii
CERTIFICATION OF ORIGINALITY	iii
ABSTRACT	iv
ACKNOWLEDGEMENTS	v
List of Figures.....	viii
List of Tables.....	ix
CHAPTER 1.....	1
INTRODUCTION	1
1.1 Background of Study	1
1.2 Problem Statement.....	2
1.3 Objective	2
1.4 Scope of Study.....	3
CHAPTER 2.....	4
LITERATURE REVIEW.....	4
2.1 Overview	4
2.2 Rule of Mixture	5
2.3 Volume fraction of composite.....	6
2.4 Woven fibre.....	6
2.5 Surface Treatments	7
CHAPTER 3.....	14
METHODOLOGY	14
3.1 Producing weaved kenaf.....	15
3.2 The alkali-silane treatment of kenaf	16
3.3 Fabrication of Polypropylene sheets.....	18
3.4 Fabrication of the composites.....	20
3.5 Cutting of the samples.	21
3.6 Testing procedures and standards.....	22
3.7 Project Milestones and Gantt chart.....	24
CHAPTER 4.....	26
RESULTS AND DISCUSSION.....	26
4.1 Analytical Result	26
4.2 Experimental Results	27
4.3 Morphological study.....	29

CHAPTER 5.....	34
CONCLUSIONS AND RECOMMENDATIONS	34
REFERENCES	35
APPENDICES	38

List of Figures

Figure 1.1: View of kenaf plant [3].	2
Figure 2.1: Stress-strain curve of a fibre and matrix [6].	5
Figure 2.2: Structure of a woven fibre.	7
Figure 2.3: Tensile strength with different treatments [8].	8
Figure 2.4: Tensile strength of alkali-silane treated composites [7].	9
Figure 3.1: Steps taken before composites can be produced.	14
Figure 3.2: Kenaf fibre is weaved using the weaving board.	15
Figure 3.3: Woven kenaf.	16
Figure 3.4: Woven kenaf after being immersed in 5% NaOH solution for 24 hours.	17
Figure 3.5: Woven kenaf being immersed in 1% concentration acetic acid solution.	17
Figure 3.6: Woven kenaf during silane treatment at 28°C.	18
Figure 3.7: The PP palettes are evenly distributed.	19
Figure 3.8: The mould is being cooled under compaction.	19
Figure 3.9: The layering of the composite.	20
Figure 3.10: The marked composites are cut into rectangular shapes.	21
Figure 3.11: The samples are milled in order to obtain required width and length.	21
Figure 3.12: The standard dimensions for tensile test Type I [19].	22
Figure 3.13: The sample is loaded into the tensile grip.	23
Figure 3.14: The test is ended when the samples break.	24
Figure 3.15: Key milestones of the project.	24
Figure 4.1: Analytical and experimental tensile strength.	28
Figure 4.2: FESEM of untreated composite with magnification of 80X.	30
Figure 4.3: FESEM of untreated composite at magnification of 40X.	30
Figure 4.4: FESEM of untreated composite at magnification of 100X.	31
Figure 4.5: FESEM of treated composite at 60 X magnification.	31
Figure 4.6: FESEM of treated composite at 50 X magnification.	32
Figure 4.7: FESEM of treated composite at 60 X magnification.	32

List of Tables

Table 3.1: The preparation of samples.....	15
Table 3.2: Dimension for test samples of ASTM D638 standards [19].....	22
Table 3.3: Activity planned throughout the project.....	25
Table 4.1: Properties of filled/reinforced polypropylene composites [2].	26
Table 4.2: Experimental results of tensile strength of the samples.	28

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Over the past few years, many scientists and engineers have showed their interests in using natural fibre in polymer composites applications. This is mainly due to vast availability, low cost, lightweight and biodegradability of the natural fibre [1].

Natural fibres are subdivided based on their origins such as plants, animals, or minerals. Plant fibres are the most popular fibres which are used as the reinforcement in composites [2]. One of the reasons for this favouritism is because natural fibres have higher specific strength than glass fibre. Natural fibres also have similar specific modulus compared to fibre glass. With these properties plus advantages like low cost and vast availability, natural fibre offers desirable strength. Kenaf is one of the examples of plant fibres with a rapid growth rate over a wide range of climate condition, and high yield. Figure 1.1 shows the pictorial view of the kenaf plants. Kenaf absorbs a significant amount of carbon dioxide in the atmosphere as well as nitrogen and phosphorus which exist in the soil, making it more favourable than other natural fibre. Polypropylene (PP) is a type of polymer which is extensively used in the industry due its notable strength, low cost and recyclability. In this study, woven kenaf was used, where the structure of kenaf was expected to improve the ability of the fibre to withstand load, thus improving the mechanical properties.



Figure 1.1: View of kenaf plant [3].

1.2 Problem Statement

Natural fibre has been used as reinforcement in polymer composite. However, the hydrophilic nature of the natural fibre and hydrophobic nature of thermoplastic polymer restrict the interfacial bonding between the polymer and natural fibre [3].

Surface treatment on the natural fibre is one of the solutions to enhance the interfacial bonding between the fibre and matrix. Therefore, alkali-silane treatment on the fibre was proposed to improve the interfacial bonding between thermoplastic matrix and fibre.

1.3 Objective

The objective of this project is to study the influence of alkali-silane treatment on the tensile strength of the PP/woven kenaf composites.

1.4 Scope of Study

Locally produced Propelinas' polypropylene type G452 was used as the matrix in this project. Local kenaf yarn was used, where the weaving process was done manually, using a weaving board. Surface treatment that had been chosen was alkali and silane treatment. Sodium hydroxide was used in the alkali treatment in order to remove impurities from the fibre. Three-aminopropyltriethoxysilane solution was used in the silane treatment that acts as a coupling agent that enables the cross linking between the matrix and fibre.

The composites were produced using the compression moulding technique. The tensile strength of the composites was determined using tensile test with ASTM D638 standard. The morphological study was done by using the Field Emission Scanning Electron Microscope (FESEM) technique.

CHAPTER 2

LITERATURE REVIEW

2.1 Overview

The natural fibre composites had been used extensively in the automotive and aerospace industry due to its low cost, availability, low density, relatively high strength and environmental friendly [3] - [5]. Since glass and carbon fibre are really expensive and consumes a lot of energy during production, industry tends to choose natural fibre reinforced thermoplastic composites. This type of composite often applied in the automotive industry for the production of car doors panels, car roofs and covers.

Limitations of the production of natural fibre reinforced composites were mainly due to the difference between hydrophobic nature of the matrix and hydrophilic nature of the fibre which caused the lack of adhesion in the interfacial bonding between the fibre and matrix. Properties of the natural fibre which depends on the harvest quality, plant's age, extraction technique and the conditions of the plant site are also one of the limiting factors of natural fibre reinforced composites.

Interest have grown throughout these past few years in order to improve the interfacial bonding between natural fibre and matrix in hope that the mechanical properties of the composite could be enhanced further for greater application.

2.2 Rule of Mixture

Rule of mixture is a rule commonly used in order to evaluate and estimate the mechanical properties of a polymer matrix composite, such as the tensile strength and the elastic modulus of the composite. Woven type of fibre could be considered as long continuous fibre. In order to calculate the tensile strength of the composite the tensile strength of fibre and the tensile strength of matrix at fibre failure, σ_f^* and σ_m' respectively should be identified. The difference between the tensile strength of matrix, σ_m^* and tensile strength of matrix at fibre failure can be seen in Figure 2.1. The tensile strength of matrix is the ultimate tensile strength of the matrix whereas the tensile strength at fibre failure is the tensile strength of matrix taken during the fibre breaks [6].

After all the values are known the tensile strength of the composite could be calculated based on Equation 2.1. V_f are the symbol used for the volume fraction of the fibre whereas V_m is the volume fraction of matrix.

$$\sigma_{cl}^* = \sigma_m' (1 - V_f) + \sigma_f^* V_f \dots\dots\dots \text{Equation 2.1}$$

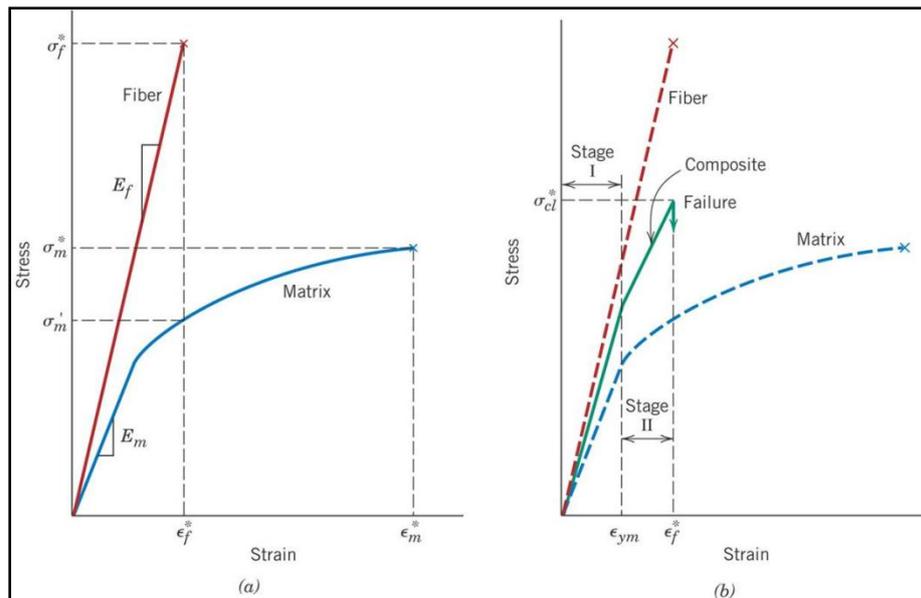


Figure 2.1: Stress-strain curve of a fibre and matrix [6].

2.3 Volume fraction of composite

The volume fraction of the fibre and matrix in a composite could influence the value of the tensile strength of the composite obtained. Volume fraction can be determined by using the formula based in Equation 2.2 [6]. The volume fraction represents the percentage of the fibre or the matrix tensile strength that will contribute to the composites' tensile strength.

$$V_m = \frac{V_m}{V_m + V_f}$$
$$V_f = \frac{V_f}{V_m + V_f}$$

..... Equation 2.2

Where $V_m + V_f = 1$

However, the volume fraction is not the only factor that can influence the composite tensile strength. If the fibre's volume fraction is higher than the matrix, the matrix may not be able to fully cover the surface of the fibre. Therefore, the volume fraction of the fibre is always limited to 0.5 only. Based on the study done by Asumani et al [7], the best volume fraction of the kenaf fibre that results in the highest tensile strength of PP/kenaf composite is at 30%. The above equation may also be used by calculating the weight fraction which is equivalent to the volume fraction.

2.4 Woven fibre

In order to improve the structure of the fibre, fibre had been weaved in order to improve the ability of the fibre to withstand the load acting on it [3]. The structure of the woven fibre is bi-directional, which means that the strength of the composite will be the same regardless in any direction. This is also known as isotropic properties of composite. Besides, woven fabric reinforcement provides greater flexibility during processing and it can maintain adequate mechanical properties, and woven fibre is suitable to be used in compression moulding processing technique. Figure 2.2 shows the structure of a woven fibre.

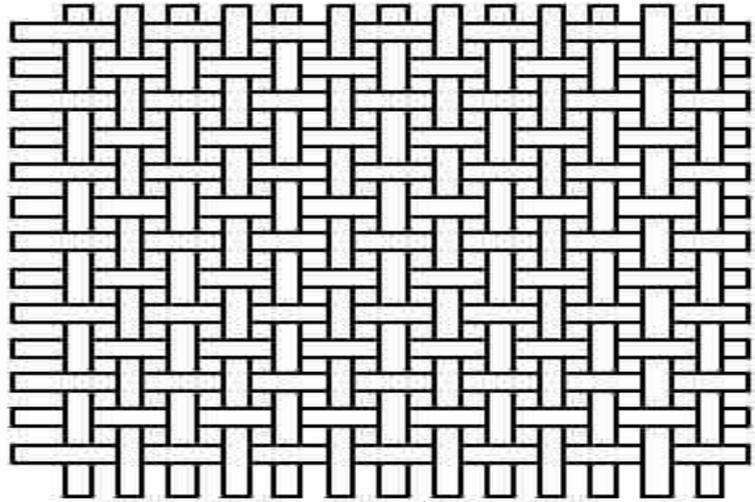


Figure 2.2: Structure of a woven fibre.

2.5 Surface Treatments

There are several studies that had been done in order to prove that by surface treatment and modification, the bonding between natural fibre and matrix could be enhanced thus improving the strength of the composites [7] – [18]. Studies done by Goripathi et al. [8], compared several types of the surface modification techniques in order to improve the adhesion between the polylactide acid (PLA) and also the jute fibre. Types of surface modification techniques used were alkali, permanganate, peroxide and silane treatments. From Figure 2.3, it could be seen that the tensile and flexural strength of the composites are their highest when being treated with silane. While the strength is the lowest when being treated with alkali and permanganate.

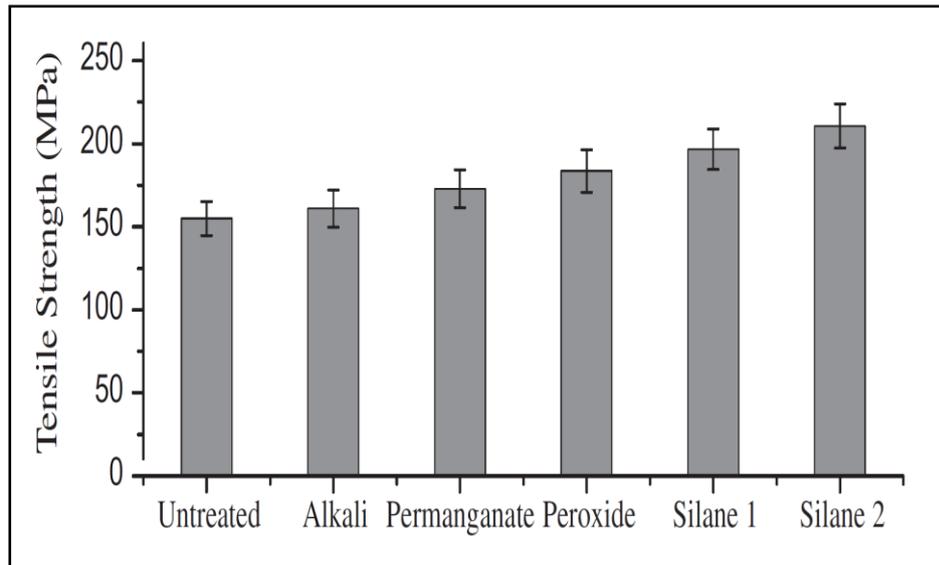


Figure 2.3: Tensile strength with different treatments [8].

Asumani et al. [7] had studied the effect of alkali-silane treatment on the tensile properties of short fibre non-woven kenaf fibre reinforced polypropylene composites. The kenaf was prepared in three ways, untreated, treated with sodium hydroxide solution only and treated with sodium hydroxide solution followed by three-aminopropyltriethoxysilane solution. The tensile and flexural strength of the composites with alkali treated fibres showed improvement over that of composites reinforced with untreated fibres.

However, it was noticed in Figure 2.4 that when the alkali concentration goes above 6%, the tensile strength drops significantly, regardless of the fibre content of the composites. This verdict is constant with the attribution of the drop in strength was due to chemical degradation of the kenaf fibre. It could be concluded that the use of the combined alkali-silane treatment gives more advantage than either the alkali or silane treatment applied alone. In particular, the specific tensile strengths of alkali-silane treated kenaf composites with 30% fibre mass fraction are, yields the most optimum value of tensile strength and flexural strength.

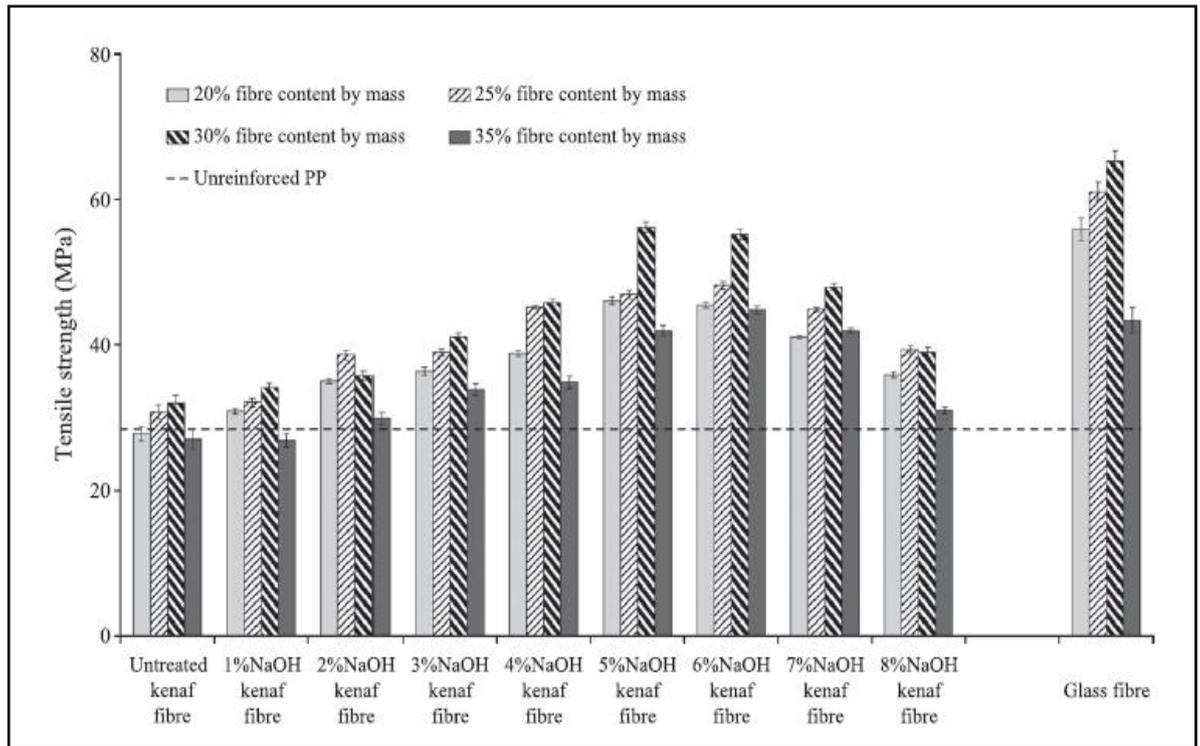


Figure 2.4: Tensile strength of alkali-silane treated composites [7].

The study done by El-Shekeil et al. [9], the kenaf fibre reinforced polyurethane was treated with alkaline and pMDI which is a silane solution. When the composites are treated with only alkali solution, the tensile strength decreases while the tensile modulus showed a slight increment. This explained that when the composite was treated with alkaline solution, the impurities are removed and the surface roughness of the fibre increase in order to bond better with the matrix. However, when the composite was treated together with pMDI solution, the tensile strength increase about 30% from the untreated composite and the tensile modulus also showed a significant increase of 42%. The silane solution actually rooted the cross-linking between the fibre and matrix which made the fibres is able to transfer more load thus increasing the tensile and flexural strength of the composites.

John et al. [10] used zein, a protein component which had been obtained from corn, as the coupling agent in order to analyse its effect on interfacial bonding in kenaf fibre reinforced polypropylene composites. The composite which had been treated with zein has showed a slight increment in

the tensile strength and flexural strength. However the impact strength is seen to decrease when the composite was treated with zein. This explained that during impact fracture the energy dissipation mechanisms operating are matrix and fibre fracture, fibre–matrix debonds and fibre is pulled out. Fibre fracture dissipates lesser energy compared to fibre pull out. Therefore, the main failure mechanism in these composites is fibre fracture, resulting in lower energy dissipation hence impact strength decreases.

Furthermore, as reviewed by Ku et al. [11] the effects of different fibre surface modifications when being treated with 2%NaOH process and then soaked with 5% NaOH and coupling agent, on jute/polybutylene succinate (PBS) bio-composites. The results showed that surface modifications could eliminate surface impurities, increased surface roughness and decreased the diameter of jute fibre, therefore significantly increased the tensile strength and modulus of the composites. It was also observed that when the bio-composites of jute fibres were treated by 2%NaOH, 2 + 5%NaOH or coupling agent, their tensile properties increased obviously when compared to those untreated and yielded an optimum value at fibre content of 20 wt.%. The results also showed that the strength and stiffness of composites were dependent on the types of treatment.

In other experiment which was also reviewed by Ku et al. [11] the composites contained 10 w/t% of fibres were processed by extrusion and injection moulding. Five surface modification methods which are alkali, silane, potassium permanganate, acrylic acid, and sodium chlorite treatments, were employed to improve the interfacial bonding between fibres and matrix. After the surface had undergone the modification, the result showed that the bio-composite tensile strengths were increased. Among these surface modification techniques, acrylic acid was found to be a relatively good method in enhancing tensile properties of both flax or high density polyethylene (HDPE) and linear low density polyethylene (LLDPE) bio-composites.

Demir et al. [12] had also suggested surface treatments on PP/luffa composites in order to improve the tensile and water sorption properties. In the experiment done by Demir et al. [12], these composites were treated with three different types of coupling agents, which are (3-aminopropyl)-triethoxysilane (AS), 3-(trimethoxysilyl)-1-propanethiol (MS) and maleic anhydride grafted polypropylene (MAPP). The composites were pre-treated with sodium hydroxide solution before being treated with the coupling agents. Both, the tensile strength of the treated and untreated composites decreased as the fiber content increased. The reduction in the tensile stress with an increase of filler content could be explained by the decrease in the effective matrix cross section.

Nevertheless, it was clearly observed that by the silane-coupling agents the tensile strength increase noticeably. The amino functional silane (AS) and mercapto silane (MS) treated fibre composites showed a reactive coupling effect that resulted in higher tensile strength rather than to the untreated composites. The increase in the tensile strength with the silane treatment can be explained by the improvement on the interfacial bonding between the filler and the matrix. Without the coupling agent, the only adhesion mechanism is inter-diffusion. Silane coupling agents yields to hydrogen and covalent bonding between hydroxyl groups of filler and polysiloxanes formed by hydrogenation of silanes which serves better adhesion between the fibre and the matrix. Higher interfacial bonding improved the stress transfer through the fibres, thus increasing the tensile strength of composites.

Aji et al. [13] had also reviewed the effect of coupling agents for enhancing adhesion thus improving interfacial bonding through producing covalent bonds between the fibre and the matrix. Coupling agents bearing two reactive groups can be used in order to chemically adjust the interaction surface of fibres and the polymer materials with a view to allow the establishment of covalent bond between them resulting in getting materials with very good mechanical properties where the interaction between the OH functional group at the fibre surface and the copolymerization of the other

reactive group with the matrix. Silane is one of the coupling chemicals which offer some major advantages, such as it is commercially available. Secondly, they tolerate an alkoxy silane group which are able of reacting with the OH-rich surface of natural fibres at one end, while at the other end they have a large number of functional groups which can be customized as a function of the matrix to be used.

On other study done by Valadez-Gonzalez et al. [14], the fibre-matrix bond strength is observed when the fibre being treated with alkali solution, silane and the surface was being pre-impregnated with polyethylene dilute solution. The interfacial shear strength between natural fibres and thermoplastic matrices has been improved by morphological modification of the fibre surface. The level of fibre–matrix adhesion is further enhanced by the presence of a silane-coupling agent. The alkaline treatment has two effects on the fibre. First, it increases the surface roughness that results in a better mechanical interlocking; and secondly, it boosts the amount of cellulose exposed on the fibre surface, thus increasing the number of possible reaction sites. The fibre pre-impregnation allows a better fibre wetting which in a normal fibre–polymer mixing procedure would not be possible because of the high polymer viscosity. Thus, the pre-impregnation enhances the mechanical interlocking between fibre and matrix. However, the fibre-surface silanization results in better interfacial load transfer efficiency but do not seem to improve the wetting of the fibre.

According to Li et al. [15], by doing chemical treatments on the fibre, the surfaces of the fibres are modified so that the bonding of the fibre and matrix are more favourable. In this paper, different types of chemical treatment had been done on the natural fibre which are alkali, silane, acetylation, benzylation, acrylation, maleated coupling agents, isocyanates, permanganates, and others are discussed. The main objective of chemical treatment is to improve the bonding between the fibre and also matrix. When treated, the water absorption properties of natural fibres is reduced, thus enabling better bonding and improving the mechanical properties of the natural fibre reinforced composite.

Study done by Kabir et al. [16], also relates to the chemical treatments on natural fibre. However, in this paper hemp fibre is being used. The hemp fibre is being treated with alkali, acetyl, and silane chemicals. When being treated with alkali and acetyl the cellulose and lignin are removed. These two constituents are unfavourable because they contributed in the hydrophilic nature of the composite. Silane treatment does not remove the cellulose and lignin, but the silane facilitate the coupling with the fibre constituents.

Studies done by Bachtiar et al. [17] and Vilay et al. [18] both had done on the treatment of natural fibre. Bachtiar [17] had done the study on treatment of alkali and compatibilizing agent on sugar palm fibre while Vilay [18] had done study on the comparison of alkali and acrylic acid treatment on bagasse fibre. All treated fibre shows improvement on their mechanical properties being compared to the untreated fibres [17]. Results obtained by Vilay [18] shows that fibre which being treated using acrylic acid shows better tensile strength compared to alkali treated.

The novelty of this project is basically there has not yet been any study done on the effect of alkali-silane treatment on woven kenaf reinforced polypropylene composites. The structured of the woven kenaf is expected to improve the load that the fibre could bare, thus increasing the overall tensile and flexural strength of the composites.

CHAPTER 3

METHODOLOGY

In this chapter, the step by step procedure in order to fabricate the test samples will be described. There will be three types of specimen that will be produced, which are neat polypropylene, untreated composite and treated composite. First, the woven kenaf will be treated with alkali then silane solution. Next, the composite will be fabricated using compression moulding technique. Lastly, the tests and the results' analysis will take place. The processes involved in order to produce the composite can be seen in Figure 3.1.

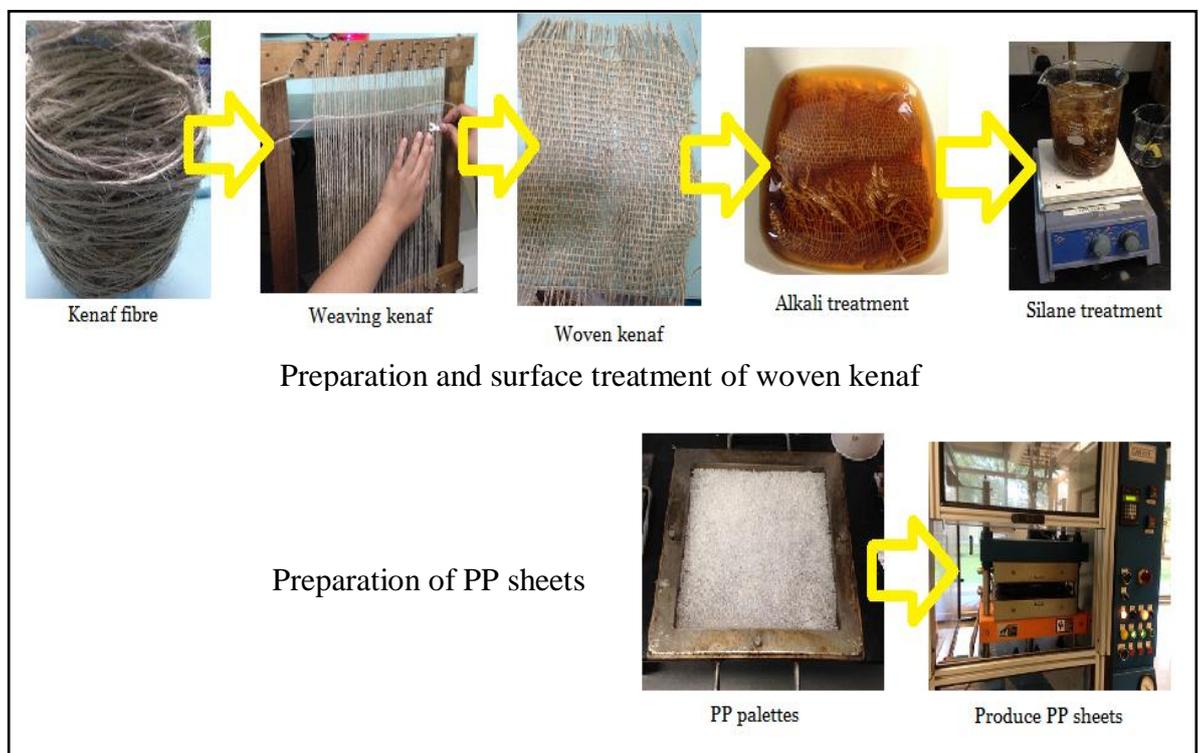


Figure 3.1: Steps taken before composites can be produced.

For this project, the samples are prepared in three ways which are the neat PP, untreated kenaf /PP composite and treated woven kenaf/PP composite. It is further explained in Table 3.1.

Table 3.1: The preparation of samples.

Specimen	Matrix (wt. %)	Fiber (wt. %)	Treatment
Neat PP	100	0	-
Untreated PP/woven kenaf	85	15	-
Treated PP/woven kenaf	85	15	NaOH + Silane

3.1 Producing weaved kenaf

By using the wooden board, the kenaf fibre is weaved. First the kenaf fibre is passed through the nails that had been situated at the both vertical end of the board. Then, the kenaf is passed through the vertical kenaf fibre perpendicularly in an ‘up and down motion’ continuously until the bottom end is reached. This action can be seen in Figure 3.2. The finished weaved kenaf could be seen in Figure 3.3.



Figure 3.2: Kenaf fibre is weaved using the weaving board.

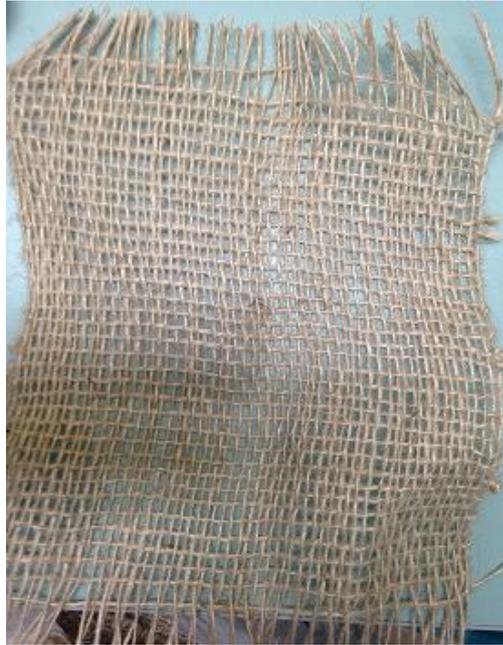


Figure 3.3: Woven kenaf.

3.2 The alkali-silane treatment of kenaf

Woven kenaf is immersed in 5% NaOH solution for about 24 hours at room temperature as seen in Figure 3.4. After immersion, the woven kenaf is washed through tap water and then immersed in water containing 1% concentration of acetic acid solution in order to neutralize the remaining alkaline molecules of NaOH in the mat as in Figure 3.5. The mat is then dried in the oven at 45°C for 12 hours [6].

Then, the fibre is immersed in a solution where 5% of weaved kenaf weight of *three-aminopropyltriethoxy* silane is diluted in a 50% aqueous solution of methanol and acetic acid is added in order to maintain the acidity of the solution [6]. The mats is immersed in the solution for about 4 hours at 28°C as in Figure 3.6. Next, the mat is washed with distilled water and dried in the oven at 45°C for another 12 hours [6].



Figure 3.4: Woven kenaf after being immersed in 5% NaOH solution for 24 hours.



Figure 3.5: Woven kenaf being immersed in 1% concentration acetic acid solution.

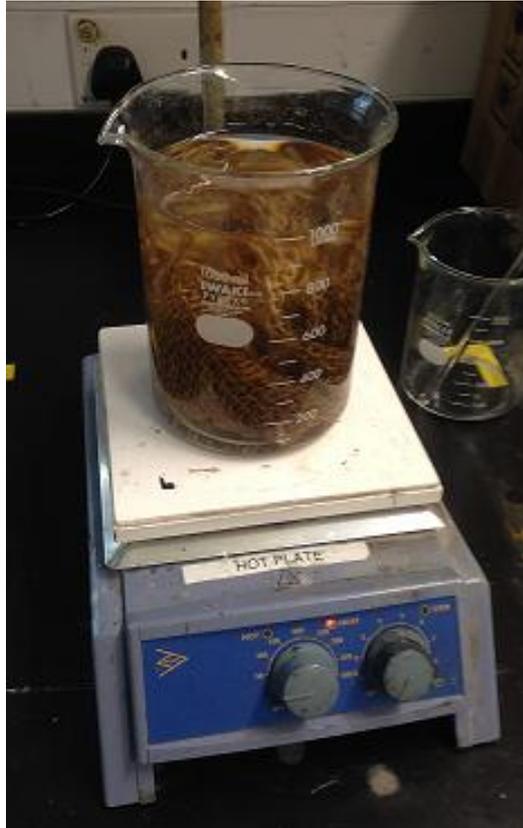


Figure 3.6: Woven kenaf during silane treatment at 28°C.

3.3 Fabrication of Polypropylene sheets

By using the compression moulding technique, the polypropylene (PP) sheets are produced. The thickness of the PP sheets should be 2 mm, therefore 2mm thickness of mould is used. First, the wax is applied thoroughly on the top and bottom part of the mould. Then, the PP palettes are distributed evenly on the bottom part of the mould as seen in Figure 3.7.

Then, the mould is closed and the mould is put under the compression plate of the machine. Then, the mould is preheated to 190°C for 10 minutes. Then, the mould is compressed under the same temperature at 10 tons force for another 10 minutes. Then, the mould is cooled under compression for about one hour until the temperature drops to at least 100°C as from Figure 3.8. Lastly, the PP sheets are removed from the mould.



Figure 3.7: The PP palettes are evenly distributed.



Figure 3.8: The mould is being cooled under compaction.

3.4 Fabrication of the composites

The composites produced will be 6 mm in thickness. Therefore, a 6mm thickness mould must be used. The bottom and top part of the mould is waxed thoroughly. Then the PP sheet is laid on the bottom part continued with a layer of kenaf mat and this is continued alternately as seen in Figure 3.9. These composites will have 3 layers of PP sheets and 2 layers of kenaf mat. Then the mould is closed and put under the compression plate.

The mould is then preheated to 185°C for 10 minutes. Then, the mould is compressed at the 185°C for 15 minutes under 10 tons force. Lastly, the composite is cooled under compression until the temperature drops at least at 80°C. This usually takes up at least 3 hours. Lastly, the composites are removed from the mould. In order to calculate the weight percentage of the composite, first, the kenaf is weighed then after the composite is produced, and the composite is weighed. Then, weight percentage of the fibre and matrix is calculated. The weight percentage of the fibre and the matrix could not be produced as desired in 30-70 ratios.

This is due to limitation of the processing technique. The desired weight percentage of the matrix and fibre is hard to produce as the percentage of the matrix flushed was hard to predict. Therefore, the weight of the percentage was determined after the composites had been produced. Since the layering of the composite and the total thickness of the composites are the same, the weight percentage of the matrix and fibre obtained are similar for the entire produced composite.

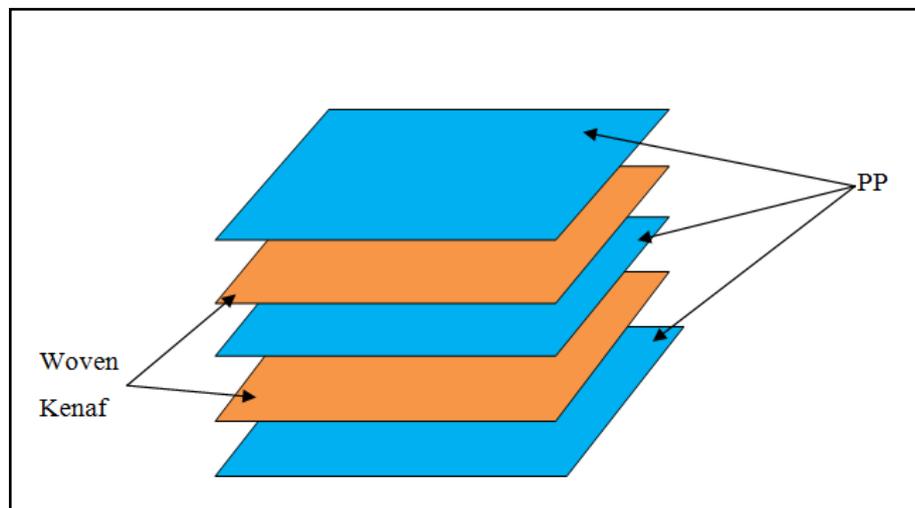


Figure 3.9: The layering of the composite.

3.5 Cutting of the samples.

Since the composites could not be cut into ‘dog bone’ shapes because the laser could not penetrate the composite, the composites will be cut into rectangular shapes. The composites are marked before cut into rectangular shapes by using a jigsaw as in Figure 3.10. Then, the width and length required is obtained by using the milling machine based in Figure 3.11.



Figure 3.10: The marked composites are cut into rectangular shapes.



Figure 3.11: The samples are milled in order to obtain required width and length.

3.6 Testing procedures and standards

In the second stage, the specimens will be tested. Tensile test under the standard ASTM D638 is used in order to test the tensile strength of the composite. The recorded results will then be analysed. There will be three types of samples which include neat polypropylene, untreated composites and treated composites. For each type of samples, there will be 5 specimens. The data will be recorded and the average reading will be taken for further analysis. The data will then be compared and discussed. Since the composite had encountered some obstacles in cutting process, the composites will be cut into rectangular shapes based on w_o and L_o based in Table 3.2. From the standard by ASTM D638 under Type I, the w_o should be 19mm, L_o should be 165 mm and the gage length, G should be 50 mm as seen in Figure 3.12 [19]. The dimension of the specimen based on ASTM D638 type I standard can be seen in Table 3.2

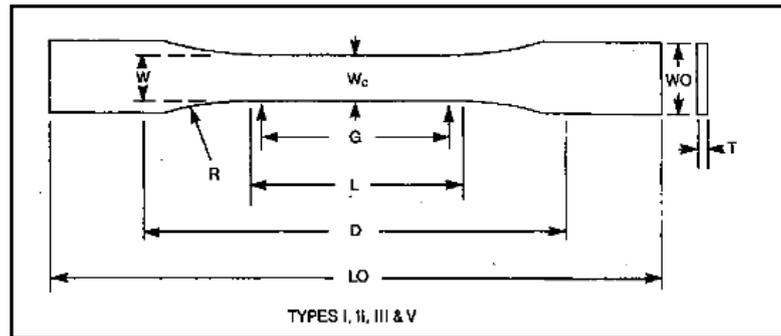


Figure 3.12: The standard dimensions for tensile test Type I [19].

Table 3.2: Dimension for test samples of ASTM D638 standards [19].

Dimensions	Length (mm)
W – Width of narrow section	13 ± 0.5
L – Length of narrow section	57 ± 0.5
WO – Width overall	19 ± 6.4
LO – Length overall	165
G – Gage length	50 ± 0.25
D – Distance between grips	115 ± 5
R – Radius of fillet	76 ± 1
T – Thickness	7 or under

ASTM D638 Tensile Test Procedure [19].

1. The width and the thickness of the specimen is taken 3 times within the gage length by using a micrometer. The data is recorded.
2. The specimen is loaded into tensile grips as in Figure 3.13.
3. The extensometer is attached to the sample.
4. The test is started by separating the tensile grips at a constant rate of speed of 5.00 mm per minute. The target time from start of test to break should be from 30 seconds to 5 minutes.
5. The test is ended after sample break (rupture) as in Figure 3.14.
6. The result obtained is recorded. And the steps 1 to 4 are repeated for 5 times for each type of samples.

After the test has ended, the ruptured part of the sample is cut into smaller parts before being analysed using the FESEM (Field Emission Scanning Electron Microscope) method.



Figure 3.13: The sample is loaded into the tensile grip.

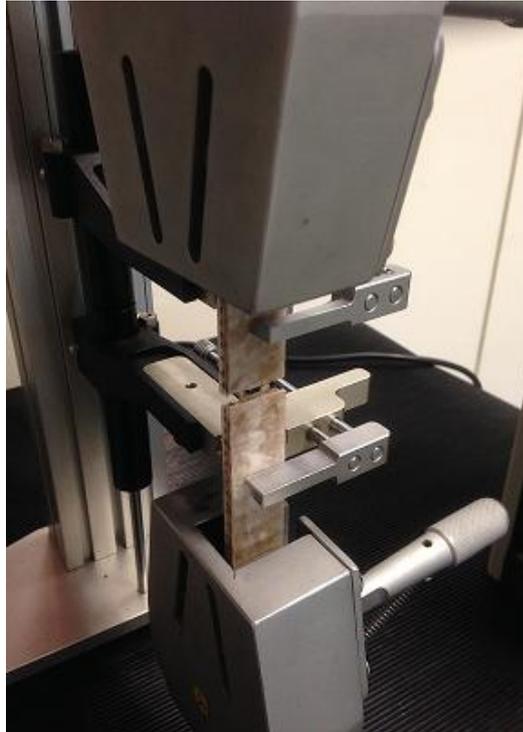


Figure 3.14: The test is ended when the samples break.

3.7 Project Milestones and Gantt chart

This project was completed within 28 weeks. Since the preparation of the sample and also the testing of the sample required a significant amount of time, key milestones and the Gantt chart as shown in Figure 3.15 and Table 3.3 has been produced in order to aid the student in managing the time for completing the project within the duration given.

Milestone List		Date
Obtained prediction data	★	9/8/2013
Acquired materials and tools needed	★	8/11/2013
Finished preparing test samples	★	9/12/2013
Finished mechanical testing	★	13/12/2013
Analyzed results	★	20/12/2013
Submitted dissertation and technical paper	★	15/1/2014

Figure 3.15: Key milestones of the project.

Table 3.3: Activity planned throughout the project.

Month	Jul-13				Aug-13				Sep-13				Oct-13				Nov-13				Dec-13				Jan-14			
Week	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28
1. Prediction of composite tensile strength																												
i. Gather material properties through literature research																												
ii. Calculate tensile strength of composite using rule of mixture																												
Predicted data obtained																												
2. Acquisition of materials and tools																												
i. Acquiring kenaf fibre, alkali, acetic acid, methanol and weaving board																												
ii. Acquiring silane solution																												
iii. Booking to use compression moulding machine, oven and cutting machine																												
iv. Booking to use tensile test equipment and FESEM lab																												
Acquired all materials and tools																												
3. Production of PP/Woven kenaf composite																												
i. Weaving kenaf manually																												
ii. Surface treatment of woven kenaf																												
iii. Producing PP sheets																												
iv. Producing composites																												
v. Preparing sample according ASTM D638 standards																												
Test samples ready for testing																												
4. Mechanical testing																												
i. Test samples- 7 samples																												
ii. Neat PP																												
iii. Untreated composites																												
iv. Treated composites																												
Finished mechanical testing																												
5. Result Analysis																												
i. Validation of predicted and experimental data																												
ii. Comparison of strength between neat PP, untreated and treated composites																												
iii. Study the morphological structure of composite from FESEM photo																												
All obtained results analyzed																												
6. Documentation process																												
i. Preparing final report																												
ii. Preparing technical paper																												
Submission of dissertation and technical paper																												
★																												

CHAPTER 4

RESULTS AND DISCUSSION

In this chapter, the analytical and experimental results are presented. A Field Emission Scanning Electron Microscope (FESEM) was used to examine the morphology structure of the surface of fractured composite samples.

4.1 Analytical Result

Analytical calculation of tensile strength is done in order to estimate or predict the possible result that could be obtained from the experiment. In order to estimate the tensile strength of the composite, some literature data were needed. The required data were given by Table 4.1 [2]. Based on Table 4.1, the kenaf was expected to fail at the elongation or strain of 2.2%.

Filler/reinforcement in PP	Units	Neat PP	Kenaf	Glass	Talc	Mica
Filler by weight	%	0	50	40	40	40
Filler by volume	%	0	39	19	18	18
Specific gravity	–	0.9	10.7	1.23	1.27	1.26
Tensile modulus	GPa	1.7	8.3	9	4	7.6
Specific tensile modulus	GPa	1.9	7.8	7.3	3.1	6.0
Tensile strength	MPa	33	65	110	35	39
Specific tensile strength	MPa	37	61	89	28	31
Flexural modulus	GPa	1.4	7.3	6.2	4.3	6.9
Specific flexural modulus	GPa	1.6	6.8	5.0	3.4	5.5
Elongation at break	%	≥10	2.2	2.5	–	2.3
Notched Izod impact	J/m	24	32	107	32	27
Water absorption (24 h)	%	0.02	1.05	0.06	0.02	0.03

Table 4.1: Properties of filled/reinforced polypropylene composites [2].

The stress of matrix at fibre failure, σ'_m was estimated to be less than σ_m^* , which is around 30 MPa. All necessary data needed for the calculation were obtained from Table 4.1. The tensile strength of the composite was then calculated by using Equation 2.1 which is based on the rule of mixture.

Other data that had been gathered prior to the calculations are as follows:

- Volume fraction of the matrix, $V_m = 0.85$
- Volume fraction of the fibre, $V_f = 0.15$
- Ultimate tensile stress of fibre, $\sigma_f^* = 65\text{MPa}$ [2]
- Stress of matrix at fibre failure, $\sigma'_m = 30\text{ MPa}$
- Ultimate tensile stress of matrix, $\sigma_m^* = 33\text{ MPa}$ [2]

With the assumption of perfect bonding between the matrix and fibre, tensile strength of the composite, σ_c could be calculated as shown below.

$$\begin{aligned}\sigma_c &= \sigma'_m V_m + \sigma_f^* V_f \\ &= 30 (0.85) + 65(0.15) \\ &= \underline{\underline{35.25\text{ MPa}}}\end{aligned}$$

4.2 Experimental Results

Before the test was done, the width and thickness of the specimens were measured using the micrometer. The width and thickness were measured three times and the average was calculated. The result obtained are tabulated and summarized in Table 4.2 and Figure 4.1 respectively.

Table 4.2: Experimental results of tensile strength of the samples.

Sample	Tensile Strength (MPa)	Average Tensile Strength (MPa)	Std deviation
Neat PP	16.53	16.12	0.32
	15.71		
	16.30		
	16.15		
	15.90		
Untreated composite	26.84	26.05	0.63
	26.43		
	26.05		
	25.29		
	25.63		
Treated composite	24.38	24.28	0.75
	23.20		
	23.92		
	24.86		
	25.05		

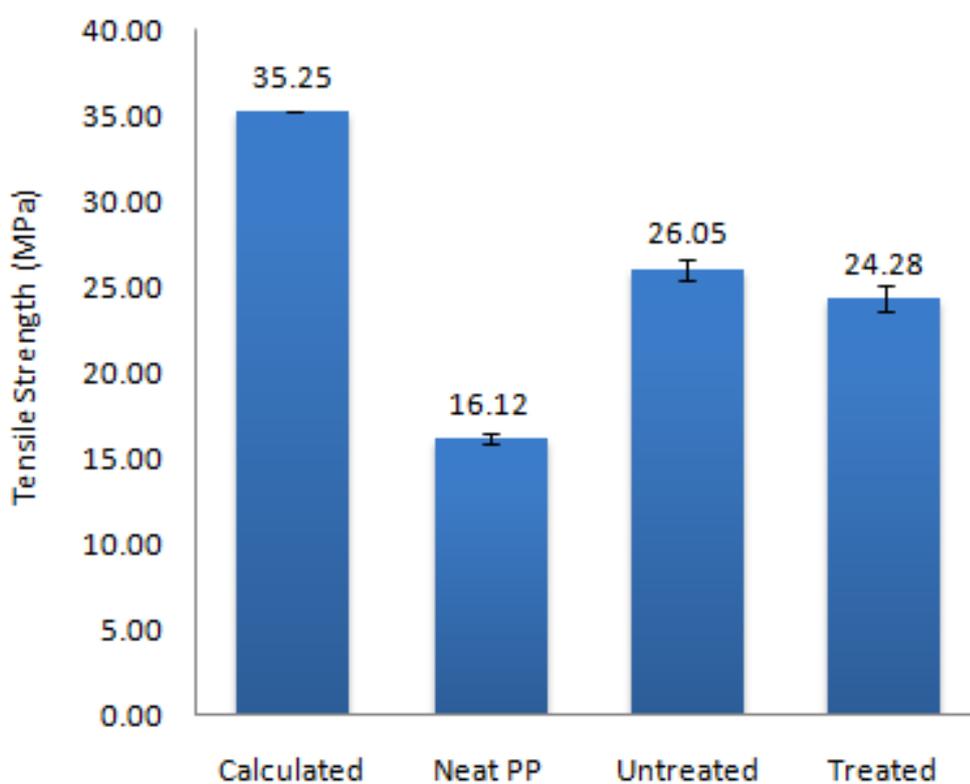


Figure 4.1: Analytical and experimental tensile strength.

From Figure 4.1, it could be seen that by adding woven kenaf in the composites, the tensile strength of the untreated composites is increased up to 62% compared to the neat PP. However, the tensile strength of the treated composite decreased slightly. The reason for this phenomenon will be discussed in the next section.

The difference between analytical and experimental result is most likely due to the assumption of perfect bonding made in the analytical calculation. In fact, there would be imperfection of the interfacial bonding between the matrix and fibre in the experimental samples. Therefore, the experimental results are always lower than the analytical ones.

The decrease of the tensile strength of the experimental value could also be associated with voids or air bubbles that were formed during the compression moulding. The existence of these air bubbles would contribute to lower tensile strength as the voids will be the rupture area during testing.

4.3 Morphological study

In this section, the results of treated and untreated composites are further discussed. Based on Figure 4.1, the untreated composites had a slightly higher value of tensile strength compared to the treated composites. In order to analyze the results in a greater depth, FESEM analysis was carried out to study the morphology of the composites. Figures 4.2, 4.3 and 4.4 show the FESEM micrographs of the untreated composites taken at different magnification. Figures 4.5, 4.6 and 4.7 depict the FESEM micrographs of the treated composites taken at different magnification.

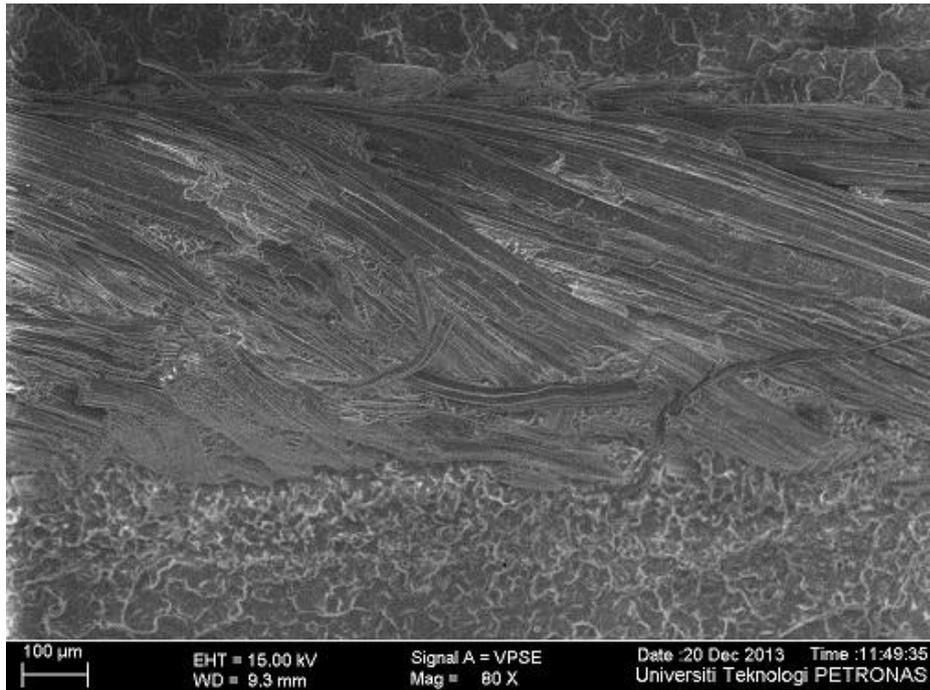


Figure 4.2: FESEM of untreated composite with magnification of 80X.

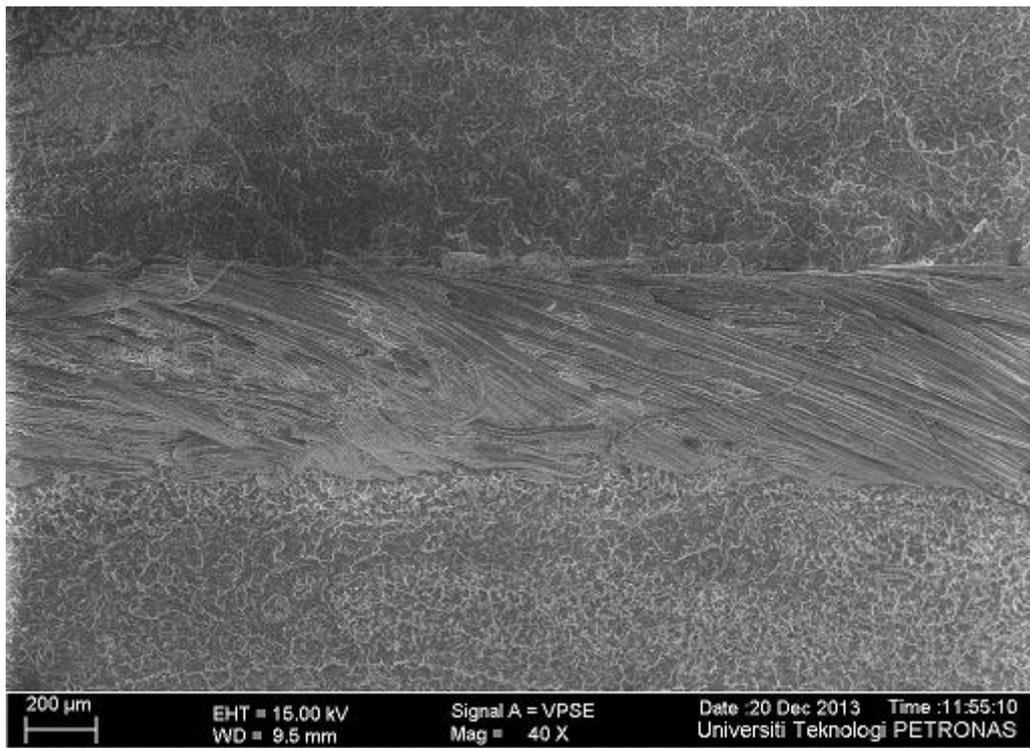


Figure 4.3: FESEM of untreated composite at magnification of 40X.



Figure 4.4: FESEM of untreated composite at magnification of 100X.



Figure 4.5: FESEM of treated composite at 60 X magnification.

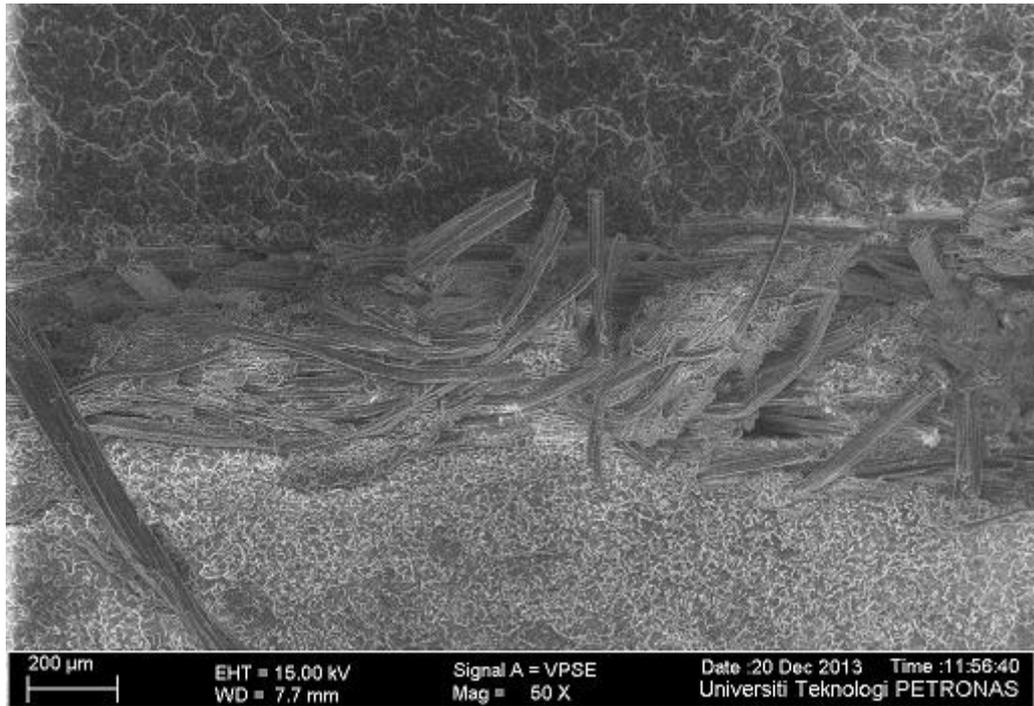


Figure 4.6: FESEM of treated composite at 50 X magnification.



Figure 4.7: FESEM of treated composite at 60 X magnification.

In Figures 4.2 and 4.3, it could be seen that the fibre was not fully covered by the matrix. In Figures 4.4 and 4.5, voids or air bubbles could be seen in the pictures and had been highlighted. However, voids existed in both untreated and treated composites. In Figure 4.6 it could be seen that the fibre deteriorated within the matrix. However, the interfacial bonding was improved as shown in Figure 4.7. This proves that the alkali-silane treatment improves the bonding between the matrix and fibre. However, the deterioration of the fibre inside the matrix occurred in the treated composite reduces the tensile strength compared to the untreated composite. When the fibres ruptured inside the matrix, the connection between the fibres is broken along the way, prohibiting the chance of load transfer from the matrix to the fibre. Therefore, the tensile strength of the composite is lower. The deterioration of the fibre may be due to the drying process that was not done continuously, allowing the fibre to be deteriorated in wet conditions.

CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS

Based on the result obtained as shown in Figure 4.1 and Table 4.2, improvement of 62% in tensile strength was achieved in the untreated composites compared to neat PP. An improvement of 51% in tensile strength was achieved in the treated composites compared to neat PP. The decrease of tensile strength of the treated composite is most likely due to the rupture of the fibre as shown in the FESEM micrographs.

There are several recommendations that could be used in the future endeavour of this project. The effect of alkali-silane treatment on the flexural and impact properties of PP/woven kenaf composites should be done. Continuous drying process should be done in order to avoid potential rupturing of the kenaf fibre. The influence of compression moulding parameters on the mechanical properties of the samples should also be further studied.

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APPENDICES

A1- Complete result of Neat PP

Sample Code	Width,w (mm)	Thickness, t (mm)	Tensile Strength (MPa)
N1	11.80	3.18	16.53
N2	11.85	3.52	15.71
N3	11.76	3.43	16.30
N4	11.70	3.41	16.15
N5	11.82	3.27	15.90
Average	11.786	3.362	16.118
Std Deviation	-	-	0.32

A2- Complete result for untreated composites.

Sample Code	Width,w (mm)	Thickness, t (mm)	Tensile Strength (MPa)
U1	19.67	6.33	26.84
U2	19.33	6.31	26.43
U3	19.30	6.07	26.05
U4	19.20	6.09	25.29
U5	19.36	6.45	25.63
Average	19.372	6.25	26.048
Std Deviation	-	-	0.62

A3- Complete result for treated composites.

Sample Code	Width,w (mm)	Thickness, t (mm)	Tensile Strength (MPa)
T1	19.21	6.66	24.38
T2	19.73	6.65	23.20
T3	19.38	6.52	23.92
T4	19.03	6.64	24.86
T5	19.31	6.72	25.05
Average	19.332	6.638	24.282
Std Deviation	-	-	0.75