Faculty of Engineering

Physical, Mechanical, Thermal and Morphological Properties of Nipah (*Nypa Fruticans*) Palm Fibre Reinforced Unsaturated Polyester Composites

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A thesis submitted

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DECLARATION

I declare that the work in this thesis was carried out in accordance with the regulation of Universiti Malaysia Sarawak. It is original and is the result of my work, unless otherwise indicated or acknowledged as referenced work. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

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ABSTRACT

The physical, chemical, thermal, crystalline, surface morphology, tensile, water absorption of untreated and alkali treated nipah (*nypa fruticans*) palm fibre were investigated in this work. There were two types of fibres extracted from the fronds, Fibre A and Fibre B, with 0.530 mm and 0.039 mm diameter respectively. The untreated and alkali treated nipah palm fibre were studied for its behaviour towards Thermogravimetric Analysis (TGA), Fourier Transform Infrared Spectroscopy (FTIR), Differential Scanning Calorimetry (DSC) and X-Ray Diffraction (XRD). Hand lay-up and cold press method were used to fabricate nipah palm composite. The highest composite strength and Young's modulus for Fibre A were at 29.36 MPa and 471 MPa respectively. For Fibre B composites, the highest strength was at 29.67 MPa and 102 MPa for its Young's modulus. The results of water absorption test showed better absorption resistance of treated fibre towards water. Future work direction of this investigation is to use vacuum bagging method to fabricate the composite and to use different types of polymer matrix.

Keywords: *Nypa fruticans*, nipah palm, natural fibre, natural fibre composites, fibre extraction

Sifat-Sifat Fizikal, Mekanik, Termal dan Morfologi Komposit Poliester Tak Tepu Bergentian Pokok Nipah

ABSTRAK

Sifat fizikal, kimia, terma, kristal, morfologi permukaan, ujian tegangan, serapan air untuk gentian nipah (nypa fruticans) tak dirawat dan dirawat alkali dikaji dalam kajian ini. Terdapat dua jenis gentian yang diperolehi daripada pelepah pokok nipah iaitu, Gentian A dan Gentian B, masing-masing berukuran 0.53 mm dan 0.039 mm diameter. Gentian-gentian tersebut dirawat menggunakan alkali. Kesan Gentian Tunggal A yang tidak dirawat dan dirawat telah dikaji untuk kesan rawatan terhadap Analisis Termogravimetrik (TGA), Fourier Transformasi Spektroskopi Inframerah (FTIR), Kalorimetri Imbasan Pembezaan (DSC) dan Pembelauan Sinar-X (XRD). Kaedah bengkalai tangan dan pengacuan tekanan – sejuk digunakan untuk proses fabrikasi komposit polimer bergentian nipah. Kekuatan komposit dan modulus Young tertinggi untuk Gentian A adalah pada 29.36 MPa dan 471 MPa. Untuk komposit Gentian B, kekuatan tertinggi adalah pada 29.67 MPa dan 102 MPa bagi modulus Young. Hasil ujian serapan air mendapati rintangan serapan adalah tinggi untuk gentian yang dirawat. Cadangan kerja penambahbaikan di masa hadapan adalah menggunakan kaedah hampagas atau beg bertekanan untuk proses fabrikasi komposit dan juga menggunakan polimer matrik yang lain.

Kata kunci: *Nypa fruticans, pokok nipah, gentian semula jadi, komposit gentian semula jadi, pengekstrakan gentian*

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LIST OF ABBREVIATIONS

ASTM	American Society for Testing and Materials
CRAUN	Crop Research and Application Unit
DSC	Differential Scanning Calorimetry
FRIM	Forest Research Institute of Malaysia
FTIR	Fourier Transformation Infra-Red
MEKP	Methyl Ethyl Ketone Peroxide
NaOH	Natrium Hydroxide / Sodium Hydroxide
PPKS/ICATS	Pusat Pembangunan Kemahiran Sarawak / International College of Advanced Technology Sarawak
SEM	Scanning Electron Microscope
TAPPI	Technical Association of Pulp and Paper Industry
TGA	Thermogravimetric Analysis
XRD	X-Ray Diffraction
5W/0N	5 wt% fibre loading / untreated composite
5W/2N	5 wt% fibre loading / 2% alkali treated composite
5W/4N	5 wt% fibre loading / 4% alkali treated composite
5W/6N	5 wt% fibre loading / 6% alkali treated composite
5W/8N	5 wt% fibre loading / 8% alkali treated composite

5W/10N	5 wt% fibre loading / 10% alkali treated composite
10W/0N	10 wt% fibre loading / untreated composite
10W/2N	10 wt% fibre loading / 2% alkali treated composite
10W/4N	10 wt% fibre loading / 4% alkali treated composite
10W/6N	10 wt% fibre loading / 6% alkali treated composite
10W/8N	10 wt% fibre loading / 8% alkali treated composite
10W/10N	10 wt% fibre loading / 10% alkali treated composite
15W/0N	15 wt% fibre loading / untreated composite
15W/2N	15 wt% fibre loading / 2% alkali treated composite
15W/4N	15 wt% fibre loading / 4% alkali treated composite
15W/6N	15 wt% fibre loading / 6% alkali treated composite
15W/8N	15 wt% fibre loading / 8% alkali treated composite
15W/10N	15 wt% fibre loading / 10% alkali treated composite

CHAPTER 1

INTRODUCTION

1.1 Introduction

Our mother earth has been providing us with abundant natural resources of fibres for the livelihood of the people to survive. These natural fibres have been used as ropes, roof, huts, brooms, hat, baskets, and many more. The potential of these natural fibres has been the topic of various research works, where they were investigated as reinforcing material in polymer composites. Extensive research has been done to investigate mechanical, thermal, chemical and physical properties of natural fibres. Literatures have provided countless research work on natural fibre. More new natural fibres have been investigated, and among the newly introduced natural fibres are the betel nut fibre (Lai & Mariatti, 2008; Yusriah et al., 2014b), the napier grass (Haameem et al., 2016; Reddy et al., 2009; Ridzuan et al., 2016) and the roselle fibre (Nadlene et al., 2016; Thiruchitrambalam et al., 2010). These newly introduced fibres are likely to have a promising future to be among the more established natural fibres such as jute, cotton, ramie, flax, hemp, kenaf and bamboo. Their strength is comparable to the established natural fibres.

Nipah palm or scientifically known as *Nypa fruticans* (Figure 1.1) is the new natural fibre source studied in this research. Little is known about this non-timber forest product that grows at a swampy area along the Sarawak Rivers. Nipah palm is locally known as "*pokok apong*" by the Sarawakian. It is well-known for its sugar sap; it has been collected by the population nearby and has become the source of income for them. The

dried or fresh young leaves of nipah palm are traditionally used to make baskets, roof thatches, food wrappers, cigarette wrapper and many other items. The large population of this type of palm has encouraged the research work to be done and to bring it to a higher level than it is now. In order to explore the possibilities of extracting fibres from nipah palm, the literatures on other natural fibres were used as guidelines and references.

Figure 1.1: Nipah palm along Sungai Kuap, Kuching

Nipah is the most abundant palm in the mangrove forest of the South, Southeast Asia and the Oceania. Historically, this palm provides a wide diversity of use towards the indigenous people living near the forest (Tsuji et al., 2011). Nipah palm is abundant along the riverbanks of Sarawak River. It grows well where salt water and fresh water meets. This palm is trunkless and has its fronds grow from the roots. The trunk submerged in the water during high tide and exposed to the air during low tide. The submerged fronds are bulky and spongy compared to the upper fronds. The spongy nature of the lower fronds is assumed to provide the palm the buoyancy during high tide. The upper frond is rigid and sturdy, supporting the towering fronds of the palm. They can grow up to 10 m tall (Teo et

al., 2010; Tsuji et al., 2011). Nipah leaves have been used for roof thatching, cigarette wrappers, raincoats, brooms, mats, baskets, umbrella, hats and ropes (Teo et al., 2010). Their young leaves have been harvested by the locals as dessert wrappers. Nipah sugar sap has also been collected and used as sweetener in local delicacies. Countries like Malaysia and Indonesia use it as the source of bio-ethanol (Hidayat, 2015; Tamunaidu & Saka, 2011). Sugar sap is used to produce vinegar, alcohol and fermented beverage in Philippines, Malaysia, Indonesia and Bangladesh. Young nipah seeds are eaten raw or preserved in syrup (Hamilton & Murphy, 1988).

1.2 Research gap

There is a growing interest in natural fibre reinforced polymer composites. Extensive research was done on jute, sisal, bamboo, coir, kenaf, oil palm, sugarcane bagasse and other natural fibre. Each natural fibre has its own common method of extraction. The extraction of natural fibres greatly depends on the structure and the fibre source. The mechanical strength of polymeric composites fabricated with these natural fibres is being investigated extensively, providing various data as the benchmark for nipah palm fibre. Various treatment methods to improve the adhesion of natural fibre with polymeric matrices can be easily available in the literature. Common treatment for natural fibre is the alkali treatment investigated at various percentages. The mechanical properties of single fibre and fibre reinforced polymer composites are abundant. All these investigations provide abundant material properties of natural fibres in the literature. There is a huge research gap between the common natural fibre and nipah palm fibre especially on its potential as reinforcing agent. Extensive investigation on nipah palm fibre extraction, the characterisation of fibre properties, its reaction with treatment and its fibre strength

behaviour as composite reinforcing agent were conducted. These are the investigations that are done in this research and its ultimate purpose is to fill and close the research gap.

1.3 Problem statement

The abundance of nipah palm along the riverbanks of Sarawak Rivers opens up a new opportunity to conduct a study on the palm for fibre extraction and to be used in composite materials. There is a gap of knowledge about nipah palm fibre that leads to the interest in studying about its fibre. The palm itself has never been the subject of research for composite materials. Previous work done on nipah palm is on its particle as medium density fibre board (Kruse, 2001). Other investigation includes its biological standpoint (Tsuji et al., 2011) status and distribution of nipah palm (Teo et al., 2010) and biofuel crop (Hidayat, 2015; Phaiboonsilpa et al., 2011). None of the investigation covers on its fibre as reinforcing agent. This investigation is to extract nipah palm fibre as reinforcing agent. The characterisation of fibre in terms of its physical properties, chemical composition, thermal behaviour and its morphology were investigated. Therefore, this research work will provide new knowledge on the extraction method, composite fabrication, characterisation of nipah fibre, mechanical test, namely, tensile test and water absorption test.

1.4 Research hypothesis

The water retting method is predicted to be the method used to produce nipah palm fibre in large scale, since most natural fibres such as jute, coir, flax, and hemp use water retting process. Alkali treatment of nipah palm fibres will be able to improve the surface

adhesion of the fibres with the polyester matrix. Physical and mechanical properties of nipah palm fibre and its composites are expected to be reliable to be used as composite materials. The prospect of nipah palm fibre as reinforcing agent in polymer composites is promising. This will help the population surrounding nipah palm to generate more income in harvesting and extracting nipah palm fibre to be sold for composite fabrication.

1.5 Scope of the research

This research focuses on the development of the extraction method of nipah palm fibre from nipah palm fronds. Two types of fibre were found on nipah fronds, designated as Fibre A and Fibre B, based on their diameter. The diameter and density for both fibres were measured. Fibre A were examined for its chemical composition, series of alkali concentration 6%, 8%, 10% and 12% were conducted on nipah palm Fibre A to examine its effects towards the fibres. The effects of these treatment were analysed by FTIR, TGA, DSC and SEM. Untreated and treated single fibre tensile tests were conducted for Fibre A. Nipah palm fibre composites were fabricated with 5 wt%, 10 wt% and 15 wt%, in short and randomly aligned fibre loading condition. Water absorption test for untreated (5 wt% and 20 wt% loading) and 8% alkali (10 wt% and 15 wt% loading) and 10% alkali (15 wt% loading) were conducted. Fibre B were treated with 2% and 4% alkali, fabricated into composites and were subjected to tensile test. The morphology of the treated and the fracture surface were captured and analysed using SEM.

1.6 Research objectives

The objectives of this research are:

1. To investigate physical, thermal, chemical and morphological properties of single nipah fibre.
2. To evaluate tensile and water absorption properties of nipah fibre composites.

1.7 Thesis organisation

This report consists of five chapters. Chapter 1 is about the introduction for this research. Chapter 2 is the literature review of the research, containing the general research background, problem statements, past studies and their findings regarding natural fibre reinforced polymer composites, and the research objectives. Chapter 3 outlines the materials and methods of this research. It explains how nipah fibres were extracted, prepared for physical, thermal, chemical, morphological investigation, treatment with alkali, testing procedures and composites fabrication to achieve the objectives of this study. The research findings and discussion where concluded in Chapter 4. Chapter 5 provides the summary and conclusion based on the findings in this study.

CHAPTER 2
LITERATURE REVIEW

2.1 Overview

This chapter explains about natural fibre, its extraction method and the common fabrication methods for polyester composites. The fibre surface modifications using alkali conducted on other natural fibres are reviewed. The thermal, chemical and mechanical properties of other natural fibres are discussed. The morphology of natural fibre under SEM were reviewed and analysed. The water absorption behaviour of natural fibre was studied. The applications of natural fibre as car component were reviewed.

2.2 Nipah palm

Nipah palm grows in the mangrove forest, it is also known as mangrove palm. The growth area of nipah palm is where the fresh water meets the salt water of the mangrove forest. The muddy and brackish water of this area are the natural habitat of nipah palm. Nipah palm are rarely seen on the seashores, but more to the estuarine area. Two forms of nipah found in Malaysia is "*nipah gala*" and "*nipah padi*". Nipah palm found in Malaysia and Papua New Guinea is bigger than the one found in Philippines.

(a)

(b)

Figure 2.1: Mangrove forest distribution (a) Sarawak (b) Kuching, Samarahan, Sibu and Sarikei (SFC, 2017)

Nipah palm largest population is found in Indonesia (700 000 ha), Papua New Guinea (500 000 ha) and Philippines (8 000 ha) (CABI, 2019). In Sarawak, mangrove forest occupying slightly more than 0.09 million hectares of (SFC, 2017). Figure 2.1(a), show the distribution of mangrove forest in Sarawak, as marked in red. Mangrove forest in Sarawak is concentrated within the Kuching, Samarahan, Sarikei and Sibu division as shown in Figure 2.1(b). The actual data for nipah palm population within the mangrove forest in Sarawak is not available. It has yet been explored by the Forest Department of Sarawak due to no requirement for the actual population. Sarawak has nine major type of forest, which is rich and diverse with flora and fauna species, some have yet to be discovered and found. This explained the lack of work done on nipah palm in Sarawak, as priority is given to other flora and fauna in the deep rainforest.

Nipah palm is the most utilised mangrove species. It is the most valued palm for the population living at the growth area. Nipah palm is known as *pokok apong* in Sarawak and *attap palm* in Singapore. The commercial value of nipah palm in Sarawak is still lacking compared to its neighbouring country. Indonesia and Philippines are conducting studies to diversify the usage of nipah palm product. For instance, Indonesia is exploring renewable energy derived from plant to meet their growing demand on energy. Nipah palm sap has been investigated for its potential as bioethanol production (Hidayat, 2015). Ethanol produced from nipah sap was reported to be better than sugarcane, cassava, coconut and potato (Hamilton & Murphy, 1988). Philippines is the third largest area with nipah plantation in Southeast Asia (Rodriguez, 2015). It is reported that a group of enterprising farmers in Philippines has come out with natural sweetener from nipah sap besides its common usage such as roofing material, vinegar (*sukang paombong*) and wine (*laksoy*)

(Peralta, 2018). Kruse (2001) reported the application of nipah particles with coconut as MDF board as shown in Figure 2.2.

Figure 2.2: Nipah particle board

An effort has been made by the Sarawak government in providing livelihood opportunities to the coastal communities by creating *Taman Agro Nipah*, which covered an area of 10 acres at Beting Maro, Betong (Nasri, 2019). This is for the community to commercialise and generate more income from the *gula apong* industry. CRAUN research centre has come out with *gula apong* powder which acts as sweeter with low glysemic index. *Gula apong* and cigarette wrappers are sold in most local market as shown in Figure 2.3(a) and Figure 2.3(b) respectively. Local *kueh* known as *penyaram* uses gula apong as its sweetener as shown in Figure 2.3(c). The leaves of nipah palm specifically its young shoot, has been collected as local dessert wrappers as shown in Figure 2.3(d). Besides that

the locals have been benefiting the nipah palm growth area as a source of income where this is where the locals catch crab, prawn and "*kompon*" to make ends meet. *Kompon* is a fish bait, famous among local anglers. *Gula apong* production has been dwindling recently due to lack of interest of the younger generation to be involved into the long cooking process to produce *gula apong*.

(a) (b)

(c) (d)

Figure 2.3: Nipah-based products (a) Gula apong (b) Cigarette wrappers (c) *Kueh penyaram* (Adibasuf, 2019) (d) *Kueh celorot* (Fazlisyam, 2011)

2.3 Natural fibre

Fibre is a thread or filament from which a vegetable tissue, mineral substance, or textile is formed as defined by the Oxford dictionary. On the basis of diameter and character, fibres are grouped into three different classifications: whiskers, fibres and wires. Whiskers are very thin single crystals that have extremely large length-to-diameter ratios, high degree of crystalline perfection and almost flaw-free. Fibres are either polycrystalline or amorphous and have small diameters. Fibres are stronger than its bulk form, and the reasons for these are their small diameters contain almost no critical defects and fibres can be aligned according to its load direction, therefore, contributing to higher stiffness to the structure (Migliaresi & Pegoretti, 2002). There are two types of fibre, namely, synthetic fibre and natural fibre. Synthetic fibre has been dominating the world of fibre well before the introduction of natural fibre as reinforcement in composites. The examples of synthetic fibres are glass, carbon and aramid. Glass fibres are the most common synthetic fibres used in polymer matrix composites due to its high tensile strength, low cost, good chemical resistance, and good insulation properties. However, glass fibres main disadvantages are low in tensile modulus, relatively high density, sensitive to abrasion, low fatigue resistance and poor adhesion to matrix resins (Migliaresi & Pegoretti, 2002).

Natural fibres originated from plants, animals and minerals. There are six basic types of natural fibres. They are classified as follows: bast fibres (jute, flax, hemp, ramie and kenaf), leaf fibres (abaca, sisal and pineapple), seed/fruit fibres (coir, cotton and kapok), core fibres (kenaf, hemp and jute), grass and reed fibres (wheat, corn and rice) and all other types (wood and roots) (Faruk et al., 2012). Mohanty et al. (2002) classified natural/biofibres as shown Figure 2.1. Based on this classification, nipah palm fits in the

bast fibres, as the fibres are extracted from nipah palm fronds. The natural fibre originating from mineral is asbestos. Natural fibre exhibit a number of excellent properties that enable them to be used as reinforcing agent in composites, and among them are their light weight properties, renewable, naturally available in abundance, high strength, high stiffness, high corrosion resistance and non-toxic, low density, low cost, biological degradability and specific resistance (Athijayamani et al., 2010; Barreto et al., 2010).

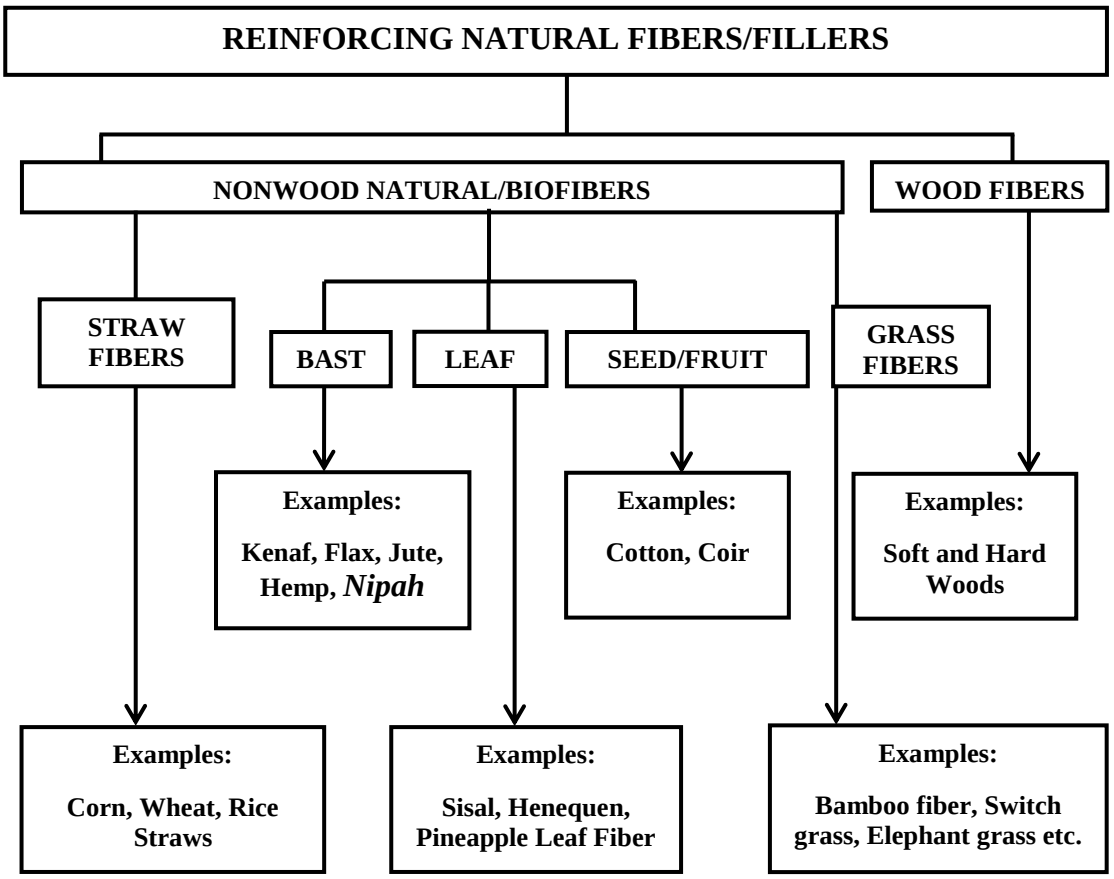


Figure 2.4: Classification of natural/biofibres. (Mohanty et al., 2002)

2.3.1 Natural fibre extraction method

Fibre extraction has been the main subject when it comes to a newly studied fibre. Proper extraction technique is important as any damage done to the fibre will affect the testing at a later stage. Various methods have been employed to extract other natural fibres and the method will be discussed. The microbiological and chemical retting have been in the literatures for natural fibre retting method. Chemical retting involves acids, bases, surfactant and chelators. This process is not sustainable and energy efficient. It was reported that chemical retting produced low quality fibres (Tamburini et al., 2004). Microbiological retting utilising the microbial action degraded the non-cellulosic substance of the plants. The most utilised microbiological retting were water retting and dew retting, and also known as field retting. Water retting process involved soaking of fibre source into water. It could either be a water tank or a pond. The water would penetrate into the bark, allowing the bacteria to degrade the pectic substances. In dew retting, the plants were spread out in the field and attacked by fungi (Liu et al., 2015). Water retting was reported to contribute to major limitation to an efficient and high quality fibre production (Tamburini et al., 2004). This was due to the long water retting process. Long period of field retting for hemp fibre leads to reduction of fibre quality, as this could be due to continuous increase of cellulose degradation (Liu et al., 2015).

Elephant grass as investigated by Rao et al. (2007) employed two methods; (a) retting and manual extraction, and (b) chemical and manual extraction. In method (a), the culms of elephant grass were cut, their base and leaves at the node and end of the culm were trimmed. The culms were then dried for a week and made into strips. The strips were soaked in water for a period of about 10 days. The strips were then beaten gently with a

plastic mallet in order to loosen and separate the fibre, this process is called the mechanical process. The resulting fibre bundle was scrapped with sharp knife and combed until individual fibres were obtained. In method (b), strips of elephant grass were soaked in 0.1-N alkali solution (4 grams of alkali crystal per one litre of water) for different periods up to 72 hours was taken, the optimum period for chemical treatment. The strips were then washed in the water and were subjected to similar mechanical process for separation of the fibres. Prasad et al. (2011) reported the method used to extract this type of grass fibres. The extraction of fibres was done by two methods, namely, (a) retting, and (b) chemical methods, and both were followed by a manual extraction. In the retting method, the grass strips were kept in water for a period of about three to five days, followed by the mechanical process of beating with a plastic mallet. In the chemical method, the strips were soaked in 0.1N alkali for an optimum period of 72 h.

Lai and Mariatti (2008) studied the betel palm frond fibres. The fibres were extracted using a sharp knife to cut according to its texture and woven into composites. A different method was employed by Hassan et al. (2010) where the betel nut were kept in dark room in moist condition for 15 days and separated by hand stripping, kept under the sun, followed by brushing to open and clean the fibres. The fibres from this method of extraction were compounded with extrusion and hot press machine to prepare the composites.

Sapuan et al. (2006) reported the extraction method of banana fibre. The banana fibre was extracted from fully-grown trunks. The trunks were then placed under the sunlight for drying process for about two weeks. After the trunks were dried, they were soaked in the water for another two weeks. After the drying process, the fibres were cut into 10 mm and 15 mm average width and about 137 cm for the longest and 77 cm for the

shortest in length. The fibres were then woven for subsequent composites preparation. Biological retting via fermentation used in extracting banana fibre has satisfactorily obtaining fibres from the plant. This process was reported to be the first to be able to produce hierarchical microstructural ordering present in banana fibre due to less fibre surface destruction (Gañán et al., 2004).

Deshpande et al. (2000) extracted bamboo fibres by using chemical and mechanical processes. Sodium Hydroxide (NaOH) was used to remove the lignin that bound the fibres together. Bamboo strips were soaked in 0.1 N alkali solutions for different periods. 72 h was the optimum hour for soaking. The strips were then washed with water and dried at room temperature for 1 hr and then subjected to the mechanical process for the completion of fibre separation. The mechanical separation process had to be operated to ensure a complete separation of the fibres. The fibres diameters from the chemical process were bigger as compared to the rolled fibre separation. This leads to higher tensile strength of the composites.

Roselle crops (*Hibiscus sabdariffa*) were harvested at the bud stage in order to get good quality fibres. The stalks were tied into bundles and retted in water for three to four days. The retted stem of the roselle plant was washed in running water. The fibres were then removed from the stem, cleaned and dried in the sunlight (Thiruchitrambalam et al., 2010). Other method reported for roselle fibre extraction is by using drive-power decorticating machine (Nadlene et al., 2016). It is also reported that water retted roselle fibres were found to be smoother and finer compared to machine extraction.

For sisal fibres, they were extracted from their leaves by a mechanical process called decortication. In this process, the leaves were crushed by a rotating wheel with blunt

knives, so that only the fibres remained. The decorticated fibres were washed before dried in the sun, proper drying condition is important as fibre quality depends on the reduced moisture content of the fibre (De Andrade Silva et al., 2008).

Coir fibre was extracted from the mesocarp, which constituted about 25% of the nut. Coir fibre could be either extracted from their husk or from the green nut. The green nut had to be kept under water for two to four months (Satyanarayana et al., 2007). This method is more time consuming as compared to if machine are to be used. The industries in Brazil were using German technology to extract fibres and were able to generate 75 tons of fibres per month. The demand of these fibres was from Mercedes Benz Company.

Satyanarayana et al. (2007) reported that sisal fibre was one of the most widely used natural fibres and was very easily cultivated. Each leaf contained 700 to 1400 technical fibres with the length of 50 cm to 100 cm, with horse shoe cross-section. Kenya and Tanzania are the second and third world sisal producers. The fibres were extracted using a primitive machine popularly known as “periquita”. Other method used to extract sisal fibres were the retting method and mechanical method by using decorticators. Mechanical process was reported to produce better fibre quality compared to retting process (Li et al., 2000).

Piassava fibres were extracted manually from leaf stalks. The stalks were picked once a year, for a longer and more flexible fibre of better commercial value and to preserve the longevity of the plants. The fibres were extracted using long knives, then the stalks were pierced to the fibres, which were then cleaned, brushed, sorted out on the basis of length and tied into bales with varied weights (D’Almeida et al., 2006).

Jute fibres were traditionally extracted using the stem retting technique. The jute bundles are immersed in clear slow flowing water in canal, tanks, ponds or ditches. The ratio of water to jute was 1:20, the bundles were kept under water for 15 to 20 days. Once the retting was completed, the fibres were extracted, washed and dried for sale (Ali et al., 2015). The recent interests of the people nowadays are towards products that are environment-friendly. This has prompted the needs to search for a jute fibre extraction method that is friendly towards the environment (Biswas et al., 2019). This research proposed a plant that will produce jute fibre in a sustainable method. Jute fibre are now extracted using jute decorticator machine meant for large production, these machines are available for retail (Alibaba, 2019).

The literature review based on the extraction method for other established natural fibre conclude that, water retting and decorticator machine are normally the method used to investigate the fibre extraction process. For instance, jute and coir were water retted before the introduction of decorticator machine. As years gone by, due to the needs of producing more jute and to speed up the extraction process, decorticator are widely used. Water retting are known to create the issue with water contamination due to massive water retting amount from its decomposed mass. The lack of space for water retting also leads to the introduction of decorticator machine. When decorticator machine is easily available, they are used to extract other types of fibre such as sisal, piassava, roselle and others. It is interesting to note that extraction process depends greatly on the source of the fibre, for instance agro waste such as oil palm and sugarcane bagasse are more straight forward process. As these crop wastes are collected from the crop field. They are cleaned, boiled and dried before they can be used as reinforcing fibre.

The extraction method used affects the quality of the fibres, water retted fibres are known to produce smoother fibres compared to the decorticated fibres (Haameem et al., 2015). The mechanical process introduced surface defects to the fibres. The optimum period for water retting varies for each type of plant. For example, jute was retted for 15-20 days (Ali et al., 2015), roselle was retted for 3-4 days (Thiruchitrambalam et al., 2010) and coir was retted for 4 months (Tomczak et al., 2007). Investigation on flax water retting in distilled water, reported that the duration of 6 days is sufficient to produce reasonable fibre properties (Ruan et al., 2015). The optimum duration of water retting varies for each fibre types as the source of the fibre whether they are of the bast fibre, straw fibre, grass fibre, seed fibre or the leaf fibre. Thicker fibre source requires more time. The water retting duration are affected by the concentration of microorganism existence in the pond or canal.

Based on the literature review on fibre extraction, it is significant to extract nipah palm fibre by using a primitive method before engaging a more industrialized method. Pond is vastly available within Kuching, Sarawak. Secondly the amount to be water retted is small so water contamination is not an issue to the ecosystem. It is predicted that, the water retting condition for nipah would require more time in the pond, as the fibre source is from the whole fronds. Nipah fibre source is considered the biggest compared to other types of natural fibres, its frond diameter can get as big as 15 cm especially the lower frond.

2.4 Fabrication method for polyester matrix composites reinforced with natural fibre

The fabrication method for natural fibre composite depends greatly on temperature sensitivity and hydrophilic nature of the natural fibre (Summerscales & Grove, 2013). The common method for natural fibre composites are injection molding (Ferreira et al., 2010; Jayaraman, 2003; Rahman et al., 2010), compression moulding (Haameem et al., 2016; John et al., 2010; Prasad et al., 2017; Rasidi et al., 2014), pultrusion (Akil et al., 2014), vacuum bagging (Lai & Mariatti, 2008; Mariatti, Jannah, Bakar & Khalil, 2008; Nadlene et al., 2016), hand lay-up (Alavudeen et al., 2015; Davallo et al., 2010; Dhakal et al., 2007; Júnior et al., 2015; Khanam et al., 2011; Srinivasababu et al., 2009), resin transfer moulding (Kabir et al., 2012; Rassmann et al., 2011) and filament winding. The hand lay-up method had been widely applied to fabricate polyester composites. This method is mostly used in research work due to the simple set up especially when equipment in the laboratory is limited. Hand lay-up method was used to fabricate the buriti fibre on silicone mould and cured for 24 hours (Monteiro et al., 2014). A combination of hand lay-up method and compression moulding method had been used to fabricate the natural fibre reinforced polyester composites as shown in Figure 2.5 (Boynard & D'Almeida, 2000; Dhakal, Zhang & Richardson, 2007; Haameem et al., 2016). Hand lay-up method and compression moulding is chosen as the method used to fabricate nipah palm fibre composite as this equipment is readily available in the laboratory.

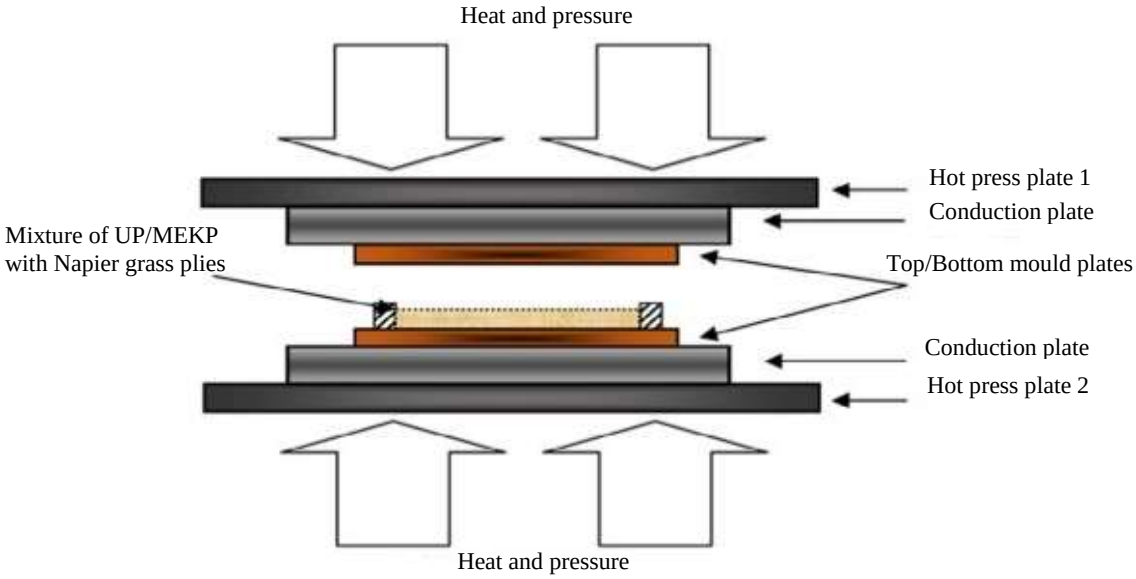


Figure 2.5: Compression moulding method (Haameem et al., 2016)

2.5 Modification of fibre surface for properties enhancement

The surface modifications of natural fibres are crucial to improve the compatibility of natural fibre with its matrices. Natural fibre is hydrophilic while polymeric resin is hydrophobic, the incompatibility between these two opens up the opportunity of studying the surface modification of cellulose fibres. The presence of water and hydroxyl group in natural fibre and high water and moisture absorption leads to poor mechanical properties (Mwaikambo, 2008). However, all these limiting factors can be improved by treating the cellulosic fibres with the appropriate chemicals. There are various common chemical treatment for natural fibres such as alkali, silane, acetylation and others.

2.5.1 Alkali treatment

Alkali treatment has been widely used to improve the properties of cellulosic fibre. The purpose of alkali treatment is to remove the lignin, pectin, waxy substances and natural oils covering the external surface of the fibre cell walls as shown in Figure 2.6 (Mwaikambo & Ansell, 2002). This will subsequently produce a closely packed cellulose chain which consequently will improve the mechanical properties of the fibre (Bledzki & Gassan, 1999).

Alkali treatment also breaks the hydrogen bonds and increases the number of free hydroxyl group (Ridzuan et al., 2016). The chemical reaction is as in Equation 2.1.

$$\text{Fibre} - OH + NaOH \text{ ---------- } \rightarrow \text{ Fibre} - O^{-} Na^{+} + H_2O \qquad \text{Equation 2.1}$$

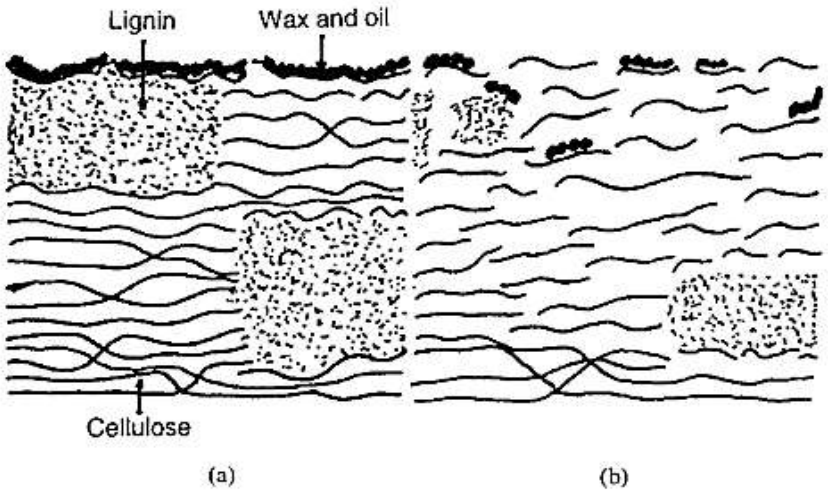


Figure 2.6: Typical structure of (a) Untreated and (b) Alkalised cellulosic fibre (Mwaikambo & Ansell, 2002)

Alkali treatment is one of the standard procedures in the pulp and paper industries for lignin removal. Alkali treatment is known to increase the surface roughness and decrease the moisture content (Haameem et al., 2016). Alkalisation process results in fibrillation that breaks the fibre bundles into smaller bundles, increasing the effective surface area of the fibre strands, therefore, improving the surface contact between fibre and the matrix (Haameem et al., 2016). Various percentages of alkali treatment had been used to study the effects of alkalisation towards natural fibres. A study on napier grass with 5%, 10%, 15% and 20% were conducted. Fibre fibrillation was observed for 5% and 10% alkali treated fibres. The structures of the fibre deteriorated and the fibre bundles were split into ultimate fibrils. This was said to enhance the tensile strength as larger amount of stress could propagate through the fibres owing to the larger surface area for energy dissipation. However, at a higher alkali concentration, the fibrillation was much more aggressive causing the cavities to develop within the fibrils, resulting in insufficient energy transfer. A higher alkali concentration and a longer exposure caused the fibres to become brittle (Hossain et al., 2011). It was reported that the highest tensile strength for napier fibres was treated with a 10% alkali treatment (Haameem et al., 2016).

Alkali treated fibre had its lumen almost disappeared which may have been the consequences of fibres swelling or the lumen collapsing in the alkali solution. Swelling was thought to be the result from the incorporation of sodium ions into the cellulose structure which broke the bonds among cellulose sheets (John & Anandjiwala, 2008). Alkali treatment caused a significant shrinkage and the weight loss increased with the increasing alkali concentration. This was reported to be the result of stripping of the lignin and hemicelluloses from the fibres in alkali solution (Cai et al, 2016). Treated fibres surface with alkali would roughen the surface, increase the number of interaction sites and

subsequently improve the fibre-matrix interlocking and its mechanical properties (Osorio et al., 2012).

Alkali treatment conducted on sugarcane bagasse revealed that 5 wt% alkali showed better lignin and hemicellulose removal and producing uniform fibres (Hossain et al., 2014). Mwaikambo and Ansell (2006) stated that 5 wt% of alkali was mild enough to dissolve all lignin and pectin. Oushabi et al. (2017) also reported that 5 wt% alkali was the optimal alkali concentration in terms of bond characteristics for date palm fibre. However, a 10% alkali treated napier grass demonstrated the highest tensile strength. Napier grass was soaked for 30 minutes in the solution (Haameem et al., 2016). The literature provides various soaking duration of fibres in alkali solution. Suizu et al. (2009) reported the alkali treatment for ramie fibre was conducted with 15 wt% alkali for two hours. Oil palm fibres were dipped in 5% alkali for 48 hours (Sreekala et al., 1997). Betel nut fibres were soaked at 6% alkali for three hours (Lai & Mariatti, 2008). The high alkali concentration will lead to delignification of fibres, which will affect its tensile strength (De Rosa et al., 2011).

The alkali treatment has two major effects towards natural fibre, that is to increase possible reaction sites by exposing more cellulose and to increase surface roughness for better mechanical interlocking between fibres and matrices (Valadez-Gonzalez et al., 1999). The rough surface of fibres will facilitate the mechanical interlocking with the resin-matrices resulting in enhanced interface as well as improving packing order of crystalline region (Mwaikambo & Ansell, 2006). Surface roughening will increase the area providing larger contact area for fibre-matrix interlocking. Alkali-treated fibres depicted compressed cellular structure specifically the lumen. This leads to reduced void content and less water absorption capacity of the treated fibres (Cai et al., 2015; Reddy et al., 2012; Ridzuan et al., 2016). Lumen disappearance was observed in alkali-treated abaca fibre.

This was due to its fibre swelling and collapsing of lumen in alkali solution (Cai et al., 2016). The fibre swelling was due to sodium ion deposited on the cellulose structure during alkali treatment (John & Anandjiwala, 2008). It was reported by Ridzuan et al. (2016) that alkali treatment had produced smaller and more uniform diameter of *Pennisetum purpureum* fibre.

2.5.2 Silane treatment

Silane works as a coupling agent which bonds organic and inorganic materials. In this context, it is to bond natural fibre and polymer matrices. Trialkoxysilanes are the most established silane used for natural fibre polymer composites. The organofunctionality of the silane interacts with the polymer matrices with their interaction modes depending on the functionality's reactivity or compatibility towards the polymer. A non-reactive alkyl group of the silane may increase the compatibility with non-polar matrix due to their similar polarities. The most reported silanes and their applied target polymer matrices are in Table 2.1.

Table 2.1: Silanes used for the natural fibre/polymer composites: chemical structures, organofunctionalities and target polymer matrices. (Xie et al., 2010)

Structure	Functionality	Abbreviation	Target matrix
$(RO)_3Si\text{-}(CH_2)_3\text{-}NH_2$	Amino	APS	Epoxy Polyethylene Butyl rubber Polyacrylate PVC
$(RO)_3Si\text{-}CH{=}CH_2$	Vinyl	VTS	Polyethylene Polypropylene Polyacrylate
$(RO)_3Si\text{-}(CH_2)_3\text{-}OOC(CH_3)C{=}CH_2$	Methacryl	MPS	Polyethylene polyester
$(RO)_3Si\text{-}(CH_2)_3\text{-}SH$	Mercapto	MRPS	Natural rubber PVC
$(RO)_3Si\text{-}(CH_2)_3\text{-}O\text{-}CH_2CHCH_2O$	Glycidoxy	GPS	Epoxy Butyl rubber Polysulfide
$R_2\text{-}Si\text{-}Cl_2$	Chlorine	DCS	Polyethylene PVC
VTS grafted plastic	Vinyl	VSPP VSPE	Polypropylene polyethylene
$(RO)_3\text{-}Si\text{-}R''\text{-}N_3$[b]	Azide	ATS	Polypropylene Polyethylene Polystyrene
$(RO)_3\text{-}Si\text{-}(CH2)_{15}CH_3$	Alkyl	HDS	Polyethylene Natural rubber

[a] R = -methyl or ethyl

[b] R" = $-C_6H_4\text{-}SO_2-$

The process involves several stages of hydrolysis, condensation and bond formation during the treatment process. Silanols form in the presence of moisture and hydrolysable alkoxy groups. During condensation process, one end of silanol reacts with the cellulose hydroxyl group and other end reacts (bond formation) with the matrix (Si-Matrix) functional group (Kabir et al., 2012). This process also provides the hydrocarbon chain that restrains the fibre swelling into the matrix. Silane provides the surface coating for natural fibres, it penetrates into pores and provide better tensile strength to the composite (Valadez et al., 1999a). Treatment of 2% silane for 3 hour shows on kenaf and pineapple leaf treated fibres shows tensile strength enhancement. Silane treatment on hemp fibre shows improvement in the tensile strength. Hemp treated with 1% treatment shows 15% and 10% higher than that of 5% alkali and untreated hemp fibre respectively (Sepe et al., 2018).

2.5.3 Acetylation treatment

Acetylation treatment involves the incorporation of acetyl function group into natural fibres. Acetylation treatment is esterification method for plasticizing of natural fibre. Pre-treatment with alkali is necessary to enable effective reactions with chemicals (Nadlene et al., 2016). The hydrophilic hydroxyl group (OH) of the fibre reacts with acetyl group (CH_3CO) to take out the moisture. This decreases the hydrophilic nature of the fibre. Acetylation provides rough fibre surface with less number of void content, that gives mechanical interlocking to the composites (Tserki et al., 2005).

2.6 Analysis of thermal properties

The effect of alkali on natural fibre is on the dissolution of amorphous parts of natural fibre. Each natural fibre reacted differently based on the concentration of the alkali, soaking time and the type of fibre. The analysis of thermal properties of untreated and treated natural fibres showed differences to be explored and knowledge to be acquired. The thermographic analysis conducted on piassava showed a low temperature degradation between 75 °C to 85 °C. The low temperature weight loss for piassava was 5.18%. A similar low temperature weight loss were reported for luffa (5%) (Boynard & D'Almeida, 2000), jute (10.2%), flax (6.3%), wheat straw (7.3%), α-cellulose (7.6%) and lignin (5.8%) (D'Almeida et al., 2006b). Thermal degradation for piassava started at 225 °C (D'Almeida et al., 2006a). The first stage of thermal degradation observed for napier grass was at 250 °C. At this stage, it was reported that napier grass had undergone moisture vapourisation and the crystalline temperature was at 320 °C (Haameem et al., 2016).

Differential Scanning Analysis (DSC) is conducted to study the effect of alkali treatment towards the glass transition temperature (T_g) and its melting point (T_m). The first heating scan in DSC was endothermic peak related to hydration of water, the second heating scan was related to decomposition of hemicellulose and lignin, and the third heating scan was the high-temperature degradation (Haameem et al., 2016). Studies done on bamboo fibre/polylactic acid composites showed a change in T_c which demonstrated that treated composites have a strong capability of cold crystallisation, which might be related to the removal of lignin, hemicellulose and other impurities (Lin et al., 2018). The glass transition temperature for untreated napier grass was reported at 66.6 °C (Haameem et al., 2016).

Treated fibres are subjected to FTIR analysis to confirm the chemical composition changes due to the effect of alkali treatment. FTIR studies suggested the reduction of hemicellulose content after alkali treatment of fibres. FTIR conducted to native napier grass resulted in the following bands, 1727 cm^{-1} and 1260 cm^{-1} corresponded to hemicellulose, the bands at around 3430 cm^{-1} and 2923 cm^{-1} corresponded to α-cellulose (Reddy et al., 2012). The disappearance of two peaks, 1742 cm^{-1} and 1242 cm^{-1} after the alkali treatment of abaca fibres indicated that either the carboxylic acid an acetyl groups were destroyed by the alkali treatment (Cai et al., 2016). Alkali treatment had removed acid, lignin and other natural fibre constituents from the fibre (Sain & Panthapulakkal, 2006; Valadez-Gonzalez et al., 1999). Treated betel palm fibres had broad intense peak at 3313.62 cm^{-1}, which indicated more –OH group existed during the alkali treatment. Peak around 1730 cm^{-1} referred to the ester and ether crosslink between cellulose and lignin or cellulose and hemicelluloses (Lai & Mariatti, 2008). The O–H bond was found to decrease by the increase of the alkali concentration. This was due to the hydroxyl group involvement in the hydrogen bonding with the carboxyl groups of fatty acids on the fibre surface. The range of peaks at 1000 cm^{-1} and 1500 cm^{-1} showed the presence of hemicellulose after the treatment (Mwaikambo & Ansell, 2002). The hydrolysis of hemicellulose in alkali was due to the breaking of C–O–C bonds between monomers. This indicated that hemicellulose is easily removed compared to lignin (Oushabi et al., 2017). According to Spinacé et al. (2009) on curaua fibres component, the intense absorptions of curaua fibres were in the range of 3500 cm^{-1} to 3200 cm^{-1} related to the O–H stretching and hydrogen bonds, 2900 cm^{-1} - 2750 cm^{-1} related to the C–H stretching in saturated hydrocarbons, 1740 cm^{-1} related to C=O stretching of acetyl or carboxylic ester and 1240

cm^{-1} related to CH angular deformation. The curaua fibres components are mainly cellulose, hemicellulose and lignin.

2.7 Analysis of chemical properties

The chemical compositions of lignocellulosic fibres are cellulose, hemicellulose, lignin, pectin, ash and moisture content. Generally, fibres contain 73.6% cellulose, 9.9% hemicellulose, 7.5% lignin and 0.9% ash (Tomczak et al., 2007). Table 2.2 shows the chemical composition of lignocellulosic fibres in Brazil and other countries. These compositions vary from each fibre, the properties of each constituent contribute to the overall properties of the fibre. Hemicellulose is responsible for the biodegradation, moisture absorption and thermal degradation of the fibre as it shows least resistance, whereas lignin is thermally stable but is responsible for the UV degradation (Saheb & Jog, 1999). Lignin also plays the role of binding the fibres of cellulose (Deshpande et al., 2000). The chemical composition analysis done on various parts of nipah palm showed the hemicellulose content is the highest in fronds at 25.7 wt% and the lowest in husk at 21 wt % (Phaiboonsilpa et al., 2011) as shown in Table 2.3 and Table 2.4. Nipah chemical composition was reported to be similar among various parts of palm oil (Shibata et al., 2008). Studies conducted on napier grass showed that lignin composition increases when the alkali concentration increases (Reddy et al., 2012).

Table 2.2: Chemical composition of lignocellulosic fibres (Satyanarayana et al., 2007)

Fiber		α-Cellulose (%)	Hemi cellulose (%)	Lignin (%)	Ash (%)	Extracts (%)
Bagasse (sugarcane)	B	54.3-55.2	16.8-29.7	25.3-24.3	1.1	0.7-3.5[@]
	O	33-44	27-32	19-24	4.26	
Bamboo	O	33-45	30[&]	20-25		
Banana	O	60-65	6-8	5-10	1.2	
Jute	B	60	22.1	15.9	1.0	
	O	59-71	12-13	11.8-12.9	0.7	0.5-2
Ramie	O	80-85	3-4	0.5		64
Piacava	B	31.6		48.4		
Rice straw	O	51-70		12-16	15-20	9-14[*]
Curaua	B	70.7-73.6	21.1	7.5-11.1	0.79-0.9	2.5-2.8[#]
Pineapple	B	83		12		
	O	80		12	0.1-1	4
Sisal	B	74-75.2	10-13.9	7.6-7.98		
	O	60.67	10-15	8-12	0.14-0.87	1.7-6.0
Coir	B	43.4-53	14.7	38.3-40.77		3.5
	O	43.77	<1	45		4.5
Cotton Lint	O	90	6			0.4
Luffa cylindrica	B	62	20	11.2	0.4	3.1

O – others, B – Brazilian; # - from unpublished work; * - silica; & - pentosans; @ - water-free sample

Table 2.3: Chemical composition of nipah palm parts (Tamunaidu & Saka, 2011)

Parts	Cellulose[a]	Hemicellulose[b]	Lignin		Starch	Protein	Extractives[c]	Ash	Total
			Klason	Acid-soluble					
Frond	35.1	26.4	17.8	1.9	0.3	1.9	1.9	11.7	97.0
Shell	45.6	23.5	17.3	2.1	n.d.	0.8	1.3	8.2	98.8
Husk	36.5	21.8	27.3	1.5	0.1	1.9	0.8	8.1	98.0
Leaf	28.9	23.6	32.0	1.8	2.8	3.0	2.3	5.1	99.5

n.d. not detected

[a] cellulose = α-Cellulose

[b] Hemicellulose = Holocellulose – (α-Cellulose)

[c] Extraction was done for 24 h using acetone.

Table 2.4: Chemical composition of nipah frond. Percentage data based on weight of oven-dried raw material[a] (Phaiboonsilpa et al., 2011)

Component	Yield (%)
Hemicellulose	18.9
Cellulose	25.6
Lignin	23.7
Protein	2.4
Starch	0.8
Extractives in	
EtOH/benzene	1.8
Hot water	26.0
Inorganics	2.9
Total	102.1

[a]Inorganic constituents in the material extracted by EtOH/benzene and hot water

2.8 Mechanical testing of natural fibre

The strength of natural fibre is an important factor when these fibres are to be used as reinforcing agent in composites. The methods for single fibre testing are normally developed based on the fibre diameter, size and length.

2.8.1 Single fibre test

Single fibre test is conducted to determine the strength of the natural fibre. The typical single fibre test method is by using a cut-out paper or thin plastic tab to mount the fibre. An individual fibre will be glued at both ends of the tab with adhesive. The tab will be cut off at the mid gauge area once tab is gripped to tensile machine. Literatures provided the tab mounted method for single fibre tensile test, with each fibre were mounted onto a thick paper. The single fibre test was conducted on snake grass fibre according to ASTM D3379-75 standards using the Instron Universal Testing machine as shown in Figure 2.7. The gauge length of each fibre was taken as 100 mm and a 1000 g load cell was used for the testing. The crosshead speed was 5 mm/min (Sathishkumar et al., 2012).

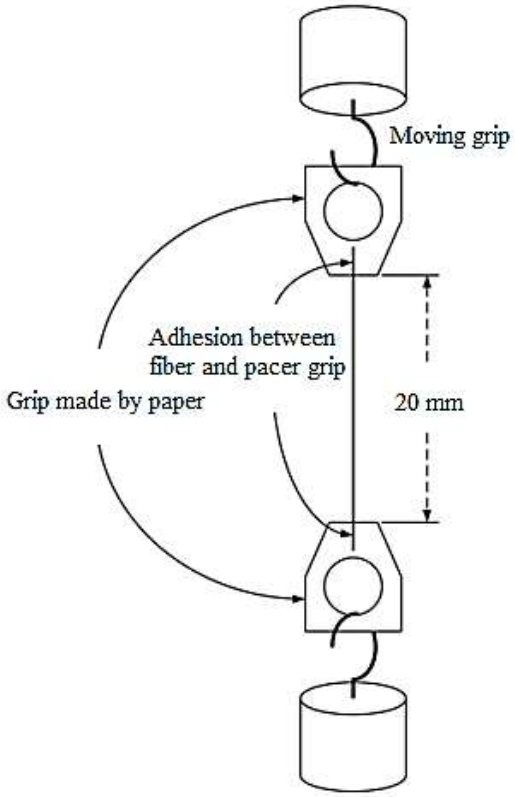


Figure 2.7: Single fibre set up for snake fibre (Lee et al., 2009)

Another method was used by Lee et al. (2009) to test the single fibre properties with a specific grip method. Small load cell (20 g) was used for the single fibre testing, and a grip made of thin paper was designed with a hole which can be attached to the upper hook in the tensile machine. The single fibre was glued to the paper in the grip and then stretched at the rate of 10 mm/m. Lee et al. (2009) also reported that the mechanical properties of natural fibres were more broadly scattered than those synthetic fibres. Natural fibres are grown naturally, therefore, their properties can vary immensely from plant to plant. It is reported that the non-linear pattern of tensile strength of the treated fibres of abaca fibres were due to the twisted fibre above 7 wt % alkali treatment (Cai et al., 2015).

2.8.2 Natural fibre composite strength

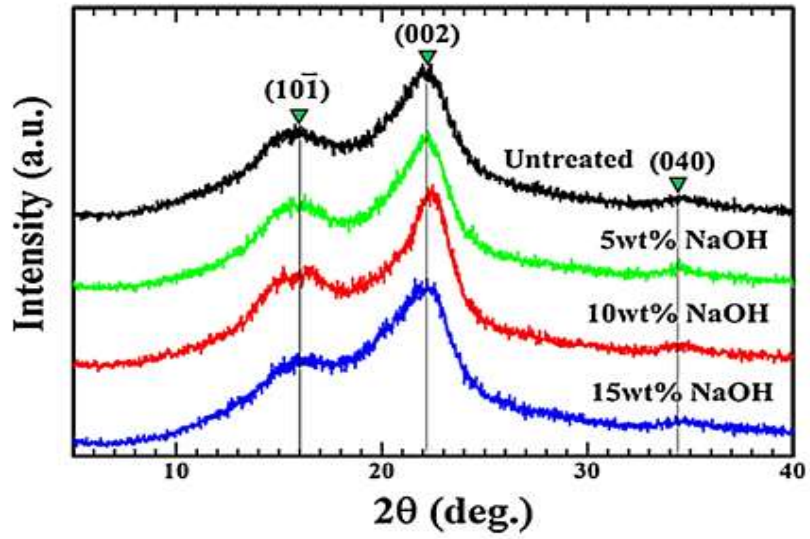
The mechanical strength of composites varies with its loading orientation. Studies conducted on long and short sisal fibres, for randomly oriented and continuously aligned long fibre showed that the latter is suitable for high strength and stiffness in a single direction. Short and randomly oriented fibres are suitable for isotropic composite properties (De Rosa et al., 2011; Joseph et al., 2002). Studies on newly introduced natural fibre, napier grass, treated with 10% alkali revealed a high tensile strength of above 80%. Napier grass composites with 25% volume fraction yielded optimum tensile and flexural strength, recorded at 18.17 MPa, 2.00 GPa respectively for long fibre and 15.64 MPa, 3.02 GPa for short fibre respectively. The tensile strength of pure polyester used in the study was 10.1 MPa (Haameem et al., 2016). Alkali treated bamboo fibres as compared to the untreated fibres, had increased its strain at break and its ductility. However, its density, yield strength, ultimate tensile strength and Young's modulus value were lowered. The tensile strength of other natural fibre reinforced polymer composites varied as the fibre diameters were not the same. It is worth to note that the important comparison that has to be made includes the fibre diameter. Studies on sisal fibre reported that the Young's modulus value and the tensile strength of composites depended on its internal structure such as cellulose content, crystallinity index and micro-fibril angle (Mwaikambo & Ansell, 2006). It was reported by D'Almeida et al. (2006a) and Nascimento et al. (2010) that tensile strength and Young's modulus value of piassava fibres reinforced epoxy composite failed to reinforced the epoxy composites up to 30% of volume fraction. The value improved higher than pure epoxy at 40% of volume fraction. Polyester composite reinforced with sponge gourd (*Luffa cylindrical*) reported low mechanical properties,

strength value of 21.3 MPa and did not improve the mechanical behaviour of the pure polyester resin. It was reported that there was a change in the failure mode of composite when luffa was introduced (Boynard & D'Almeida, 2000). Coir fibre has to be loaded up to 45% or even higher in order to obtain a significant reinforcing effect when tested in tension. However, its flexural strength showed no improvement even in a higher loading condition. Lack of efficient reinforcement is due to low modulus of elasticity as compared to pure polyester (Monteiro et al., 2008). It was reported that the tensile strength were 16 MPa (untreated) and 23 MPa (treated) of sisal fibre reinforced polyester composites failed to improve the strength of pure polyester, around 38 MPa (Melkamu et al., 2018).

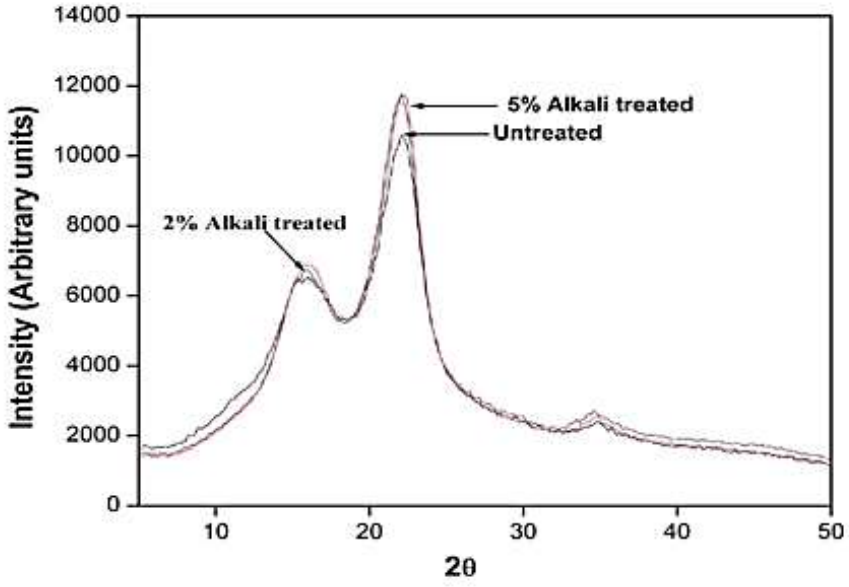
2.9 Crystallinity of natural fibre

The crystallinity index of natural fibre is contributed by its cellulose content. Cellulose consists of amorphous and crystalline regions. The crystallinity is affected by the presence of cellulose and non-cellulosic constituents in natural fibre (Reddy & Yang, 2005). Treated natural fibre are subjected to realignment of it structure due to the removal of the amorphous region. The realignment of cellulose structure increased its crystallinity percentage and thus enhancing the tensile properties (Haameem et al., 2016). The crystallinity index calculation was made by referring to Segal et al. (1959). The impact of raw and alkali treatment natural fibre was studied by using XRD to measure the degree of crystallinity. The XRD spectra of treated abaca fibre showed three main peaks around 15.7°, 22.2° and 34.5°, and crystallinity may be increased by increasing the alkali concentration as shown in Figure 2.8(a) (Cai et al., 2015). The degree of crystallinity of alkali treated napier grass was slightly higher than the untreated fibre because of the

removal of amorphous hemicellulose. The degrees of crystallinity for the untreated, 2% and 5% alkali-treated fibres were found to be 38.3%, 42.7% and 42% respectively as shown in Figure 2.8 (b) (Reddy et al., 2009).



(a)



(b)

Figure 2.8: X-ray diffractogram (a) Abaca (Cai et al., 2015) (b) Napier grass (Reddy et al., 2009)

2.10 Analysis on water absorption impact towards natural fibre composites

The water absorption test is the significant parameter when the composite has the potential of outdoor application. Water absorption leads to swelling of materials, dimensional changes, rigidity, stiffness and strength reduction (Mittal & Chaudhary, 2018). Moisture absorption leads to swelling due to the breaking of hydrogen bonds and the hydroxyl group form new hydrogen bond with water molecules (Kabir et al., 2007). Water absorption characteristics depend on the type of fibre, fibre orientation, surface exposure of fibre, permeability of fibre, temperature, humidity, viscosity of matrix, hydrophilicity behaviour and void content (Akil et al., 2011; Jawaid et al., 2011; Nosbi et al., 2010). It is also reported that the rate of water absorption increases when the fibre content increases (Melkamu et al., 2018). This finding is due to the increased amount of fibre and naturally this leads to more water absorbed. It is reported that water absorption of napier grass composites significantly impairs the mechanical properties (Haameem et al., 2016). Water absorption has two patterns; first step is absorption up to its saturation level due to capillary effect. Second step is the reduction of absorption due to hydro-elastic property of fibre (Dhakal et al., 2007). Studies done by Melkamu et al. (2018) reported on the reduction of water absorption rate of sisal fibre after the alkali treatment. It was suggested to be caused by the formation of outer surface layer that resisted the water absorption. Alkali treatment of other fibre also shows improvement in terms of good moisture resistance compared to untreated fibres (Haameem et al., 2016; Ramadevi et al., 2012; Wang et al., 2008). Based on the extensive work on alkali treatment to other fibre, it is expected that alkali treatment towards nipah palm fibre would have significant impact on the water absorption.

2.11 Application of natural fibres in various automotive component

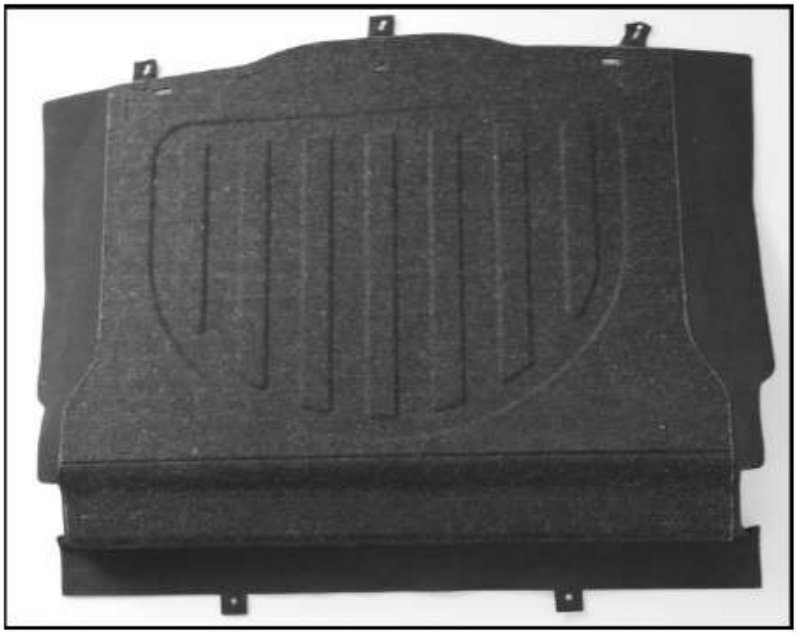
(a)

(b)

(c)

Figure 2.9: Automotive components (Behlouli, 2014; Holbery & Houston, 2006) (a) 50 Mercedes- Benz E-Class components (b) A front-end grill opening (Ford Montageträger) (c) The underbody of a Daimler Chrysler.

(d)



(e)

Figure 2.9: (d) NF composite door panel (e) KIA spare wheel cover (continued**)**

(f)

(g)

Figure 2.9: (f) Wheel arches for Opel Insigna (g) An example of a headliner (continued)

The automotive production rate throughout the world is expected to increase up to 76 million cars in 2020. This will lead to price hikes of petroleum-based product due to the depletion of petroleum reserves. It is reported that the reduction of car weight by 25% is equivalent of saving 250 million barrels of crude oil (Davoodi et al., 2010). Automotive manufacturers are looking for a replacement for the heavy petroleum based materials of the

automotive parts by using light biomaterials. This can be achieved by using natural fibre composite as the panels. Natural fibre composites have experienced rapid growth over the last decade in the European automotive market. In terms of recyclability of automotive parts, manufacturers are replacing glass fibres with natural fibres. However some consideration such heat resistance and loading requirement have to be taken into account for the natural fibre to be used as automotive parts.

There are a few natural fibres that have been used for automotive applications. Jute was used in the 90s as Mercedes-Benz door panels. Flax has been used in German automotive industry, followed by hemp (Fogorasi & Barbu, 2017). Flax, hemp, sisal, wool and other natural fibres were used to make fifty Mercedes Benz E-Class components as shown in Figure 2.9(a). They were used to make the vehicle interior parts such as glove box, door panels, seat coverings, sear surface/backrest, trunk panel, trunk floor and insulation, and the exterior part, namely the floor panels (Holbery & Houston, 2006). Natural fibres were also utilised by Toyota in their models (Crown, Camry, Mark X), using the natural fibre-polypropylene non-woven mat for their parcel shelves (Figure 2(b)). Other than that, they were used as front end grille of FORD (Figure 2(c)), door panels (Eco Technilin SAS) as shown in Figure 2(d), instrument panels (SMART Fortwo vehicle) using 50 wt% flax and 50 wt% PP and for spare part wheel cover by PSA, Toyota and KIA (flax-PP) as shown in Figure 2(e), trunk area of Opel Figure 2(f), parcel shelves or headliners previously made with polyester non-woven mats, as shown in Figure 2(g) (Behlouli, 2014). Recent work done on the hybrid of kenaf/glass epoxy composites in a passenger car bumper beam shows some mechanical property advantage compared to the common bumper beam material even though the impact properties still need improvement (Davoodi et al., 2010).

The contribution of natural fibre composite in developing sustainable automotive industry is increasingly getting more attention from researchers. There are still a lot of investigations need to be done in selecting suitable natural fibre for specific automotive component and also to make sure that the performance of the new component is comparable to petroleum-based product while benefiting the light weight criteria of natural fibre composite.

2.12 Chapter summary

Natural fibre has been the topic of interest in replacing glass fibre and petroleum-based fibre especially in the automotive sector. The abundance of nipah palm along the Sarawak river banks has open up the opportunity to investigate the potential of its fibre as reinforcing agent in polymer composites. In Sarawak, nipah palm sugar is the main product that is obtained from nipah palm besides cigarette and local dessert wrappers. Other effort done by the Sarawak government is to increase the trade value of nipah palm sugar to improve the livelihood of its people.

Natural fibre such as jute, hemp, flax and kenaf has been investigated extensively in improving its performance as reinforcing agent. Various treatment processes are available in the literature such as alkali treatment, silane treatment, acetylation treatment and many more. Alkali treatment is the most common and oldest method used as chemical treatment for natural fibre. Alkali treatment has proven its effectiveness in improving fibre and matrix adhesian and thus enhancing the tensile properties as well as its thermal degradation and water absorption resistance.

Numerous researches on natural fibre have gained the interest to investigate new potential of natural fibres for reinforcement in polymer composites. This is the pioneer research work done on nipah palm as reinforcing agent. There is no other work found in the literature on the investigation of nipah palm fibre as reinforcing agent. All the literatures on natural fibre were investigating the other new fibre possibility and also the improvement of other established natural fibre. The literature review done shows the research gap of nipah palm fibre as reinforcing agent. Therefore this investigation was done is to closed the gap and to provide new knowledge to the natural fibre family.

CHAPTER 3

MATERIALS AND METHODS

3.1 Overview

This chapter explains in detail how the raw materials are prepared. It includes the methods involved in fibre extraction from nipah fronds, fibre preparation for characterisation analysis, composites sample preparation, mechanical testing and surface morphology of the samples.

3.2 Preparation of raw materials

Figure 3.1: Nipah palm (arrow indicated the fronds of the nipah)

(a) (b)

Figure 3.2: (a) Fresh Nipah fronds (b) Nipah fibres from fresh frond

The source of fibres for this research is from nipah palm as shown in Figure 3.1. Nipah palm were collected along Sungai Kuap. At the beginning of the initial investigation of the leaves and fronds, the fronds as shown in Figure 3(a), were found to contain the desired fibres as shown in the cross-section in Figure 3.2(b). Nipah fronds are found to have two distinctive parts, the lower fronds and the upper fronds. The lower fronds are the submerged fronds during high tide, and it has a spongy structure inside, surrounded by hard and waxy dark brown bark. The upper fronds contain more rigid bundles of fibres as compared to the lower fronds as they are less spongy.

3.2.1 Fibre extraction

Nipah palm fibres were extracted using two methods, namely dry extraction and wet extraction. The flow chart of the process is in Figure 3.3.

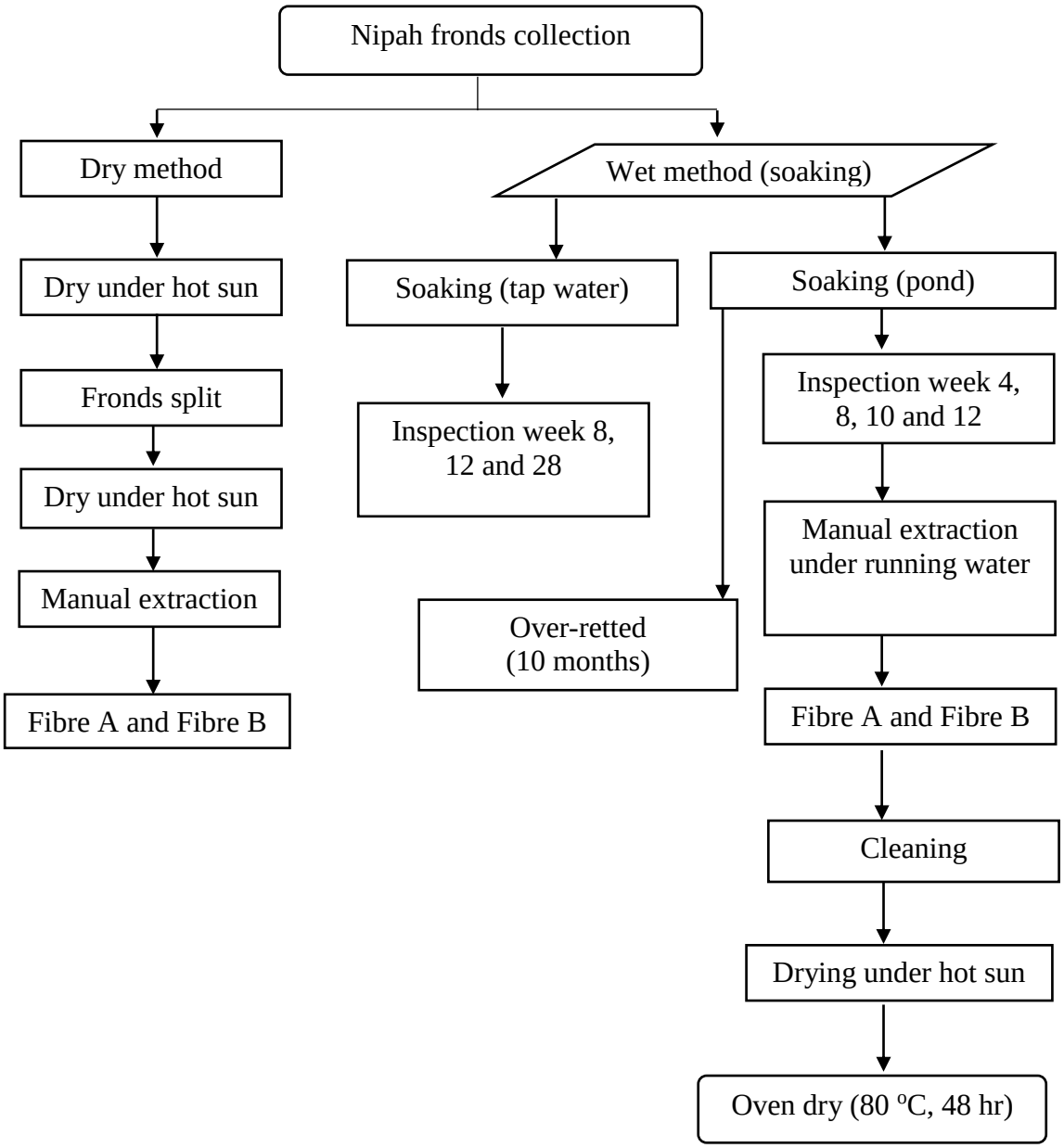


Figure 3.3: Process flow chart showing the fibre extraction process

3.2.1.1 Dry extraction

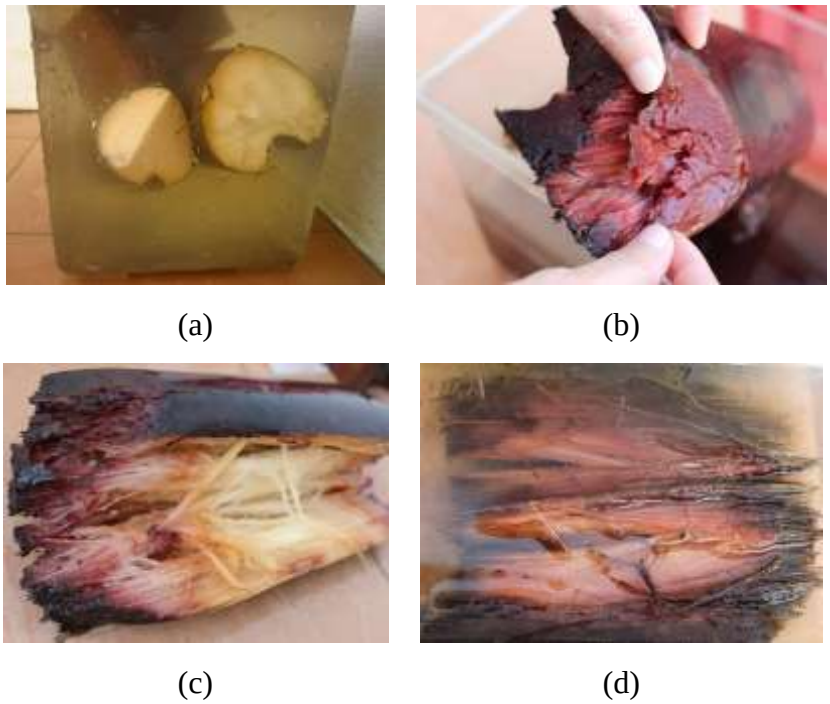
(a) (b)

(c) (d)

Figure 3.4: Dry extraction (a) Whole upper fronds (b) Split upper fronds (c) Split lower fronds (d) Manually cleaning extracted fibres

Collected fronds were initially dried under the hot sun (30 °C to 33 °C) for 168 hours (7 days) as shown in Figure 3.4(a). The fronds were examined every day to monitor possible changes of the fronds. In order to accelerate the drying process, the fronds were split up as shown in Figure 3.4(b) to Figure 3.4(c). The fibres were then extracted from the dried fronds manually as in Figure 3.4(d) with hand glove. Two types of fibre were extracted from the fronds, concurrently, designated as Fibre A and Fibre B according to the fibre diameter as explained in section 3.4.1. Each strand of fibres was cleaned at the same time to remove the dark decay substance on the fibre. Extraction process from the dried frond was time consuming and fibre breakage was observed during the process. Therefore, dry extraction was discontinued.

3.2.1.2 Wet extraction



(a) (b)

(c) (d)

Figure 3.5: Simple wet retting test (a) Day 1 (b) 8 weeks (c) 12 weeks (d) 7 months

The next step was to test the wet extraction. It is called wet extraction, as the fronds were soaked in water for certain period of time to soften the structure to ease the fibre extraction. This process is also known as water retting process. This is the more common method engaged by other natural fibre researchers (Haameem et al., 2016; Prasad et al., 2011; Reddy et al., 2009; Satyanarayana et al., 2007). Since nipah palm fibre was fairly new in this field, the optimum time for water-retting process has to be determined as over-retted sample will affect the properties of the fibre.

Simple test for water retting process was conducted, which act as the control sample for the water retting process. The process flow is as shown in Figure 3.3. Nipah fronds were soaked in a plastic container containing tap water as shown in Figure 3.5(a).

The tap water was changed periodically every two to three days. When there were no changes of the fronds condition, the water was never again being changed, just added in more water as the water level getting low in the container. After eight weeks (Figure 3.5(b)), the frond turned reddish, slightly softer at the end of the fronds, even the previously clear tap water turned reddish, the fronds were still hard enough to press, and at 12 weeks the nipah fronds were soft but still quite hard to be pressed. The extraction of fibres was still not easy as the whole nipah fronds were soft but the decayed viscid substance still firmly attached to the fibres. This was observed with the samples were opened as shown in Figure 3.5(c) and the control sample after 7 months (Figure 3.5(d)).

Figure 3.6: Wet retting (a) Day 1 (b) 4 weeks (c) 8 weeks (d) 12 weeks-upper fronds (e) 12 week-lower fronds (f) Extracting process (g) Extracting both fibres at the same time (h) Freshly extracted Fibre A (j) Freshly extracted Fibre B

Based on the initial findings of the control sample soaked in tap water, about 70 kg of nipah fronds were prepared for retting process in a fresh water pond. Fresh water pond was chosen to allow biodegradation of the non-cellulosic component of the fronds. Nipah fronds were cut to about 50 cm, placed into a plastic bag, weighted down with big stone and dropped into the fish pond as shown in Figure 3.6(a). These samples were inspected at 4 weeks, 8 weeks, 10 weeks, 12 weeks and more. At 4 weeks (Figure 3.6(b)), the fronds were hard, and the tips of the fronds were slightly softer compared to the centre of the fronds. The fronds were returned to the pond for further retting process. At 8 weeks as shown in Figure 3.6(c), the fronds started to emit decomposed odour, the fronds were getting softer and the whole fronds were able to be opened easily exposing the internal structure of the fronds. The viscid substance still looked fresh and rigid. The fronds were placed back into the pond for further water retting process as some fronds were still hard. At 12 weeks as in Figure 3.6(d) to Figure 3.6(e), at this point of checking the fronds emitted an extremely strong decompose odour. When the fronds were opened and cleaned under running water, the fibres were separated easily from the fibre bundles as shown in Figure 3.6(f). All the fronds were inspected to determine the condition of each frond. Some fronds were soft enough to be extracted while some needed to be returned to the pond as they were still hard. Some of the fronds were left until 10 months in the pond, and those were the fronds that were difficult to be extracted.

The extraction process of the fibre was done manually under running tap water. The source of the tap water is directly from the water catchment area at Mount. Sadong, Serian. The water-retted fibres were easier to be pulled out from its fibre bundles as the viscid substance has loosened up. Figure 3.6(f) to Figure 3.6(g) show the extraction process of both fibres. Fibre bundles were easily separated once the water retting process is optimum.

Fibres were pulled out one by one and cleaned at the same time. The extracted Fibre A are as shown Figure 3.6(h), while Fibre B are the remaining of the fibre bundle once Fibre A had been pulled out. It was placed in a basin that contains water as it was easier to remove the dirt. Figure 3.6(j) shows the clean Fibre B.

3.2.2 Cleaning and drying process

The cleaning process of the fibre after the extraction process was carried out by soaking the fibres in detergent (Sunlight Dishwashing Liquid) for one hour at 70 °C as shown in Figure 3.7(a) to Figure 3.7(b), for Fibre A and B (Rout, 2001). The fibres were then cleaned under running water for about 15 minutes and dried under the hot sun as shown in Figure 3.5(c) and proceeded to oven drying at 80 °C for 48 hours.

(a) (b) (c)

Figure 3.7: (a) Cleaning of Fibre A (b) Cleaning of Fibre B (c) Dried Fibre A and B

3.2.3 Unsaturated polyester resin

Commercial unsaturated polyester resin supplied by Reseng Trading Sdn Bhd, Kuching, Sarawak was used as the matrix for the composites as shown in Figure 3.8. The polyester was mixed with 2% of methyl ethyl ketone peroxide (MEKP) as hardener.

(a) (b)

Figure 3.8: (a) Polyester resin (b) Hardener

3.3 Sample preparation

The sample preparation method for single fibre test, composite fabrication, loading parameters, and fibre surface treatment method are explained in detail. The process flows are as in Figure 3.9.

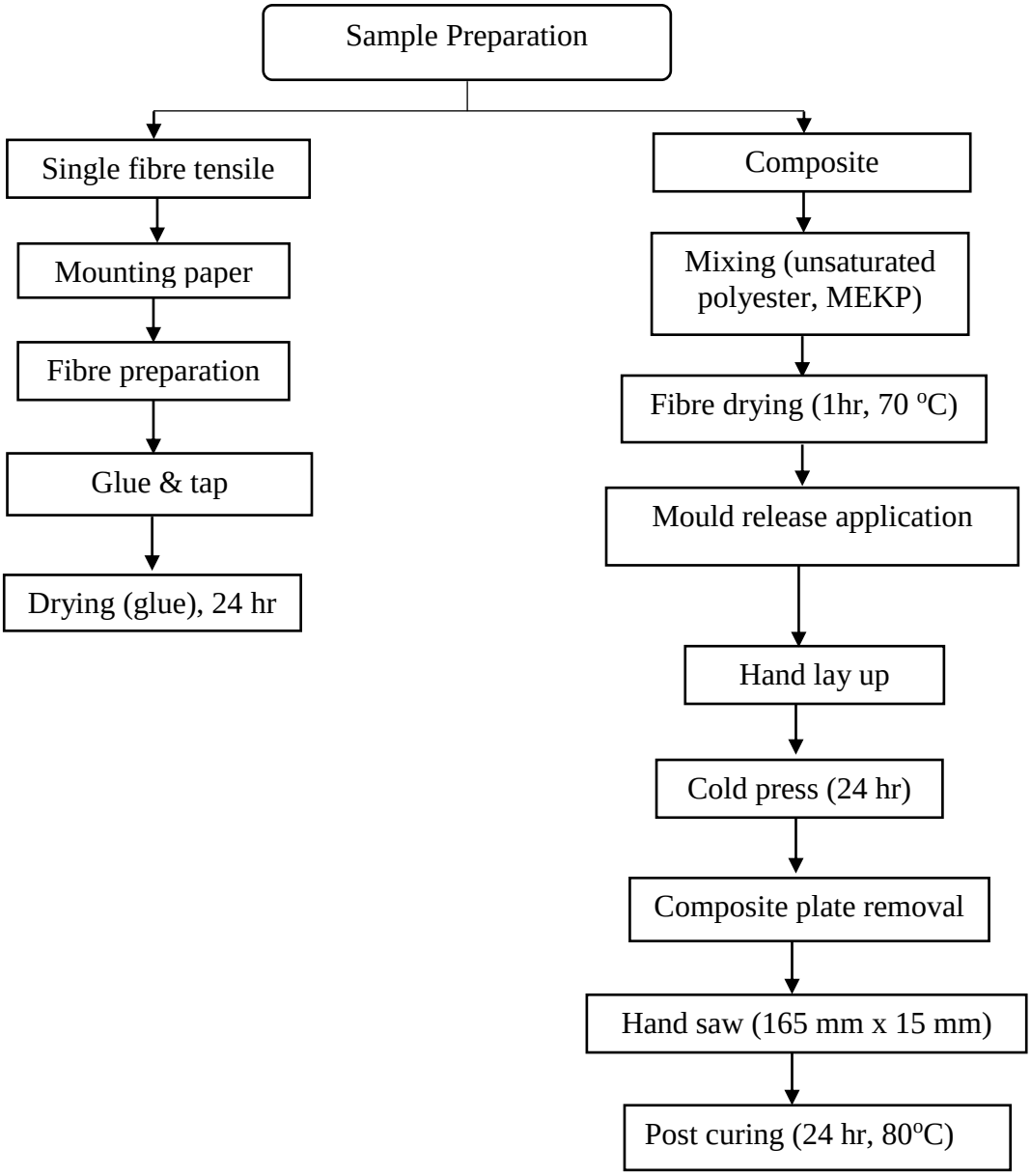


Figure 3.9: Process flow chart of sample preparation

3.3.1 Single fibre test sample

The single fibre test was performed on nipah fibre to determine its axial tensile modulus, ultimate strength and failure strain according to ASTM D3379-75 (Prasad & Rao, 2011; Sathishkumar et al., 2012). The gauge length was set at 50 mm. The single fibre test was performed using cardboard mounting method as shown in Figure 3.10. The overall cardboard length was 150 mm, and the width was 25 mm as shown in Figure 3.10(a). The test speed was set at 0.5 mm/min. The single fibre was mounted to the tap-shaped cardboard at both ends of the fibres using super glue as shown in Figure 3.10(b). This was to secure the bond and to provide a better grip for clamping. The test was performed once the glue had dried up completely. The cross-head speed was set at 1 mm/min. The gauge length was set to 50 mm. The supporting cardboard was cut in the middle after the paper tab was fixed to the tensile machine as shown in Figure 3.10(c). The actual samples for this test are as shown in Figure 3.10(d). The average value of 10 samples was taken for the results.

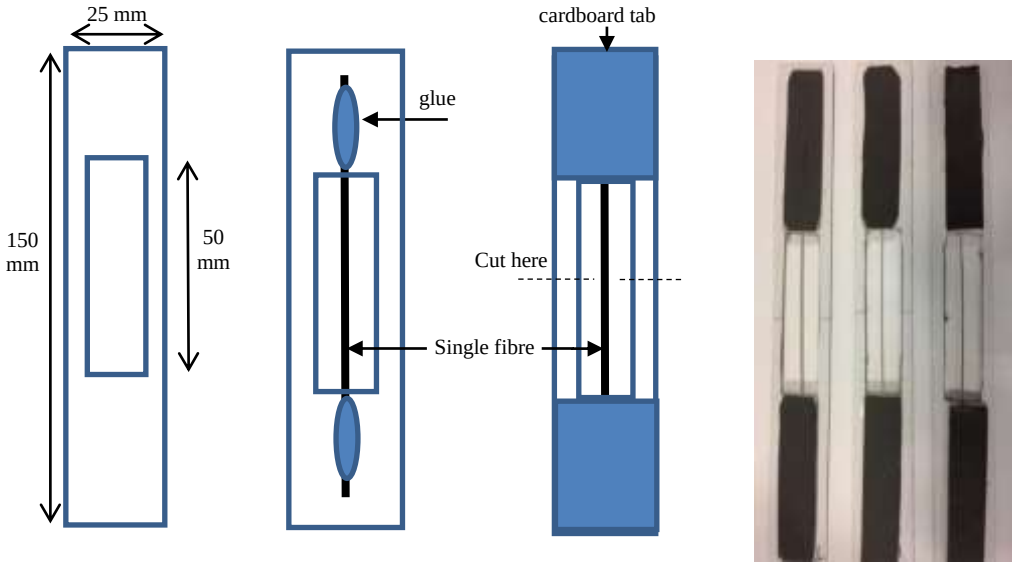


Figure 3.10: Single fibre test based on ASTM D3379-75 (a) Dimension of cardboard (b) Fiber and glue application (c) Paper tab application (d) Actual photo of samples

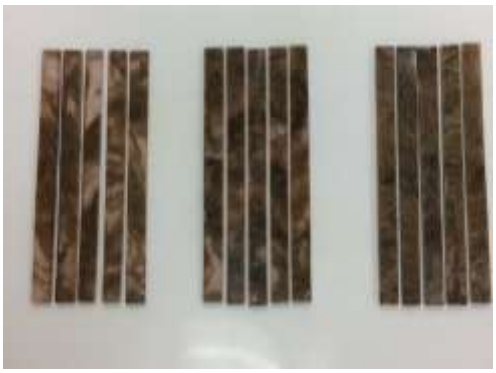
3.3.2 Composite fabrication

The composites were fabricated using hand lay-up and compression moulding technique. The composites were fabricated using a 230 mm x 230 mm aluminium mould. Vaseline pure petroleum jelly was used as a release agent to ease the removal process of composites. Vaseline was used as it was effective in releasing the composite plate compared to other mould release agent and the cost was so much lower than the mould release agent. The mould cavity was brushed with Vaseline pure petroleum jelly. Fibres were dried in the oven at 70 °C for one hour prior to the fabrication process. The polyester resin mixture was prepared while the fibres were in the final drying stage in the oven. The resin was mixed with two to three drops of hardener, manually stirred to homogenise the mixture. This was to make sure the unsaturated polyester and the catalyst were well mixed.

(a)

(b)

(c)



(d)

Figure 3.11: (a) Cold press machine during pressing (b) Cold press machine after 24 hours (c) Composite plate of fibre B (d) Tensile sample of fibre B

The dried fibres were then arranged on the mould; the resin was poured into the mould cavity. The fibres were pressed against the resin to ensure proper wetting as shown in Figure 3.11(a) to Figure 3.11(b). The mould was covered when the resin had reached the gelling stage. Gelling stage is the abrupt change of viscosity of the resin. Gelling stage is important to reduce the formation of air bubbles in the composite plate (Haameem et al., 2016). The composite plate was removed and hand-sawed to 165 mm x 15 mm x 3 mm as shown in Figure 3.11(c) to Figure 3.11(d). The samples were then post-cured at 80 °C for 24 hours. Table 3.1 shows the fibre loading condition for composites fabrication. The fibre loading were only up to 15% due to improper wetting condition between resin and fibre

when the composite were loaded above 20%. The fibre condition for Fibre A was short fibre with fibre length of 15 mm to 20 mm. Fibre B composites were fabricated using long fibre due to the agglomeration of fibres during the mixing process. The agglomeration was found to be minimal for long fibres although agglomeration was still observed as discussed in Chapter 4.

Table 3.1: Composite loading condition for nipah palm fibre.

Loading (%)	Composite ratio (matrix: fibre)	Fibre A Weight (g)	Fibre B Weight (g)	Treatment condition					
				Untreated	NaOH (%)				
					2	4	6	8	10
5	95:5	8	7	Fibre A & B	Fibre B	Fibre B	Fibre A	Fibre A	Fibre A
10	90:10	16	14	Fibre A & B	Fibre B	Fibre B	Fibre A	Fibre A	Fibre A
15	85:15	24	21	Fibre A & B	Fibre B	Fibre B	Fibre A	Fibre A	Fibre A

3.3.3 Alkali treatment

Table 3.2: Alkali treatment (NaOH) calculation

NaOH Concentration (%)	NaOH pellet (g)	Volume of distilled water (ml)
2	20	1000
4	40	1000
6	60	1000
8	80	1000
10	100	1000
12	120	1000

(a) (b)

Figure 3.12: (a) Bulk fibre soaking (b) Individual fibre soaking

The alkali treatment of the fibre was done by using sodium hydroxide (NaOH), at different concentrations as shown in Table 3.2. The alkali was prepared by diluting the pre-measured alkali pellet in distilled water. Fibre A was soaked in alkali solution for two

hours, followed by cleaning in distilled water. Figure 3.12 shows the bulk soaking of fibres and individual soaking of fibres for area shrinkage measurement. The fibres were dried in the oven at 80 °C for 24 hours. Fibre B was soaked for 30 minutes in alkali solution and dried at 80 °C for 24 hours. The dried samples are shown in Figure 3.13.

(a) (b) (c) (d)

Figure 3.13: Fiber A, after alkali treatment (a) 8%, (b) 10%, (c) 15% (d) Fibre B, after alkali treatment

3.4 Nipah fibre characterisation techniques

The diameter measurement method, fibre cross-section area measurement, density measurement, chemical composition analysis, fourier transforms infrared spectroscopy (FTIR), thermogravimetric analysis (TGA), differential scanning calorimetry (DSC), X-ray diffraction (XRD) and scanning electron microscope (SEM) are explain in detail. The process flow chart is as shown in Figure 3.14.

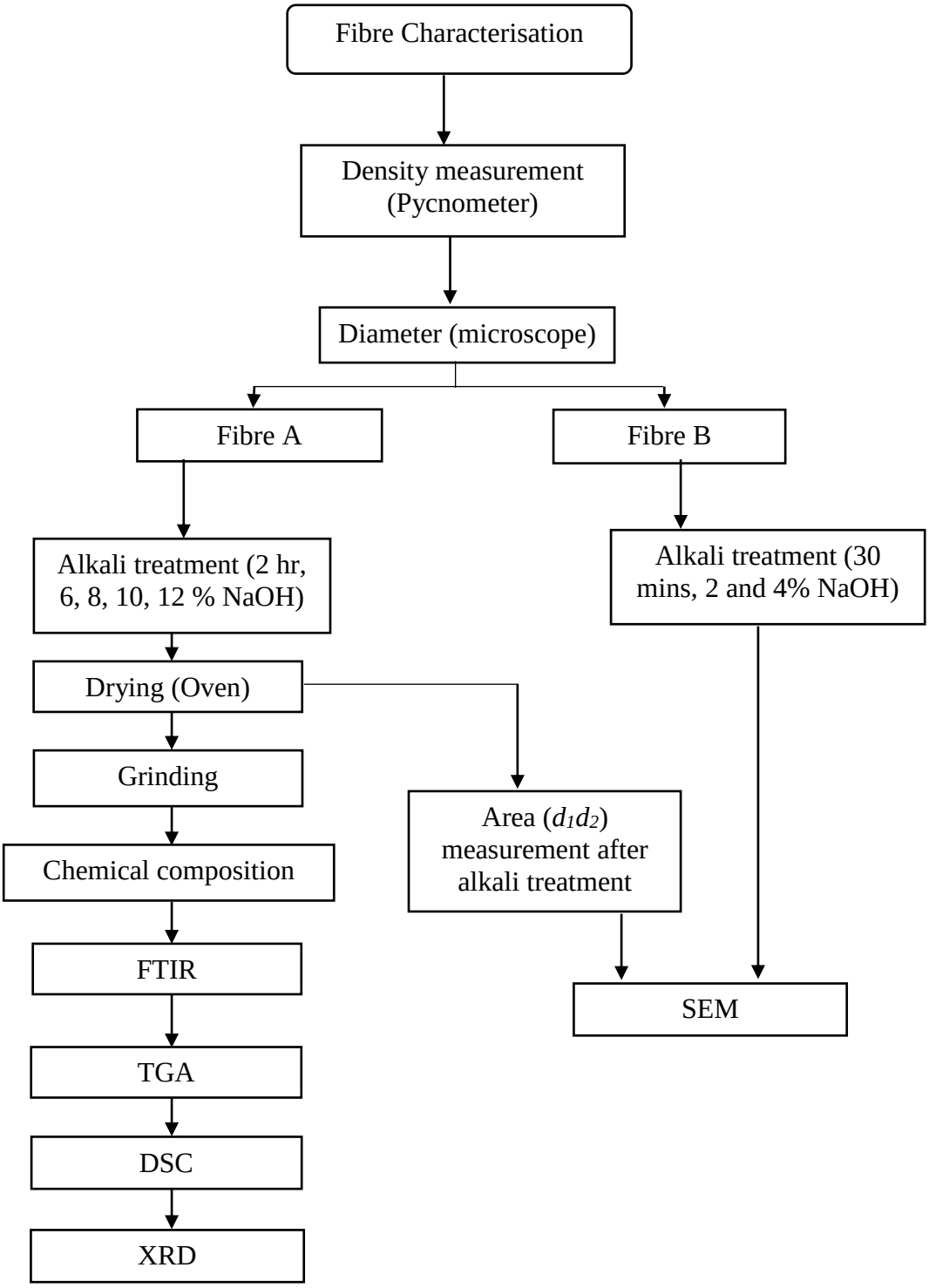
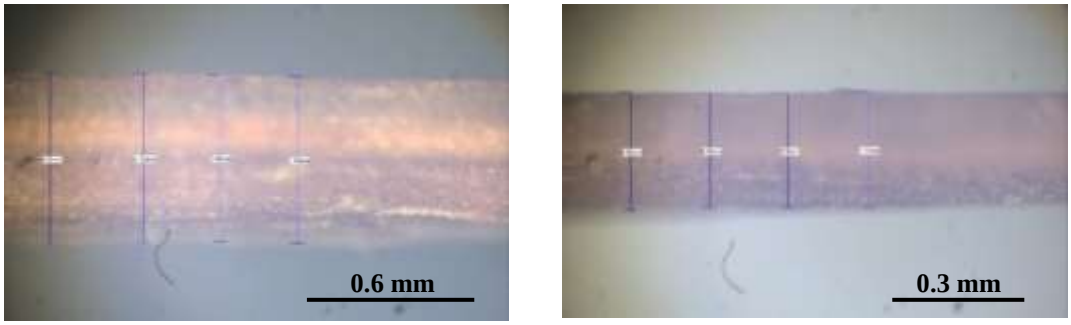


Figure 3.14: Process flow chart for nipah palm fibre characterisation

3.4.1 Diameter measurement

The diameter of the fibre was evaluated from optical observation under the microscope (SPG Fluorescent Microscope). Fibre A and B were measured from the images captured using the attached camera. The measurement was taken at 100 different locations along each fibre, and four fibre samples were measured for each type of fibre. The images from the microscope are shown in Figure 3.15.



(a) (b)

Figure 3.15: Microscopes images of diameter measurement of Fibre A (250x) (a) d_1 (b) d_2

3.4.2 Fibre cross-section area measurement

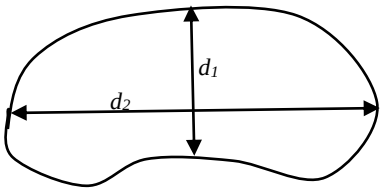


Figure 3.16: Fibre diameter, d_1 and d_2

The fibre cross-section area measurements were measured to determine the shrinkage of the area due to the effect of alkali treatment. Since the fibre is not spherical in shape, the cross-section area was taken as the measurement instead of the diameter readings (Cai et al., 2016). The average cross-sectional area (A) was calculated from two fibre perpendicular diameters as shown in Figure 3.16, d_1 and d_2, $A = \pi d_1 d_2/4$.

3.4.3 Density measurement

(a) (b) (c)

Figure 3.17: Part of density measurement steps (a) Sieving (b) Adding distilled water (c) Heating

The density of Fibre A and B were measured using water pycnometer procedure according to the American Society of Testing and Materials (ASTM) D 854. The term density refers to mass per unit volume. The particle density measures the actual particle which made up the nipah powder mass and defined as the ratio of the mass of the particles to the mass of water they displaced. The fibres were ground using a blender into powder form. The powder was sieved using sieves of different sizes (212 µm, 150 µm and 75 µm). Only powder that could pass through the 75 µm sieve was used for the density test. About 10 grams of powder sample was used for this test. The powdered nipah was placed into a 50 ml bottle. The masses of the bottle and the powder were measured and filled with

distilled water. The final step was to heat the bottles until it bubbles. Figure 3.17 shows the steps involved in the density measurement and Table 3.3 shows the formula for the density calculation.

Table 3.3: Density test calculation

Density Test of Nipah Fibre A				
Pycnometer number	**Formula**	**Unit**	**1**	**2**
Mass of bottle + powder + water	M_3	g	75.45	76.30
Mass of bottle + powder	M_2	g	22.98	23.90
Mass of bottle full of water	M_4	g	75.45	76.30
Mass of bottle	M_1	g	22.50	23.08
Mass of powder	$M_2 - M_1$	g	0.48	0.82
Mass of water in full bottle	$M_4 – M_1$	g	52.95	53.22
Mass of water used	$M_3 – M_2$	g	52.47	52.40
Volume of powder particles	$(M_4 – M_1) – (M_3 - M_2)$	mL	0.48	0.48
Particle density, p_s	$(M_2 - M_1) / [(M_4 – M_1) – (M_3 - M_2)]$	Mg/m^3	1.0	1.0
Average value, p_s		Mg/m^3	1.0	

3.4.4 Chemical composition analysis

(a) (b)

Figure 3.18: (a) Heavy duty commercial blender (b) Blended fibres

The chemical composition was carried out at the Forest Research Institute of Malaysia (FRIM) and ENVIC Laboratory Sdn. Bhd. using the Technical Association of Pulp and Paper Industry (TAPPI) method. This was to determine the α-cellulose, hemicellulose and lignin. The standards are TAPPI T 203CM-99 (α-cellulose), TAPPI T 222 om-02 (lignin) and aqueous alkali extraction (hemicellulose). The equipment for the analysis was weighing balance, water bath and burettes. The samples were oven-dried before blended to powder form by using a heavy duty commercial blender as shown in Figure 3.18.

3.4.5 Fourier Transforms Infrared Spectroscopy (FTIR)

Figure 3.19: FTIR instrument

Fourier transforms infrared spectroscopy was carried out using Nicolet iS10, as shown in Figure 3.19. It was to derive the FTIR spectra of the untreated and 6%, 8%, 10% and 12% alkali treated Fibre A. All the spectra were recorded in the wavenumber range between 400 cm^{-1} and 4000 cm^{-1}.

3.4.6 Thermogravimetric Analysis (TGA)

The thermogravimetric analysis was carried out using Mettle Toledo for untreated and treated nipah fibre, with the nitrogen was used as the carrier gas, and the heating rate used was 10 °C/min. The thermal properties of the fibres were observed at the temperature of 30 °C to 600 °C. Grinded untreated and 6%, 8%, 10% and 12% nipah treated samples of nipah fibre powder were used for the measurement.

3.4.7 Differential Scanning Calorimetry (DSC)

The Mettle Toledo differential scanning calorimetry was used to analyse the thermal characteristic due to the effect of alkali treatment towards moisture and inorganic volatile content in the fibres. The samples were placed in an alumina crucible. About 10 mg of sample was used. The testing was carried out in high purity nitrogen gas at a heating rate of 10 °C/min heated ranging from 30 °C to 700 °C.

3.4.8 X-Ray Diffraction (XRD)

XRD (P8Advan-Bruker) analysis was carried out to analyse the crystallinity of Nipah fibre before and after the NaOH treatment. The measurement was taken from 2θ of 10º to 90° using Cu Kα X-ray source. The fibre crystallinity index (CI) was calculated based on the Segal Empirical method;

$$C_{Ir} = (I_{200} - I_{am}) / I_{200} \times 100\% \qquad \text{Equation 3.1}$$

where I_{200} is the peak intensity corresponding to cellulose *I*, and I_{am} is the peak intensity of the amorphous fraction.

3.4.9 Scanning Electron Microscopy (SEM)

(a) (b)

Figure 3.20: (a) Scanning Electron Microscope (b) Gold sputter

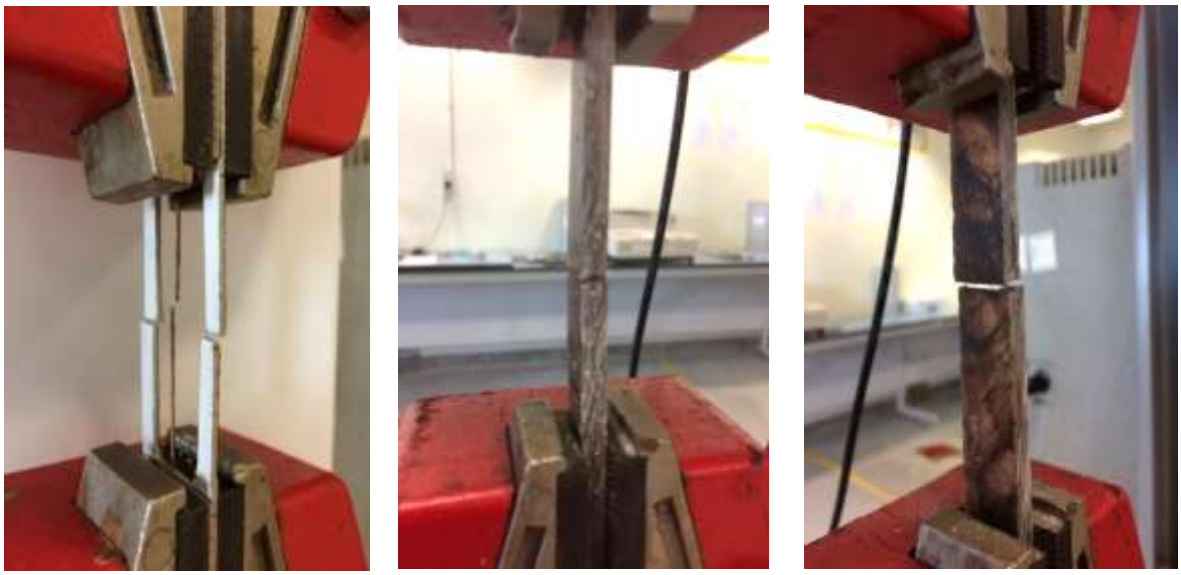
The fibre surface and the fractography of the single fibre test and composites were studied with Hitachi TM3030 at an acceleration voltage of 15 kV as shown in Figure 3.20. Prior to the analysis, the samples were gold-coated using a sputtering machine to improve the surface conductivity. The digital optical microscope, Hirox KH8700 was used to capture the coloured images of the untreated fibre surface of fibres and its cross-section.

3.5 Mechanical testing

Mechanical testing was conducted to determine nipah single fibre strength and its composite strength.

3.5.1 Tensile test

The tensile test was performed using the Testometric (25 kN) tensile machine, according to the standard test method for plastic ASTM D638-03. The crosshead speed was set at 1 mm/min (single fibre test) and 2 mm/min (composite sample), with the gauge length of 50 mm. The average of 3 and 5 values were taken as the results. The test set up is shown in Figure 3.21.



(a) (b) (c)

Figure 3.21: Tensile test (a) Single fibre (b) Fibre A composite (c) Fibre B composite

The formula used to calculate the tensile test, tensile strain, and elongation at break and tensile modulus are:

$$\text{Stress, } \sigma_{max} = \frac{P_{max}}{A} \qquad \text{Equation 3.2}$$

Where, P_{max} = Maximum force applied.

A = Cross – section area.

Strain, $\epsilon = \frac{L - L_0}{L_0}$ Equation 3.3

Percentage of Elongation, % = $\frac{L - L_o}{L_o}$ x 100 %

Where, L = The distance between gage marks at any time at any load

L_o = The original distance between gage marks

Young Modulus, E = Stress / Strain = $\frac{\sigma}{\epsilon}$ Equation 3.4

3.6 Water Absorption

The water absorption test was conducted based on ASTM D570-98. A long-term immersion in distilled water up to 28 days was conducted and the measurements were taken on daily basis. The samples were wiped with cloth before the measurement was taken. The temperature of the water was maintained at the normal room temperature. The samples were all pre-weighted before the immersion using analytical balance.

Water Absorption = $(\frac{Wt - Wo}{Wo})$ 100% Equation 3.5

Where W_o = dry weight of the sample, W_t = weight at a specific time

3.7 Chapter summary

In this chapter, the extraction process of nipah palm fibre from fresh nipah fronds was explained. The sample preparation for single fibre tensile test and composite fabrication was discussed. The surface treatment and fibre characterisation techniques for diameter measurement, density measurement, chemical composition analysis, FTIR, TGA, DSC, XRD and SEM were explained in detail. The methods for mechanical test and water adsorption for composite sample were also discussed.

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Overview

This chapter explains the results and discussion of nipah palm fibre extraction from the fresh Nipah fronds. The results of the diameter and density measurement of Fibre A and Fibre B, fibre surface characteristics and its cross-section are presented. The effect of various percentage of NaOH towards Fibre A, its fibre morphology, fibre diameter, surface morphology, chemical composition, TGA, FTIR, DSC, XRD and tensile strength characteristics are discussed. The results of loading condition of Fibre A composites, fracture surface morphology and water absorption characteristics are discussed. Lastly, the results of surface treatment for Fibre B and its composites, surface morphology and fracture morphology are presented and discussed.

4.2 Fibre extraction and raw nipah fibre characterisation

Nipah palm fibre extraction was carried out by using two methods. The details of the results for both methods, were discussed in the subsequent section. Nipah palm fibre extraction method must be able to produce reliable fibres that were free from pre-mature damages.

4.2.1 Fibre extraction result

(a) (b) (c)

Figure 4.1: (a) Fibres from dry extraction process (b) Fibres from wet extraction process (c) Over-retted fibres

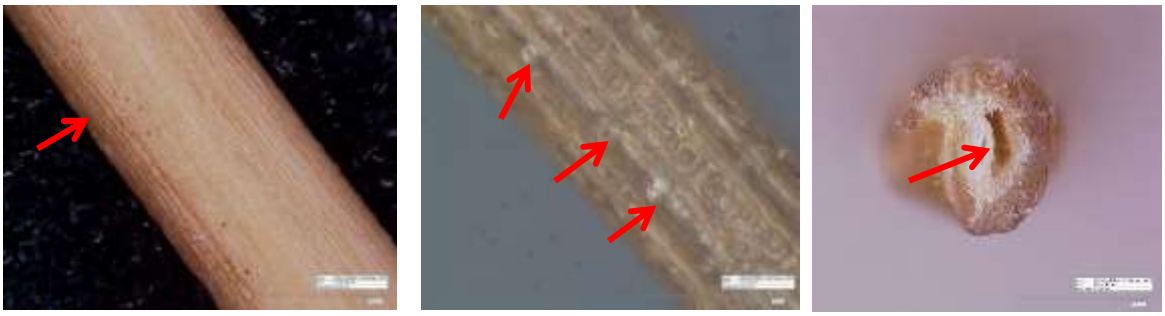
Two fibre extraction methods were conducted to extract the nipah palm fibre. The dry fibre extraction took about a week of drying process before the fibres could be scrapped from the fronds. However, it could be observed that the process of fibre scrapping tends to break the fibres as most of the dried fibres were tightly glued to its fibre bundles. It was found that the process of scrapping and extracting were time consuming and the process had introduced premature defects such as breakage. The defects were due to excessive force exerted during the pulling process from the dried fronds. The amount of fibres produced from the dry method was very limited, as shown in Figure 4.1(a).

Due to the poor results of dry fibre extraction attempt, a simple water retting test was conducted inside a small container. This water retting test, confirmed that the microbiological action in a pond was needed to speed up the decaying process of the non-cellulosic substance of nipah fronds. The condition at 12 weeks and 7 months showed that the viscid substance still looked fresh and strongly attached to the fibres.

Wet retting process for nipah fronds was conducted at a pond, which took about 12 weeks to be completed. As reported in the literature, the wet retting or water retting process is a time consuming process (Tamburini et al., 2004). This was when the fibres could be easily extracted without too much force and most unlikely to introduce premature defects to the fibres. At this particular point, the fibre separation was very easy, because each fibre strand could be pulled, leaving behind the non-cellulosic substance. The whole fronds emitted strong decomposed odours, as compared to the previous checking points at week 8 and week 10. The wet extraction process produced two types of fibres, namely Fibre A and Fibre B, which was not possible for the dry extracting process without breaking the fibres apart. The fibres from wet extraction method were cleaner as the extracting process was done under running water. It was observed that lower fronds of nipah palm were easier to be extracted due to the larger amount of spongy media as compared to the upper fronds. The lower fronds were normally bigger in diameter compared to the upper fronds and were observed to be softer once they reached the 12 weeks water retting process. They were easily split up manually requiring less force. The less exposed part of the fronds was at times difficult to be extracted even though the 12 weeks of water retting process were completed. This could be due to the age of the palm itself. The effects of harvest time were studied on hemp fibres (Liu et al., 2015). During the collection process, the fronds were not collected from one single palm tree but also from other nipah palms. Another reason could be the effect of fronds diameter, where the lower fronds could reach 200 mm to 300 mm in diameter while the upper fronds were around 100 mm. The yield of this process was about 2%. The fibres extracted from this process are shown in Figure 4.1(b). Some of the nipah fronds were left in the pond for more than 12 weeks, to observe the over retting condition of the fibres. It was observed that fibre

breakage was prominent as shown in Figure 4.1(c). The fibres were brittle and broke into tiny pieces due to the excessive water retting process. Fibre separation was difficult as the tiny pieces of Fibre A entangled within the Fibre B mesh. Since this is a pioneer research works on nipah palm fibre. There is no specific method available in the literature regarding nipah fibre extraction. Therefore, both dry and wet methods were completely based on trials especially on the optimum water retting period. This study concluded that 12 weeks is the optimum period for nipah fibre to be easily extracted.

4.2.2 Fibre characteristic



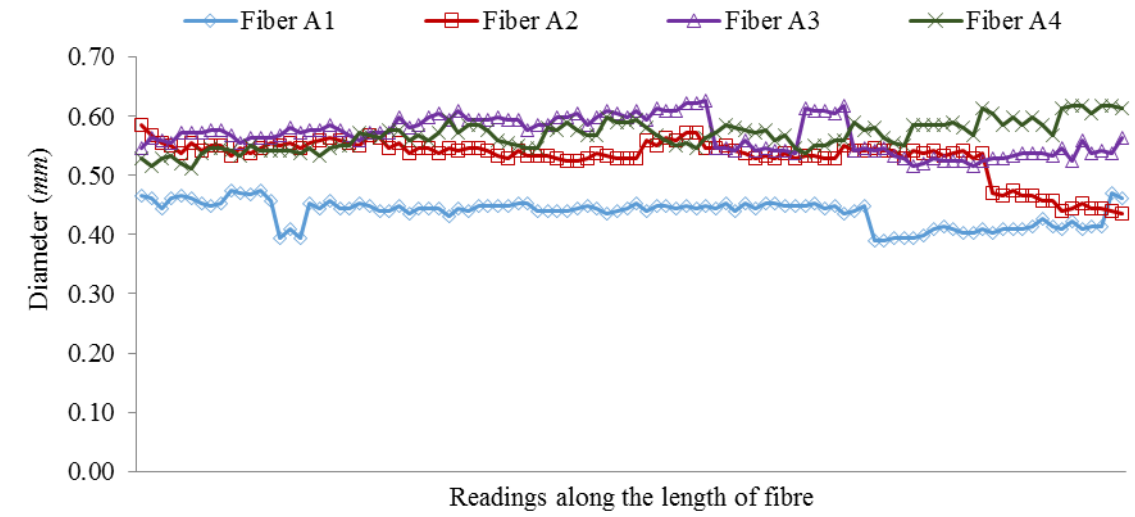
(a) (b) (c)

Figure 4.2: Optical images of (a) Fibre A (b) Fibre B (c) Fibre A cross-section

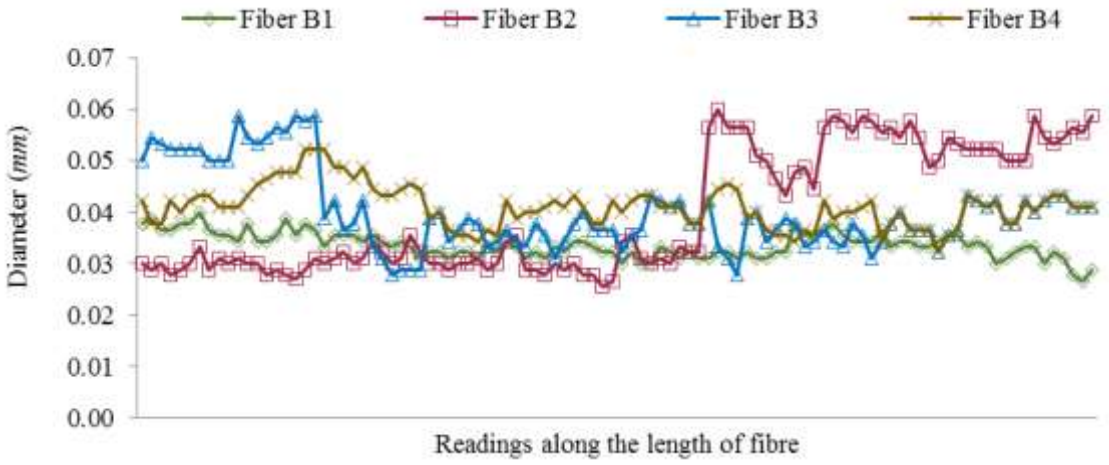
As observed in Figure 4.2(a) to Figure 4.2(b), Fibre A and Fibre B have the characteristics of natural fibre, both has rough and uneven surface. At a closer look of both fibres, Fibre A has concave structure throughout its length while Fibre B has protruding surface as shown by arrows in the figures. Fibre B was found encapsulating Fibre A during the extraction process, providing the interlocking mechanism for both fibres. The cross-

section of Fibre A revealed a hollow structure as shown by an arrow, and the actual shape of Fibre A is elliptical. Figure 4.2(c) is the cross-section of Fibre A.

4.2.3 Diameter measurement



(a)



(b)

Figure 4.3: Variation of fibre diameter (a) Fibre A (b) Fibre B

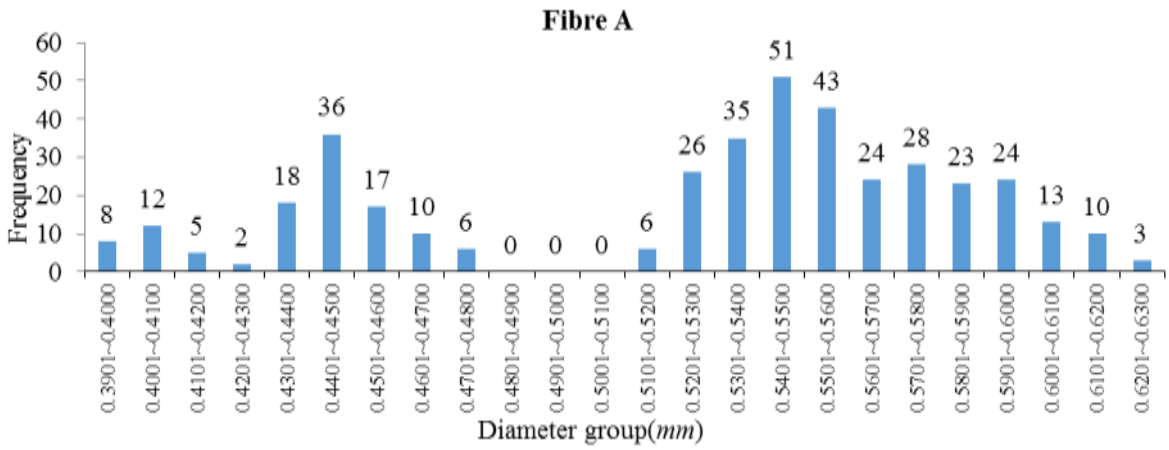
Fibre A1, A2, A3 and A4 are the four randomly chosen of type A fibre for the evaluation, whereas, Fibre B1, B2, B3 and B4 are type B fibres. From Figure 4.3, it is observed that the diameter for Fibre A is between 0.38 ~ 0.64 mm with an average value of 0.53 mm. The diameter for Fibre B is between 0.025 ~ 0.059 mm and the average value is 0.039 mm as in Table 4.1. The average value for Fibre A and Fibre B are calculated by dividing the total diameter with 100 readings. Fibre B is much smaller than Fibre A, about 10 ~ 15 times smaller. The variation for Fibre B is much greater due to the existence of the protruding surface of Fibre B. It is assumed that the protruding surface of Fibre B provides interlocking action supporting Fibre A therefore giving structural support for nipah fronds.

Figure 4.4(a) shows the frequency of distribution of Fibre A, with the diameter readings showing a bimodal distribution. It shows that the diameter of the measured fibre has two maximum readings. The diameter group 0.4401 ~ 0.4600 mm and the diameter group 0.5401 ~ 0.5600 mm contribute to these two peaks. The later diameter group has the highest frequency. The distribution of Fibre B, Figure 4.5(a) shows multimodal results, which has three maximum readings. The diameter groups 0.0341 ~ 0.0360 mm, 0.0421 ~ 0.0440 mm and 0.0521 ~ 0.0540 mm contribute to the multimodal results. However, the diameter group 0.0341 ~ 0.0360 mm shows the highest frequency. The diameter variability observed for nipah fibre is the characteristic of natural fibre, and there are a lot of factors that may contribute to the variability such as the plant factor and age of plant itself.

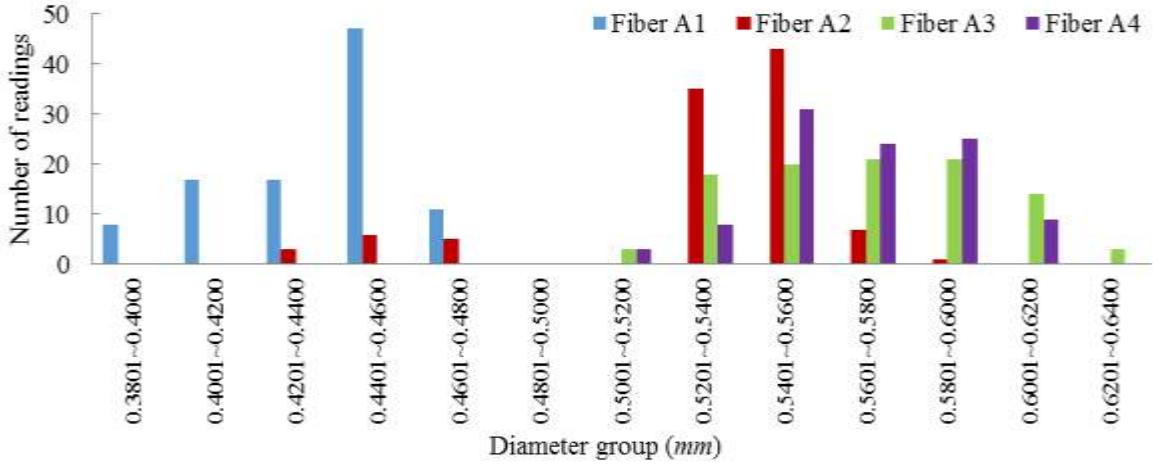
Referring to Figure 4.5(b), Fibre B2 shows a multimodal distribution, related to the diameter variability of each fibre even though the fibre originates from the same plant. This result can be linked with Figure 4.2(b) for its protruding surface. Figure 4.4(b) and Figure 4.5(b) show the frequency distribution of an individual fibre. Fibre A1 and A2 show a

bimodal distribution and Fibre A3 and A4 show a normal distribution towards the higher value on the right side of the histogram.

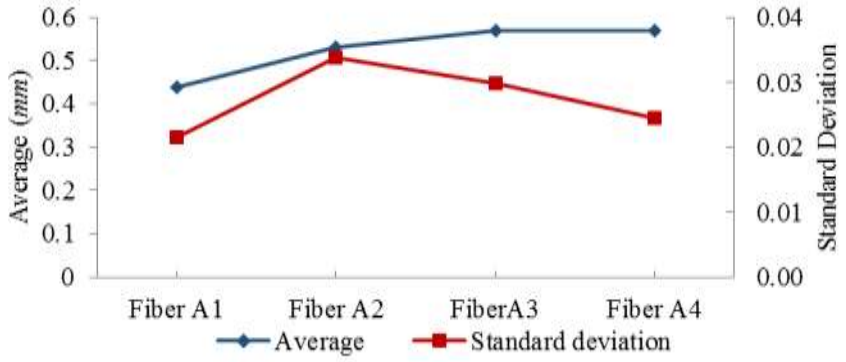
For Fibre B, Fibre B1 shows a normal distribution, Fibre B2 and B3 show multimodal distribution and Fibre B4 shows a bimodal distribution. From these figures, it is able to determine which fibre contributed the most data to certain diameter group and the diameter range of individual fibre. Out of the four fibres measured, Fibre A1 has the smallest diameter range compared to the other three fibres. Figure 4.4(c) and Figure 4.5(c) show the standard deviation plotted against the average of diameter. The standard deviation is to show the dispersion from the mean value. These figures established that the standard deviation decreases with an increase in diameter of both fibre types, concluding that coarser fibres are more regular. This result is also reported for banana fibre (Mukhopadhyay et al., 2008).



(a)

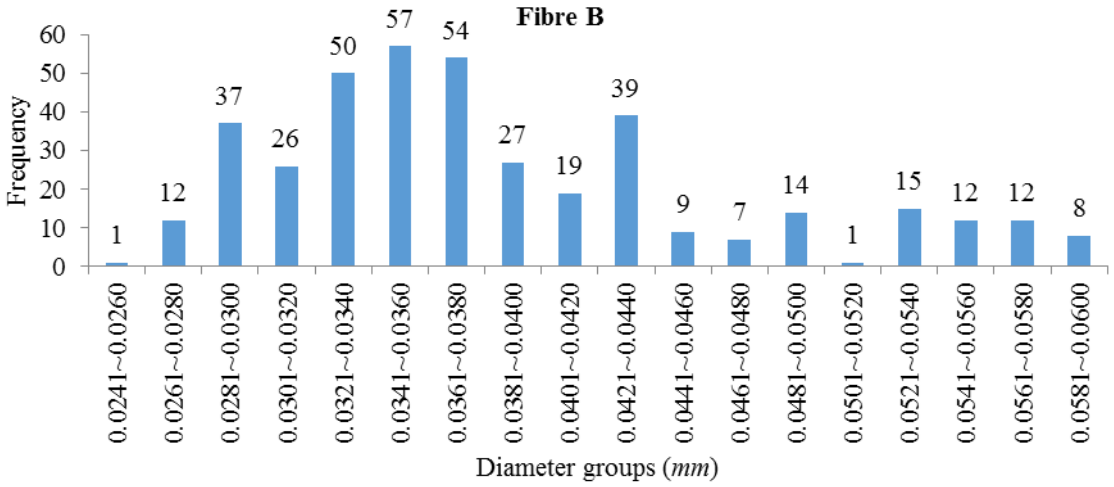


(b)

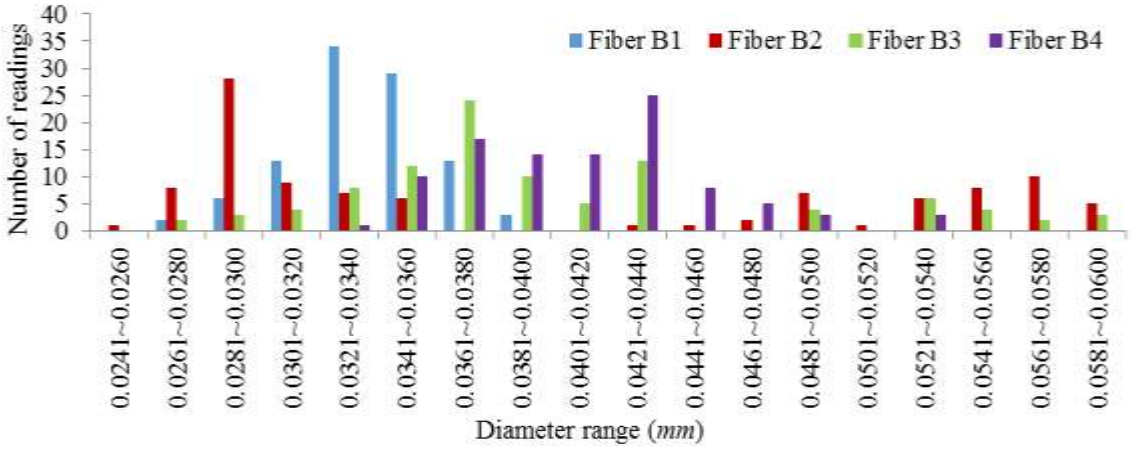


(c)

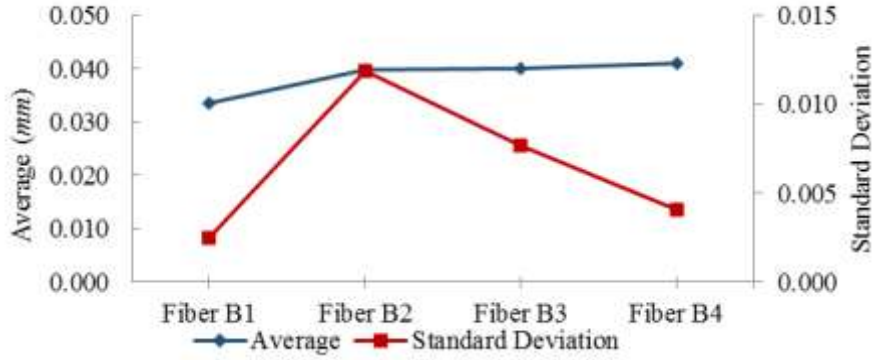
Figure 4.4: Fibre A (a) Frequency distribution of nipah fibres based on diameter (b) Frequency distribution of diameter along the length of four individual fibres (c) Average diameter vs standard deviation



(a)



(b)



(c)

Figure 4.5: Fibre B (a) Frequency distribution of nipah fibres based on diameter (b) Frequency distribution of diameter along the length of four individual fibres (c) Average diameter vs standard deviation

4.2.4 Density measurement

Table 4.1: Nipah fibre physical properties (average)

Physical properties	Nipah	
	Fibre A	**Fibre B**
Density (g/cm^3)	1.00	0.89
Diameter (mm)	0.53±0.06	0.039±0.08

The nipah fibre density for fibre is shown in Table 4.1. The density value of Fibre A is higher than Fibre B, at around 12%. The difference is considered small as compared to the diameter of both fibres, presumably due to the existence of vessel (Figure 4.2(c)) in the fibre.

4.2.5 Chemical analysis

Table 4.2: Chemical composition of Fibre A

Natural fibre	**α-Cellulose (% w/w)**	**Hemicellulose (% w/w)**	**Lignin (% w/w)**	**Reference**
Nipah Fibre A	27.0	23.0	21.0	-
Oil palm (frond)	39.5	29.8	21.2 (Klason)	Shibata et al. (2008)
Oil palm (trunk)	30.6	33.2	24.7 (Klason)	

The chemical analysis of nipah fibre is shown in Table 4.2. As stated in the literature, the nipah palm chemical composition is said to be very similar to oil palm (Shibata et al., 2008; Tamunaidu & Saka, 2011). Nipah and oil palm are from the same

palmae family, therefore, it is expected that there are some similarities in terms of its chemical composition.

4.3 Effect of alkali treatment towards nipah fibre (Fibre A)

4.3.1 Fibre physical form after alkali treatment

Figure 4.6: (a) Untreated and 6% alkali treated fibres (b) 8% alkali treated fibres (c) 10% alkali treated fibre (d) 12% alkali treated fibre

It is clear that alkali treatment has significant physical changes towards nipah palm fibre. The effect of 6%, 8%, 10% and 12% alkali treatment had changed the physical appearance of the fibres. As can be observed in Figure 4.6, the colour of the fibres had turned from a light shade of brown to dark brown due to the alkali treatment. All the treated fibres showed fibre bending and twisting. The higher the alkali concentration, the

greater the effect of fibre twisting. The 12% alkali treated nipah fibres were badly twisted and fibre breakage was noticeable. This indicates that nipah fibres undergone destruction due to high alkali concentration. This could be due to excessive stripping of undesirable outer layer of the fibres such as hemicellulose and lignin. Hemicelluloses are amorphous and hydrophilic and soluble in alkali solution. Lignin is known to provide structural strength towards the fibre. Lignin is amorphous and hydrophobic in nature and soluble in alkali solution (Ridzuan et al., 2016). Both hemicellulose and lignin can be removed by alkali solution. Once removed, the bulk lignin will be disrupted, causing disaggregation of micro fibril of the fibres (Yusriah et al., 2014b). This is concluded by some shrinkage of the fibres. The results of the shrinkage are shown in Figure 4.7 and the values are in Table 4.3.

The shrinkage values are almost similar for 6% alkali, 10% alkali and 12% alkali, which are between 54% to 55%. Fibre treated with 8% alkali shows shrinkage at 59%. The value of area reduction showed fluctuating pattern, it did not show a continual decrease pattern as the alkali concentration increase. This result is in agreement with the finding by Kabir et al. (2013) on hemp fibre. These reductions were due to fibre degradation and delignification of the treated fibres. Sugarcane fibre bundles were reported to show better lignin and hemicellulose removal at 5% alkali (Hossain et al., 2014; Yue et al., 2015), while Napier grass experienced 12% to 45% diameter reduction after alkali treatment (Ridzuan et al., 2016). Screw pine (*Pandanus Odoratissimus*) fibre reported the highest cross-sectional area reduction of 42.1% for alkali concentration of 15% (Abral et al., 2012). Kenaf fibre cross-sectional area was reported to show rapid decrement pattern when subjected to various immersion time, alkali concentration and temperature (Hashim et al., 2017).

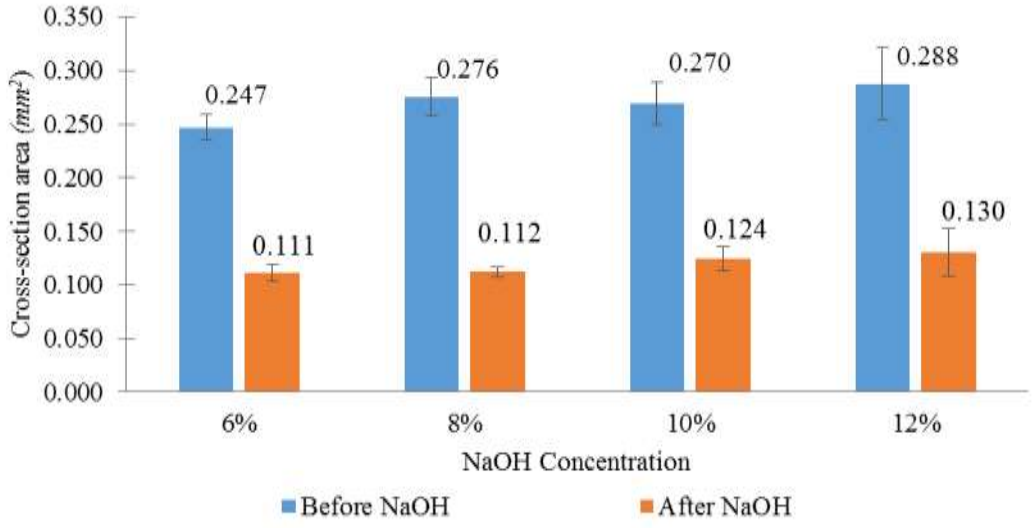


Figure 4.7: Fibre cross-section area before and after alkali treatment

Table 4.3: Fibre cross-section area shrinkage percentage

Alkali Concentration (%)	Cross section area shrinkage (%)
6	55
8	59
10	54
12	55

4.3.2 Chemical composition

The chemical composition of untreated fibre and alkali treated nipah fibre is as shown in Table 4.4. The composition of α-cellulose was slightly higher than hemicellulose content. As can be observed, the composition of α-cellulose and lignin is increased along with alkali concentration while the hemicellulose remained similar except for the 6% alkali

treated fibre. This finding is in agreement with the finding for napier grass, similar trend of the chemical composition before and after treatment was observed for napier grass (Reddy et al., 2012). The α-cellulose content was increasing as the amorphous structure had been removed by the alkali treatment and thus enabling more α-cellulose to be deposited on the fibre surface. This finding is supported by research work done by Valadez-Gonzalez et al. (1999b). The hemicellulose content was almost similar between untreated and treated fibres. The nipah frond fibre composition is comparable to nipah frond as presented in the literature (Tamunaidu & Saka, 2011). The α-cellulose content of treated nipah showed the value between 37% to 44%. Similar value was reported for coir fibres (Rout et al., 2001). Therefore, nipah fibres can be grouped into natural fibres with high mechanical properties.

Table 4.4: Chemical composition of nipah fibres before and after alkali treatment

NaOH Concentration (%)	α-Cellulose (% w/w)	Hemicellulose (% w/w)	Lignin (% w/w)
Untreated	27	23	21
6	37	26	24
8	37	24	24
10	36	23	24
12	44	23	29

4.3.3 Thermogravimetric Analysis (TGA)

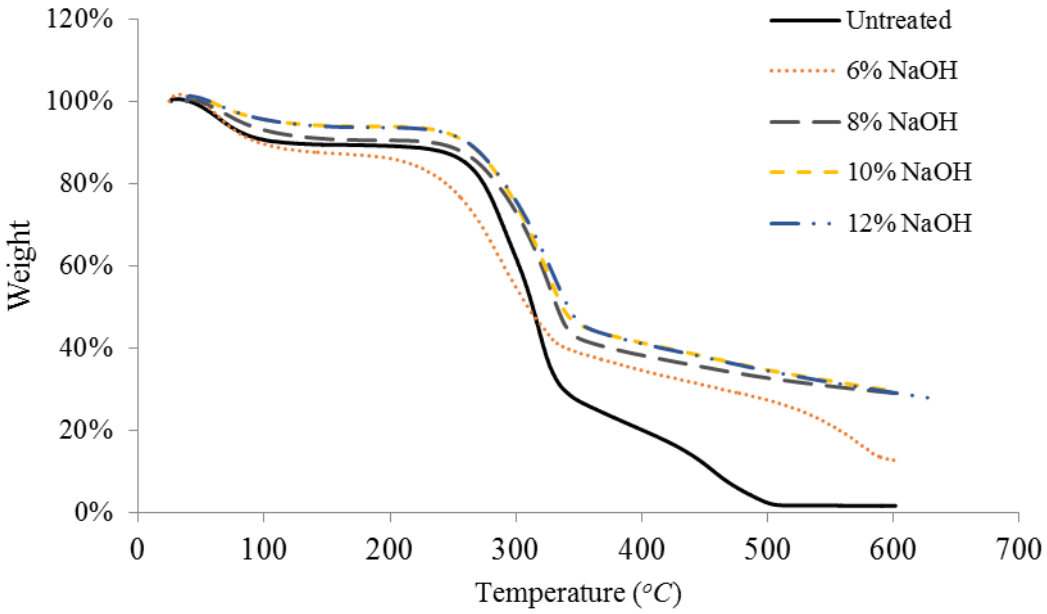


Figure 4.8: Thermogravimetric analysis of untreated and alkali treated nipah fibre

The thermogravimetric analysis result for untreated and alkali treated nipah fibres are shown in Figure 4.8. As can be observed, there are three main stages of weight loss for untreated and 6% alkali treated fibres, while 8%, 10% and 12% alkali treated samples show a two-stage graph. The weight losses at the range of 50 °C to 200 °C can be attributed to water in the form of absorbed moisture or combined water. The weight loss for untreated nipah fibre was 10%, while the treated nipah of 6%, 8%, 12% and 9% respectively. The 10% and 12% alkali treated both showed 6% weight loss. The weight losses improved as the alkali concentration gets higher, indicating thermally stable nipah. As a comparison, it was reported that the weight loss for luffa is 5%, flax (6.3%), wheat straw (7.3%) and jute (10.2%) (D'Almeida et al., 2006a). This stage represents moisture weight loss contributing to the sudden change drop, especially below 100 °C, where the water starts to evaporate above 100 °C. At the temperature of 200 °C to 300 °C, initial decomposition temperature

for untreated and 6% alkali is 280 °C and 260 °C respectively, while 8%, 10% and 12% alkali had the similar value of 320 °C. This value was close to that of luffa (250 °C), jute (282 °C), piassava (225 °C) and lignin (213 °C) (D'Almeida et al., 2006a).

The temperature when 50 wt% of mass has decomposed (D ½) was at 315 °C for untreated fibres and 310 °C, 338 °C, 340 °C, 342 °C for 6%, 8%, 10% and 12% NaOH treated respectively. The TGA showed that a residue of about 20% for untreated, 35% for 6% alkali, 38% for 8% alkali and 45% for both 10% and 12% alkali remains at 400 °C. These were similar to the value of 32.8% reported for lignin. The final residue at 600 °C for untreated was about 3% and 12% for 6% alkali treated fibres and similar values of 30% for 8%, 10% and 12% alkali treated fibres. More residues indicating that the alkali treatment has indeed increased the temperature stability of the treated fibres even at a higher temperature.

It was observed that the untreated line shows two decomposition steps, 300 °C and 470 °C and the 6% alkali treated fibres also shows two decomposition steps at 250 °C and 550 °C. The two steps exists may be due to partial removal of hemicellulose, lignin and waxes. However, at 8%, 10% and 12% alkali, it shows one decomposition step, which was the decomposition of α-cellulose and lignin. This indicates that a higher alkali percentage completely removes hemicellulose, lignin and waxes (Mwaikambo & Ansell, 2002). Similar observation was made in other natural fibres (Mwaikambo, 2008; Reddy et al., 2009; Wang et al., 2016). In order to probe further, spectral analysis were carried for the untreated and alkali treated fibres.

4.3.4 Fourier transform infrared spectroscopy (FTIR)

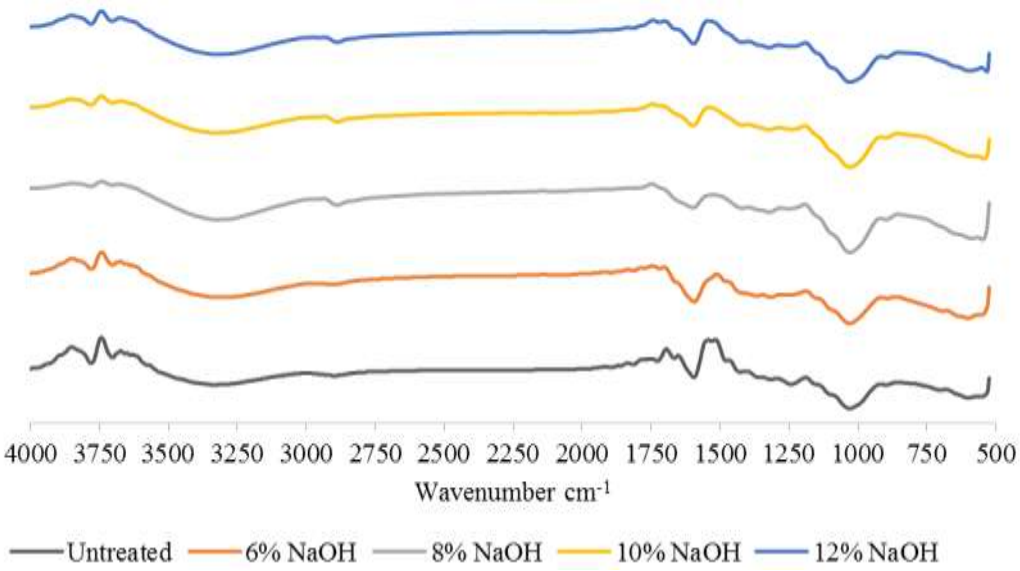


Figure 4.9: FTIR spectra of untreated and alkali treated fibres

The FTIR spectra of nipah palm fibre are as shown in Figure 4.9. Untreated, 6%, 8%, 10% and 12% alkali treated FTIR spectra show peaks dominated at 3335.03 cm^{-1}. These spectra could be attributed by O–H stretching vibration. The peak for treated nipah is broader and more intense indicating more –OH group existed due to the treatment. The peak at 1726.26 cm^{-1} for treated nipah fibre slowly disappears after the treatment with an increasing alkali aqueous solution. This is due to the removal of acid, lignin and other natural fibre constituents (Sain & Panthapulakkal, 2006; Valadez-Gonzalez et al., 1999b).

Hydrolysis occurs after the alkali treatment. This breaks down the ester bond, which explains the disappearance of 1726.26 cm^{-1} peak. The disappearance of the 1726.26 cm^{-1} and 1665.81 cm^{-1} peaks after alkali treatment indicates that either the carboxylic acid and acetyl groups were destroyed by the alkali treatment or the macromolecules containing

these functional groups were selectively dissolved from the fibre bundles under strong alkali condition. The peaks observed the 1000 cm^{-1} and 1500 cm^{-1} range show the presence of hemicellulose (Mwaikambo & Ansell, 2002). Based on peaks for nipah fibre between untreated and 6%, 8%, 10% and 12% alkali treated, the intensity of the peaks is slightly reduced, which indicates a slight removal of hemicellulose compounds, while the peaks at 1031.20 cm^{-1} and 1030.90 cm^{-1} of the untreated and treated fibre indicate the stretching vibrations of C-O. The peak at 1241.48 cm^{-1} can also readily assigned to the C–O stretching mode of acetyl groups in lignin (Cai et al., 2016). All the major peaks of nipah palm fibre are similar with the findings found by Rasidi et al. (2014). The peaks are shown in Table 4.5.

Table 4.5: Functional groups of *Nypa fruticans* (Rasidi et al., 2014)

Nypa fruticans	
Wave number (cm^{-1})	**Functional group**
3339	Hydroxyl group (-OH) of NF and absorbed moisture
2919	C-H stretching vibration
1727	Carboxyl (C=O) from hemicellulose
1607	C=C stretching from hemicellulose
1515	C=C stretching from lignin
1440	CH_2 group deformation from cellulose or C-H deformation of lignin
1368	C-H group deformation in cellulose and hemicellulose
1315	C-H group vibration in cellulose
1243	C-O-C groups from acetyl group in lignin
1000-1150	C-O-C and C-O groups from main carbohydrates of cellulose and lignin
700-900	C-H vibration in lignin

4.3.5 X-ray diffraction (XRD)

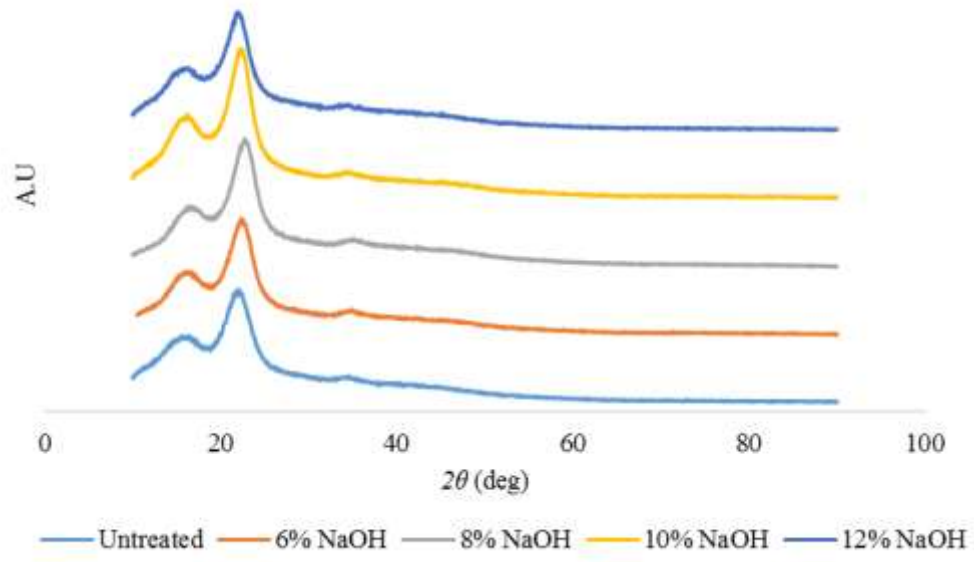


Figure 4.10: XRD of untreated and alkali treated fibres

The diffractogram obtained for nipah fibres are shown in Figure 4.10. The major peaks observed for all fibre samples were at 2θ diffraction angles of 16°, 22° and 35°. The overall pattern shows amorphous structure of the nipah fibres. The sharpest peak was observed for 8% alkali, indicating highest crystallinity value of 50%, due to the removal of amorphous structure (Júnior et al., 2015). This result is supported by the single fibre tensile strength, where the 8% alkali depicted the highest tensile strength as shown in Figure 4.12(a). The alkali treatment has removed fractions of amorphous in the fibre, leaving behind the crystalline structures in the fibres. This explains the sharpest peak at 8% alkali. The crystallinity index (CI) of the untreated and treated nipah fibres is shown in Table 4.6, the calculation was based on the Segal Empirical method (Segal et al., 1959). The degree of crystallinity of untreated fibre was 39.7% and the 8% alkali treated fibre was 50%. This shows that the percentage of crystallinity index of treated 8% alkali fibre was 11% higher

than the untreated nipah fibre. The increase of the percentage of crystallinity contributes to the enhancement of the tensile properties of the 8% alkali nipah fibre due to the restructuring of cellulose (Haameem et al., 2016).

4.3.6 Differential scanning calorimetry (DSC)

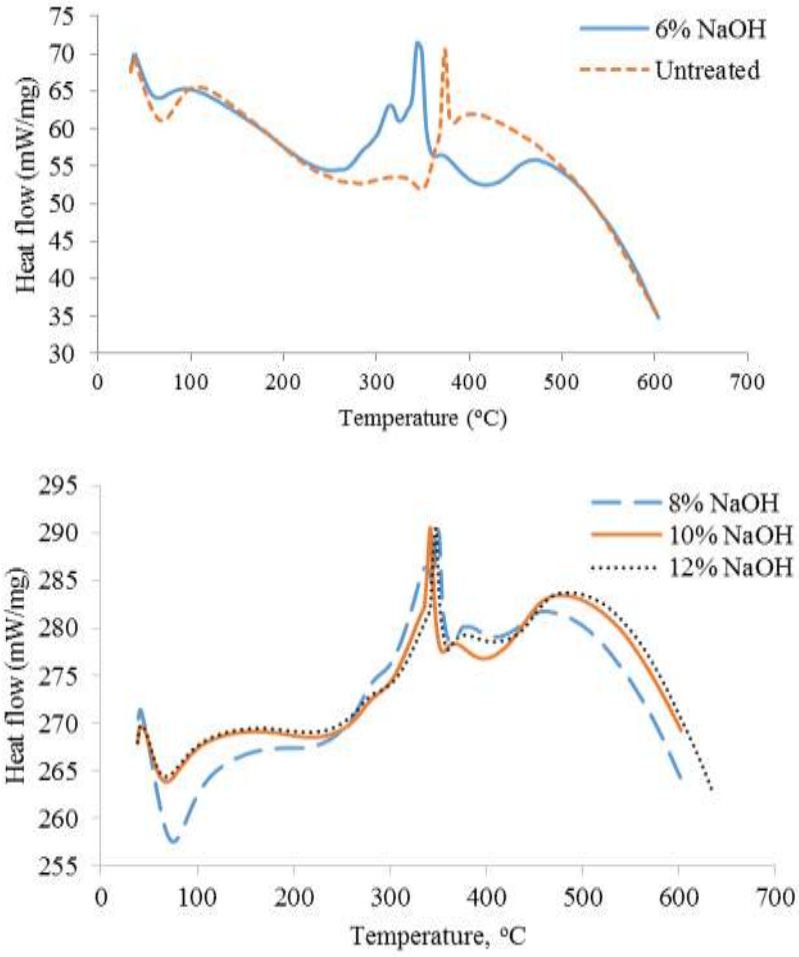
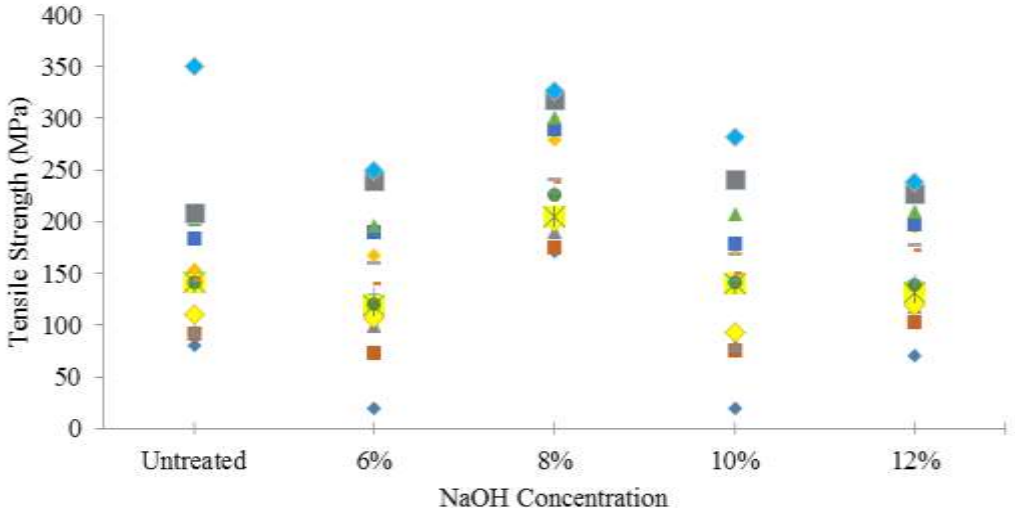


Figure 4.11: Differential Scanning Calorimetry

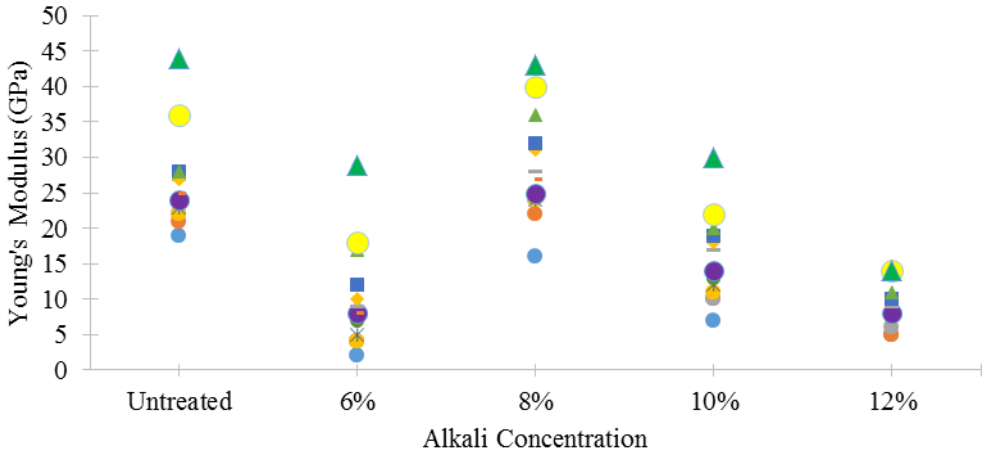
Figure 4.11 shows the thermal response of untreated and treated nipah palm fibre as a function of temperature. Untreated fibres showed a broad endothermic peak at 62 °C and

exothermic peak at 380 °C. The endothermic peaks corresponded to the glass transition temperature (T_g) of the fibres. The 8% alkali treated fibres showed higher endothermic peak at 80 °C and lower exothermic peak at 345 °C. Higher T_g was due to the removal of lignin and hemicellulose through the alkali treatment (Lin et al., 2018). The variation of the T_g result was due to the evaporation of absorbed moisture after the alkali treatment, therefore, the amount of absorbed moisture depends on the moisture resistance of the treated fibre. This result implied that the thermal stability of treated fibres was improved by the alkali treatment. However, the exothermic peak of treated fibre showed the opposite result where the treated fibre showed greater temperature stability as compared to all the treated fibres. This could be the plant factor, as the value gets even lower as the alkali concentration was increased.

4.3.7 Tensile strength



(a)



(b)

Figure 4.12: (a) Average single fibre tensile strength (b) Young's Modulus

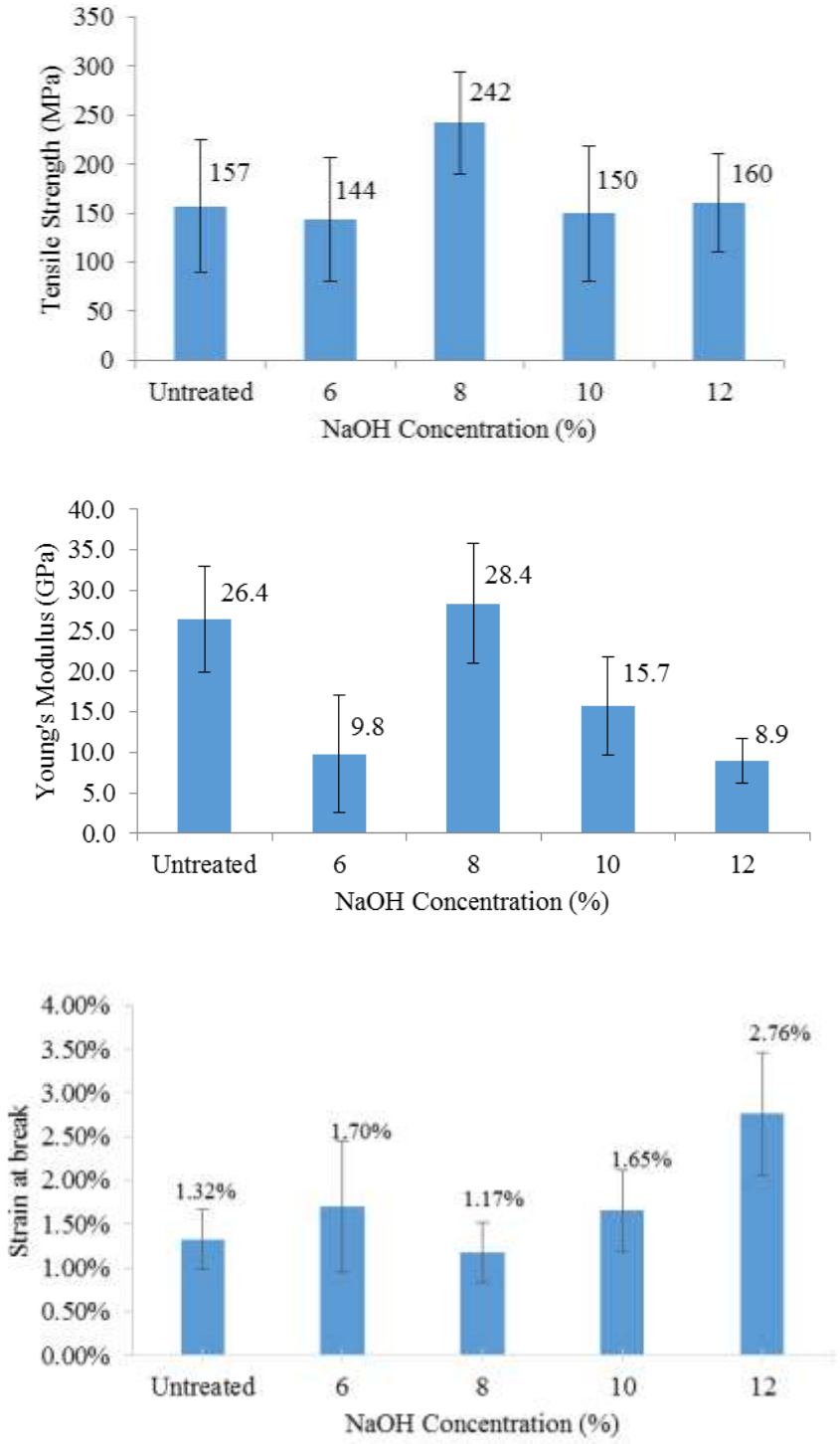


Figure 4.13: Mechanical properties of nipah fibres before and after alkali treatment (a) Tensile strength (b)Young's modulus (c) Strain at break

The tensile strength as depicted in Figure 4.12(a) shows the overall tensile strength between untreated and treated single fibres. All the symbols show individual result from each of the test. This shows the variation of the result instead of the average result. The result clearly showed that 8% alkali treated fibres had a higher tensile strength compared to the other treated fibres. The alkali treatment caused the removal of non-cellulosic content in the fibre. This removal allowed the cellulose to position itself when subjected to loading and facilitate better load transfer (Vardhini et al., 2016). The cellulose chains were no longer in constraint state, therefore the fibrils were able to position itself towards closer packing arrangement to improve fibre strength and tensile properties (Reddy et al., 2012). However, at higher alkali concentration, the tensile strength drops. This was due to fibre damage caused by extreme reaction of the alkali towards the fibres. This could also due to the existence of deep pores at higher alkali concentration. The increase number of deep pores leads to the decreasing of tensile strength (Han & Choi, 2010). The tensile properties of untreated and treated nipah fibres are summarised in Figure 4.13 and Table 4.6.

As observed in Figure 4.13(b), the Young's modulus values of treated nipah fibres were generally lower than the alkali treated fibres. However, the 8% alkali treated fibres were comparable to the untreated fibres. The alkali treatment was expected to remove the lignin from the fibres. This can be observed in the FTIR result in Figure 4.9. However, the chemical analysis test conducted on the fibres showed increment of lignin value. Lignin composition was also found to increase with the alkali percentage in napier grass (Reddy et al., 2012). A study had reported other method that was able to effectively remove lignin in sugarcane bagasse (Wunna et al., 2017). Lignin acts as cementing materials that provides stiffness to fibre (Wong et al., 2010; Yusriah et al., 2014a). This explains the similarity between untreated and treated fiber in terms of its stiffness. At higher alkali percentage, the

lignin fails to improve the stiffness as fibre breakage was obvious. Figure 4.13(c) shows the strain at break for treated nipah fibres. The strain at break for 6%, 10% and 12% alkali were higher than the untreated nipah fibres and has almost the similar value of the 8% alkali. This showed that the fibres were affected by the alkali treatment. The treatment resulted in a less dense and rigid fibres (Yusriah et al., 2014a). The tensile strengths of other natural fibres are in Table 4.6. The closest mechanical properties with nipah fibre was napier grass, the 6% alkali nipah was comparable with 5% alkali napier grass. Comparison was made with napier grass, due to the fact that napier grass was fairly new fibre investigated as reinforcing agent and napier grass used in the investigation was grown in Malaysia. Betel nut, coir and kenaf properties were also within the range of nipah fibre properties. Betel nut and coir are from the same palmae family with nipah palm.

Table 4.6: Mechanical properties of nipah fibres before and after alkali treatment.

Alkali Concentration	Tensile Strength (MPa)	Young's Modulus (GPa)	Strain at break (%)	Crystallinity Index (%)	Reference
Untreated	157	26.4	1.32	39.7	-
6	144	9.8	1.70	42.3	-
8	242	28.4	1.17	50.0	-
10	150	15.7	1.65	42.0	-
12	160	8.9	2.76	43.9	-
Napier Grass (5% alkali)	141	4.86	2.30	-	Ridzuan et al. (2016)
Betel Nut	124	1.28	22.49	-	
Coir	106 – 175	4 – 6	17 – 47	-	
Kenaf	293 – 920	22 – 60	2.7 – 6.9	-	

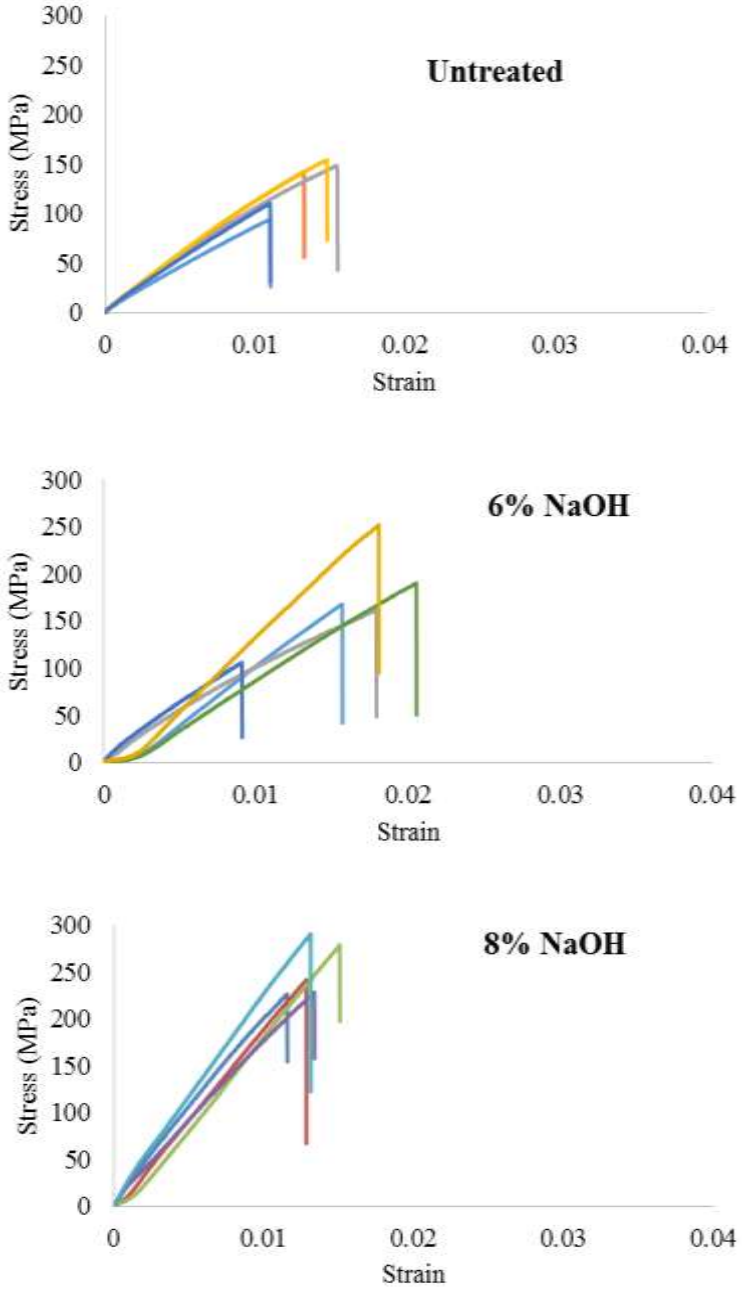


Figure 4.14: Stress-strain diagram for single fibre tensile test of the untreated fibres, alkali treated (6%, 8%, 10%, 12%) and the stress-strain summary.

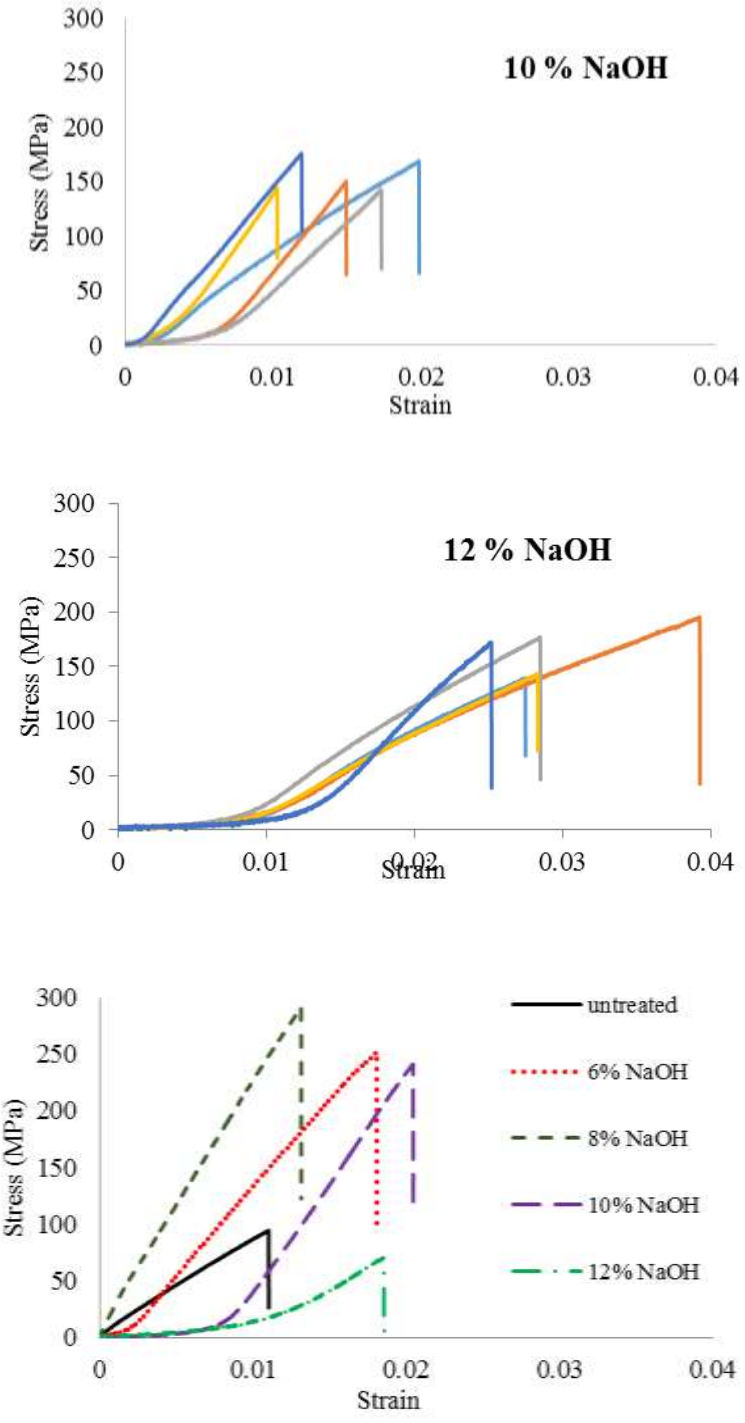


Figure 4.14: Stress-strain diagram for single fibre tensile test of the untreated fibres, alkali treated (6%, 8%, 10%, 12%) and the stress-strain summary. (continued)

The stress-strain curve for 6%, 10% and 12% alkali treated fibres displayed a non-linear behaviour, while the stress-strain curve for 8% alkali treated fibres shows a linear behaviour with higher strength as compared to the untreated fibre as depicted in Figure 4.14. It can be observed that the non-linear starts to show at 10%, 12% alkali and a few at 6% alkali. This could be attributed by the condition of the treated individual fibres as shown in Figure 4.6. The 10% and 12% alkali fibres were twisted and curly. This leads to the toe region at the beginning of the tensile test. This behaviour was also observed in single fibre test of abaca fibres (Cai et al., 2016, 2015).

4.3.8 Morphological study

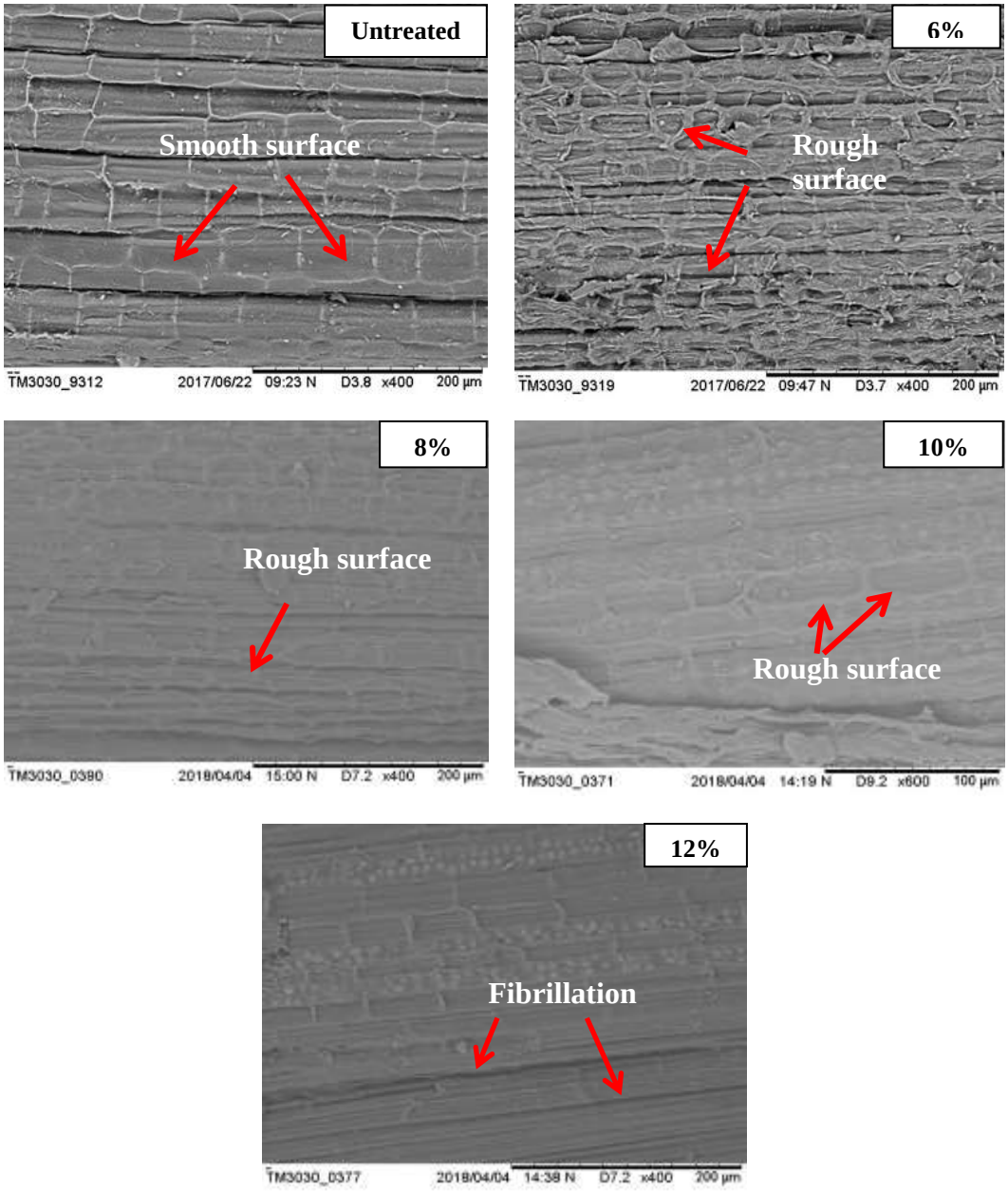


Figure 4.15: SEM images of untreated and alkali treated fibres (fibre surface)

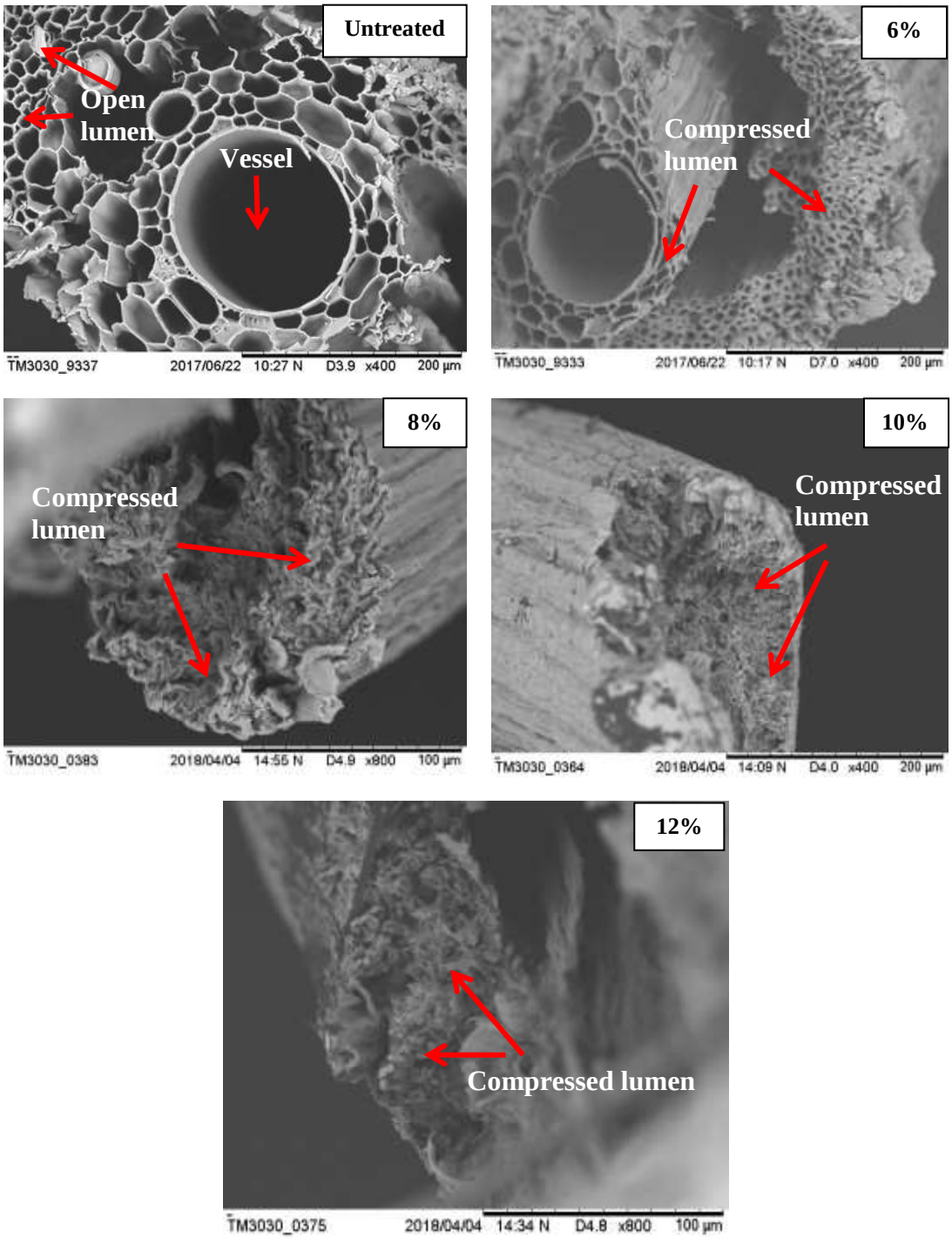


Figure 4.16: SEM images of fracture surface of untreated and alkali treated single fibres

The SEM images in Figure 4.15 show the effect of alkali treatment towards nipah fibre surface. As can be observed, the fibre surface was naturally rough. The 6% alkali treated fibres show a rougher fibre surface, and the surface somehow shows that it had been eroded by the alkali solution, leaving behind a concave surface. At 8% alkali the fibre surface appeared smoother, and the alkalisation was reported to modify the fibre surface and made the surface looks smoother (Valadez-Gonzalez et al., 1999; Wong et al., 2010). As the alkali concentration gets higher, the alkali treatment made the fibre surface looks smoother, but actually the surface had been roughened (Aziz & Ansell, 2004). This can be observed in Figure 4.15 of the 8%, 10% and 12% alkali treatment. Rougher surface will enhance the interlocking of fibres and matrix in composite fabrication.

As for the fracture surface of the fibres shown in Figure 4.16, they were almost similar, literary not flat surface, basic criteria for fibrous material. The vessel of the fibre for untreated and 6% alkali was round at the centre and the fibre lumen was elliptical for nipah fibre, and this can also be observed in bagasse fibre (Guimarães et al., 2009). The presence of vessel and lumens at the fibre surface explained the light weight of the fibre. The vessel had a very smooth internal surface. The elliptical lumen for treated fibres showed some compression. However, no change was observed for the vessel, and this might be due to the location of the vessel deep inside the fibre, which was affected by the alkaline solution. The compressed lumen could be due to the sodium ion deposited on the wall, subsequently increasing the thickness and thus, reduces the lumen size (Cai et al., 2015). The compressed lumen reduces the void content and fibre water absorption (Reddy et al., 2012; Ridzuan et al., 2016). The compressed lumen was obvious in the 8%, 10% and 12% alkali treated fibres, and the similar result was also observed in abaca treated fibres (Cai et al., 2016).

Fibrillation could be observed in 12% alkali treated fibre, and the strong alkali concentration removed the binding material at the primary nipah fibre bundle. This explained the low tensile strength of the 12% alkali fibres. The fibrillation in treated fibres could increase the surface contact areas for matrix interlocking as reported by Abral et al. (2012). However, for nipah fibre, fibrillation was only observed clearly in the 12% alkali fibres and this leads to poor tensile strength result. Fibrillation could exist in other treated nipah palm fibre, but its existence may not be obvious to be observed and captured by SEM.

4.4 Effect of alkali treatment towards Fibre A composites

The effect of alkali towards Fibre A was investigated by analysing its impact on tensile strength, fracture morphology and water absorption characteristic.

4.4.1 Tensile strength

The variation of tensile strength as a function of fibre loading and the alkali concentration is presented in Figure 4.17. It was observed that the 5W/10N and 15W/8N composites showed tensile strength at 29.36 MPa and 28.29 MPa respectively as in Table 4.7.

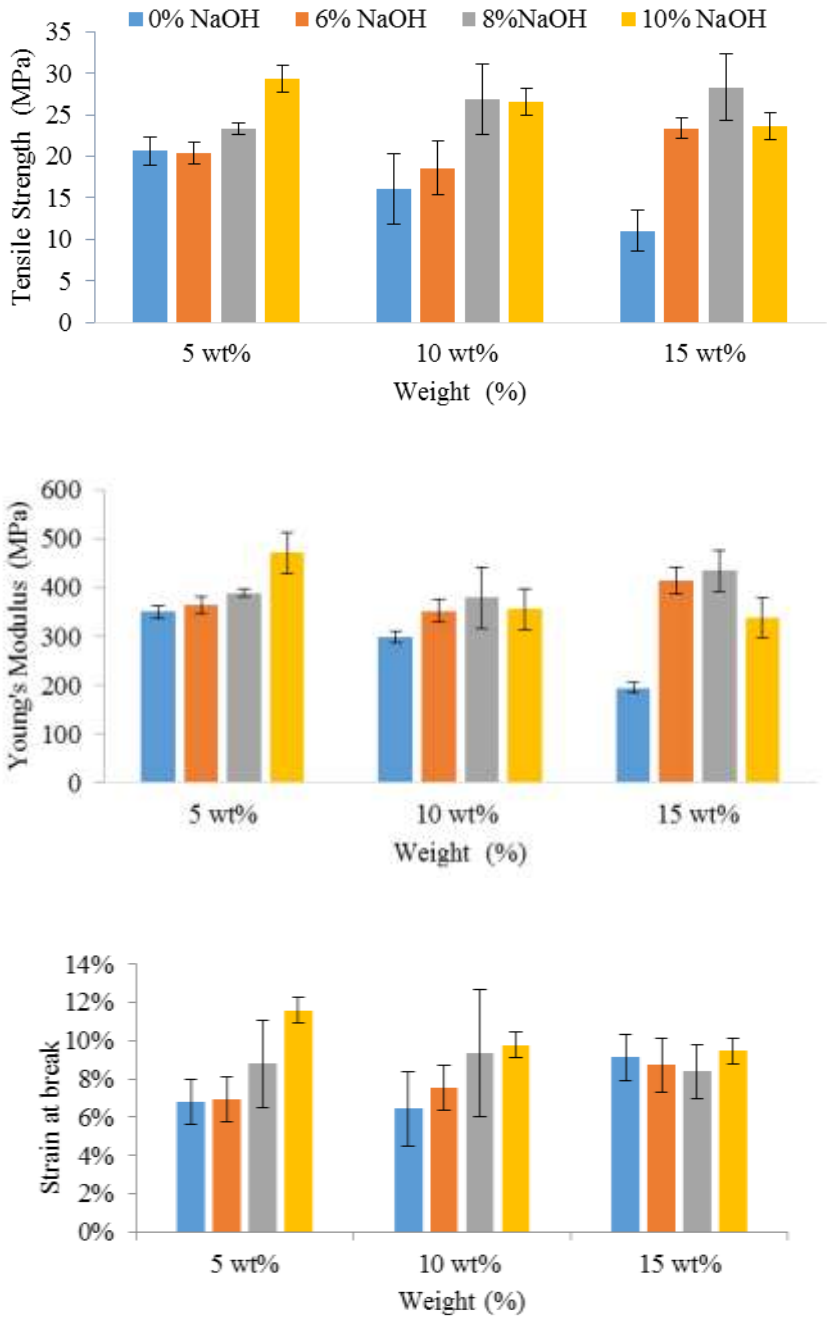


Figure 4.17: Mechanical test result for Fibre A

Similar pattern was observed for the Young's modulus value for the composites, and the highest was contributed by the 5W/10N composites at 471 MPa as shown in Table 4.8. The strain at break in Table 4.9 indicates that the 15W/8N shows the highest value of 11.58 %. There was an improvement of 42% of tensile strength, 35% of Young's

modulus value and 70% of strain at break between 5W/0N composites and 5W/10N composites.

It was noted that the 10% alkali treated fibre composites at a lower loading condition produced the best tensile strength, whereas, the 8% alkali treated fibre composites produced the best result at the highest loading condition. The single fibre test result showed 8% alkali had the highest tensile strength value and it was expected that the nipah fibre reinforced polyester composites would yield the same result. However, the result showed the opposite. It was believed that the 5W/10N composites had better interlocking and load transfer between fibres and polyester matrix. At higher loading the fibre direction might not be favourable to the load direction and packed fibre agglomeration may result to improper wetting condition. It was reported that alkali treatment can have different effect towards the mechanical properties of the fibres and its composites when they were used as reinforcement agent (Mwaikambo & Ansell, 2002). This could be the reason of fibre strength variation. Since the single fibre test of the 8% alkali showed the highest value, its composites strength showed a significant increase from 5 wt% to 15 wt % loading. The 10% alkali nipah fibre reinforced polyester composites showed a reduction in strength as the loading gets higher. It was believed that a rougher fibre with a lower loading leads to a greater strength, but as the amount of fibre increases, it leads to improper wetting condition. From this result, it should be noted that the incorporation of nipah fibre failed to reinforce polyester composites as compared to the neat polyester strength as in Table 4.10. There were a few possible factors contribute to this result. First, the existence of air bubbles in the fabricated composites as the polyester mixture was not vacuum prior to pouring into the mould. Second, the brittleness of the polyester might leads to the formation of micro cracks during cutting process. The samples

were hand saw, even though great care was taken into account during the cutting process. Third, the compatibility between nipah palm fibre and the polyester matrix may not ideal to provide the reinforcement to the composites, other type of polymer should be investigated. Fourth, the fabrication method, hand lay-up may not suitable to produce this composite. A good cold or hot press machine that is able to maintain its pressure for 24 hr and equip with a vacuum pump to suck out all the air bubbles during the pressing process might produce a good plate of composite. However, the alkali treatment had successfully increased the tensile strength of the composites compared to the untreated nipah fibre composites.

This observation was also observed in other natural fibre polyester composites (Boynard & D'Almeida, 2000; Melkamu et al., 2018). It was reported that coir fibre has to be loaded up to 45% or higher in order to obtain a significant reinforcing effect, and lack of reinforcing effect was due to low modulus of elasticity as compared to neat polyester (Monteiro et al., 2008). Piassava fibres reinforced epoxy composites was reported to show similar findings as the composites failed to reinforce up to 30% of volume fraction (Nascimento et al., 2010). The napier grass fibre was reported to reinforce the polyester composites at a value of 15.64 MPa for short fibre. The tensile strength of the pure polyester is 10.1 MPa (Haameem et al., 2016). The strength value obtained in nipah fibre composites was much higher than the value reported by napier grass. However the alkali treatment had indeed improved the wetting between nipah fibres and polyester matrix. The removal of surface impurities, non-cellulosic material and waxes resulted in cleaner and rougher fibre surface and improves the composite strength. This observation was observed somewhere else (Abral et al., 2012; Osorio et al., 2012).

The stress-strain diagrams for 5W/10N and 15N/10N composites, as well as the stress-strain diagram for neat polyester are shown in Figure 4.18. As can be observed, the stress-strain diagram for the composites were more on the brittle manner and lack of elasticity, and a similar finding was reported by Monteiro et al. (2008).

Table 4.7: Tensile strength (MPa) of nipah fibre reinforced polyester composites

Composite loading (wt %)	Untreated	6% NaOH	8% NaOH	10% NaOH
5	20.64 ± 1.64	20.38 ± 4.29	23.31 ± 2.47	29.36 ± 1.29
10	16.09 ± 1.34	18.59 ± 3.24	26.85 ± 1.28	26.58 ± 2.50
15	11.04 ± 0.73	23.39 ± 4.26	28.29 ± 4.03	23.60 ± 0.46

Table 4.8: Modulus Young's (MPa) of nipah fibre reinforced polyester composites

Composite loading (wt %)	Untreated	6% NaOH	8% NaOH	10% NaOH
5	350 ± 13	364 ± 11	389 ± 11	471 ± 33
10	298 ± 17	353 ± 23	379 ± 28	356 ± 11
15	195 ± 7	414 ± 64	434 ± 41	338 ± 27

Table 4.9: Strain at break (%) of nipah fibre reinforced polyester composites

Composite loading (wt %)	Untreated	6% NaOH	8% NaOH	10% NaOH
5	6.81 ± 1.17	6.92 ± 1.94	8.80 ± 1.22	11.58 ± 0.72
10	6.44 ± 1.19	7.54 ± 1.15	9.36 ± 1.39	9.77 ± 0.95
15	9.12 ± 2.30	8.72 ± 3.31	8.38 ± 1.42	9.46 ± 0.80

Table 4.10: Mechanical properties of neat polyester

	Tensile strength (MPa)	Young's Modulus (MPa)	Strain at break (%)
Neat polyester	36.44 ± 4.02	372 ± 42	28.40 ± 2.2

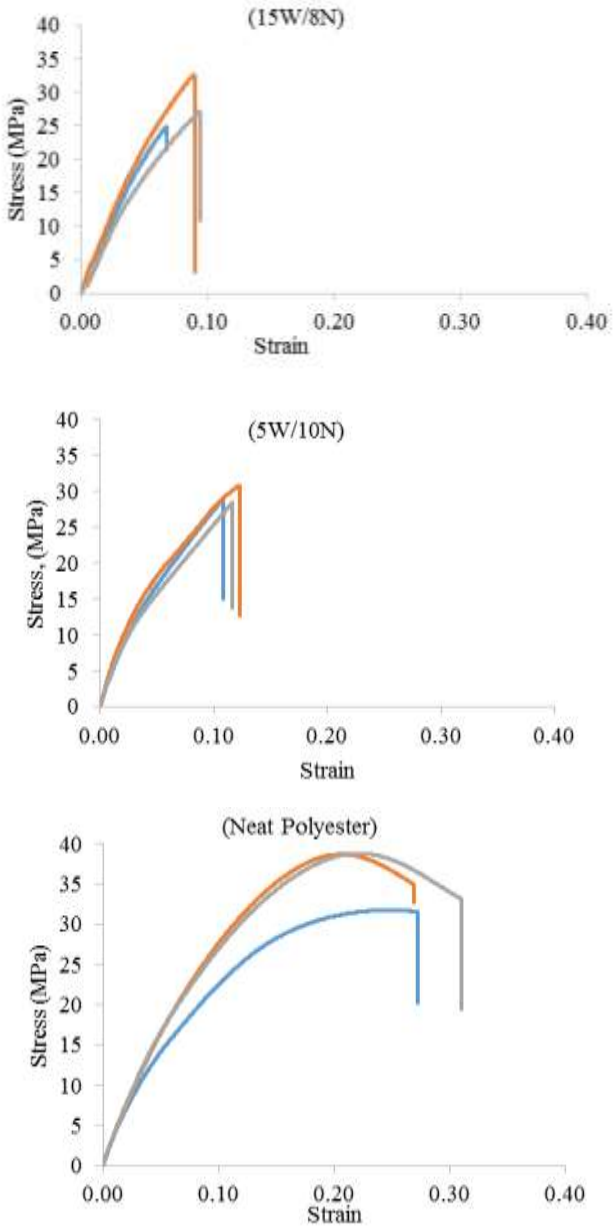
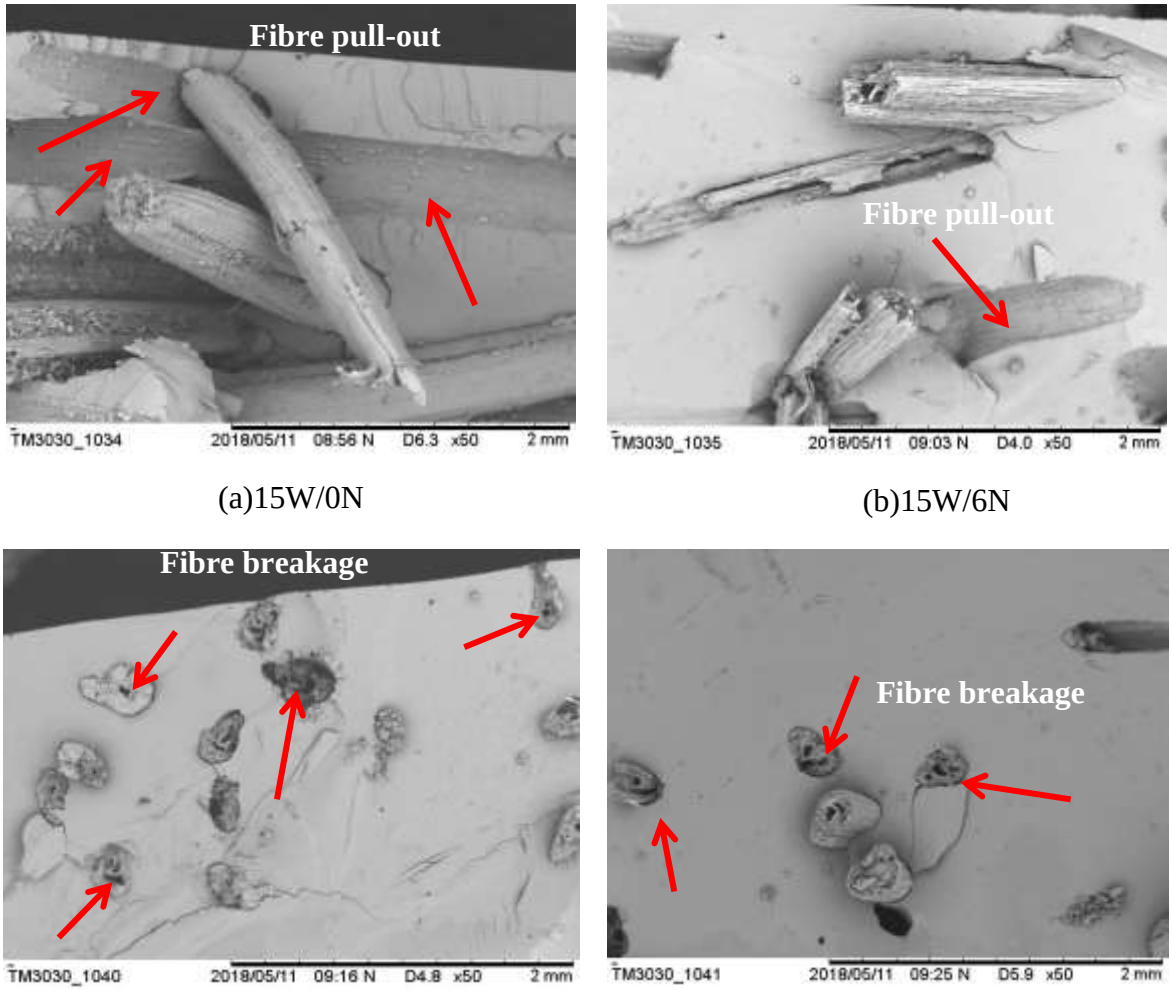


Figure 4.18: Stress-strain behaviour of nipah fibre composites and neat polyester

4.4.2 Fracture surface morphology



(a)15W/0N (b)15W/6N

(c)15W/8N (d)15W/10N

Figure 4.19: SEM images Fiber A composites (fracture surface)

As can be observed from Figure 4.19, there was a great difference between untreated and treated fibres and its composites. Poor interfacial bonding between untreated fibres and matrix was obvious, and it failed mainly due to this failure mode as shown by arrows in Figure 4.19(a). Treated fibres showed improvement in the interfacial bonding as the tensile strength had doubled the value of untreated fibres. The composites mainly failed due to fibre breakage even though some fibre pull-out was observed as shown by arrows in

Figure 4.19(c) to Figure 4.19(d). The matrix fracture surface showed a flat fracture mode, and this indicated that the matrix had undergone a brittle failure.

4.4.3 Water absorption characteristic

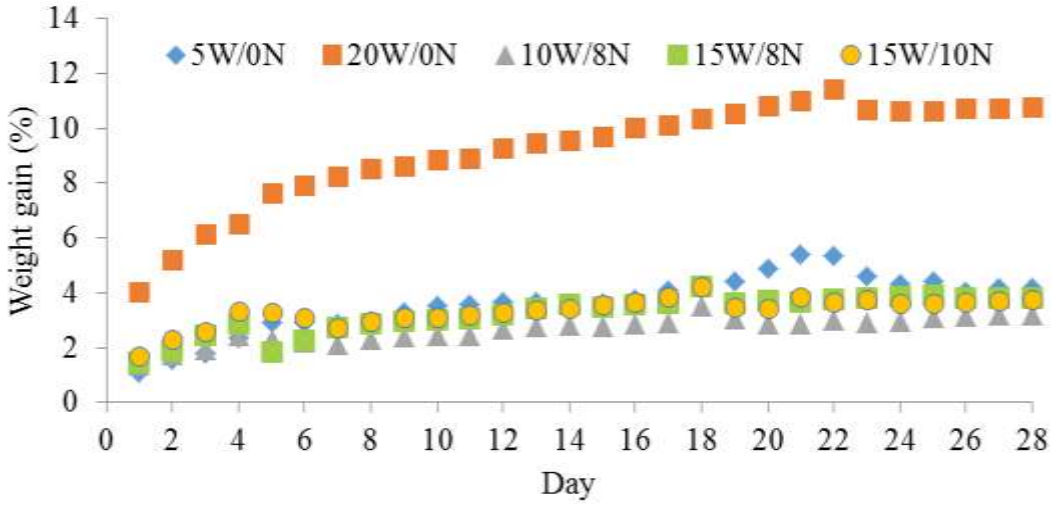


Figure 4.20: Result of water absorption test

The water absorption curve tested in distilled water at normal room temperature (28 ^{o}C) is presented in Figure 4.20. The results showed an increase in total absorption for up to 28 days, and as can be observed, the weight increased as a function of its fibre loading. This was expected as natural fibre like nipah fibre is hydrophilic in nature. The water absorption of nipah fibre composites followed the Fickian's behaviour, and this was also observed in kenaf fibre reinforced polyester composites as reported by Akil et al. (2011). The highest fibre loading conducted was at 20 wt% of the untreated fibres. There was an increase of 7% weight gain between the 15 wt% and 20 wt% fibre loading.

It should be noted that the untreated fibres had a maximum peak at day 22 while the treated fibres had its maximum peak at day 18. The surface treatment has shortened the peak period for treated fibres. The treated fibres regardless of its loading condition, its weight gain curve falls below the untreated fibres. The alkali treatment had significantly reduced the water absorption rate in treated fibres. It had created a protective layer on the fibre surface that was resistant to water absorption. This result is supported by the XRD result in Table 4.6. The crystallinity index had increased the crystallinity of the treated fibres, resulting in better packing arrangement of the cellulose chain and thus creating a shield for the treated fibres (Symington et al., 2008). The compressed lumen of treated fibres also reduced the water absorption capacity. It was also reported in the literature that alkali treated fibres showed a compressed lumen structure, and this caused the reduction of void content and less water absorption capacity of the treated fibres (Cai et al., 2015; Reddy et al., 2012; Ridzuan et al., 2016).

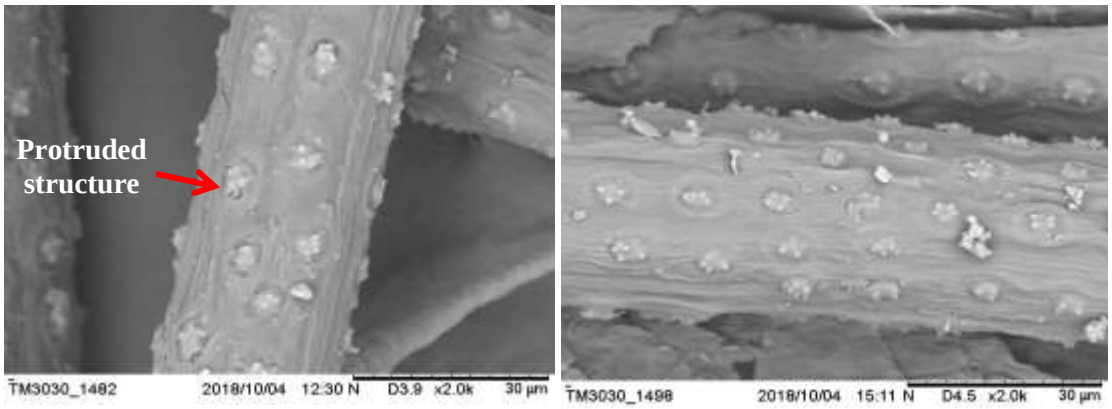
4.5 Effect of alkali treatment towards Fibre B and its composites

The effect of alkali treatment towards Fibre B and its composites was investigated by analysing its impact on physical appearance, tensile strength and fracture morphology.

4.5.1 Physical appearance

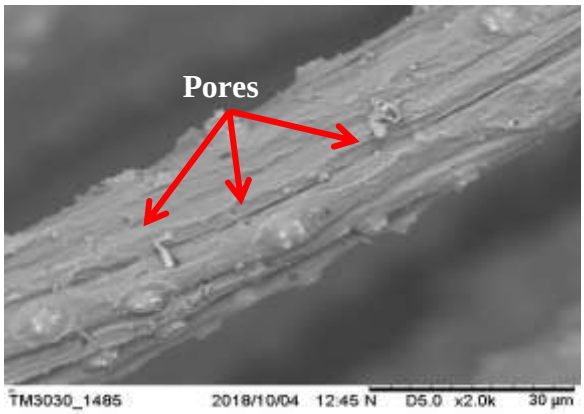
Figure 4.21 shows the physical appearance of Fibre B, viewed under SEM. It is revealed in this figure that the untreated fibre appears smooth, whereas, the treated fibres show a fibrillated structure and its pores were obvious as in the 4% alkali fibre. The

protruding surface of Fibre B was at a consistent interval throughout the entire fibre. The structure was expected to contribute to a better interlocking mechanism in polymer composites. As can be observed, the treated fibres were coarser compared to the untreated fibre. This observation was also reported by Mwaikambo and Ansell. (2006) for hemp fibres. As for the 4% alkali fibre, the existence of pores was obvious as shown arrows and no pores are visible in the 2% alkali. The severity of surface reaction increases as the concentration of alkali increases (Alawar et al., 2009). The increase number of pores in 4% alkali composites was expected to decrease the single fibre strength. However, this was beneficial in terms of better interlocking in its composites (Yusriah et al., 2014a).



Untreated

2% NaOH



4% NaOH

Figure 4.21: SEM images of Fibre B

4.5.2 Tensile strength

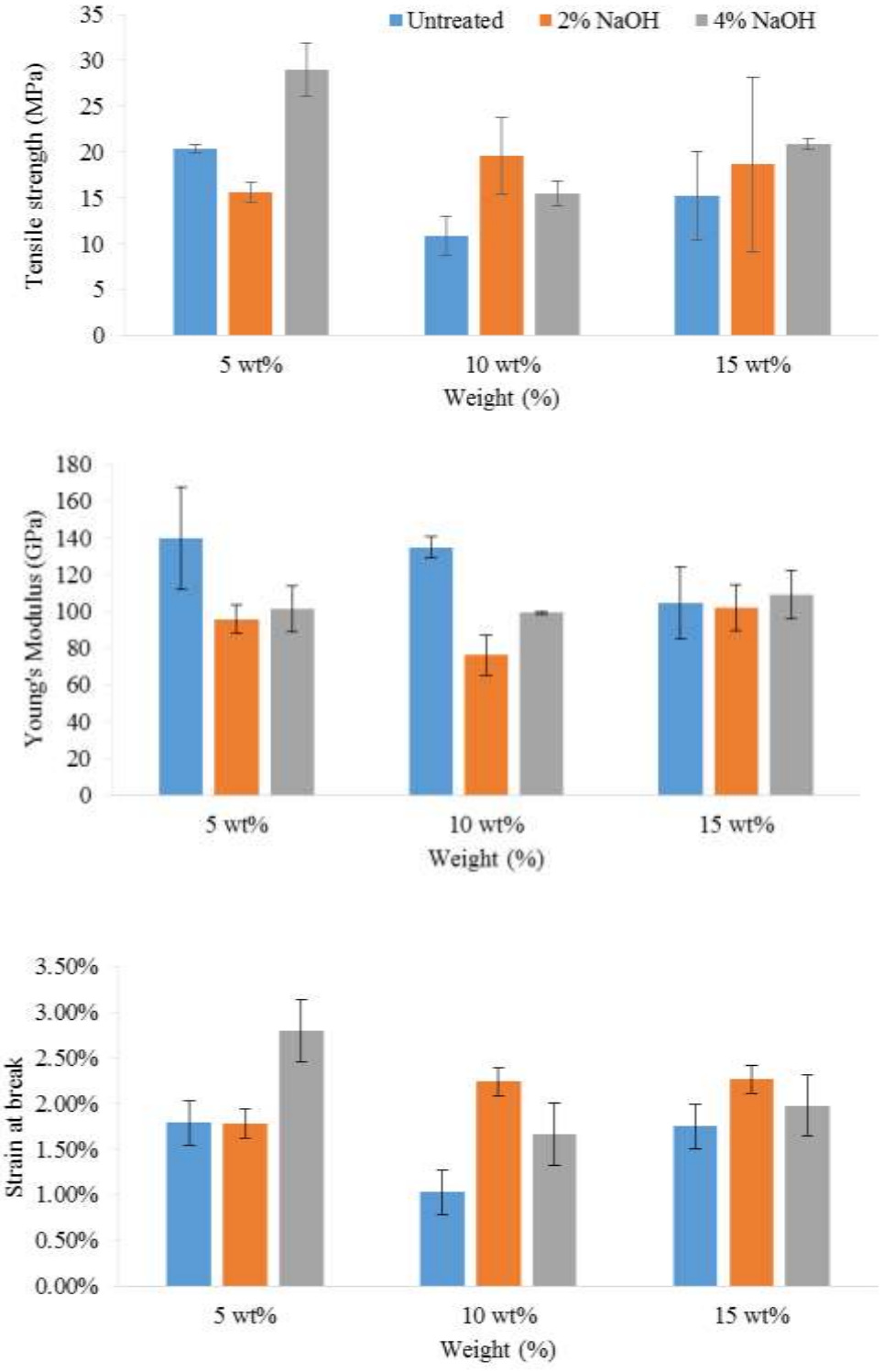


Figure 4.22: Mechanical test result of Fibre B

As can be observed from Figure 4.22, the tensile strength of 5W/4N composites contributes to the highest strength at 29.67 MPa as in Table 4.11, which was 45% improvement from the 5W/0N composites. The highest Young's modulus value was from the untreated fibre, 5W/0N at 140 MPa as in Table 4.12. Strain at break for 5W/4N showed the highest value at 2.8% as in Table 4.13.

It was apparent that the strength value for the untreated and 4% alkali composites reduces as the loading increases, even though the value actually fluctuated. However, for the 2% alkali composites, the strength increases as the loading increases.

The strength fluctuation pattern could be due to the fibre distribution within the polymer matrix. An observation through SEM reveals that fibre agglomeration was observed in most composites. The agglomeration of fibres especially for Fibre B was due to its extremely fine fibres, and when resin was poured during fabrication, these fibres were most likely to agglomerate. This might due to the formation of hydrogen bond between hydrophilic fibres (Raj et al., 1989). Agglomeration also caused poor load transfer and acted as stress concentration region. The result of Fibre B composite strength of 5W/4N was similar with Fibre A composite of 5W/10N. This indicated that 10% alkali treatment towards Fibre A was needed to produce a comparable surface roughness to 4% alkali of Fibre B that would improve the interlocking regardless of its fibre diameter. In other words, higher alkali concentration was needed to treat Fibre A in order to produce comparable results.

The stress-strain diagram in Figure 4.23 shows the 5W/4N composite with the highest strength value. The other diagrams were plotted based on its loading to show the effect of alkali concentration towards the untreated fibre composites. The treated fibre

composites showed a better strength compared to the untreated fibres as discussed earlier. The findings of Fibre B composites were similar with Fibre A composites, showing a brittle failure pattern. However, Fibre B composites were more elastic compared to Fibre A.

Table 4.11: Tensile strength (MPa) of nipah fibre B reinforced polyester composites

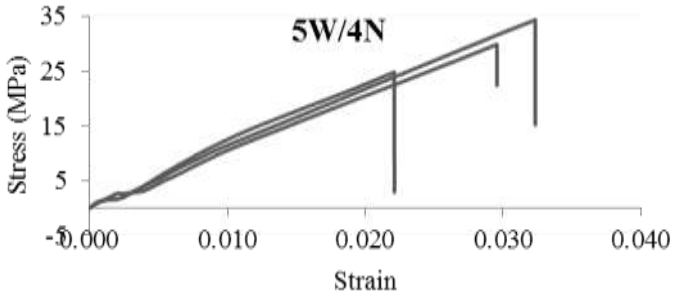
Composite loading (wt %)	Untreated	2% NaOH	4% NaOH
5	20.35	15.56	29.67
10	10.87	19.59	15.44
15	15.24	18.63	20.86

Table 4.12: Young's modulus (MPa) of nipah fibre B reinforced polyester composites

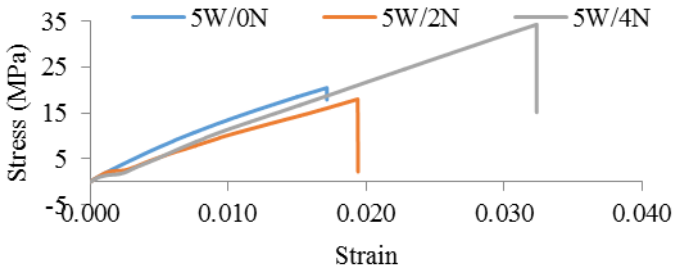
Composite loading (wt %)	Untreated	2% NaOH	4% NaOH
5	140	96	102
10	135	76	99
15	105	102	109

Table 4.13: Strain at break (%) of nipah fibre B reinforced polyester composites

Composite loading (wt %)	Untreated	2% NaOH	4% NaOH
5	1.787	1.782	2.801
10	1.031	2.239	1.659
15	1.751	2.266	1.979



(a)



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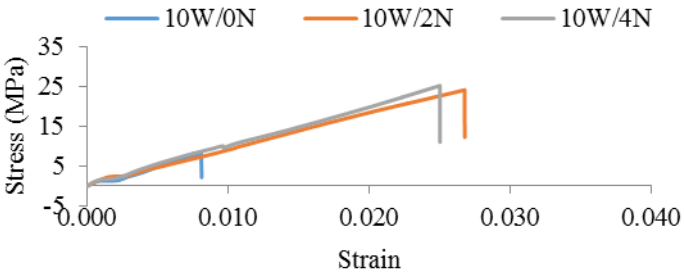
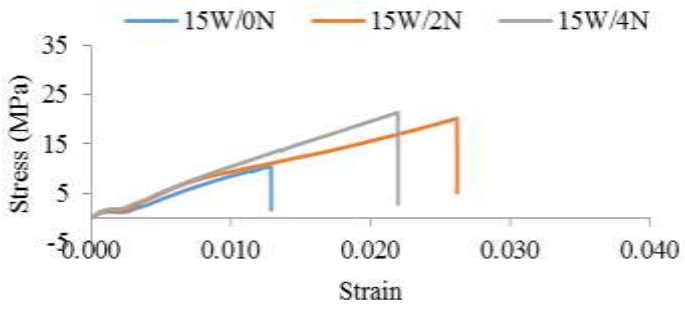


(d)



(d)

Figure 4.23: Stress-strain diagram of Fibre B composites (a) 5W/4N (b) 5 wt% loading composites (c) 10 wt% loading (d) 15 wt% loading

4.5.3 Fracture surface morphology

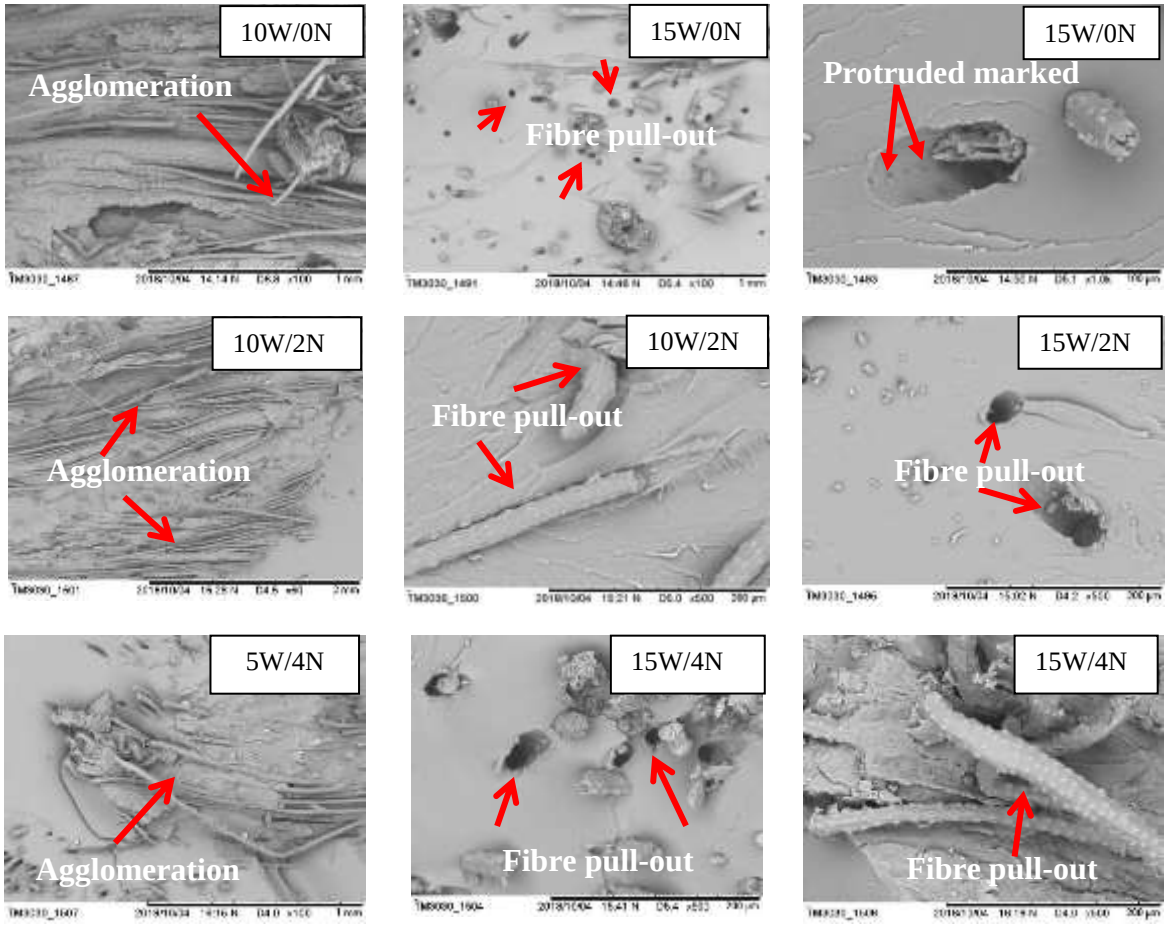


Figure 4.24: SEM images of Fibre B composites (fracture surface)

The fracture images of Fibre B composites are shown in Figure 4.24. As can be observed, fibre agglomeration is noticeable at the fracture surface of each loading condition. Fibre pull-out was observed at 15W/0N composites. The interfacial bonding for untreated fibres was weak without treatment. However, it is interesting to note that the protruding surface of Fibre B leaved marks on its interface as shown by the arrows. This actually had some effect towards the interfacial interlocking but was not strong enough to resist the load. The 2% alkali composite was observed to display the similar fracture

surface characteristic of fibre pull-out and fibre agglomeration. SEM images of 5W/4N composite showed less fibre pull-out. This was an indication that there was a good interfacial interaction between fibres and polyester matrix. 5W/4N composites contribute to the highest tensile strength for Fibre B.

4.6 Chapter Summary

Nipah palm fibre can be easily extracted using the wet method; the 12-week period of soaking process under the water degrades the non-cellulosic substance. Two types of fibre, different in sizes are measured for its diameter and density. The chemical composition analysis of Fibre A consists of 27% of α-cellulose, 23% of hemicellulose and 21% of lignin.

The fibres treated with alkali showed better thermal properties as reflected in TGA and DSC result. FTIR spectra further revealed the removal of other natural fibre constituent at higher concentration level. The 8% alkali treated fibre showed the highest crystallinity value and the highest single fibre tensile strength and the Young's modulus value at 242 MPa and 28.4 GPa respectively. A rougher fibre surface was observed for treated fibre which improved its composite strength.

The highest composites strength and Young's modulus value for Fibre A was achieved by 5W/10N composites at 29.36 MPa and 471 MPa respectively. For Fibre B composites, 5W/4N composites contributed to the highest strength at 29.67 MPa and 102 MPa for its Young's modulus value. However, the strength of both composites was lower than the neat polyester. The application of nipah fibre composites is suitable for non-load bearing structures.

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

The main objective of this research work is to investigate the physical, thermal, chemical and morphological properties of nipah fibre. This research work provides a new knowledge on nipah palm fibre characterisation and its composites properties.

The alkali treatment conducted on nipah fibre had shown better interfacial properties on its composite properties. Similar improvement reported by other natural fibre treated with alkali is also observed in nipah palm fibre. This provides a full potential for nipah palm fibre as reinforcing agent in composites. The 8% alkali treatment showed the highest strength on single fibre, while the 10% alkali treatment displayed the highest strength of its composites. The tensile strength of Fibre A and Fibre B composites showed similar value even though their fibre surface morphology was distinctively different. The 4% alkali concentration was found to be suitable for Fibre B while Fibre A needed higher alkali percentage, which was 10% and longer soaking period to achieve 29 MPa of its composite strength.

Microstructural changes of the treated fibres showed improvement due to the alkali treatment, where the roughened surface provides a good site for fibre interlocking and improving the strength of its composites. The collapsed lumen of treated fibres had improved its water absorption capacity. The water resistance layer provided by the alkali treatment helped in resisting water absorption of the composites.

Nipah palm fibre composite is recommended to be used as a reinforcing agent in a non-load bearing structure, or as filler in composite as the strength of its composites is

much lesser than its neat polyester. Non-load bearing structures for automotive components are the internal door cover, dashboard, tyre cover and many more.

There are a few future recommendations for future research such as to conduct the various fibre loading condition such as long and continuous for Fibre A and woven Fibre B composites. Long and continuous was not investigated in this research work, due to the difficulty to align the mould. Proper equipment to align the fibre during fabrication is needed to successfully produce long and align nipah fibre composites. For Fibre B, woven Fibre B is more suitable as based on its small diameter; they are susceptible for agglomeration when resins are poured into the mould. Woven fibres are able to minimise agglomeration as the fibres are firmed to its woven pattern.

Impact and flexural strength for nipah composites are recommended to be investigated. It is also recommended to use vacuum bagging method to fabricate the composites. This is to eliminate issues with air bubbles. The air bubbles are due to the reaction between unsaturated polyester resins with MEKP hardener.

The fabrication of nipah fibre composites with other polymer matrices is also recommended such as polypropylene. This will open more research opportunities for nipah fibre.

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APPENDICES

Appendix A: Publications/award from this thesis

1. Dickie, T., Syazali, S. T. B. S., & Noor Mohamed, N. H. B. (2020). Development of Nipah Palm Fibre Extraction Process. In *Materials Science Forum* (Vol. 997, pp. 57-65). Trans Tech Publications Ltd.
2. Dickie, T., Syazali, S.T.B.S., & Noor Hisyam, N.H.B. (2018). Recent development of Nipah Palm Fibre Extraction Process. *Proceedings of International UNIMAS STEM 11th Engineering Conference 2018*. Kuching, Sarawak.
3. Dickie,T., Syazali, S.T.B.S., (2016). Impact Behaviour of Nipah Palm Fibre Reinforced Polyester Composite. *Best Presenter Award, North Borneo Research Colloquium, Curtin University, Miri, Sarawak.*
4. Dickie, T., Syazali, S.T.B.S., (2016). Tensile behaviour of Nipah fiber / polyester composites. Proceedings of IMTCE 2016, *10th International Materials Technology Conference & Exhibition*, Putra World Trade Centre (PWTC) Kuala Lumpur.
5. Dickie, T., Osman M.S., (2012). Extraction process of Nipah palm fibre. *Proceedings of EnCon 2012, 5th Engineering Conference, "Engineering Towards Change - Empowering Green Solutions"*, Kuching, Sarawak.

6. Dickie, T., Osman M.S., (2012). Sample Preparation and Material Characteristic on Nipah *"GAPPS 2012" Seminar Kayu Getah, Acacia, Palma & Spesies Perintis Sarawak*
7. Dickie, T., Osman M.S., (2012). Mechanical Performance of Nipah Based Composites. Poster presentation at *1st International Conference on Design and Innovation 2012: Sustainable Ground*, Unimas

Appendix B: Equipment calibration certificates

GLOBAL VENTURE METROLOGY SDN BHD
(Company No. 1020870-D | GST No: 000694796288)
No. 57-A, Jalan Bayu Tinggi 7/KS6, Batu Unjur, 41200 Klang, Selangor
www.gvm.com.my | tel/fax: 03-33221471 | enquiry@gvm.com.my

MS ISO/IEC 17025
CALIBRATION
SAMM NO. 662

Certificate of Calibration

Certificate No	GVM180834/ S
Issue Date	20 July, 2018

Customer : ENVIC LABORATORY SDN BHD
Address : 1st Floor, Lot 13193, Sublot 11 & 12,
Samajaya Commercial Centre,
Jalan Setia Raja,
93350 Kuching, Sarawak.

Description : Analytical Balance

Manufacturer : Sartorius
Model / Type : ENTRIS224I-1S
Capacity / Range : Max. 220g
Serial No : 33908500
ID No : EL/092

Received Date : 13 July, 2018
Calibration Date : 13 July, 2018
Recalibration Date : 13 July, 2019
Specified by Customer
*The user should be aware that any number of factors may cause this instrument to drift out of calibration before the specified calibration interval has expired.

Environment Condition
Ambient Temperature : Min 26.4 °C
Max 28.2 °C
Relative Humidity : Min 52 %RH
Max 54 %RH

Condition Upon Receiving : Good in Physical Condition
Condition Upon Returning : Calibrated and Tested Serviceable
Calibration Method : In-house procedure LCP-M-02
Calibration Venue : This Instrument has been calibrated at above customer address
Calibration Result : The result as following page(s). The expanded uncertainties are based on an estimated confidence probability of approximately at 95% and have a coverage factor of k=2 unless stated otherwise.

Calibration Standards / Equipment Used

Description of Standards	ID No	Due Date	Traceable To
Standard Weight	SW-02-Site	16 January 2019	NMIM

Approved Signatory :
LIM WEI SHENG
(Head of Laboratory)

Premier calibration

MS ISO/IEC 17025
CALIBRATION
SAMM NO. 773

CERTIFICATE OF CALIBRATION

Date of Issue : 20 July 2017 **Certificate No :** **EC01-S0213**

Submitted By : ENVIC LABORATORY SDN BHD
1st FLOOR, LOT 13193, SUBLOT 11&12,
SAMAJAYA COMMERCIAL CENTRE, JALAN SETIA RAJA,
93350 KUCHING, SARAWAK.

Instrument : Water Bath
Brand : Memmert
Model : WB7
Serial No. : 1200.0398
Related No. : N/A EL/054

Receiving Date : 19 July 2017
Calibration Date : 19 July 2017
Recalibration date specified by client : -

Environment Condition
Temperature : Max: 24.7 °C / Min: 23.5 °C
Relative Humidity : Max: 58 %r.h. / Min: 52 %r.h.

The User should be aware that any number of factors may cause this instrument to drift out of calibration before the specified calibration interval has expired.

Condition of Instrument
Before Calibration : Physically good.
After Calibration : Calibrated and tested serviceable

Calibration Venue : On site at the address as shown in above.
Calibration Method : CM/T 10 with reference to ASTM E715-80(2016)

Reference Standard Used

Reference Instrument	Serial No.	Cert. No.	Traceability	Due Date
Temperature Recorder	ET-TR1	PSYP-17009665	NML-SIRIM	28-Feb-18
Type K Thermocouple Wires	ET-TKA1~9	PSYP-17009655~17009663	NML-SIRIM	21-Feb-18

Approved Signatory

Yiew Ching Tuck
Technical Manager

Premier calibration Sdn Bhd (1122788-A)
12A, Jalan Anggerik Vanilla Z 31/Z, Kota Kemuning, Sek. 31, 40460 Shah Alam, Selangor Darul Ehsan, Malaysia.
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