SYNTHESIS OF NANOCELLULOSE FIBRES FROM RUBBER WOOD FIBRES USING CHEMO-THERMO-MECHANICAL TREATMENT

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Thesis submitted in partial fulfilment of the requirements
for the award of the degree of
Bachelor of Chemical Engineering

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JAN 2014

ABSTRACT

This study aims to synthesize nanocellulose from Hevea brasiliensis fibres or commonly known as rubber wood fibres. Rubber trees are able to grow abundantly in Malaysia due to its close proximity to the equator (within 15° latitude). When the production of latex dwindles considerably, the trees are felled and are replaced with newer saplings. Hence, the end –of-cycle trees can be used as a rich source of synthesis of nanocellulose. The wood fibres obtained were pre-treated using sodium anthraquinone. This was followed by a series of treatment with a mixture of sodium chlorite and acetic acid as well as sodium hydroxide and hydrogen peroxide. The bleached pulps were then air dried and soaked in distilled water for 8 hours. It was then mixed in a mechanical blender for 15 minutes. Diluted suspensions (0.3%) are prepared and put through the sonicator for a period of four hours at 65^0C. Fibres obtained were then examined by the Mastersizer to determine the morphological size of the wood fibres. After sonicating for about four hours, the modal range of fibres obtained were 158.49 - 181.97 (μm). Fibres were then run through the homogenizer for a period 10, 20, 30 and 40 runs. After 40 runs, the modal range was between 0.142 – 0.200 (μm). The nanocellulose fibres were studied further by examining with Fourier Transform Infrared Spectroscopy (FTIR). There was a disappearance of vibration peaks at 1730.28 and 1234 cm^{-1} from spectra of treated fibres which shows the removal of hemicellulose and lignin components respectively. Images obtained from the FESEM shows that the chemical treatment resulted in increased roughness and striations of fibres. It also showed the average diameter achieved after chemo-mechanical treatment to be 160 nm.

Keywords : Wood fibres, Cellulose, Chemo-mechanical process, Nanocellulose

ABSTRAK

Kajian ini bertujuan untuk mensintesis nanosellulosa daripada gentian Hevea brasilienis atau lebih dikenali sebagain gentian kayu getah. Pokok getah lazimnya dapat tumbuh dengan subur di Malaysia kerana kedudukannya yang berdekatan dengan garisan khatulistiwa (dalam lingkungan 15° latitud). Apabila penghasilan/produksi susu getah mula berkurangan dengan ketara, maka pokok getah akan ditebang dan digantikan dengan anak pokok yang baru. Justeru, pokok yang berada di akhir kitaran boleh digunakan sebagai sumber yang kaya untuk mensintesis nanosellulosa. Gentian kayu di rawat dengan mengunakan natrium anthraquinone. Ini diikuti dengan beberapa siri rawatan dengan campuran natrium klorida dan asid asetik dan juga natrium hidroksida dan hydrogen peroksida. Pulpa yang terluntur kemudian direndam di dalam air suling selama lapan jam. Rendaman tersebut kemudian di kisar mengunakan pengisar mekanikal selama 15 minit. Campuran yang mengandungi 0.3% fiber kemudian diletakkan dalam mandian sonicator selama empat jam pada suhu 65 °C. Gentian yang diperoleh kemudian diperiksa menggunakan Mastersizer untuk menentukan saiz morfologi. Selepas melalui proses sonifikasi, modal saiz yang diperoleh adalah dalam lingkungan 158.49-181.97µm. Gentian kemudian melalui homogenizer untuk tempoh10, 20, 30, 40 kitaran. Selepas 40 kitaran, modal saiz yang diperoleh adalah dalam lingkungan 0.142-0.200 nm. Gentian nanosellulosa juga dikaji dengan menggunakan Fourier Transform Infrared spectroscopy (FTIR). Gentian yang dirawat menunjukkan kehilangan puncak getaran pada 1730 cm^{-1} dan juga 1234 cm^{-1} yang membuktikan penyingkiran hemisellulosa dan lignin. Imej yang diperoleh daripada kajian FE-SEM menunjukkan rawatan kimia telah meningkatkan kekasaran pada permukaan gentian tersebut. Imej juga menunjukkan diameter gentian sebanyak 160nm selepas rawatan kimia serta mekanikal.

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LIST OF ABBREVIATIONS

WF	Wood Fibres
MCC	Microcrystalline structure
MFC	Microfibrillated structure
NFC	Nanofibrillated crystals
CNC	Nanocrystals
H_2O_2	Hydrogen Peroxide
$NaOH$	Sodium Hydroxide
$NaClO_2$	Sodium Chlorite
$NaSO_3$	Sodium Sulphite
AQ	Anthraquinone
CH_3COOH	Acetic Acid
FTIR	Fourier Transform Infrared Transform
X-RD	X-Ray Diffraction
FESEM	Field Emission Scanning Electron Microscope
TGA	Thermogravimetric Analysis

APPENDICES

1 INTRODUCTION

1.1 Motivation and statement of problem

Cellulose fibres are the constituents of plant fibres (Kalia et al., 2010). It is considered to be one of the most abundant organic compounds derived from biomass according to Dufresne et al. (2005). They also stated that the worldwide production of this polymer is estimated to be around 10^{10} to 10^{11} tonnes each year. According to Yano et al. (2005), cellulose is used widely in various industries such as the paper, films, as well as the pharmaceutical industry.

Cellulose is a long chain polymer which is formed through the linking of smaller molecules or repeating units of D-glucose which is a type of simple sugar (Habibi et al., 2010).Kalia et al. (2009) stated that cellulose consists of helically wound cellulose microfibrils, bound together by an amorphous lignin matrix which is believed to keep water in fibres as well as to give the stem support from physical forces such as strong winds as well as gravitational effects. According to Kalia et al. (2009) besides cellulose and lignin, hemicellulose is also present. It is said to be the compatibilizeer between lignin and cellulose. Natural fibres also contain pectin, waxes and water soluble substances. Cellulose, lignin and hemicellulose is said to be the basic components of natural fibres which contribute to the physical properties such as relatively high strength, high stiffness and low density (Mitchell, 1989).

Nanocellulose refers to the cellulose fibres having diameters ranging from 10-200nm (Verma et al., 2011). There are two main types of nanocellulose which are cellulose nanocrystals and cellulose microfibrils according to Habibi et.al (2010). Based on their journal, microfibrils occur when 36 individual cellulose molecules are brought together by biomass into larger units. Nanocellulose can be extracted from the cell wall of lignocellulosic materials using different methods, such as mechanical, chemo-mechanical and enzymatic techniques (Jonoobi et al., 2009; Panthapulakkal et al., 2006). In previous studies, nanocellulose was extracted from many different fibres such as coir, hemp, banana stem, cassava bagasse and rice husk. Nevertheless, there is still relatively fewer studies on the production of nanocellulose from rubber wood. The use of rubber wood is extremely beneficial to Malaysia as it is one of the main commodities. Hence, once the latex production decreases, these trees can be processed to produce nanocellulose which has a wide array of uses. Besides that in the previous study performed by Jonoobi et al. (2011), they did not perform thermo-chemo pre-treatment on the wood fibres. Hence this study would also like to inculcate pre-treatment before performing chemo-mechanical analysis. Based on previous studies of synthesis of nanocellulose from other plant base, pre- treatment of fibres is believed to be able to clean and chemically modify the fibre surface as well as increase surface roughness (Kalia et al., 2009).

1.2 Objectives

The following is the objective of this research:

- To synthesis nanofibres from rubberwood (***Hevea brasiliensis***) by means of pre-treatment prior to chemo-mechanical process.

1.3 Scope of this research

The following are the scope of this research:

i) Preparation of wood fibre sample.

ii) Pre –treatment on wood fibres. There are two types of pre- treatment that would be examined. First method would be using a mixture of sodium hydroxide and anthraquinone and the second method would be using sodium sulphite.

iii) Chemo-mechanical treatment to further separate the cellulose fibres into smaller fibres known as nanocellulose.

iv) Verification analysis on the quality of product formed was done by studying the morphology of the cellulose fibres using Scanning Electron Microscope (SEM). The tracking of functional group changes were done using Fourier Transform Infrared Spectroscopy (FTIR). The structure and phase analysis were done using X-ray diffraction .Thermogravimetric analysis was carried out to determine the potential and extent of thermal decomposition.

1.4 Main contribution of this work

Previous studies have demonstrated the synthesis of nanocellulose from a variety of plant source that is native to the researchers' homeland. For example, jute from India and hemp from the Philippines. The current study extends these techniques to a local Malaysian variety, the rubber tree. Addressing the problems with manufacturing nanocellulose fibres from this study would also be the main focus of this study.

1.5 Organisation of this thesis

The structure of the reminder of the thesis is outlined as follow:

Chapter 2 provides an overview of the history of the rubber industry in Malaysia, characteristics as well as uses of the rubber wood tree. Composition of wood fibres especially cellulose were also discussed in detail. The advantage of cellulose fibres compared to synthetic fibres were also looked into. Besides that, most importantly a concise description of nanocellulose from its characterisation to the synthesis were also discussed in detail

Chapter 3 gives a review of the materials used as well as the preparation of the chemicals. Besides that the methodology was also discussed in detail. This includes the pre-treatment, thermo-chemo and thermo-mechanical treatment. Characterization analysis were also discussed.

Chapter 4 is devoted to the results and discussion of the characterization and analysis of the sample of wood fibres.

Chapter 5 gives a summary of the whole study performed which is divided into two parts which are the conclusion and recommendation

2 LITERATURE REVIEW

2.1 Overview

Chapter 2 provides an overview of the history of the rubber industry in Malaysia, characteristics as well as uses of the rubber wood tree. Composition of wood fibres especially cellulose were also discussed in detail. The advantages of cellulose fibres compared to synthetic fibres were also looked into. Besides that, a concise description of nanocellulose from its characterisation to its synthesis were also discussed.

2.1.1 The history of the rubber industry in Malaysia

Sir Henry Wickham is known as the father of the rubber industry in Malaysia. In 1876, seeds were brought by him from Brazil to the Kew Garden in the United Kingdom. Some of the seedlings were then transported to the Singapore Botanical Garden through Ceylon (now known as Sri Lanka). Initially, cultivation of rubber in Malaysia began in Kuala Kangsar in 1879 (Ratnasingam, 2000).

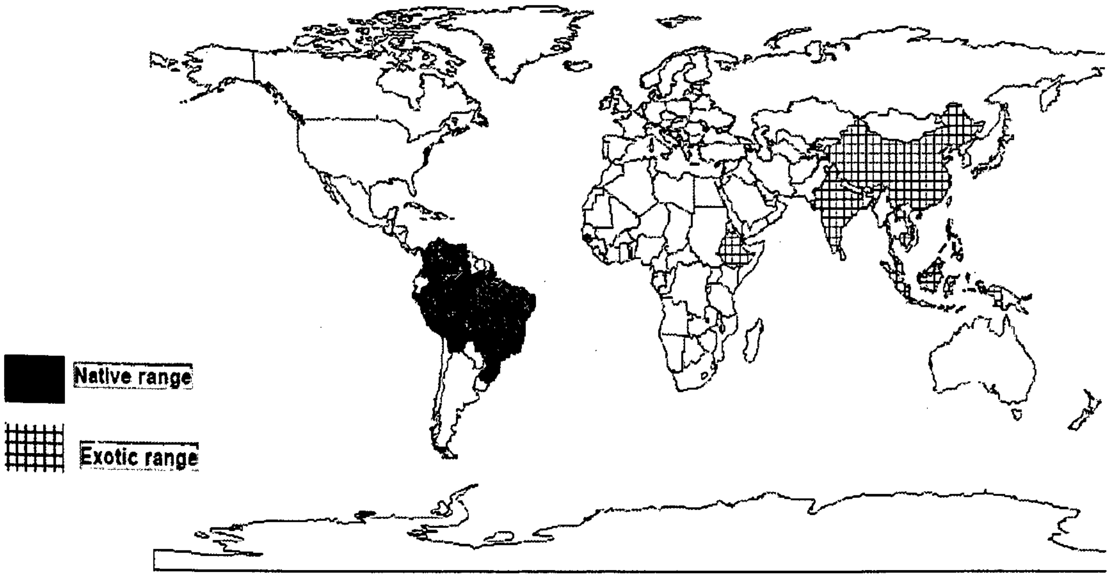

Figure 2.1: Agroforestry Database 4.0 (Orwa et al.2009)

The map in Figure 2.1 shows the countries where the species has been planted. Native range includes countries such as Bolivia, Brazil, Colombia, Peru, Venezuela. Whereas, exotic range includes countries such as Malaysia, Brunei, Cambodia, China, Ethiopia, India, Indonesia, Laos, Liberia, Myanmar, Philippines, Singapore, Sri Lanka, Thailand, Uganda, Vietnam.

According to Shigematsu et al. (2011), more than 80% of total rubber plantation areas in the world are in Asia, with Malaysia, Indonesia and Thailand covering almost 70% of the total rubber cultivation. Malaysia is currently the 3rd most important country in the world for rubber cultivation (Shigematsu et al., 2011).

2.1.2 Rubber Wood Tree

Hevea brasiliensis is a quick-growing tree. It rarely exceeds 25 m in height in plantations, but wild trees of over 40 m have been recorded (Balsiger et al., 2000). It has a well-developed taproot system and far-spreading laterals to absorb water. According to Ratnasingam et al. (2012), rubber wood was formerly regarded as a by-product of the rubber plantations and used for the production of charcoal or as fuel wood, for brick making, tobacco drying and rubber drying. Other rubber wood residues have been used successfully in Malaysia for the production of particle board, wood-cement board, and medium-density fibreboard.

The importance of the timber from the rubber plantations is now fully recognized, and in Southeast Asia it is planted solely for timber production (Ratnasingam et al., 2012). Most of the timber is used to manufacture furniture. Other uses include interior finish, moulding, e.g. for wall panelling, picture frames, drawer guides, cabinet and other handles, parquet flooring, many household utensils, crates, coffins, veneer, and glue-laminated timber.

The economic life cycle of a rubber plantation is 30-35 years, after which replanting is necessary (Ratnasingam et al., 2012) Hence , the trees that need to be felled to replant newer saplings provides a rich source of rubber wood fibre waste that can be transformed into useful products such as nanocellulose which can be then used in various compounds. Synthesising nanocellulose from agricultural remnants provides copious , cheap and available resource of inexhaustible lignocelluloses materials. Ecological worry has resulted in increased interest in renewable- based resources. Hence, materials such as rubber wood fibre is considered as an environmentally secure substitute (Digabel et al., 2006)

2.2 Lignocellulose

Cellulose, lignin and hemicellulose is reported to be the basic components of natural fibres (A.J. Mitchell, 1989). Bismarck et al (2002) in their journal stated that the lignocellulosic fibres have different categorised microstructures. The elongated single cell fibres consist of a primary wall and three other secondary walls. In the center of this, there is the lumen which transports water and other dissolved ions.

The primary cell wall consists of pectin which will be hardened when lignin deposits. Wood cell walls consists of 3-4 nm wide cellulose that are embedded in hemicellulose and lignin network (Stamm, 1964). Cellulose consists of helically wound cellulose microfibrils that form part of the secondary cell wall (Bismarck et al). Mitchell (1989) stated that lignin is the adhesive system or the gluing agent that holds the cell structure together which contribute to good physical properties.

Besides that, binding of lignin matrix is believed to keep water in fibres as well as to give the stem support from physical forces such as strong winds as well as gravitational effects. According to Kalia et al. (2011), hemicellulose is said to be the compatibilizer between lignin and cellulose. Natural fibres also contain pectin, waxes and water soluble substance.

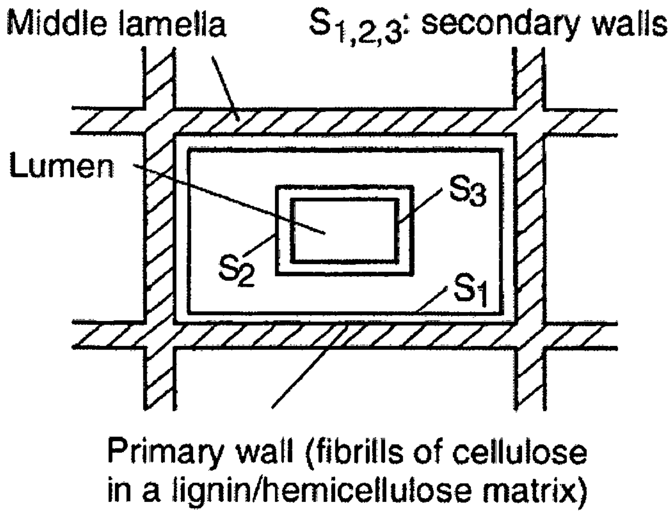

Figure 2.2: Cross Section of an elongated cell

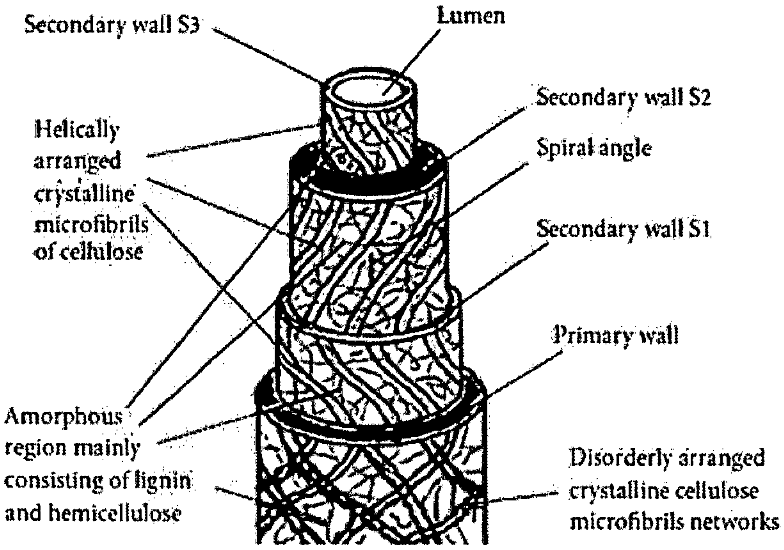

Figure 2.3: Three Dimensional Cross Section of an elongated cell (Rong et al., 2001)

Cellulose is a long chain polymer which is formed through the linking of smaller molecules or repeating units of D-glucose which is a type of simple sugar (Habibi et al., 2010). These glucose units are linked when water is eliminated by combining the hydroxyl group and hydrogen. According to the Merck Index (1968), linking two units produces a dissacharide called cellobiose. In the cellulose chain, the glucose units are in six-membered rings, called pyranoses. There are joined by single atoms (acetal lingkages) between the C1 of one pyranose ring and the C-4 of the next ring. A molecule of water is lost due to the reaction of an alcohol.(Bradfield et al., 2006).

The interchain hydrogen bonds in the crystalline regions are strong, giving the resultant fibre good strength and insolubility in most solvents. This prevents cellulose from melting. Hence, cellulose is a relatively stable polymer (Adriana et al., 2011) . Most cellulose structures can absorb large quantities of water thus the cellulose swells but does not dissolve in water (Visakh et al., 2010).

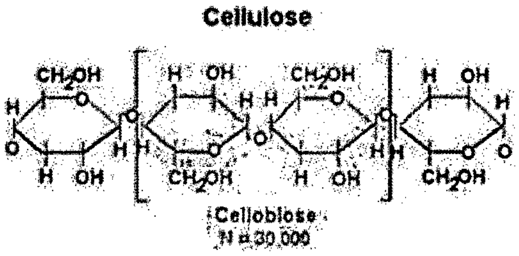

Figure 2.4 : Cellobiose unit (Bradfield et al., 2006)

In contrast with mineral fibres such as glass and carbon fibres, cellulose fibres have good flexibiltity and elasticity (Adriana et al., 2011). These characteristics allow the fibres to maintain a high aspect ratio in the manufacturing process. Cellulose fibres are the main constituents of plant fibres (Kalia et al., 2011) and it is considered to be one of the most abundant organic compounds derived from biomass according to Dufresne et al. (1997). It is also advantageous to use it as it is biodegradeable, renewable and relatively cheaper to produce when compared to synthetic materials. It is also relevant as it is derived from non-food agricultural based economy. Besides that, cellulose has low abrasivity which would provide greater durability of the processing equipment (Adriana et al., 2011).

Table 2.1 : Lignocellulose Fibres vs Synthetic fibres

Lignocellulose	Characteristic	Synthetic
Yes	Renewable	Non-renewable
No	Abrasive	Abrasive
Biodegradable	Biodegradability	Non-Biodegradable
Relatively cheaper	Cost	Expensive
Lower	Density	Higher
No	Effect on environment	Potentially harmful
Higher (138 GPa)	Youngs Modulus	Lower (65 GPa)
Low coefficient	Thermal expansion	High coefficient

2.3 Nanocellulose

Nanocellulose refers to the cellulose fibres having diameters of tens of nm (Adriana et al.,2011). Many studies have reported cellulose fibres within the nano range using different terminologies. Amongst them include, cellulose nanofibre (CNF), nanofibril, nano-cellulose, nanofibrillated cellulose (NFC), and microfibrillated cellulose (MFC). Hydrolysis of amorphous regions of these materials yields crystalline celluloses, which are known as cellulose nanocrystals (CNC), nanorods, whiskers, or nanowhiskers. Compared to microfibrils, these are typically more crystalline and have smaller aspect ratios. Although the different terms used describe variances in material properties, Moon et al. (2011) have established a nomenclature consistent with current trends.

Wood Fibres Microcrystalline Microfibrillated Nanofibrillated Nano Crystals
WF MCC MFC NFC CNC

Figure 2.5 : Comparison of structure of wood fibre till nano crystal structure (Moon, R.J., 2011, Dufresne et al., 1997 and Saito et al., 2007)

Table 2.2 : Summary of cellulose particle type characteristics (Moon et al., 2011)

Particle Type	Length (μm)	Diameter	Crystallinity (%)
WF and PF	>2000	20-50 μm	43-65
MCC	5-10	10-50 μm	80-85
MFC	0.5-10	10-100 nm	51-69
NFC	0.5-2	4-20 nm	
CNC	0.05-0.5	3-5 nm	54-88

Nanocellulose materials are suitable reinforcement materials for nanocomposites. This is because of its low coefficient of thermal expansion along the longitudinal direction as well as the high Youngs Modulus along the longitudinal direction in the crystal region (Nishino et al., 2004 and Sakurada et al., 1962). This is said to be relatively strong which can be seen by the high Youngs Modulus (Dufresne et al.,1997). Nanocellulose-based materials can be stronger than steel and stiffer than Kevlar. Having great strength as well as being light weight results in good physical properties that can be used in many applications.

Table 2.3 : Young's Modulus(GPa) for various materials

Material	Density	Tensile Strength (GPa)	Axial Elastic Modulus	Reference
Kevlar-49	-	3.06	144	(Bunsell, 1975)
Nylon 66	-	1	12.5	(Bunsell, 1975)
Carbon fibre	1.18	1.5-5.5	150-500	(Callister Jr, 1994)
Steel wire	7.8	4.1	210	(Callister Jr, 1994)
Clay nanoplatelets	-	-	170	(Hussain et al.2006)
Carbon nanotubes	-	11-36	270-950	(Yu et al., 2000)
Crystalline cellulose	1.6	7.5-7.7	110-220	(Moon et al.2011

Composite materials reinforced with nanocellulose fillers have potential applications in important fields like electronic and electrical industry, construction, biomedicine, cosmetics, paper industry, packaging, building materials, textile industry and others (Kamel, 2007). These nanocellulose reinforced polymer composites can be used in a wide array of product from lightweight armor and ballistic glass to wound dressings and scaffolds for growing replacement organs for transplantation in tissue engineering in the medical field (Korhonen et al., 2011). In the foam form, it can be used for oil recovery as well as storage. It can also be used in the pharmaceutical line, cosmetic bases and pigments, food modifiers, sensors as well as biomedical devices. Nanocellulose also provides smoother surface and enhances glossy properties. MCC have been extensively used in pharmaceuticals and food industries. Due to the highly crystalline structure, they are functionally applied as a binder, rheological modifier, or as reinforcement fillers. WF and PF are predominately applied in paper and textile products with much larger dimensions with relatively low crystallinity (Moon et al. 2011).

Many researches' have been done on the generation of nanocellulose from various sources. Sassi et al. (1995) and Torres et al. (2004) stated that cellulose fibres can be grouped according to the leaf, bast, fruit, grass and stalk. Type of leaf thus far examined includes pineapple, sisal, banana flax, hemp, jute are examples of bast. Fruits include oil palm. Grass includes bamboo and bagasse whereas straws are types of stalk. Out of all the plants, they concluded that the bast and leave are the two most commonly used in the applications as it is hard fibres.

Rubber wood is a type of hard fibre. It is also the main commodity in Malaysia due to the latex production. Nevertheless, very few studies have been performed on the synthesis of nanocellulose from rubberwood fibres. Hence, using nanocellulose derived from rubber wood fibres would be extremely beneficial to Malaysia especially since nanocellulose has a wide array of uses in various fields.

2.4 Pre-treatment

2.4.1 Surface modification

Based on previous studies, the main obstacle in the preparation of the nanocellulose composite is due to the extremely strong hydrogen bonding within the fibrils itself (S. Panthapulakkal et al., 2011). Due to their polar and hydrophilic nature, they are generally poorly compatible with non-polar matrices (Adriana et al., 2011). Polar matrices include polyolefins which are usually the base used to spin fibres into sheets at a large scale. Specific surface modifications would be able to reverse these negative drawbacks (Adriana et al., 2011).

There are numerous modifications to cater to different needs. The common methods for fibre surface modification include silylation, mercerization, peroxide, benzoylation, graft polymerisation and bacterial cellulose treatment (Kalia et al,2011). Surface modification to form ionic groups at cellulose surfaces include sulfonation, carboxylation and grafting (Hubbe et al., 2008).

a) Acetylation and silane treatment

Reactions to create hydrophobic surfaces include acetylation and silane treatments (Hubbe et al., 2008). Silane –coupling agents usually improves the degree of cross-linking in the interface region, hence enhancing bonding. Silane coupling agents were said to be the most effective amongst other coupling agents in altering the natural fibre-matrix interface (Kalia et al., 2011). Performance of silane treatment are believed to be higher for alkaline-treated fibre than for the untreated fibre. This is because there are more reactive sites that can be created for reaction (Kalia et al., 2011). Acetylation on the other hand uses surfactants and polyelectrolytes (Hubbe et al., 2008).

Though the usage of silanes such as toluene dissocyanate and triethoxyvinyl silane could improve the surface of the fibres for consequent treatments, there is still the issue of contamination of water source if these chemicals are used in large amounts.

b) Peroxide treatment

According to Sreekala et al., (2002) , peroxide treatment involves treating fibres with benzoyl peroxide or dicumyl peroxide in acetone before alkali pretreatment. These reactions require high temperatures for the effective decomposition of peroxide. Nevertheless, organic peroxides tend to decompose easily, producing free radicals. Free radicals produced in bulk may pose a threat to the ozone layer.

c) Benzoylation

In benzoylation treatment, benzoyl chloride is usually used to include the benzoyl group in the fibre which makes it responsible for the drop in hydrophilic nature of the fibre (Joseph et al., 2002). Fibres have to be subjected to alkaline pre-treatment prior to benzoylation in order to excite the hydroxyl groups of cellulose and lignin in the fibre. After treatment, fibres need to be soaked in ethanol to remove the reacted and unreacted benzoyl chloride (Wang, 2004) or fibres maybe denatured.

d) Mercerization

Mercerisation is a commonly used method to produce high quality fibres (Ray et al, 2001). It leads to the breaking down of fibre bundles into smaller fibres. Mercerisation reduces the fibre diameter. This results in better fibre-matrix adhesion and an increase in mechanical properties (Joseph et al., 2000). Mercerisation also increases the number of possible reaction sites and allows better fibre wetting. It is also said to have an effect on the chemical compostion of the fibre, degree of polymerisation as well as the molecular orientation of the cellulose crystals due to the removal of cementing substances such as lignin and hemicellulose. Hence mercerisation has long lasting effects on mechanical properties of flax fibres (Gassan et al., 1999).

Hence, this study would be performing mercerisation using two different types of chemicals. The first method would be pre-treatment with sodium sulphite (5%)(Lavoine et al). The second method with a mixture of sodium hydroxide and anthraquinone (12% + 0.1%) (Jonoobi et al, 2011) . These chemicals were chosen after due considerations as they are not too abrasive to the structure of cellulose when compared to using silanes which may damage cell structure or peroxides and benzoyls which may be harmful to the environment if used in excess.

2.4.2 Delignification

Treatment of lignocellulosics intends to reduce the crystallinity of cellulose, increase the biomass area, break the tough outer lignin layer as well as to remove the hemicellulose (Verma et al., 2011) . Delignification would ensure that cellulose is more available for acid hydrolysis. Sodium hydroxide (NaOH) is an excellent pre-swelling agent. It increases the accessibility of the core material to further hydrolysis action. Besides that, it would also aid the conversion of carbohydrate polymers into fermentable sugars swiftly as well as increase the yield (Kalia et al., 2011). Pre-treatment also helps to lower the lignin and hemicellulose contents of the fibres . Hence, it is a form of pulping and bleaching as the lignin and hemicellulose which gives fibre its distinct color are removed.

Delignification includes physical, chemical and thermal methods or a combination of the aforementioned three. Different lignocellulosic materials have different physical, chemical and biological attributes (Verma et al., 2011). Hence, it is vital to arrange appropriate technology based on their properties. Hydrothermal treatment, wet oxidation, microwave-assisted treatment as well as alkali treatments have been used widely in the delignification of previous studies of various fibres other than rubber wood.

Hydrothermal pre-treatments results in major lignin re-localisation and aids in removal of major wax as well as a small portion of hemicellulose (Verma et al., 2011). On the other hand, during steam pre-treatments, water in the cells evaporates and as the pressure drops, it explodes to provide increased specific surface area. Part of the hemicellulose decomposes to acids, which catalyses the decomposition of hemicellulose and lignin, and releases the cellulose. In wet oxidation pre-treatment, the material is treated with water and air at around 120°C as reported by Martin et al (2008).

According to Xiong et al. (2002) , microwave irradiation changes the structure of cellulose, degrades lignin and hemicellulose . Compared to conduction/convection heating, microwave directly interacts between a heated target and the electromagnetic field to generate heat. Therefore, heating is rapid when microwave is used to treat the lignocelluloics. This unique heating feature has an explosion effect among particles, and improves disruption of obstinate structure of lignocelluloses. Nevertheless, the electromagnetic field used in the microwave may intensify the destruction of crystal line structures (La Hoz et al., 2005).