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EXTRACTION AND CHARACTERIZATION OF NANOCELLULOSE FROM (OIL PALM WOOD)

by

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A report submitted in partial fulfillment of the requirements for the degree of Bachelor of Applied Science (Materials Technology) with honours.

**FACULTY OF EARTH SCIENCE
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DECLARATION

I declare that this thesis entitled EXTRACTION AND CHARACTERIZATION OF NANOCELLULOSE FROM (OIL PALM WOOD) is the result of my own research except as cited in the references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

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EXTRACTION AND CHARACTERIZATION OF NANOCELLULOSE FROM OIL PALM WOOD

ABSTRACT

In this study, the objective of this work was to develop a simple extraction and characterization of regenerated cellulose from oil palm wood. The purpose of this research is to extract cellulose from oil palm wood according to TAPPI test method. The extracted cellulose was treated with ionic liquid, BMIMCL at different weight percentages (2 wt % and 5 wt %). The chemical, structural, morphology and thermal properties of cellulose samples (untreated and treated cellulose) were characterized using X-Ray Diffraction (XRD), Fourier Transform Infrared Spectroscopy (FTIR), Field Emission Scanning Electron Microscope (FESEM) and Thermogravimetric Analysis (TGA) respectively. In this study, the cellulose treated with BMIMCL is found to have more amorphous properties compared to untreated cellulose. This proves that BMIMCL ionic liquid is effectively in converting cellulose into amorphous cellulose. However, the BMIMCL is less efficient in conversion from cellulose to nanocellulose.

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PENGESKTRAKAN DAN PENCIRIAN NANOSELULOSA DARI KAYU KELAPA SAWIT

ABSTRAK

Kajian ini adalah untuk mengekstrak dan mencirikan serat selulosa daripada kelapa sawit. Tujuan penyelidikan ini adalah untuk mengekstrak serat daripada kelapa sawit mengikut kaedah TAPPI. Serat selulosa yang diekstrak telah dirawat dengan cecair ionik BMIMCL, pada peratusan yang berbeza mengikut kandungan berat (2 wt% dan 5% berat). Sifat kimia, struktur, morfologi dan sifat haba sampel selulosa (tidak dirawat dan selulosa yang dirawat) telah dicirikan menggunakan pembelauan Sinar-X (*XRD*), Spektroskopi Inframerah Transformasi (*FTIR*), Paparan Emisi Mikroskopi elektron penskanan (*FESEM*) dan Analisis Termogravimetri (*TGA*). Dalam kajian ini, serat selulosa yang dirawat dengan BMIMCL menunjukkan ciri-ciri amorfus berbanding selulosa yang tidak dirawat. Ini membuktikan bahawa cecair ionik, BMIMCL adalah berkesan dalam menukarkan selulosa ke selulosa amorfus. Walau bagaimanapun, BMIMCL adalah kurang berkesan dalam penukaran dari selulosa kepada nanoselulosa.

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LIST OF ABBREVIATION

BMIMCl	1-Butyl-3-methylimidazolium chloride
IL	Ionic liquid
CNC	Cellulose nanocrystal
XRD	X-ray diffraction
FTIR	Fourier transforms infrared spectroscopy
TGA	Thermogravimetric analysis
DTG	Differential thermogravimetric
TAPPI	Technical Association of the pulp and paper industry
CNF	Cellulose nanofibril
OPT	Oil palm wood
OPF	Oil palm front
EFB	Empty fruit bunch
MFC	Microfibrilated cellulose
NCC	Nanocrystalline cellulose
BNC	Bacterial nanocellulose
PPF	Palm presed fiber
CY	Char yield
kV	Kilovolt
JEOL	Couple to a special freeze-manufacturing device

LIST OF SYMBOLS

K	0.94 correction factor
λ	0.154 nm
β	Corrected angular width in radius at half max intensity of peak
Gpa	Young modulus strength
$J g^{-1}$	Young Modulus strength
Kg / m^3	Density
I_{002}	The peak intensity corresponding to crystalline
I_{am}	The peak intensity corresponding to amorphous

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Nanocrystalline is cellulose in crystalline form which is extracted from woody and non woody plant. Cellulose is a natural high molecular weight linear homopolymer consist of repeating β -D-glucopyranosyl units joined by 1-4 glycosidic linkages in order. In the cell wall of lignocellulosic materials, cellulose exists as aggregates of nano-size fibrils (Bettaieb *et al.*, 2015). Cellulose is the most abundant constituent in plant. It has wide application in biomedical, packaging, automobile sector and many other application (Maiti *et al.*, 2013).

Furthermore, cellulose becomes most abundant source for renewable polymer. Nowadays, cellulose has been invented to make new product, for example in nanocomposite, nanofiber and nanobiocomposite. Demand for this application has higher since there are a lot of new product and need to compete in this field. Product from nanocellulose from agricultural residues possesses economic and environmental advantages. Research has been run which already identify that characterization and properties of nanocrystal have a good future in making new product. Example extraction in nanocomposite is transfer cellulose whiskers from an aqueous into an organic polymer matrix and in nanofiber was prepared from cotton stalks by different chemical treatments followed by ultrasonic.

The main reason in using cellulose nano-fibers in composite materials is due to the potentially high stiffness of the cellulose fibers for reinforcement. This property can be achieved with the reducing amount of amorphous material by breaking the structure of the plant in individualized nano-fibers with high crystallinity (Frone *et al.*, 2011). However, this nanocrystalline has a few disadvantages due to their polar and hydrophilic character and despite being unique characteristic. These limitations make them poorly compatible with non-polar matrices, such as polyolefins, and induce loss of mechanical properties of composite material upon moisture adsorption (Frone *et al.*, 2011).

Cellulose fibrils are structural entities formed through a cellular manufacturing process, cellulose biogenesis, stabilized by hydrogen bonds and van der Waal forces. The fibrils contain crystalline and amorphous regions that can be generally separated from the given cellulose source into amorphous and/or crystalline components, by mechanical, chemical or a combination of mechanical, chemical or enzymatic processes, yielding cellulose nanofibrils (CNF) or cellulose nanocrystals (CNC) (Salas *et al.*, 2014).

Nanotechnology is the understanding and controlling of matter at dimensions of roughly 1-100 nm, where unique phenomena enable novel applications. Nanomaterials from cellulose and lignocelluloses play a role in the nanotechnology field. The development of cellulose nanofibers (CNFs) has attracted significant interest in the last few decades due to the unique characteristics they endow such as high surface area-to-volume ratio, high Young's modulus, high tensile strength and low coefficient of thermal expansion (Chirayil *et al.*, 2014).

1.2 Problem statement

As an agriculture plant, the oil palm wood has become one of the major crops that supply to the economic growth of Malaysia. The total biomass of 95 million tons are generated annually, this lignocellulosic material produces continuous supply for the new oil palm biomass industry. The trunks become available during the replanting season on cycle of every 25-30 years. Holocellulose in oil palm wood range from 72 to 78 % (Lamaming *et al.*, 2015). This make cellulose suit for raw material in cellulose nanofiber application. Several researches have been done with microstructure of cellulose and provide useful knowledge of oil palm wood (Lamaming *et al.*, 2015).

Acid hydrolysis is the most suitable method for nanocellulose production. The side effect of acid hydrolysis contributed to serious environmental threats due to acid recovery and reuse issues into the effluent stream (Harmsen *et al.*, 2010). The unique property of ionic liquids is that they have essentially no vapour pressure, which makes them optimal replacements for volatile organic solvents traditionally. A nice feature of ionic liquids is that yields can be optimized by changing the anions or properties of the cation. In addition, several ionic liquids show enhancement in reaction rates and selectivity, compared to organic solvents with the added advantages of ease of recovery and reuse of these ionic solvents (Yadav *et al.*, 2003).

Due to fast development and growth in this few decades, has led to using nanocrystalline as a new product continuously. The market for nanocrystalline cellulose is expected to be mainly driven by end-user industries such as automotive, paint and coating. In this study, the extraction and the characterization of nanocellulose from oil palm wood will be carried out to maximize the use of nanocellulose in varies field of industries.

1.3 Objectives of Study

The objectives of this study are:

- a) To extract cellulose from oil palm wood.
- b) To convert cellulose to nanocellulose using ionic liquid as pre-treatment solvent.
- c) To characterize physical, chemical and thermal properties of 1-butyl-3-methylimidazolium chloride (BMIMCl) treated cellulose.

1.4 Expected Outcomes

Tappi test were used to extract cellulose from oil palm wood. Ionic liquid pre-treatment methods also can increase the production of nanocellulose. In this study, it was expected that there will be some differences in the physical and chemical appearance of the cellulose obtained from this pre-treatment. Many studies have been made by several scientists but not all they can solve the problem of biomass. Utilization of waste material such as palm frond also being studied. The competition in industries for producing biodegradable products has increased tremendously. Different approaches have been attempted to use biomass as natural biopolymer for production of biodegradable plastics. Recently, biomass is became a massive problem to many country due to its effect to environment.

So, this research will help palm oil sector to solve the biomass problem of oil palm frond and turn them into valuable product which is nanocellulose. The treated and untreated cellulose were analysed using X-Ray Diffraction (XRD), Field Emission Scanning Electron Microscope (FESEM), Thermogravimetric Analysis (TGA), and Fourier Transform Infrared Analysis (FTIR).

CHAPTER 2

LITERATURE REVIEW

2.1 Non Wood/Oil Palm

The differences in chemical composition between the various types of oil palm fibres are shown in Table 2.1. Extractive content is considered to play the most important role in obstructing the formation of microbes in the early stage of cloning. OPT fibre is also believed to have a high dimensional stability because its high extractive content (especially oil and wax) will reduce the hygroscopic fibre property (Taylor *et al.*, 2008).

	Extractive (%)	Holocellulose (%)	Cellulose (%)	Lignin (%)	Ash (%)
Empty fruit bunch (EFB)	3.21	80.09	50.49	17.84	3.4
Oil palm frond (OPF)	4.40	83.54	56.03	20.48	2.4
Oil palm trunk (OPT)	5.35	73.06	41.02	24.51	2.2
Hardwood	0.1-7.7	71-89	31-64	14-34	<1.1
Softwood	0.2-8.5	60-80	30-60	21-37	<1.1

Table 2.1: Chemical composition of oil palm wood fibres (Taylor *et al.*, 2008).

The utilization of OPT fibre in paper production will also reduce the pulp yield. This is because most extractives have a bad impact on chemical and mechanical pulping. Chemical analysis of oil palm fibres shows that the principal component is cellulose. OPF fibre contains the highest percentage of holocellulose (83.54%) and α -cellulose content (56.03%). The amount of cellulose in fibres will affect the property and economic production of fibres for various uses. In pulp and paper technology, the strength of paper depends on the content of cellulose in the natural plant. Plant materials with 34% and over cellulose content are characterized as promising for pulp and paper manufacture from a chemical composition point of view. From the result shown in table 2.1, OPT fibre showed the highest percentage of lignin (24.51%), followed by OPF (20.48%) and EFB (17.84%). This was expected since mature tissues at the trunk accumulate higher amounts of metabolic products compared to the frond and branches (Taylor *et al.*, 2008).

Lignin is a preferable polymer and its removal during pulping requires high amount of energy and chemicals. EFB fibres had the lowest lignin content, which indicates that this material can undergo bleaching more easily and with the utilization of fewer chemicals than OPT fibres. However, this result was not in agreement with previous studies. Those researchers found that EFB contained the highest percentage of lignin. This discrepancy may be attributed to different methodologies and different plant varieties used in our analyses. Chemical composition varies from plant to plant and within plants from different parts of the same plant. It also varies within plants from different geographic locations, ages, climate, and soil conditions. Oil palm fibres were found to have higher ash content compared to wood fibres. EFB fibre showed the highest percentage (3.4%) of ash. Ash consists of calcium and magnesium, but certain plants contain high levels of silica that cause the knife to

become blunt during the cutting process. This inorganic element will also create problems in a chemical sense, causing tearing during the pulping process, especially in the chemical recovery stage (Taylor *et al.*, 2008).

2.2 Composition of Non Wood Plant

The composition of non-wood plant can be divided into cellulose, lignin and hemicellulose. Cellulose have 2 type which are holo cellulose and α - cellulose. Lignin is one of the component of nanocrystalline that consist of H lignin, S lignin and G lignin, whereas hemicellulose is differ from cellulose in which it contains sugar units in composition.

2.2.1 Cellulose

Cellulose possess distinctive structural properties for example it has tension resistance is comparable to that of steel (Festucci-buselli, Otoni, & Joshi, 2007). However, cellulose have bright future commercial for the pulp, paper, and textile as well for chemical industries which supply to polymer product, cellulose biosynthesis in tree still not be fully discover. Due to incoming economic growth, abundant of tree cellulose for forest product industries, cellulose biosynthesis in trees is crucial (Festucci-buselli *et al.*, 2007). This unique cellulosic structure can be extracted from plants by a top-down approach into well-defined and multi-functional architectures that are available in reinforcing polymer (Ng *et al.*, 2015). Cellulose contains the most abundant renewable polymer resource available today. As a chemical raw material, it is generally famous that it has been used in the form of fibres or derivatives about 150 years for a wide spectrum of products and materials in daily life.

What has not been known until relatively recently is that when cellulose fibres are subjected to acid hydrolysis, the fibres yield defect-free, rod-like

crystalline residues. Cellulose nanocrystals (CNs) have collect in the materials community a tremendous level of attention that does not appear to be abandon. These biopolymer assemblies warrant such attention not only because of their better physical and chemical properties but also because of their inherent renewability and sustainability in addition to their abundance. They have been the subject of a wide array of research efforts as reinforcing agents in nanocomposites due to their low cost, availability, renewability, light weight, nanoscale dimension, and unique morphology. Indeed, CNs is the fundamental constitutive polymeric motifs of macroscopic cellulosic-based fibres whose sheer volume dwarfs any known natural or synthetic biomaterial (Habibi, Lucia, & Rojas, 2009). Acid hydrolysis of cellulose fibres yields highly crystalline rod like particles through selective degradation of the more accessible material. The cellulose nanocrystals that result from this degradation are of colloidal dimensions, and when stabilized, they form aqueous suspensions, the properties and applications of which are reviewed in a recent article. Using acid hydrolysis, native cellulose suspensions have been prepared from a variety of sources, including bacterial cellulose, microcrystalline cellulose, sugar beet primary cell wall cellulose, cotton, tunicate cellulose, and softwood pulp. The hydrolysis conditions are known to affect the properties of the resulting nanocrystals.

For example, a longer reaction time leads to shorter nanocrystals. Different acids also affect the suspension properties: hydrochloric acid hydrolysis yields cellulose rods with minimal surface charge, whereas the use of sulphuric acid provides highly stable aqueous suspensions, due to the esterification of surface hydroxyl groups to give charged sulfate groups. The rod like shape of the charged cellulose nanocrystals will leads to the formation of anisotropic liquid crystalline phases, above a critical concentration which have been extensively studied.

Nanocrystal size, dimensions, and shape are also determined to a certain extent by the nature of the cellulose source. The degree of crystallinity of the cellulose within the organism, as well as the dimensions of the micro fibrils, varies widely from species to species. Nanocrystal size, dimensions, and shape are also determined to a certain extent by the nature of the cellulose source. The degree of crystallinity of the cellulose within the organism, as well as the dimensions of the micro fibrils, varies widely from species to species (Beck-candanedo *et al.*, 2005).

2.2.2 Lignin

Lignin is one of the components in nanocrystalline. It has its own role in plant cell and makes the system run well. Lignin is a polymer of aromatic subunits usually derived from Phenylalanine. Function of lignin is to serve as matrix to provide additional rigidity and compressive strength as well, maintain the cell wall and water permeable. Lignin made up of hydroxycinnamyl alcohols, coniferyl alcohol and sinapyl alcohol (Vanholme *et al.*, 2010). In addition to developmentally programmed deposition of lignin, its biosynthesis can also be induced upon various biotic and abiotic stress conditions, such as wounding, pathogen infection, metabolic stress, and perturbations in cell wall structure. Because lignin protects cell wall polysaccharides from microbial degradation, thus imparting decay resistance, it is also one of the most important limiting factors in the conversion of plant biomass to pulp or biofuels. The removal of lignin from plant biomass is a costly process; hence, research efforts are now aimed at designing plants that either deposit less lignin or produce lignin that are more amenable to chemical degradation (Vanholme *et al.*, 2010).

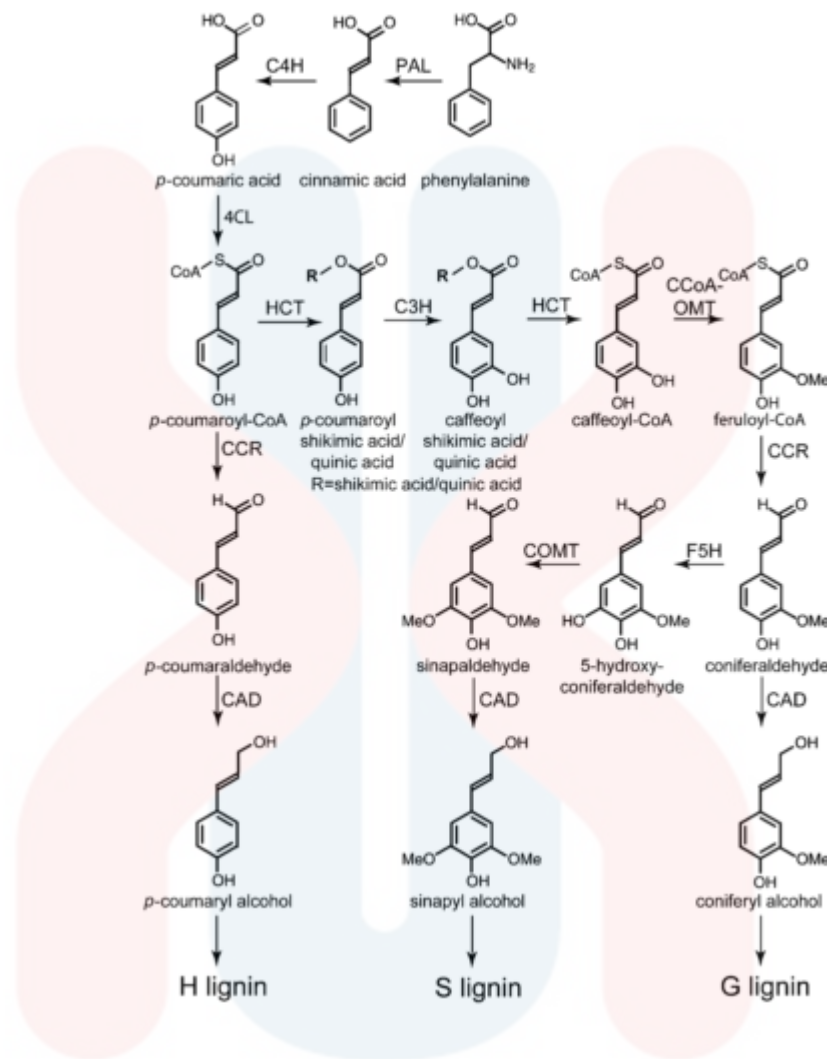


Figure 2.1: The main biosynthetic route toward the monolignols *p*-coumaryl, coniferyl and sinapyl alcohol (Vanholme *et al.*, 2010).

In freeze-drying these CNC suspensions to solid forms, cellulose nanocrystals were observed to self-assemble into micrometre-long, highly crystalline (91.2%) and nonporous or macroporous fibres with an average diameter of 386 nm that remained in the assembled fibrous form in suspensions with hand shaking and mechanical stirring for prolonged time. Cellulose nanofibers have been generated by electro spinning of cellulose and cellulose derivative solutions, a process relying on chemicals in dissolution and derivatization of cellulose which resulting in the loss of the native cellulose $I\beta$ crystalline structure. The intriguing self-assembling behaviour

of CNCs suggests this cryogenic process to be an attractive non-chemical alternative for fabricating ultra-fine cellulose fibres from aqueous nanocellulose suspensions while achieving highest crystallinity surpassing even its source (Jiang & Hsieh, 2013).

2.2.3 Hemicellulose

Hemicelluloses are polysaccharide which is differing from cellulose which contains sugar units in composition (D- xylose, D- mannose, D- glucose, L- arabinose, etc). They are branched polymers (amorphous) and shorter chains (degree of polymerization of up to 200 sugar units) compared with cellulose. Monosaccharides comprised of structure of hemicellulose are consist of by hexose sugars comprising glucose, mannose and galactose and pentose comprising arabinose and xylose, and produce many quantity of uronic acids and deoxy-hexose in vegetables (Cristina *et al.*, 2015). Hemicelluloses, which rank second to cellulose in plant cell walls, are heterogeneous polymers mainly containing xylose, ara-binose, galactose, mannose, 4-O-methyl-d-glucuronic acid and soon. Because of their significant amount, usage of hemicellulose is limited on an industrial scale because of their complex and heterogenous structure. Improvement ideal techniques to get more homogenous hemicellulose portion from lignocellulosic the efficient conversion of hemicellulose to high-value added chemicals will be aided by biomass (Li *et al.*, 2016).

2.3 Nanocellulose

Nanocellulose can be categorised into few types, including microfibrillated cellulose (MFC), nanocrystalline cellulose (NCC) and bacterial nanocellulose (BNC). Each type of nanocellulose has different structure.

2.3.1 Structure

Cellulose is the important compartment which been produce in the biosphere, been estimate the production be over 7.5×10^{10} tons. The first cellulose been discovered and isolated by Anselme Payen in 1838, later on physical and chemical properties have been discovering. Further discovered are continuing with respect to its biosynthesis, assembly, and structural features. Regarding of this source, cellulose can be characterized as a high molecular weight homopolymer of -1,4-linked anhydro-D-glucose units in which every unit is corkscrewed 180° with respect to its neighbours, and the repeat segment taken to be a dimer of glucose, known as cellobiose (*see* Figure 2.2) (Habibi *et al.*, 2009) .

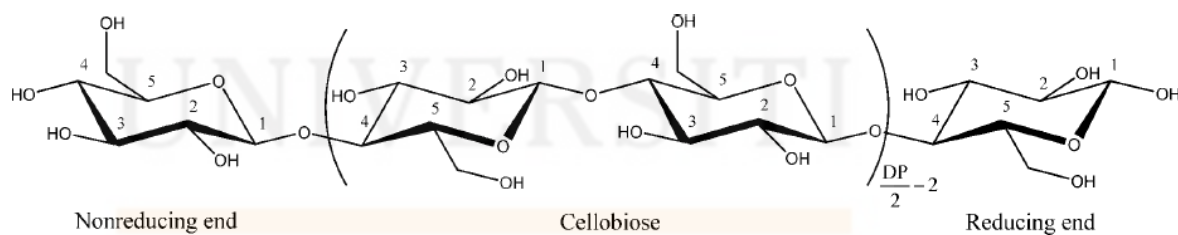


Figure 2.2: Chemical structure of cellulose (Habibi *et al.*, 2009).

2.3.2 Microfibrillated cellulose

Microfibrillated cellulose (MFC) is formed from delamination of wood pulp by mechanical pressure before and/or after chemical or enzymatic treatment. Its diameter range from 5 – 60 nm and its length consist of several micrometres (Gray *et al.*, 2011).

The production of MFC by fibrillation of cellulose fibres into nano-scale elements requires intensive mechanical treatment. However, depending upon the raw material and the degree of processing, chemical treatments may be applied prior to mechanical fibrillation. These chemical processes are aimed to produce purified cellulose, such as bleached cellulose pulp, which can then be further processed. There are also examples with reduced energy demand in which the isolation of cellulose micro fibrils involves enzymatic pre-treatment followed by mechanical treatments. Depending upon the raw materials and fibrillation techniques, the cellulose degree of polymerization (Plackett, 2010).

Micro fibrillated cellulose nanofibers (MFC) disintegrated from wood pulp can be used in polymer nanocomposites of high mechanical performance. These nanocomposites show a Young's modulus approaching 20 GPa. The reported strength is also high, although the data should be interpreted with care since flexural strength of brittle materials depends strongly on specimen geometry. Still, nanocomposites based on MFC nanofibers from wood pulp are a new class of materials with potential for exceptionally high mechanical performance. MFC nanofibers are expected to show high stiffness since the Young's modulus of the cellulose crystal is as high as 134 GPa. The MFC typically consist of disintegrated micro fibril aggregates with a lateral dimension in the scale of tens of nanometres (Henriksson *et al.*, 2007).

2.3.3 Nanocrystalline cellulose

Nanocrystalline cellulose (NCC) is formed from acid hydrolysis of cellulose from many sources. Its diameter range from 5 – 70 nm and its length is from 100–250 nm (from celluloses of tunicates, algae, bacteria) (Gray *et al.*, 2011). Nanocrystalline cellulose derived from acid hydrolysis of native cellulose possesses different morphologies depending on the origin and hydrolysis conditions. Acid treatment (acid hydrolysis) is the main process used to produce nanocrystalline cellulose, which are the smaller building blocks released from the original cellulose fibres. Native cellulose consists of amorphous and crystalline regions, and the amorphous regions have lower density compared to the crystalline regions, so when cellulose fibres were subjected to harsh acid treatment, the amorphous regions break up, releasing the individual crystallites (Peng *et al.*, 2011).

The properties of NCC depend on various factors, such as cellulose sources, reaction time and temperature, and types of acid used for hydrolysis. Nanocrystalline cellulose derived from acid hydrolysis using various forest product sources can disperse in water due to their negative charged surfaces. At low concentration, NCC particles are randomly oriented in aqueous suspension as an isotropic phase, and when the concentration reaches a critical value, they form a chiral nematic ordering, where NCC suspensions transform from an isotropic to an anisotropic chiral nematic liquid crystalline phase. The phase behaviour of NCC is sensitive to the presence of electrolytes and their counter ions, as well as macromolecules. The effect of added electrolyte on the phase separation of NCC (Peng *et al.*, 2011).

2.3.4 Bacterial Nanocellulose

Bacterial Nanocellulose (BNC) is formed from bacterial synthesis. Its diameter range from 20 - 100 nm and it's have different type of nanofiber network (Gray *et al.*, 2011) . Bacterial cellulose (BC) is produced as an extracellular primary metabolite by bacteria belonging to the genera *Acetobacter*, *Agrobacterium*, *Alcaligenes*, *Pseudomonas*, *Rhizobium*, *Aerobacter*, *Achromobacter*, *Azotobacter*, *Salmonella* or *Sarcina*. Its most efficient producers are gram negative acetic acid bacteria *Acetobacter xylinum* which has been reclassified and included within the novel genus *Gluconacetobacter*, as *G. xylinus*. Culture is normally carried out in static conditions at temperatures around 28 - 30°C. The system initially becomes turbid and later on a white pellicle appears on the surface of the fermentation vessel. Since bacteria used are aerobic, cellulose pellicle is formed only in the vicinity of the oxygen-rich air-liquid surface, and it adopts its shape (Charreau *et al.*, 2013).

Several reasons for the formation of the cellulose pellicle by bacteria have been proposed, such as a mean of maintaining their position close to the surface of culture solution and also as a protective coating to guard bacteria from ultraviolet radiation, or from enemies and heavy-metal ions whereas nutrients diffuse easily along the pellicle. It is believed that cellulose molecules are synthesized in the interior of the cell and spun out to form protofibrils of ca. 2-4 nm diameter, which are crystallized into micro fibrils, these into bundles and the latter into ribbons. BC ribbons have been reported to have a thickness of approximately 3-4 nm, 70-100 nm in width, and 1-9 µm in length. Produced BC ribbons form a dense reticulated structure stabilized by extensive hydrogen bonding whose solid portion is less than 1%, and the rest is water (Charreau *et al.*, 2013).

2.4 Isolation of cellulose

Generally, the biomass recalcitrance is mainly due to the presence of highly complex phenolic polymer lignins, which resists degradation of plant cell wall polysaccharides and thus reduces the accessibility to cellulose. Hence, removal of lignin is the key processing step during the extraction of cellulose fibres. In chemical pre-treatment, non-cellulosic components (lignin and hemicellulose) needs to be dissolving from the lignocellulose and subsequently separate from the production process. However, the fact that non-linear polymer lignin is built in chemically diverse and poorly reactive linkages, which require costly and harsh pre-treatment to degrade this component. The major influence on the operation costs are involved reduction of lignocellulose particle size and subsequent depolymerisation process. Thus, effectively overcoming the biomass recalcitrance structure and releasing the locked polysaccharides is the crucial task for the emerging of nanocellulose industries (Yahya *et al.*, 2014).

The biomass pre-treatment is aims to reduce the degree of polymerization of cellulose (glucose congregate forming a polymer molecule) by breaking the lignocellulose complex, solubilized the lignin and hemicellulose, increase porosity and surface area of hidden cellulose, and reduce the crystallinity of cellulose. This fractionation process makes the cellulose accessible to hydrolysis reaction and improves depolymerisation of cellulose chain into nano-dimension. The biomass pre-treatment process is categorized into 3 main classes in terms of pH, which is acidic, alkaline and neutral (organosolv and ionic liquid treatments). However, most of the processes suffer from unsatisfactory separation of cellulose and lignin, where over-degradation lead to the formation of by-product (polysaccharide is change to sugars

rather than nanocellulose). Furthermore, the usage of severe reaction conditions (high temperature and pressure) makes the operation cost higher and increase the investment risks, as larger capital investment are required compared to pulp and paper manufacturing (Yahya *et al.*, 2014).

2.5 Application and properties of nanocellulose

Nanocellulose can be used in various field of application due to its unique characteristics and properties. Nanocellulose exhibits excellent mechanical properties, biodegradable and non-toxic. For example, BNC is used in medical application; MFC is used in films due to its optical properties.

2.5.1 Application of nanocellulose

The function of the plant cell is to cure internal and external wound care. Bacterial Nano Cellulose (BNC) use in medical application such as wound dressing. Bacterial strain and on drying procedure of the membrane are the properties of bacterial cellulose for wound dressing. So, focusing on network parameters by IR spectroscopy, gas permeability, and Young`s modulus measurement are important. For some case which use BNC inside the body (implant) for cardiovascular diseases which is the no 1 illness in the worldwide and the important application area. Coronary bypass (inner diameter < 6 mm) only autograph (vena saphena and arteria mammaria) can be used. Using the BNC vessel substitutes in the cardiovascular field implantable tubes with inner diameter of 3-5 mm and a length of more than 10 cm are needed. Bio formed hydrogel tubes are characterized by mechanical stability, good surgical handling, and a smooth inner surface. Inner surface of the BNC have better smoothness than common synthetic implant material (Klemm *et al.*, 2009).

The major advantages of nanocellulose are the excellent mechanical strength,

nontoxic, biodegradable, broad availability with various functionality. Like the characteristic of cellulose, the nanoscale of this biopolymer changes dramatically. It is stronger than steel, stiffer than Kevlar (with specific Young's modulus 65 J g⁻¹ for micro fibrils and 85 J g⁻¹ for nanocrystals) high elasticity, lighter in weight, conductive, highly absorbent and so on. With all these superior properties, nanocellulose has great potential in applications such as strength enhancers in paper, as additives to composites, polymer reinforcement, in emulsions, films and barrier coating, water treatment, as oxygen barriers for food packaging, electronics, cosmetic, pharmaceuticals and biomedical devices (e.g. scaffolds in tissue engineering, artificial skin and cartilage, wound healing and vessel substitute (Yahya *et al.*, 2014).

Oil palm produces copious amounts of recyclable biomass that are generated from various processing stages. Only 10% of the oil palm plant is economically valuable, used for the production of oil palm oil and palm kernel oil, whereas the remaining 90% is constituted of oil palm trunk (OPT), oil palm fronds (OPF), empty fruit bunches (EFB), and palm pressed fibres (PPF). Malaysia, the leading producer of palm oil, produced and estimated 56.9 million tonnes of oil palm in 2000. Recently, nanotechnology was introduced in producing bio-fibres and nano-size fibres with high strength and surface area. Therefore, there is a great need to be able to introduce the fibres from the oil palm trunk in order to sustain wood industry sector in Malaysia (Surip *et al.*, 2012).

2.5.2 Properties of nanocellulose

Mechanical properties for natural fibres are influenced by chemical composition and location in plant. Factor that affect the fibre properties are maturity, separating processes, microscopic and molecular defects. The mechanical properties

for micro fibrils must be enormous and less dispersed than lignocellulose fibres from which they are released from because of more homogenous. These magnificent mechanical properties make cellulose nanoparticles suitable for reinforced polymer composite. The optical properties of nanocellulose films are obtain by determining the regular light transmittance with a UV-visible spectrometer. Films made from micro fibrils cellulose (MFC) can be optically transparent if the cellulose nanofibers are densely packed, and to avoid the light scattering, the interstices between the fibres are small (Dufresne, 2013; Ioelovich, 2016).

The value of the elastic modulus of pure crystalline cellulose is not yet well-defined. However, several theoretical molecular model and X-Ray diffraction measurements have been done in order to measure the elastic modulus of crystalline cellulose. In 1962, a value of around 140 GPa is evaluated by X-Ray diffraction. Recent molecular simulation techniques have also affected the elastic modulus value. Thus, mechanical properties of crystalline cellulose can compete with current engineering materials such as glass or steel, especially when the density is taking into account. The lower density of cellulose (1500 kg/m^3) gives to cellulose a very high specific modulus. It allows cellulose fibres to compete with current engineering reinforcement materials. However, the elastic modulus value of cellulose fibre inside a sheet of paper is lower than 140 GPa because fibres are generally damaged during the paper making process which decreases their elastic modulus (Denoyelle, 2011).

2.6 Ionic Liquid

Ionic liquid has shown great potential in becoming the eco-friendly, as well as free of both arsenic and pentachlorophenol, and yet they also show effectiveness as nanocellulose preparation. A research has been done on this subject ILs in wood

has been addressed in and protic ILs with organic anion as nanocellulose has been discussed in. Some ILs with a nitrate counter ion, such as 3-alkoxymethyl-1-methylimidazolium tetrafluoroborate, 1-butyl-3-methylimidazolium chloride (BMIMCl), hexafluoride-phosphates, didecylmethyl ammonium and benzalkonium nitrates. The mechanical properties of the wood treated with the former two ILs were lower than natural wood. Wood treated with the chloride and acesulfamate was, however, characterized by lower absorption of free water than the control wood. Only in the case of treatment with acesulfamate was the hygroscopicity of the treated wood distinctly lower than that of control wood. Due to the high interest in the applications of ILs, new, cheaper, multifunctional ILs which are easy to prepare are highly desired (Abu-eishah, 2015).

The existence of water in ionic liquid solution may have also contributed to the homogenous swelling. However, pretreatment on the oil palm biomass fibre by removing lignin, hemicellulose, and some other components contained in the fibre have been attribute to the subsequent swelling and dissolution mechanism of fibre. On the basis that ionic liquid are thermostable and non-volatile, and can be easily prepared and recycled this process of dissolution and regeneration of cellulose seems to be promising 'green' process for the preparation of regenerated cellulosic materials, and can overcome the inherent environment problems of waste (toxic) gases in the current industrial process for manufacturing cellophane and viscose rayon. In this study, BMIMCl is used to dissolve the cellulose (Surip *et al.*, 2012).

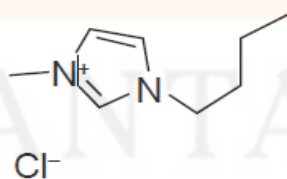


Figure 2.3: The structure of 1-butyl-3-methylimidazolium chloride (BMIMCl) (Surip *et al.*, 2012).

CHAPTER 3

MATERIALS AND METHOD

3.1 Materials

Materials used in this research were divided into apparatus and chemical.

3.1.1 Apparatus & Chemical

Chemicals: Sodium hydroxide (NaOH), Ethanol, benzene/toluene, acetone, Sodium chlorite (NaClO₂), 10% of acetic acid (CH₃COOH), acetone and 1-butyl-3-methylimidazolium chloride (BMIMCL).

Apparatus: Grinder, 70-mesh stainless steel sieve, beaker, extraction timble, buckner funnel, Soxhlet set, pipe, round bottle (500 ml), heating mentle, conical flask (250 ml), hot plate stirrer, vacuum pump, filtering flash and crucible and filter paper.

3.2 Methods

The methods for this experiment consist of sample preparation followed by extraction process, production of holocellulose, production of α -cellulose and production of nanocellulose.

3.2.1 Sample preparation

The oil palm wood was dried under the sun for 7 hours to prevent the growth of microorganism and to reduce the moisture content of the sample. After that, the samples were crushed, grinded and sieved until the size of 210 micrometer was obtained.

3.2.2 Extraction process

10 gram of powder sample (oil palm wood) was used for this extraction. The powder sample was inserted into timber. Ethanol and benzene was prepared ration 2: 1

(200ml: 100ml) solvent. The solvent was placed in round bottle. Soxhlet set was set using a tube which had water in and water out. Soxhlet set and round bottle was placed together which soxhlet set is on top and round bottom flask bottom shown in Figure 3.1. Round bottom was placed at heating mantle maximum temperature for the first 30 minutes. Later, the temperature were reduced to half until complete (sample turn to yellow/green). Solvent will turn clear (inside the timber) were took 5-6 hours. The end products consist of 2 product which was free extractive (timber) and extractive (round bottle). The product that was chosen is free extractive. Then, the mixture was allowed to cool, which were then washed with distilled water and acetone. After that, the samples were dried at oven less than 50 °C for 24 h to remove residual solvents and the solid in powder form were collected. The powder sample was known as free extractives sample and use for next process.



Figure 3.1: Extraction process set up.

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3.2.2.2 Holocellulose

5 gram of free extractive powder was put in conical flask. The chemical used are 1.5 gram NaClO_2 , 10% acetic acid (5ml), magnetic stirrer and distilled water (dH_2O) (100ml). The sample was heated on hot plate with cover the conical flask at 70°C (boiling point NaClO_2) about 30 minutes (in fume chamber) were covered with small conical flask to prevent the strong odour of chemicals gas release from the mixture and not cover with aluminium foil. First 30 minute, 10% acetic acid (5ml) was added inside conical flask. After 30 minutes, 1.5 gram NaClO_2 was added inside conical flask. Both step considered 1 step. This step repeated for 4 times (4 hours) should be standard TAPPI and bleached until completed (5 times). Cooling process took place inside the ice box for 30 minutes with ice. After that, the filtering flush and vacuum pump were used. The sample is washed with distilled water and acetone (25ml) to remove any excess of residual chemicals. Then, sample was dried in oven for 50°C for 24 hours. Lastly, the holocellulose was obtained.

3.2.2.3 α -cellulose

The production of α -cellulose was obtained by ridding of hemicellulose from the holocellulose sample. Briefly, about 2 g of sample and 15 ml of 17.5 % of NaOH were placed in a conical flask and stirred for 1 min. After that, 10 ml of 17.5 % of NaOH was added and stirred for 45 s. 10 millilitres of 17.5 % of NaOH was then slowly added into mixture again and stirred for 3 min. This step was repeated for 4 times in 10 min, each addition was about 2.5 min. The mixture in conical flask was then covered and stirred for 30 min by using magnetic stirrer. Later on, 100 ml of distilled water was added and the mixture was stirred at room temperature for another 30 min. The precipitated material was then filtered using a Buchner funnel,

washed thoroughly with 8.3% of NaOH and cool distilled water as well as followed by soaking with 2 N of acetic acid. Lastly, the residue was dried in an oven at 50 °C for 24 h. All these steps were allowed the elimination of hemicelluloses, while leaving the α -cellulose.

3.2.3 Nanocellulose preparation

A mass percentage of 2 % alpha cellulose was added in a beaker containing BMIMCl ionic solution and a magnetic stirrer. Then, the mixture was heated in an oil bath at 90 °C under vigorous stirring on a magnetic hot plate stirrer for 12 h. After 12 h, the mixture was cool at room temperature for 30 min (Tan *et al.*, 2015). The precipitate of nanocellulose was then filtered and washed with distilled water. Generally, the washing process was repeated for several times until the off-white precipitate is achieved and the water is not oily. The whole procedures were repeated with the mass fraction of 5 % extracted cellulose and ionic liquid solution. The nanocellulose precipitates were kept in -80 °C freezer for 24 h followed by freeze drying for 24 h to obtain nanocellulose in powder form. The nanocellulose samples must be in frozen state (Figure 3.12) before undergoing the freeze drying process. Finally, the nanocellulose samples of the oil palm were labelled NCC 2 wt% and NCC 5 wt% depending on the mass fraction. Table 3.1 show the cellulose and IL content.

Table 3.1: Cellulose and IL content in IL treatment.

Mass fraction	Total amount (cellulose)	Total amount (IL)
2 wt %	1g	49g
5 wt %	1g	19g

3.3 Analytical Method

The analytical method for this experiment consists of characterization using XRD, FTIR, FESEM and TGA.

3.3.1 X-ray Diffraction (XRD)

An X-Ray generator with CuK α radiations ($\lambda = 1.5406 \text{ \AA}$) were generated at a voltage of 45 kV and current of 30 mA was used to characterize the crystalline structure of the nanocrystals isolated. All samples were scanned in 2θ ranges varying from 5° to 50° at $2^\circ/\text{min}$. The crystallinity index was calculated based on Segal, Creely, Martin, & Conrad (1959) the following equation:

$$C_I (\%) = 100 \times (I_{002} - I_{am}) / I_{002}$$

Where I_{002} is the peak intensity corresponding to crystalline ($2\theta = 18^\circ$) and I_{am} is the peak intensity of the amorphous region ($2\theta = 22^\circ$).

The crystallite size of each sample perpendicular to planes, w (nm), was calculated by using Scherer Equation:

$$w = k \lambda / \beta \cos \theta$$

Where θ is the diffraction angle, $k = 0.94$ (correction factor), $\lambda = 0.154 \text{ nm}$ and β is the corrected angular width in radians at half maximum intensity of the peak.

3.3.2 Fourier Transform Infrared (FTIR)

The presence of any changes in functional group during treatment in the samples was scanned by FTIR spectroscopy. The sample was recorded using Thermo Scientific model Nicolet I S10 spectrometer. Dried sample were pounded together with KBr and pressed into a pallet. The pallet was then viewed in the spectrometer in the range from $500\text{--}4000 \text{ cm}^{-1}$.

3.3.3 Thermogravimetric analysis (TGA)

Thermogravimetric analysis was performed to determine the thermal decomposition of the cellulose after each treatment. The thermal stability data were collected on a Perkin Elmer TGA 7. The sample of 7 mg were burned under temperature ranging 25 ° C -550 ° C at a heating rate of 10 k / min under a nitrogen gas flow 20 cm³ min⁻¹.

3.3.4 Field Emission Scanning Electron Microscope (FESEM)The morphology of the sample was examined by JEOL (JSM – 7800 F) Fesem at an acceleration voltage of 5 kV. The sample was sputtered coated with gold using sputter coater.

3.4 Flowchart

This research involves sample preparation, extraction process, chemical treatment and characterization. Overall experiment procedure for this research is shown in Figure 3.2.

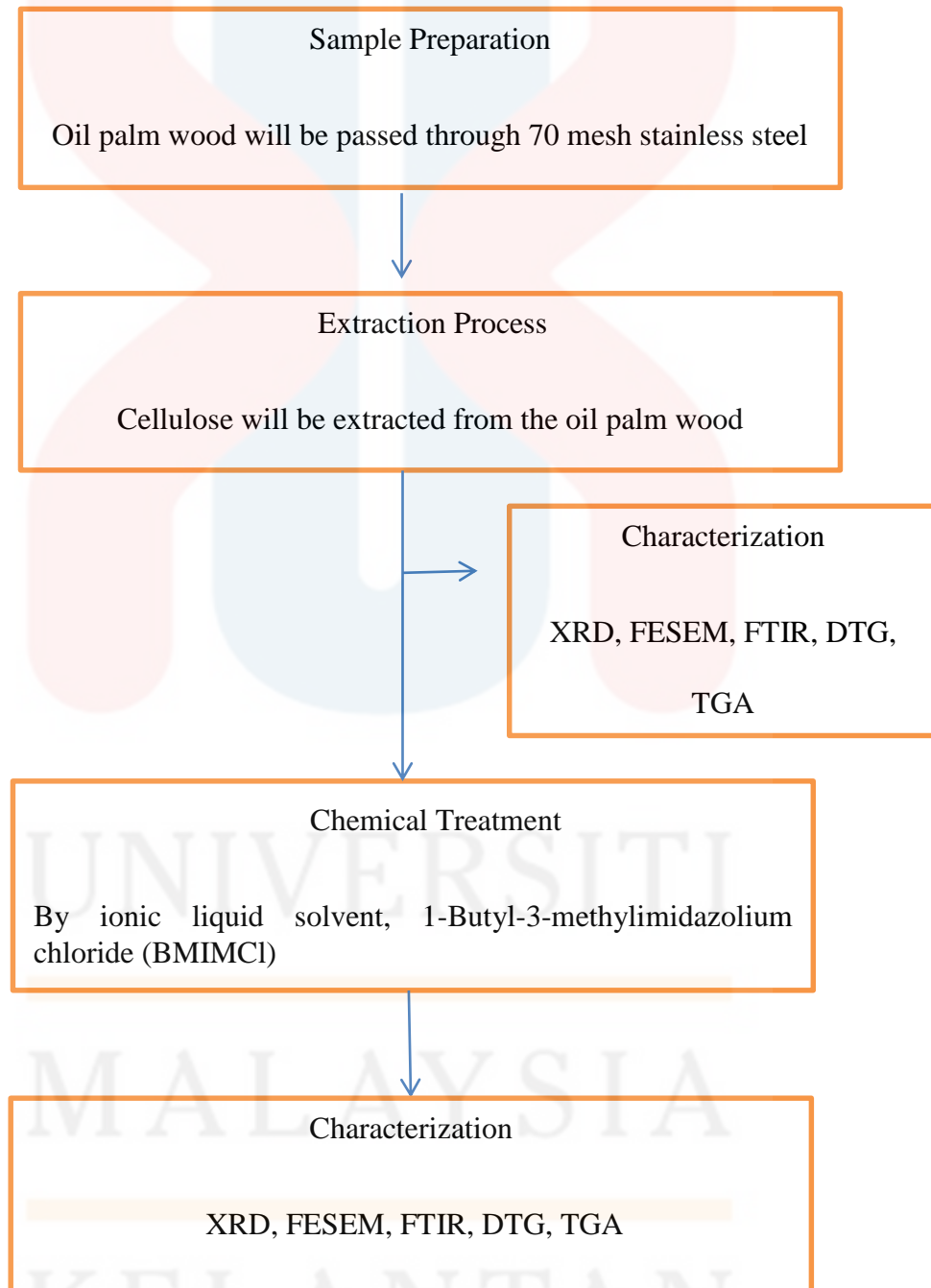


Figure 3.2: Overall experiment procedure in this research.

Chapter 4

RESULTS AND DISCUSSION

4.1 XRD ANALYSIS

The X-Ray Powder Diffraction (XRD) method was used to determine the percentages of crystallinity index and crystallite size of the produced cellulose from the alpha cellulose, pure cellulose, 2 wt% and 5 wt% ionic liquid treated cellulose. Figure 4.1 shows X-Ray diffractograms obtained for all samples and all been listed intensity at 2θ value of 22° to their crystalline structure of cellulose and a shoulder peak in the region $2\theta = 18^\circ$. These 2 peaks of diffraction intensity indicated that all of the cellulose produced was cellulose 1 type. Cellulose crystallinity is an important factor which determines the rigidity, elasticity, absorption-desorption of fibres and also the thermal stability of the cellulose (Lamaming, Hashim, Peng *et al.*,2015).

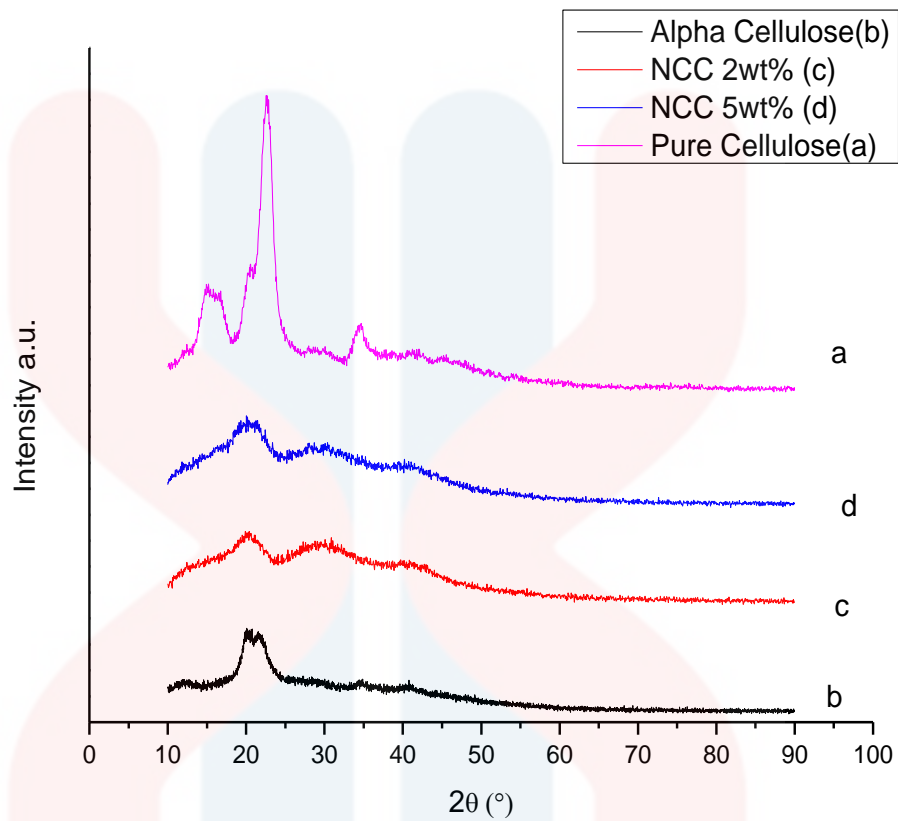


Figure 4.1: XRD curve for oil palm wood

The crystallinity index for the samples of pure cellulose, alpha cellulose, 2wt% and 5 wt% of BMIMCl treated cellulose are 72.69 %, 46 %, 6.82 % and 7.97 % respectively. The crystallinity index of alpha cellulose and pure cellulose is higher than BMIMCl cellulose. The crystallite size for the pure cellulose, alpha cellulose, 2 wt% and 5 wt% of BMIMCl treated cellulose are 4.68 nm, 2.74 nm, 2.23 nm and 1.93 nm respectively.

Table 4.1: Crystallinity index (CrI) and crystallite size of untreated cellulose and treated cellulose.

Samples	Crystallinity index (%)	Crystallite size (nm)
Pure cellulose	72.69	4.68
Alpha cellulose	46	2.74
2 wt %	6.82	2.23
5 wt %	7.97	1.93

The result shows the crystallite sizes of the BMIMCl treated cellulose (2 wt% and 5 wt%) smaller than alpha cellulose and pure cellulose because of the incomplete growing of crystallites after regeneration (Gao *et al.*, 2011). Referred to Table 4.1, it can be concluded that after treatment crystallinity index and crystallite size are lower than before treatment. Efficient removal of non-cellulosic polysaccharides such as hemicelluloses and lignin matrix that attached to the celluloses fibres would also contribute to increase crystallinity.

These results indicate that transformation from highly crystalline to amorphous occurred after the dissolution and regeneration of cellulose in BMIMCl. In comparison with the original cellulose, the intensity of diffraction peaks of the regenerated cellulose reduced significantly the crystallinity of the regenerated cellulose was lower than the original cellulose. This means that, the dissolution process, BMIMCl rapidly broke intermolecular and intermolecular hydrogen bonds and destroys the original crystalline form. Moreover, the regeneration process was so not permanent that it was unfavourable to the formation of cellulose crystallization.

4.2 FTIR ANALYSIS

Figure 4.2 illustrates the Fourier transform infrared spectroscopy (FTIR) spectra of the alpha cellulose, pure cellulose, In general, the spectra for all sample are similar. The FTIR identifies the functional groups that are present in the sample. As a lignocelulosic material, oil palm composed mainly hemicellulose, lignin and celluloses. Therefore, alkenes, aromatics, esters, ketones and alcohols with different oxygen-containing functional group are the main composition of these material (Abraham *et al.*, 2011). All the spectra dominated by signals in the peak ranging 3390 - 3232 cm^{-1} due to stretching vibrations of OH groups. The hydrophilic tendency of materials also reflected in the broad absorption band in ranging from 3521 - 3078 cm^{-1} region. The existence of O-H is attributed to the moisture content where hydroxyl is found in cellulose, hemicellulose and lignin (P.Sherman Hsu *et al.*, 1997). Cellulose being a linear polymeric compound has some important functional group within the cellulose units. At the range of 2910 - 2874 cm^{-1} which indicates the aliphatic saturated C-H stretching associated with methylene groups in cellulose.

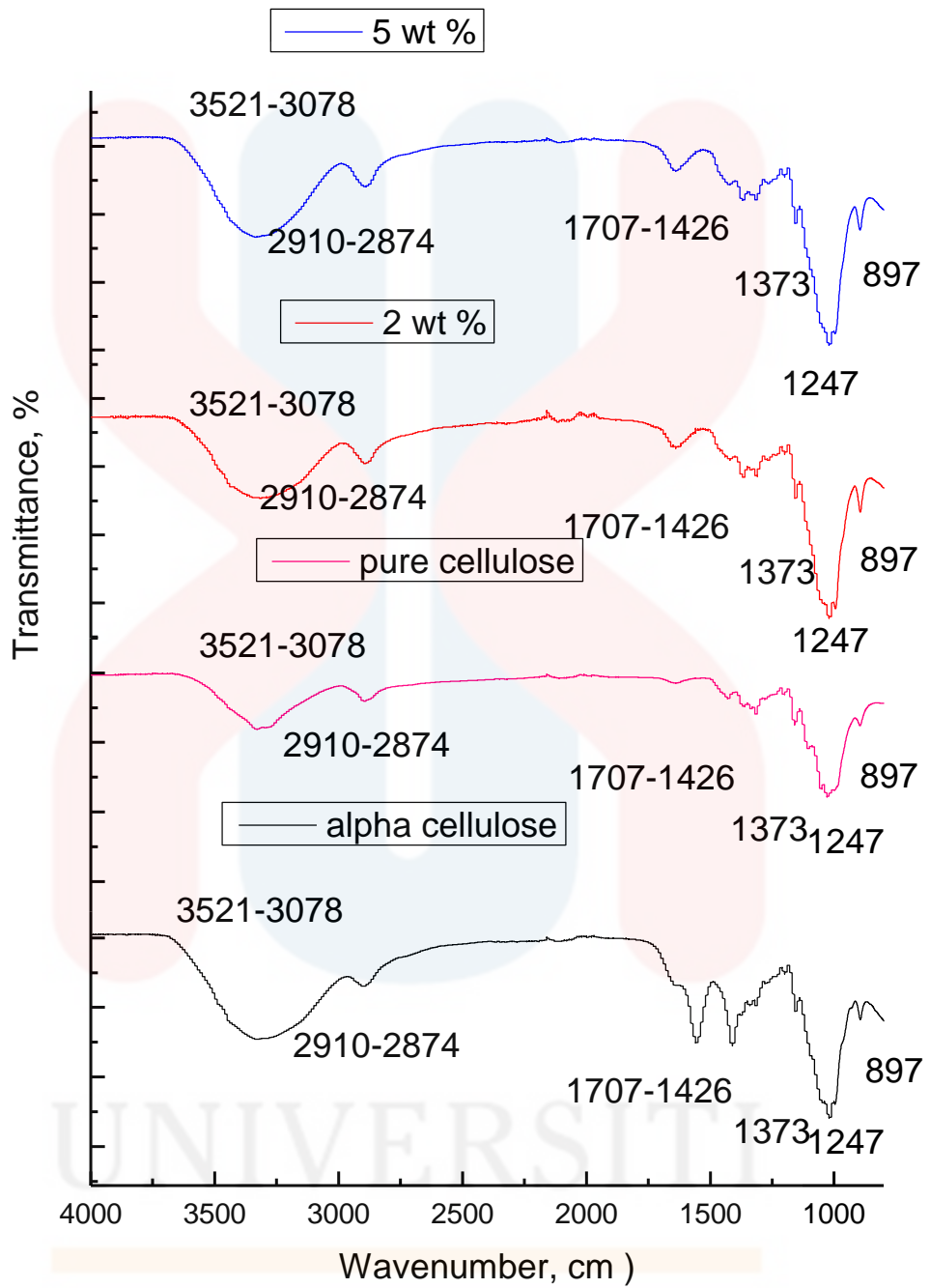


Figure 4.2: Infrared spectra for oil palm wood.

It is well known, that the lignin is a multifunctional natural polymer built up by oxidative coupling of three major C6-C3 phenylpropanoid units, which is randomized structure in a tridimensional network by certain interunit linkages. The peak at 1707 cm^{-1} and 1723 cm^{-1} in the spectra and treated sample is assigned to the $\text{C}\equiv\text{O}$ stretching of the acetyl. Uronic ester groups of hemicellulose or to the ester linkage of carboxylic group of the ferulic and *p*-coumaric acids of lignin (Sun *et al.*, 2005). The peak at 1640 cm^{-1} may attribute to the bending mode of the absorbed water and a contribution of some carboxylate groups. Aromatic hydrocarbons of lignin showed absorption in the peak 1510 cm^{-1} and 1426 cm^{-1} correspond to carbon-carbon stretching vibrations in the aromatic ring. Deformation of C-H asymmetric can be found at peak around 1373 cm^{-1} .

These two peaks were found to vanish in the produced nanocrystal spectra indicating the removal of lignin. The intensity of the peak at 1247 cm^{-1} sharply decreased proving that the removal of hemicellulose. The increase of band at 897 cm^{-1} can be attributed to the typical structure of cellulose due to the β -glycosidic linkages of glucose ring of cellulose and rotation of glucose residue around the glucosidic (Gan *et al.*, 2004). From the figure we can see both untreated cellulose and regenerative cellulose shows familiar FTIR spectra, which means there was no significant difference between the structure of untreated cellulose and regenerative cellulose. There were only some physical changes during the whole dissolving and process.

4.3 GA ANALYSIS

Figure 4.3 shows the TGA curves for all samples. From Figure 4.3, the weight loss of the samples starts at temperature levels ranging from 50°C to 100°C due to evaporation of the moisture in the material tested. Being a lignocellulosic material, the composition of sample degraded below 400°C. Wax, pectin and hemicellulose also degraded at temperature of 180°C while cellulose and lignin degraded at temperature levels of 300°C and 400°C, respectively (Johar *et al.* , 2012). According to Hasan, (2013), the degradation of natural fibres takes place in two stages starting from the degradation of the amorphous phase consisting hemicellulose, lignin and crystalline phase, where the cellulose is dominant in the phase.

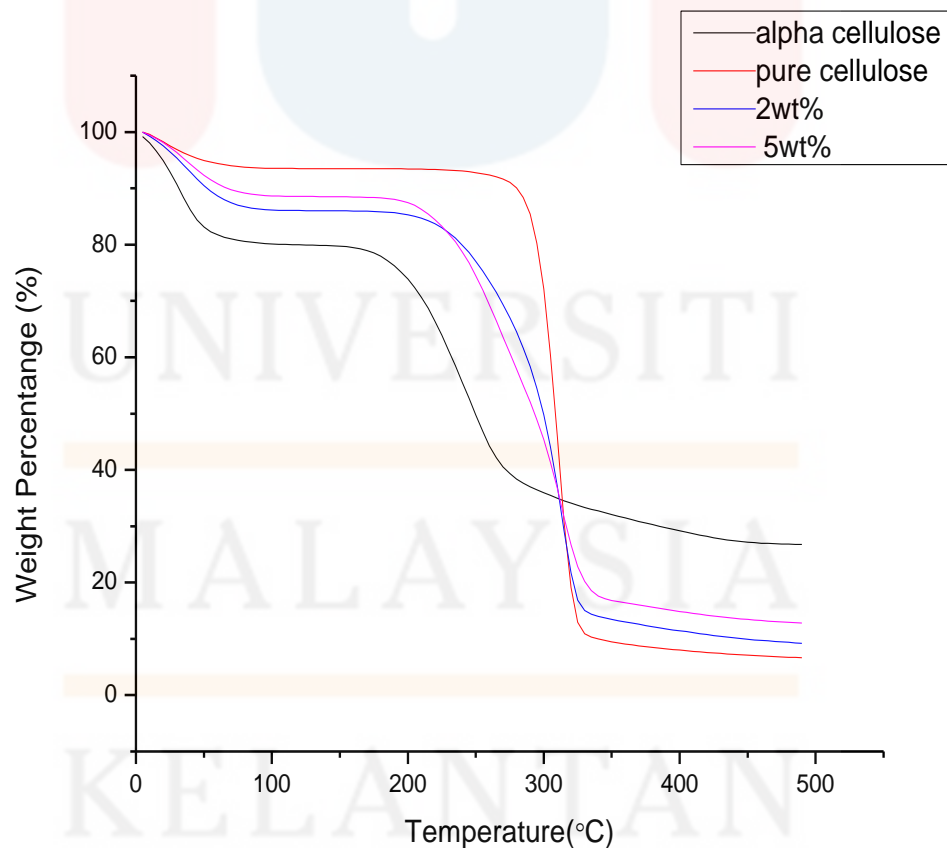


Figure 4.3: TGA analysis for oil palm wood

After ionic liquid treatment with BMIMCl, the thermal stability of the lignocellulosic materials increased due to the removal of hemicellulose and lignin during the treatment process. The chemical treatment attacks the amorphous region in the cellulose and the increase the degree of crystallinity. Higher crystallinity values resulted in greater resistance towards heat and increase in the maximum temperature for thermal degradation. At temperature levels ranging from 180°C to 350°C, the degradation of the pure celluloses took place.

Decomposition of pure cellulose took place within a relatively narrow range of temperatures (280°C – 320°C) and showed only one-step pyrolysis process revealed by DTG curve. The thermal decomposition temperature of pure cellulose was because of the compact and highly dense structure of cellulose. The decomposition of BMIMCl ionic liquid (2 wt % and 5 wt %) took place within a wider range of temperatures with two pyrolysis process separated in close proximity as revealed by the DTG curve. The first pyrolysis occurred from 200°C – 250°C with the maximum temperature peak at 240°C, while the second process had greater curve over the first pyrolysis, ranging from 280°C – 320°C with the maximum temperature peak at 310°C.

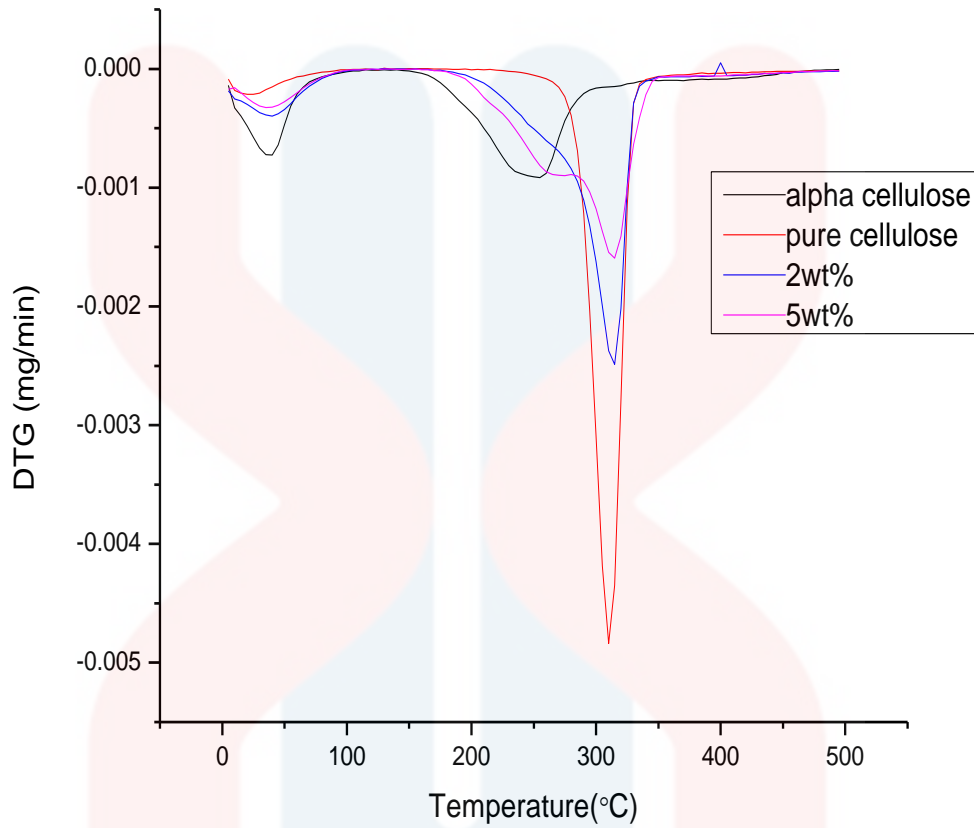


Figure 4.4: DTG analysis for oil palm wood

Hence, it can be deduced that one step pyrolysis process occurred for the pure cellulose, while the two step pyrolysis process occurred for the BMIMCl treated cellulose. The different in the thermal decomposition was assumed due to the different decomposition gasification processes or degree of crystallinity. It was suggested that the decomposition temperature of BMIMCl treated cellulose was found to be lower than the pure cellulose because of the reduction in molecular weight of treated cellulose. The first pyrolysis happened at lower temperature (240°C) showed the decomposition of highly accessible and amorphous regions

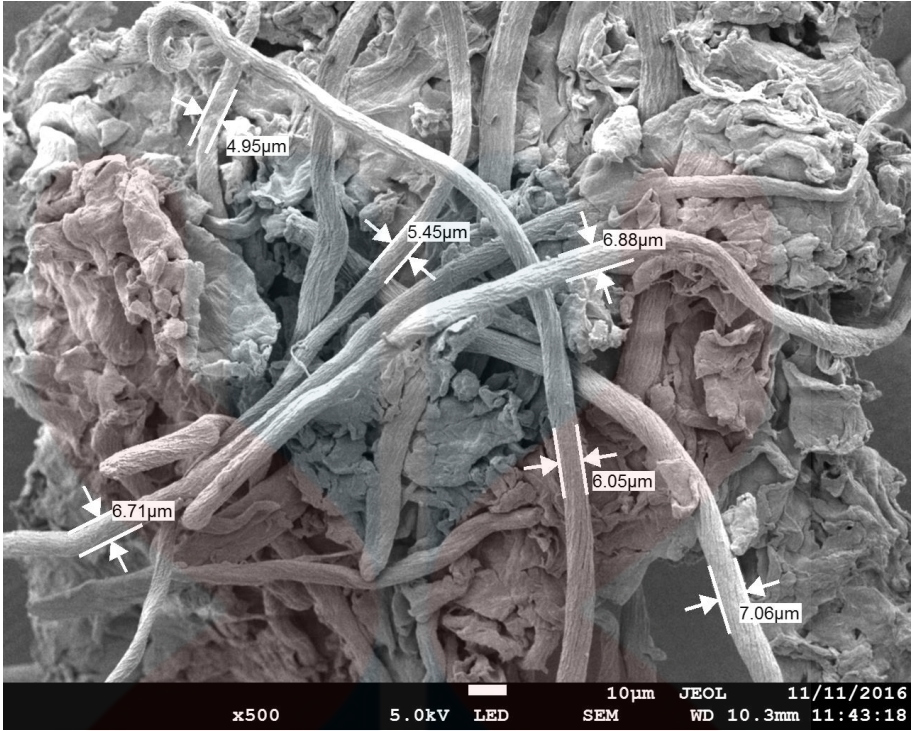
while the second pyrolysis took place at higher temperature (324°C) was attributed to the breakdown of the crystal part.

A decreasing trend of decomposition temperature suggests that the thermal stability of BMIMCl regenerated cellulose is much better than alpha cellulose. Reduction in thermal stability of treated cellulose causes by several conditions. This demonstrated that after cellulose regeneration by BMIMCl, its thermal stability decreased, this was consistent with the decreased in molecular weight of cellulose dissolved in BMIMCl. These phenomena can be explained by nothing that cellulose is attracted by Cl⁻ during the dissolving process in BMIMCl, and most of the hydrogen bonds and part of the molecular cellulose chain are degraded, resulting in decreasing in molecular weight and thermal stability. Although the regenerated cellulose has a lower temperature for decomposition, the regenerated cellulose thermal decomposition residues are more than that original cellulose which are alpha cellulose and pure cellulose according to the char yield (CY) at 550°C. Based on the result, the char residue of regenerated cellulose (14-18 %) was greater than untreated cellulose (12-13 %).

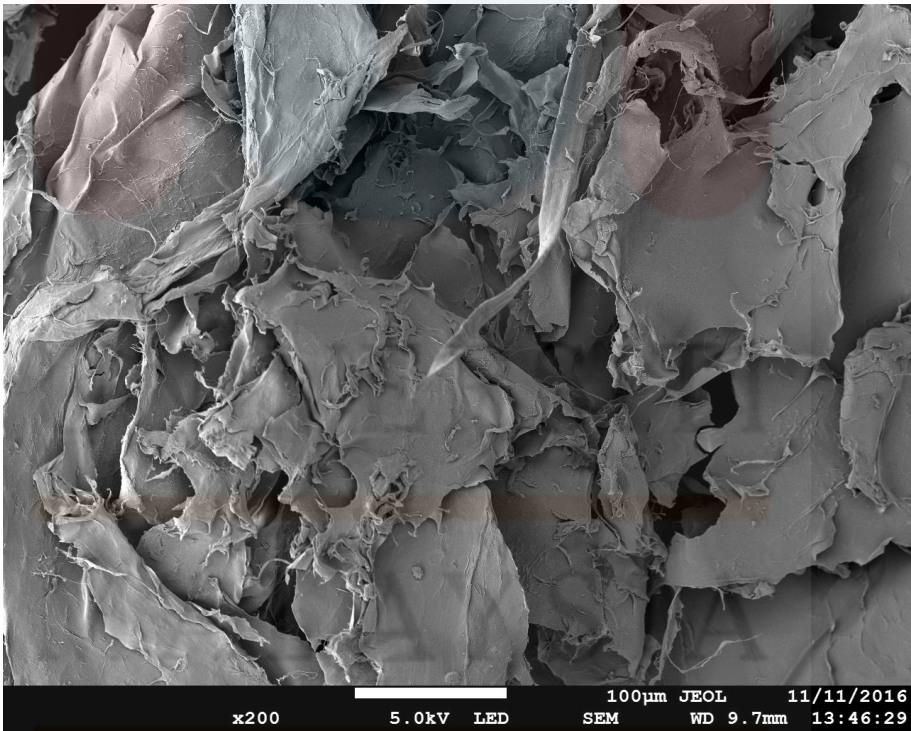
As shown in Figure 4.3 the thermal stability of pure cellulose were stable than ionic liquid regenerated cellulose. The partial destruction of crystalline part and hydrolysis were probably the reason for this decrement. In addition, the residual of regenerated cellulose was higher than untreated cellulose. Pyrolysis residues of cellulose were the primarily indecomposable inorganic salts. It demonstrated that more inorganic salt were involved into cellulose in the dissolution and regeneration process. So, thermal stability of untreated cellulose was better than treated cellulose.

4.4 FESEM

Figure 4.5 shows the Field Emission Scanning Electron Microscope (FESEM) micrograph for alpha cellulose and BMIMCl treated cellulose at 2 wt%. The chemical treatment seems to affect the morphology of the fibres in terms of size and surface smoothness of the fibres. The alpha cellulose fibre is fibrils while the 2 wt % is in agglomerate shape. The BMIMCl treated cellulose is more not smooth and irregularities because removal of lignin and hemicellulose. Therefore when putting this sample in the grinder, the alpha cellulose is easily break compared to the spherical shape. Cellulose 2 wt % shows a higher average length and diameter, 8.93 nm.



a



b

Figure 4.5: FESEM analysis of oil palm wood (a) alpha cellulose (b) 2 wt%.

Chapter 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

Based on the findings in this work, cellulose was successfully extracted from oil palm wood and underwent treatment with ionic liquid BMIMCl. BMIMCl is environmentally friendly and green solvent. In this study, the properties of BMIMCl treated cellulose (2 wt % and 5 wt %) and untreated cellulose (alpha cellulose and pure cellulose) were compared. The structural morphology changes were characterized using X-ray diffraction (XRD), Fourier transform infrared spectroscopy, Thermogravimetric analysis (TGA) and Field emission scanning electron microscopy (FESEM). XRD analysis showed that crystallinity index and crystallite size decreased after pre-treatment. BMIMCl treated cellulose becomes more amorphous after BMIMCl pre-treatment. This was further confirmed by the FTIR result that show higher and intense peak at 2 wt% and 5 wt% compared to untreated cellulose.

Besides that, TGA analysis displayed that have a good thermal stability for untreated cellulose than treatment cellulose. Since cellulose exhibits some characteristics such crystallinity index, crystallite size, functional groups, thermal stability, shape and size depending on their source. The FESEM image of alpha cellulose fibre is fibrils shape while the 2 wt % is in agglomerate shape. Alpha cellulose is more smooth and regularities than BMIMCl treated cellulose because of the ionic liquid removing the cellulose. In this study, the cellulose treated with BMIMCl is find out more amorphous properties compared to untreated cellulose instead of nanocellulose. This proves that BMIMCl ionic liquid is effectively in

convert the cellulose into amorphous cellulose. However, the BMIMCl is less efficient in conversion from cellulose to nanocellulose. The detail research will be helpful in application of cellulose. The produced cellulose has multifunctional purpose. Application of regenerative cellulose was as clothing textile, this class of materials is also used in production of disposable medical devices as well as fabrication of artificial membrane.

5.2 Future Recommendation

Nanocellulose can be obtained by extraction process which is TAPPI Test. However, this research only manages to get amorphous cellulose instead of nanocellulose. The production of nancellulose can be improved by combining physical treatment (Homogenization) and chemical treatment which physical treatment will help to convert the cellulose into nano size. Several time of extraction process should be carried out to make sure the lignin and the extractive is completely removed. The characterization of morphology must be also supported by Transmission Electron Microscopy (TEM) and other characterization instrument.

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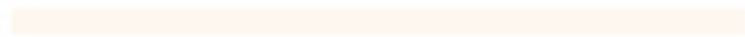
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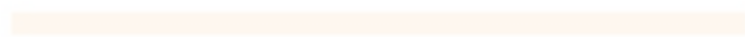
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