UNIVERSITI TEKNIKAL MALAYSIA MELAKA

OPTIMIZATION STUDY ON PROCESS CONDITION OF DURIAN SHELL PULPING TOWARDS HYDROPHOBICITY

This report submitted in accordance with requirement of the Universiti Teknikal Malaysia Melaka (UTeM) for the Bachelor Degree of Manufacturing Engineering (Engineering Material)(Hons)

by

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This report is submitted to the Faculty of Manufacturing Engineering of UTeM as a partial fulfillment of the requirements for the degree of Bachelor of Manufacturing Engineering (Engineering Materials) (Hons.). The members of the supervisory are as follow:

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ABSTRAK

Hidrofobik lignin serat dinding sel disimpan di dalam sel-sel khusus untuk membuatkan ia tidak mudah terpengaruh kepada air dan mengelakkan daripada keruntuhan sel. Perubahan yang berlaku kepada komposisi lignin di dalam spesis serta tisu-tisu yang berbeza menunjukkan bahawa komposisi lignin berbebeza-beza ini bergantung kepada fungsi yang tepat. Lignin dan sifat plastik ini berkait rapat kerana lignin mampu bertindak dan mempunyai sifat pemplastikan di dalam sistem. Oleh yang demikian, penyelesaian alternatif dibuat dengan menggunakan sisa biomas daripada sumber semula jadi seperti kulit durian. Kulit durian adalah salah satu bahan yang berpotensi untuk menghasilkan pelbagai jenis kepingan komposit. Serat semula jadi adalah bahan yang mudah terurai dan mampu menyumbang kepada pembangunan yang mampan tetapi ia mudah menyerap kelembapan dan air. Jadi, dalam kajian ini, kepingan komposit direka dengan mengekalkan kuantiti lignin ditahap yang terbaik bagi mencapai ciri-ciri keplastikan. Dalam usaha untuk mencapai ciri-ciri keplastikan kepingan komposit, beberapa parameter perlu di optimumkan. Peratus alkali digunakan, masa memasak dan suhu memasak adalah parameter yang akan dioptimumkan dengan menggunakan dua tahap reka bentuk faktorial. Kandungan lignin yang optimum iaitu 57.67 % telah didapati daripada parameter jangka nombor 4. Daripada peratusan lignin tertinggi tersebut juga terhasilnya sudut sentuhan air yang paling tinggi iaitu 70.33°. Dengan terhasilnya kepingan komposit yang mempunyai nilai kandungan lignin optimum yang menyumbang kepada nilai sudut sentuhan air yang tinggi, kajian ini akan menyumbang kepada aplikasi kepingan komposit yang mempunyai kebolehan kelebihan penolakan air sepertimana ciri-ciri yang terdapat dalam lignin itu sendiri. Oleh itu, kepingan komposit tersebut boleh digunakan dalam pelbagai jenis persekitaran termasuk persekitaran lembap dan sekaligus menjadi produk mesra alam yang mengurangkan kesan persekitaran berbahaya kepada generasi akan datang.

ABSTRACT

The hydrophobic cell wall fiber lignin is deposited in specialized cells to make them impermeable to water and prevent cell collapse. The variation in lignin composition that exists among different species, and among different tissues within the same species suggests that lignin composition varies depending on its precise function. Lignin and the plasticity is related because lignin can acts as the plasticizer in the system. An alternative solution is made by using the biomass waste from natural resources like durian shell. Durian shell is one of the potential material to produce variety type of composite sheet. Natural fiber is a biodegradable material that can easily decompose and contribute to the sustainable development but it easier to absorb the moisture and water. So, in this study, a composite sheet is fabricated by sustaining the maximum amount of lignin from the durian shell fiber to achieve plasticity property. In order to obtain the plasticity property of the composite sheet, few parameters had been optimized. The percent of alkali used, time of cooking and cooking temperature are the parameters that had been optimized using two-level factorial design. Optimum amount of lignin which is 57.67 % has been found from the run number.4. The highest percentage of lignin is also the higher degree of water contact angle which is 70.33 °. Be produced from a composite sheet which has the optimum lignin content which contributes to the high water contact angle. This study will contribute to the composite sheet that has the ability excess water rejection characteristics as contained in lignin itself. Thus, composite sheet can be used in a variety of environments including humid environment and at the same time being eco-friendly products that reduce harmful environmental effects on future generations.

DEDICATION

To my beloved parents and family, I have devoted all my effort in order to accomplish Projek Sarjana Muda (PSM) report. The reason why I devote all my effort in this report is because I want my family to know especially my beloved mother and father that I have done my best in order for me to fully fill the bachelor degree program. On top of that, I dedicate this report to my supervisor and co-supervisor which has helped me throughout this whole semester to complete my full thesis.

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LIST OF ABBREVIATIONS, SYMBOLS, AND NOMENCLATURES

AFM	-	Atomic Force Microscope
AQ	-	Anthraquinone
BET	-	Brunauer–Emmett–Teller
CMC	-	Carboxymethylcellulose
DOE	-	Design of Experiment
DSAC	-	Durian Shell Activated Carbon
EFB	-	Empty Fruit Brunch
IPA	-	Isopropyl Alcohol
KOH	-	Potassium Hydroxide
NaOH	-	Sodium Hydroxide
SEM	-	Scanning Electron Microscopy
SMCA	-	Sodium Monochloroacetate
TAPPI	-	Technical Association of the Pulp and Paper Industry
WCA	-	water contact angle

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Composite sheet is a flat piece of two or a lot of part material that can be obtained from two sources which are artificial and natural fiber (Jochen, 2002). Composite sheet is built by blending the heterogeneous pulp and slag-wool fiber, and also can be contributed with or without composite additive. Some of the benefits in using composite sheet are low density, nonporosity and affordability. Some of the composite sheet application are paper.

Recently, research most are working in producing composite sheet are from natural fiber. For example, composite sheet from kenaf, bamboo and paddy straw. Durian is also one of the potential material for producing it. Durian (*Durio zibethinus* Murray) is that the hottest seasonal fruit in South East Asia countries. Durian is a tropical fruit of trees species of Malvales order in Bombaceae family and genus of *Durio* (Nanthachai, 1994). It also widely known to the locals as the "King of Fruits". Durian flesh will be exported outside the country as commercial food product. Example of commercial products are the flesh itself, biscuit, ice cream and cake. According to Department of Agriculture Malaysia, (2009) reported that, in Malaysia, around 376,273 metric tonne of durian are raised in year 2008. Malaysian Agriculture Research Development Institute (MARDI) (1997) reported that, there are several state in Malaysia that have durian season which are Pahang, Kelantan and Perak. According to MARDI (1997) the durian season happen between June until August. During that

season, the dumping of durian shell will happened. Therefore, it is a good advantage to convert the durian shell waste to be another product that can give profit in return such as producing composite sheet. Besides, the cellulose from the durian shell can be exchanged to carboxymethylcellulose (CMC) by carboxymethylation using sodium hydroxide (NaOH) and sodium monochloroacetate (SMCA) in isopropyl alcohol (IPA) (Rachtanapun, 2008). Durian shell has its own special properties that suite it to be employed in composite sheet. According to Am *et al.* (2011) contains of 9.24 % of moisture content, 4.34 % of ash content, and 6.43 % of fixed carbon. By using the durian shell in creating composite sheet, the waste can be reduced and the price are saved (Wilaipon, 2011). Thus, it can decrease the landfill area for disposal which may maintained the health of environment.

Natural fiber consists of lignin. Lignin is an amorphous polymer but unlike hemicellulose, lignin is comprised mainly of aromatics and has little effect on water absorption (Holbery, 2006). Lignin is also the most abundant large molecule polymer in the cell wall. From a chemistry side, phenylpropanoid derivatives are the basic units of the lignin, they are mix into high molecular substance carbon-carbon bond or either bonds (Chen, 2014). Usually, the cell wall of plants with a mechanical action and enduring function always contain a largest lignin contents. The lignin contents is about 14-25 % in herbaceous plant and 27-32 % in woody (Chen, 2014).

Hydrophobic composite sheet may be produced by maintaining the lignin inside the structure. The hydrophobic composite sheet is used for resisting the water absorption. A surface is hydrophobic in the event that it tends not to assimilate water or be wetted by water. A surface is hydrophilic in the event that it has a tendency to adsorb water or be wetted by water. All the more especially, the terms depict the communication of the limit layer of a strong stage with fluid or vapor water. Hydrophobic is actually related with the contact angle. If the contact angle of water is less than 30°, the surface is selected hydrophilic since the forces of fundamental interaction between water and the also surface nearly equal the cohesive forces of bulk water, and water does not cleanly drain from the surface (Butt, 2003). Sanchin *et al.* (2010) claimed that, on a hydrophobic surface water forms distinct droplets because the plasticity property will increase.

In this study, composite sheet will be produced by using soda pulping as the "pulping" method. Soda pulping is actually known as soda anthraquinone (AQ) which is a modified chemical process for wood pulp production with sodium hydroxide as the cooking chemical as a pulping additive to decrease the carbohydrate degradation (Ramadan *et al.*, 2014). The advantages of AQ are having a high pulp yield and conjointly leading to higher mechanical strength properties of the yield comparable at the side of its environmental friendly approach likewise as its reduced active alkali consumption (Ramadan *et al*. 2014).

This project investigates the optimum process condition of parameters in durian shell pulping towards the hydrophobicity composite sheet. The composite sheet may be achieved through the optimum amount of lignin content inside the sheet by optimizing the optimum value of pulping parameters. The parameters involved percent of alkali used, cooking period and cooking temperature. Next, it will characterized using Scanning Electron Microscopy (SEM) to study the surface morphology and Technical Association of the Pulp and Paper Industry (TAPPI). TAPPI standard method T 222 os-74 to study the lignin content. Other than that, the analyses is water contact angle and the composition of element of lignin.

1.2 Problem Statement

Mostly the composite sheet nowadays are made from polymer based materials. The disadvantages of polymer based materials are non-biodegradable, expensive, can occur pollution and also high cost. Therefore, to overcome that advantages from polymer based materials, the natural fiber which is durian shell is use in this research. Sheet will expand and contract more in the cross-grain direction when exposed to moisture changes. Nowadays, hydrophobic composite sheet application is all about tends not to adsorb water or be wetted by water. A surface is hydrophilic if it tends to adsorb water or be wetted by water. According to Roy (2001), moisture absorption depend upon void content in the composite and any increase in the water absorption. When have a lot of moisture or water was absorbed, the mechanical properties of hydrophobic composite sheet is decrease. That is due to the high hygroscopicity in

natural fiber itself. Like a sponge, it absorbs or loses moisture relative to the extremes of exposure and the surrounding atmosphere. Lignin is the one part in durian shell that have thermal properties characteristic. The softening temperature of absolutely dried lignin ranges from 127 °C to 129 °C, which remarkably decreased with increased water content, indicating that water acts as a plasticizer in lignin (Song, 2008). Therefore, finding suitable amount of lignin content is very important in this research to support the process condition of durian shell pulping toward hydrophobicity since lignin act as natural plasticizer.

1.3 Objective

There are a few aim of this study:

a) To investigate the relationship between percentage of NaOH used, cooking period and cooking temperature of lignin content towards hydrophobicity property using two-level factorial design method.
b) To verify the effect of lignin as natural plasticizer towards hydrophobicity.
c) To characterize the durian shell composite sheet in terms of lignin content, surface morphology, water contact angle and composition element of lignin.

1.4 Scope

The study will focus on process condition of durian shell pulping towards hydrophobicity. The plasticity property is obtained from the optimum amount of lignin inside the sheet since the lignin is a natural plasticizer. The pulp from durian shell is used as the main raw material. In order to find the best lignin content for producing the most successful hydrophobic composite sheet, response surface methodology by two-level factorial design method is used as the optimization tool. The parameter that will be optimized are percentage of alkali used, cooking period and cooking temperature. Soda pulping is used as the method of pulping. Finally, the hydrophobic composite sheet is characterized in terms of lignin content, surface morphology, water contact angle and composition element of lignin.

CHAPTER 2

LITERATURE REVIEW

2.1 Durian

The Durian is native to Brunei, Indonesia and Malaysia. Some of aficionados called durian as “King of Fruits” (National Library Board, 2005). The root comes from the word "Duri" which means spine. Durian or the scientific name Durio zibethinus Murray is that the most well-liked seasonal fruit in South East Asia countries. The word durian is actually refer to the fruit but it also mean the flesh that can be eat or the durian pulp and also to the tree of durian itself. The appearance of durian is has covered by sharp of spines or thorns, has a thick fibrous husk and for the flesh is white, creamy or golden yellow which is sharply aromatic and strongly tasted. According to Morton, (1987) there are more than 300 named assortments of durian in Thailand. Just a couple of these are in business development. In Malaysia, 100 sorts are evaluated for size and quality. In peninsular Malaya, there are 44 clones with little contrasts in time and degree of blossoming, botanical and natural product morphology, efficiency and palatable quality. In Malaysia has one fruiting seasons for durian which are in early June until August. During the seasons the fruits are exists in very larger numbers in local markets.

Durian tree started from seed and can be reached up to 25- 50 m in height and 120 cm with a trunk in diameter. The durian’s leaves are about 8-20 cm long and also

2.5-7.5 cm wide and has shape in elliptic to oblong. Upper surface of leaves is smooth, shiny, or dark green. The bottom surface is scaly but sometimes is brown and sometimes with a golden shine.

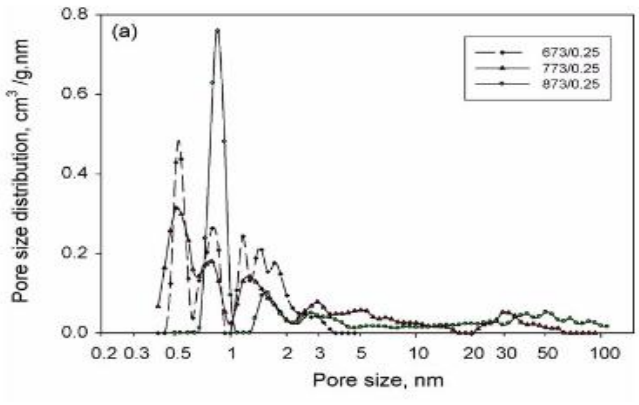
2.1.1 Durian shell

According to Jongjit *et al.* (2011) it can be seen that durian shell contains lignin and hemicellulose which are the primary cause of the change in the characteristics of the natural fibers in the composite. The alkaline pore water in the composite dissolves the lignin and hemicellulose and thus breaks the link between the individual fiber cells. Development of composite materials for buildings using durian shell with low thermal conductivity can be an alternative way to solve simultaneously energy and environmental concerns (Khedari *et al.* 2003). As mentioned, durian shell is comprise of cellulose, hemicellulose and lignin. It can be done by converting durian biomass to sugar for potential production of ethanol and also for reducing environmental problem and pollution. In addition, durian shell also can be enhanced to soil quality. This can be defined by experiment when durian shell were applied in the soil at different percentages can increase the soil quality parameters. That means the durian shell was efficient in improving the soil quality.

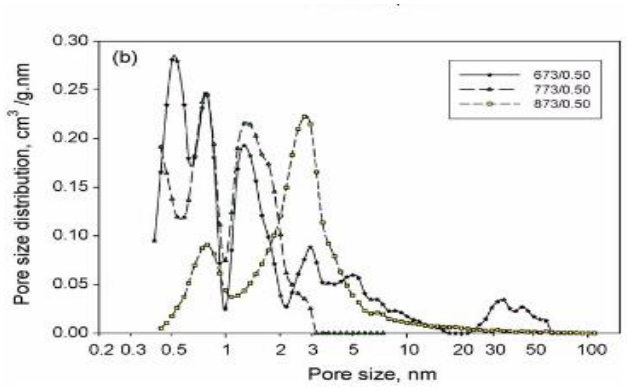
2.1.1.1 Pore distribution

Chandra *et al.* (2007) claimed that the pore structure and pore size distribution of activated carbon commonly varies based on the nature of raw materials and activation method. In the same research, the pore size distribution was largely dependent on the Potassium Hydroxide (KOH) to durian shell ratio. The pore size distribution delineates a model of solid internal structure that assumes that a similar set of non-interacting and frequently formed model pores will represent the complicated void areas inside the important solid. The pore size distribution is kindly associated with each kinetic and equilibrium properties of porous material and maybe is that the most significant facet for characterizing the structural nonuniformity of porous materials applied in industrial application. Figure 2.1 shows the graph of pore size distribution

of durian shell for the activated carbons shows the event of recent micropores and mesopores.



(a)



(b)

Figure 2.1: Pore size distributions of durian shell activated carbons. (a) KOH to durian shell ratio 0.25 and (b) KOH to durian shell ratio 0.50 (Chandra *et al.* 2007)

2.1.1.2 Pore characteristic

The pore properties of the carbons including the Brunauer–Emmett–Teller (BET) surface area, pore volume and pore size distribution were characterized using nitrogen adsorption. The KOH to durian shell ratio of 0.5 and activation temperature of 773 K was found as the optimum conditions to acquire high surface area activated carbon from durian shell (Jaka *et al.* 2009).