OPTIMIZATION OF PROCESS CONDITION ON LOADING DEGREE OF MAGNETIC COMPOSITE SHEET BY LUMEN LOADING TECHNIQUE

AFIQ SYAZWI BIN AHMAD NIZAM
B051210045

UNIVERSITI TEKNIKAL MALAYSIA MELAKA
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This report submitted in accordance with requirement of the Universiti Teknikal Malaysia Melaka (UTeM) for the Bachelor Degree of Manufacturing Engineering (Engineering Materials) (Hons.)

By

AFIQ SYAZWI BIN AHMAD NIZAM

B051210045

930525-07-5381

FACULTY OF MANUFACTURING ENGINEERING

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APPROVAL

This report is submitted to the Faculty of Manufacturing Engineering of UTeM as a partial fulfillment of the requirements for 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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TAJUK: **Optimization of Process Condition on Loading Degree of Magnetic Composite Sheet by Lumen Loading Technique**

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ABSTRAK

Sejak generasi lalu, penggunaan kepingan komposit semakin meningkat kerana aplikasi kepingan tersebut semakin dipelbagaikan. Pada masa kini, kebanyakan kepingan komposit magnetik diperbuat daripada bahan berasaskan polimer. Bahan polimer mengambil masa yang lebih lama untuk reput, lebih mahal dan tidak mesra alam. Dengan teknologi yang lebih canggih sekarang, kepingan komposit magnetik boleh diperbuat daripada bahan buangan biomas daripada sumber semula jadi seperti kulit durian. Berdasarkan statistik yang dibuat oleh Jabatan Pertanian Malaysia 2012, ladang durian adalah yang terbesar di Malaysia pada keluasan kira-kira 100,614 hektar. Untuk mengelakkan kulit durian daripada menjadi bahan buangan, ia boleh digunakan untuk menghasilkan kepingan komposit magnetik. Dalam kajian ini, kulit durian akan dicampurkan dengan zarah magnet untuk menghasilkan kepingan komposit magnetik. Kepingan komposit magnetik ini lebih mudah untuk dilupuskan dan lebih mesra alam sekitar berbanding kertas konvensional. Parameter yang perlu diambil kira untuk mencapai sifat-sifat magnetik yang maksimum adalah jumlah magnetit (Fe_3O_4), kelajuan pergolakan dan masa pergolakan. Pengoptimuman ini akan dijalankan dengan menggunakan dua tahap reka bentuk faktorial. Maklum balas daripada reka bentuk ini eksperimen adalah tahap pemuatan. Kemudian, lembaran magnet akan menjalani analisis beberapa seperti analisis morfologi dan X-ray pengukuran pembelauan untuk mengesahkan dan menyokong maklumat. Dari eksperimen yang dijalankan, didapati bahawa sampel 3 dengan jumlah magnetit (Fe_3O_4) sebanyak 3g, kelajuan pergolakan 1300 rpm dan masa pergolakan 30 min mempunyai tahap pemuatan yang paling tinggi sebanyak 92%. Sampel 3 kepingan komposit menjalani beberapa analisis untuk mengesahkan kehadiran zarah magnet dalam kepingan tersebut. Kepingan komposit yang dihasilkan boleh digunakan untuk mengelakkan elektromagnet dari gangguan alat elektronik sensitif.

ABSTRACT

From the past generations until now, the usage of composite sheet keep increasing as the applications of the sheet are increasingly diversified. Nowadays, most of the magnetic composite sheets are made from polymer based material. Polymer material takes longer time to decompose, more expensive and not environmental friendly. As the technology evolved, magnetic composite sheet can be produced by using biomass waste from natural sources such as durian shell. According to the statistics done by The Department of Agricultural of Malaysia in 2012, durian plantation is the largest in Malaysia of about 100,614 hectars. To prevent the durian shell from waste, it can be used to produce magnetic composite sheet. In this research, durian shell is combined with magnetic particle to produce magnetic composite sheet. This magnetic sheet is more easily degraded and more environmental friendly compare to conventional paper. The magnetic sheet is prepared by using lumen loading method. The parameters that are important in order to achieve maximum magnetic properties are the amount of magnetite (Fe_3O_4), agitation speed and time of agitation. The optimization was carried out using 2 level factorial design method. The response of the design is degree of loading. Then, the magnetic sheets undergo a few analyses such as magnetic measurement, morphology analysis and X-ray diffraction measurement to confirm and support the result. From the experiment, it was found that the sample 3 with the amount of Fe_3O_4 of 3g, the agitation speed of 1300 rpm and the agitation time of 30 min has the highest loading degree of 92%. The sample 3 magnetic sheets undergo a few analyses to confirm the presence of the magnetic particles in the sheet. The magnetic sheet produced can be used for electromagnetic shielding that can prevent electromagnetic interference from sensitive electronics.

DEDICATION

This report is dedicated to my beloved parents, my supervisor, my family members and also to all my friends who have been supported and inspired me to complete this project.

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In The Name Of Allah (S.W.T), The Most Gracious and The Most Merciful. First and foremost, praise to Allah for His Wisdom and blessing in giving me the strength, patience and motivation to complete my PSM project successfully.

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Fe_3O_4	-	Magnetite
KOH	-	Potassium Hydroxide\
NaOH	-	Sodium Hydroxide
PCC	-	Precipitated Calcium Carbonate
SEM	-	Scanning Electron Microscopy
XRD	-	X-ray Diffraction
VSM	-	Vibrating Sample Magnetometer
DC	-	Direct current
Ms	-	Magnetization value
Hc	-	Coercivity
%	-	Percent
PEI	-	Polyethylenimine
emu	-	Electromagnetic Unit
g	-	Gram
min	-	Minute
rpm	-	Revolutions per Minute

CHAPTER 1

INTRODUCTION

This chapter describe the background, problem statement, objectives and scope of the study.

1.1 Background of study

In the last several years, composite sheet have gained growing attention due to the wide application range and low cost of production. Composite sheet is a flat piece of combination of two sources which are artificial and natural fiber. Magnetic composite sheet is probably the most common application from natural fiber composite sheet (Madsen and Gamstedt, 2013). Magnetic sheet is generally made of cellulose pulp and magnetic particles, derived mainly from wood, rags, biomass waste and certain grasses, processed into flexible sheets or rolls by depositing them from an aqueous suspension. Magnetic sheet exhibits some superiorities in properties, such as softness, renewable use, and folding resistance. Besides that, magnetic sheet can be used for storage information, security paper purposes, paper handling, reprographic functions such as magneto graphic printing substrate, electromagnetic shielding and magnetic separation of antibodies based on selective adsorption (Rioux et al. 1992).

The most common methods used for preparing magnetic composite sheets are the in situ synthesis and the lumen-loading approach (Chia et al. 2006). Lumen loading is a technique whereby the fillers are introduced into the fiber lumen while leaving the fiber surfaces free of filler. Lumen loading techniques have been used to

making $CaCO_3$ lumen loaded paper (Middleton et al. 1989; Middleton & Seallan 2003) and magnetic paper (Zakaria et al. 2004,2005). Lumen loaded pulp has two advantages over the conventional method, which the fillers are protected by the cell wall in the fiber lumen from dislodgement during papermaking process and the other advantage is that the outer surfaces of fiber free of filler which form a better inter-fiber bonding and has higher strength than conventional-loaded paper (Middleton & Seallan 1985). Furthermore, magnetic sheet produced by lumen loading technique has better magnetic properties compared to sheet produced by in situ method (Zakaria et al., 2004), although in situ method have better control on the magnetic particles size, greater uniformity and better dispersion of magnetic particles that producing good quality of magnetic sheet (Garcia et al., 1995 &1997).

Recently, lot of studies have been done about producing magnetic composite sheet from natural fiber such as kenaf (Zakaria et al., 2006) and rice straw (Kuo and Shen.,1992). This is because natural fiber is environmental friendly and very low cost. One of the potential natural fiber to make magnetic sheet is durian shell. Durian is widely recognized and revered in Southeast for its unique aroma and shape with great taste. The aril colour of durian is variety usually yellow or red, depending on the species. Apparently, durian shells have gone to waste after the flesh is consumed. According to a statistic accomplished by The Department of Agricultural of Malaysia in 2012, durian plantation is one of the largest plantation in Malaysia of about 100,615 hectares. There is a huge waste of durian shells as a result of large durian plantation area. The waste of durian shell can be reduced because it can be used as a raw material to produce magnetic composite sheet by adding magnetic substances. Chandra et al. (2009) said, the durian shells have high water content (40%), hard shell texture and also strong durian flavor. Durian shell is designed to be breathable and moisture controlled, thus the cell microstructure of durian shell contains many interconnecting pores to allow gasses to move through even as keeping the proper quantity of water molecules inside. The high porous surface structure of durian shell ensures a large quantity of magnetic particles to enter the lumen of fibers. Durian shell has the very best absorption ability amongst all, signifies that it has the absolute best filling of pores in a magnetic composite sheet.

Magnetic composite sheet have been successfully produced from unbleached kenaf (*hibiscus cannabinus)* kraft pulps through the lumen loading technique and in situ method (Zakaria et al., 2006). For the lumen loading technique, commercial magnetite (Fe_3O_4) powders have been presented into the lumen by mixing the pulp and magnetic powder. For the in situ method, nanosized magnetite particles have been deposited in the presence of pulp fibres and placed inside the lumen of fibres by chemical coprecipitation process. Magnetic composite sheet obtained via lumen loading technique have better magnetic properties compare to in situ method (Zakaria et al., 2004). This is because small (nm) size of particles synthesized by using chemical coprecipitation method, shows super paramagnetic manner. The loading degree of lumen loading sample is higher compare to in situ sample due to aggregation process that caused particles in lumen loading mound together (Shahril,2015).

Magnetic properties of magnetic composite sheet are affected by the magnetite particles used. Rioux et al., (1992) suggested magnetite as magnetic pigments in producing the magnetic sheet. The magnetic properties of the magnetic composite sheet increased when the magnetic particles increase (Gao *et al.*, 2014). Therefore, the magnetic particles will easily fill into the lumen of wood fiber and between the fiber itself when magnetic particles are increased. Polyethylenemine (PEI) is reported to be an excellent retention aid to improve filler retention and has been used as wet end additive to improve drainage, and wet strength of composite sheet (Alince, 1987). PEI acts as flocculent for magnetite particles and fibers which will enhance the retention of filler in lumen fiber. The loading degree of magnetic sheet increase with the addition of PEI. Therefore by increasing the percentage of PEI, the loading degree of sheet will improved.

Therefore, this study will focused on optimizing the process condition in lumen loading parameters in achieving the maximum magnetic properties of durian shell magnetic composite sheet. The optimization process will be carried out using 2 level factorial design method by Design Expert software. The responses of this design of experiment are degree of loading. Lastly, the magnetic sheet will undergo a

few analysis such as morphology analysis and X-ray diffraction measurement to confirm and support the result.

1.2 Problem statement

Previously, magnetic sheet was produced using pure magnetic particles such as magnetite and maghemite combined with polymer. However, there are many disadvantages of using polymer based material. These material needs to undergo complicated process to make the magnetic sheet and the polymer material takes decade to degrade and this will cause pollution. Furthermore, the polymer materials are expensive and this will lead to higher cost production of the magnetic composite sheet. Nowadays, a magnetic composite sheet can be produced by the combination of raw material such as durian shell and magnetic particles that are low in cost and more environmental friendly. This magnetic sheet is more easily degrade and have better strength compared to polymer sheet. The magnetic composite sheet will be prepared by lumen loading method. This is because, magnetic composite sheet obtained from lumen loading technique have higher magnetic properties compare to in situ method (Zakaria et al., 2004). However, lumen loading method has poor magnetic retention inside the fiber. Commercial magnetic particle in micron size can only move through lumen of fibers if the diameter is smaller than the pits diameter (Zakaria et al., 2005). The diffusions of magnetic particles during lumen loading process are influenced by various factors, such as flocculation of magnetic particles into bigger size. This factor will make magnetic particles experienced difficulty to move into the lumen fiber and the particles will less evenly distributed in the lumen fibers (Zakaria et al., 2004). Besides that, large size particles are easier to disengage due to their lower surface energy during washing process. These factors will lead to low magnetic retention inside the fiber lumen. Therefore, the solution to improve the magnetic retention of fiber using lumen loading method is by doing optimization of magnetic properties.

1.3 Objective

The main objectives of this study are listed below :

i. To determine the compatibility of durian shell waste combines with magnetic particle to produce magnetic composite sheet.
ii. To optimize the process condition in lumen loading parameters in achieving maximum magnetic properties of durian shell magnetic sheet.
iii. To analyze the characteristic of the magnetic paper in terms of surface morphological, X-ray diffraction measurement, degree of loading and magnetic measurement.

1.4 Scope

This research focus on the compatibility of durian shell waste combine with magnetic particle to produce magnetic composite sheet. The method used to make the paper pulp is by soda pulping. After pulp has been produced, magnetic particles will be induced using lumen loading technique by various different values of parameters in order to achieve maximum magnetic properties of the composite sheet. The parameters are amount of magnetite (Fe_3O_4), agitation speed and time of agitation. The optimization will be carried out using 2 level factorial design method. Finally, the magnetic sheet will undergo a few analysis conducted such as morphology analysis and X-ray diffraction measurement, while the loading degree and magnetic measurement are the responses for the optimization design.

CHAPTER 2

LITERATURE REVIEW

A review on previous research work in several areas relevant to this research is presented in this chapter.

2.1 Durian

Durian (Durio zibethinus) called the "king of fruits" is a members of the family Bombacaceae and genus Durio (Brown, 1997). It is the most popular seasonal fruit and most highly valued fruits in South East Asian countries. In 2007, Thailand produced 686,478 tonnes of durian consequently volumes of durian shell residue in excess of 48,800 tonne per year are produced (Sangkaeo, 2011). The exotic fruit durian, originating from tropical areas, has unique and great flavour. The fruit is ovoid to nearly circular shape with an natural size weighing 2.0-4.5 kg depending on the varieties (Hokputsaetal, 2004).

2.1 Durian shell

Durian shell is a plant waste of durian fruit consumption. Durian has been widely used in food processing industries (Foo and Hameed, 2011), but only one-third of durian is safe to be eaten, whereas the shell will no longer be useful (Amid and Mirhosseini, 2012). The shell commonly weighs more than half of the total fruit weight is green to yellowish brown, thick and semi-woody with sharply pointed pyramidal thorns. Aril, the edible portion of the fruit only accounts for about 15–

30% of the mass of the fruit. Durian shell has exceptional properties that can be used in making the magnetic composite sheet. The durian shell consists of 9.24 % of moisture content, 4.34 % of ash content, and 6.43 % of fixed carbon (Wilaipon, 2011). Durian shell give a good source of cellulose (Rachtanapun et al., 2012). Chandra et al. (2009) stated that the durian shells have high water content (40%), rigid shell surface and also strong durian flavour. The high porous surface structure of durian shell ensures a large quantity of magnetic particles to enter the lumen of fibers. Durian shell has the very best absorption ability amongst all, signifies that it has the absolute best filling of pores in a magnetic composite sheet.

2.2 Pore distribution

Chandra et al., (2009) discovered the special characteristics of durian shell in term of pore distribution where the durian shell is use as raw material in producing activated carbon. The pore size distribution delineates a model of solid internal structure, which assumes that an identical set of non-interacting and often shaped model pores can represent the complex void area within the real solid. The pore measurement distribution is intently concerning each kinetic and equilibrium properties of porous material and perhaps is the important aspect for characterizing the structural heterogeneity of porous materials employed in industrial applications. Pore widening more commonly begins when a number of opened pore exists in the structure which is the case when the chemical ratio is reasonably excessive (Mohanty et al., 2006). Figure 2.1 show the size of pore distribution of durian shell with potassium hydroxide (KOH).