UNIVERSITI PUTRA MALAYSIA

EFFECTS OF PULPING CONDITIONS AND PROCESSING VARIABLES ON THE PERFORMANCE OF KENAF MEDIUM DENSITY FIBRE BOARD

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By

MAJID DEHGHAN NAYERI

Thesis submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfillment of the Requirements for the Degree of Doctor of Philosophy

July 2014

Dedicated To

This thesis is dedicated to my lovely children Amin, Ali and Mohammad, my dear wife, and my parents that I owe them all of success in my life.

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of the requirement for the degree of Doctor of Philosophy

EFFECTS OF PULPING CONDITIONS AND PROCESSING VARIABLES ON THE PERFORMANCE OF KENAF MEDIUM DENSITY FIBRE BOARD

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July 2014

Chairman: **Professor Paridah Md. Tahir, PhD**

Institute: **Institute of Tropical Forestry and Forest Products**

Kenaf, *Hibiscus cannabinus* L., is a environmentally friendly crop which is recognized as one of the potential lignocellulosic material to replace wood in different kinds of wood based products. Kenaf has excellent properties for pulp and paper, medium density fibre board (MDF), and other composites, as it has a low density, little abrasion during processing, high filling levels, and high specific mechanical properties. The kenaf stem is composed of an outer layer (bast) and a core which either by chemicals and/or by enzymatic retting it is easy to separate. The bast constitutes 25 to 40% of the stem dry weight and shows a dense structure. Alternatively, the core is wood-like and makes up the remaining 60 to 75% of the stem. Both portions are greatly different ties in terms of anatomy, physical and chemical content.

Studies concerning the production of medium density fibre board (MDF) with kenaf as an alternative fibrous material have been carried out as an attempt to provide a sustainable and viable destination for this lignocellulosic material. This work aimed to evaluate the properties of kenaf fibres at different refining conditions and to produce kenaf MDF of acceptable strength. In this dissertation, MDF was prepared from kenaf and rubber wood and their mixture. The parameters of preparation and the resulting intermediate products as well as the final products, MDF, were characterized and compared at each experimental step.

The investigation of fibre dimensions based on pulping of bast and core fibre resulted in different behavior of the two classes of fibre. There are significant variations in all aspects of fibre morphology of bast fibre at different pulping temperatures and pulping time, and a significant interaction was detected between both parameters. The bast fibre produced longer and thinner fibre, compared to the core fibre, thus yielding fibre of higher aspect ratio. The changes in fibre morphology were clear when the pulped temperature increased. The core fibre exhibited significant variations in fibre length, fibre width, and wall thickness in all parameters. The lumen diameter and aspect ratio

fibre width, and wall thickness in all parameters. The lumen diameter and aspect ratio were not significantly affected by differences in pulping temperature, pulping time, and the interaction between both parameters. The fibre width was reduced at an increasing pulping temperature and time, but lumen diameter was not significantly affected. The aspect ratio also decreased with increasing pulping temperature and time. The length of core fibre decreased with increasing pulping temperature and pulping time. The fibre width shows constant reduction as the pulping condition become more severe. Consequently the Runkel ratio was decreased also. The pH value of both fibres was reduced as the temperature increased; core fibre was more acidic (pH= 3.8) compared to bast fibre (pH= 4.5). It is evident from results that bast fibre is more resistant to acid and displayed greater acid buffering capacity compared to core fibre.

The investigation of the effect of refining pressure (6 and 8) and resin content (10, 12, 14 %) on physical (WA and TS), and mechanical (MOE, MOR, IB) properties MDF made from a mixture of biomass kenaf (*Hibiscus cannabinus* L.) stem and rubberwood showed thermo-mechanical refining and resin content were influential in the increment of physical and mechanical properties of the MDF. For 8 bar of refining pressure with 14% resin content, the MDF recorded optimum WA of 83.12%, TS of 20.2%, MOR of 25.3 MPa, MOE of 3450 MPa and IB of 0.51 MPa [density 700 (kg/m^3)].

Response surface methodology was used to establish the optimum process variables (resin content, and pressure) for effect on board properties. By using response surface and contour plots, the optimum set of operating variables can be obtained graphically, in order to achieve the desired board properties. Therefore, it was recommended that the board properties increase when the resin content and pressure increased. It can be inferred that any parameters, individually, had a positive effect on the increase of board properties. The main effects of parameters are in following order: main effect of resin content> pressure.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk Ijazah Master Sains

KESAN PEMBOLEHUBAH PENAPISAN KE ATAS SIFAT GENTIAN KENAF DAN PENGARUH TERHADAP PERSASTI PAPAN GENTIAN BERKETUMPATAN SEDERHANA

Oleh

MAJID DEHGHAN NAYERI

Julai 2014

Pengerusi: **Professor Paridah MD Tahir, PhD**

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Kenaf , Hibiscus cannabinus L. , adalah tanaman mesra alam yang diiktiraf sebagai salah satu bahan lignoselulosik yang berpotensi untuk menggantikan kayu dalam pelbagai jenis produk berasaskan kayu . Kenaf mempunyai ciri-ciri yang sangat baik untuk pulpa dan kertas, papan gentian berkepadatan sederhana (MDF), dan komposit lain kerana ia mempunyai ketumpatan yang rendah, sedikit lelasan semasa pemprosesan , tahap pengisian yang tinggi, dan sifat-sifat mekanikal yang tinggi. Batang kenaf terdiri daripada lapisan luar (bast) dan teras yang mudah dipisahkan sama ada dengan menggunakan bahan kimia atau enzim pengeratan. Bast yang membentuk 25 hingga 40% daripada berat batang kering mempunyai struktur yang padat. Teras kenaf pula mempunyai sifat seperti kayu dan membentuk 60-75 % daripada batang kenaf. Kedua-dua bahagian adalah sangat berbeza dari segi anatomi, fizikal dan kandungan kimia.

Kajian mengenai pengeluaran papan gentian berkepadatan sederhana (MDF) dengan kenaf sebagai bahan berserabut alternatif telah dijalankan sebagai satu usaha untuk menyediakan destinasi mampan dan berdaya maju untuk bahan lignoselulosik ini. Kajian ini bertujuan untuk menilai sifat-sifat gentian kenaf pada keadaan penapisan yang berbeza dan untuk menghasilkan MDF kenaf dengan kekuatan yang boleh diterima. Di dalam disertasi ini, MDF telah disediakan daripada kenaf dan kayu getah dan juga campuran kedua-dua bahan. Parameter penyediaan dan produk perantaraan serta produk akhir, MDF, telah disifatkan dan dibandingkan pada setiap langkah eksperimen.

Siasatan dimensi ke atas serat berasaskan pulpa daripada bast dan teras menunjukkan tingkah laku yang berbeza antara kedua-dua serat tersebut. Terdapat variasi yang ketara dalam semua aspek morfologi gentian bast pada suhu dan masa pempulpaan yang berbeza, dan interaksi yang nyata telah dikesan di antara kedua-dua parameter tersebut.

Bast menghasilkan serat yang lebih nipis dan panjang berbanding dengan teras, dengan itu menghasilkan serat yang mempunyai nisbah aspek ratio yang lebih tinggi. Perubahan dalam serat morfologi adalah jelas apabila suhu pulped meningkat. Serat teras mempamerkan variasi ketara dalam panjang gentian , lebar serat, dan ketebalan dinding pada semua parameter. Garis pusat lumen dan nisbah aspek ratio tidak terjejas ketara oleh perbezaan dalam suhu pulpa , masa pulpa , dan interaksi antara kedua-dua parameter tersebut. Lebar serat berkurangan dengan peningkatan suhu dan masa pempulpaan, tetapi diameter lumen tidak terjejas dengan ketara. Nisbah aspek ratio juga menurun dengan peningkatan suhu dan masa pempulpaan. Panjang serat teras menurun dengan peningkatan suhu dan masa pempulpaan. Lebar serat menunjukkan pengurangan berterusan kerana keadaan pulpa menjadi lebih teruk. Oleh yang demikian, nisbah Runkel turut menurun. Nilai pH bagi kedua-dua serat berkurangan apabila suhu meningkat; gentian teras adalah lebih berasid (pH = 3.8) berbanding dengan gentian kulit (pH = 4.5). Ianya jelas bahawa gentian kulit adalah lebih tahan asid dan memaparkan kapasiti buffering asid yang lebih besar berbanding dengan gentian teras.
Siasatan daripada kesan tekanan penapisan (6 dan 8) dan kandungan resin (10, 12, 14 %) pada sifat fizikal (WA dan TS) , dan mekanikal (MOE , MOR , IB) MDF dibuat daripada campuran batang kenaf (Hibiscus cannabinus L.) dan kayu getah menunjukkan bahawa penapisan termo-mekanikal dan kandungan resin mempengaruhi kenaikan sifat-sifat fizikal dan mekanikal MDF. Pada tekanan penapisan 8 bar dengan kandungan resin 14%, MDF merekodkan WA yang optimum iaitu 83.12 % , TS sebanyak 20.2 %, MOR sebanyak 25.3 MPa, MOE sebanyak 3450 MPa dan IB sebanyak 0.51 MPa [kepadatan 700 (kg/m^3)].

Kaedah gerak balas permukaan telah digunakan untuk mewujudkan pemboleh ubah proses yang optimum (kandungan resin, dan tekanan) untuk kesan terhadap ciri papan. Dengan menggunakan permukaan sambutan dan plot kontur, set optimum pembolehubah operasi boleh diperolehi secara grafik , untuk mencapai ciri papan yang dikehendaki. Oleh itu , adalah dicadangkan bahawa ciri papan meningkat apabila kandungan resin dan tekanan meningkat. Kesimpulan boleh dibuat bahawa mana-mana parameter, secara individu , mempunyai kesan yang positif terhadap peningkatan ciri papan. Kesan utama parameter diatur seperti berikut: Kesan utama kandungan resin > tekanan.

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This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfillment of the requirement for the degree of Doctor of Philosophy. The members of the Supervisory Committee were as follows:

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LIST OF ABBREVIATIONS

AFM	Atomic force microscopy
ANOVA	Analysis of variance
AR	Aspect ratio
BC	Buffering capacity
CCD	Central composite design
CMP	Chemical mechanical pulp
CR	Compression ratio
CTMP	Chemical thermo-mechanical pulp
D/IF	Delamination/internal fibrillation
EFB	Empty fruit bunch
HCL	Hydrochloric acid
IB	Internal bond strength
INTROP	Institute of Tropical Forestry and Forest Products
JANS	Japan, Australia and New Zealand Standard
L/D	Length to diameter ratio
LE	Linear expansion
LSD	Least significant difference
MC	Moisture content
MDF	Medium Density Fiberboard
MOE	Modulus of elasticity
MPOB	Malaysian Palm Oil Board
MUF	Melamine modified urea-formaldehyde
NaOH	Sodium hydroxide
NSSC	Neutral sulphite semi-chemical
RMP	Refiner mechanical pulping
RR	Runkel ratio
RSM	Response Surfaces Methodology
RW	Rubber wood
SAS	Statistical analysis system
TMP	Thermomechanical Pulping
TS	Thickness swelling
UF	Urea–formaldehyde
WA	Water absorption

CHAPTER 1

INTRODUCTION

1.1 Background

The available supply of wood is becoming limited in many parts of the world as a result of environmental and green movement, landfill regulations and recycling trends. Due to poor resources of wood for composite board manufacturing, non-wood fibres play a major role in providing the equilibrium between supply and demand (Kalaycıoglu and Nemli, 2006a). However many factors such as politic, trade and industry, social, geographic and ecology determine the accessibility and end use of these renewable bio-resources throughout the world (Aji et al., 2009).

Medium density fibreboard (MDF) is manufactured by reconstituting fibres through a flexible and wide range of manufacturing conditions; any non-wood fibre, including various agricultural crops, could be potential raw material for the manufacture of MDF (Xing et al., 2006). Wide varieties of crops from many different regions around the world have been studied for this purpose (Papadopoulos, 2003). In general, lignocellulosic non-wood fibres are a relatively inexpensive substitute to higher-quality wood fibres (Ye et al., 2007).

Kenaf, *Hibiscus cannabinus* L., is a biodegradable and environmentally friendly crop with one of the highest rates of CO2 absorption of any plant (Kalaycıoglu and Nemli, 2006b). It is very effective in mitigating the effects of global warming and is currently being grown in many continents (tropical and subtropical) for its beneficial properties.

Nowadays, there are various new applications for kenaf including paper products (newsprint, bond paper, and corrugated liner board) (Mossello et al., 2010). Recent researches to have further increased the diversity of uses for kenaf by demonstrating its suitability in building materials (fibreboards), textiles, adsorbents, and fibres in new and recycled plastics (Russo et al., 2012). One of the most rapidly expanding applications for kenaf fibres at present is in composites. The interest in using natural fibres in composites has increased during recent years due to their combustible, non-toxic lightweight, non-abrasive, low-cost and biodegradable properties (Jawaid and Abdul Khalil, 2011).

In Malaysia, realizing the diverse possibilities of commercially exploitable derived products from kenaf, the National Kenaf Research and Development Program has been formed in an effort to develop kenaf as a possible new industrial crop for Malaysia. The government has allocated RM12 mil for research and further development of the kenaf-based industry under the 9th Malaysia Plan (2006–2010) in recognition of kenaf as a commercially viable crop (Abdul Khalil et al., 2010).

1.1.1 Kenaf anatomy and its influence in pulping condition

The kenaf stem is composed of an outer layer (bark) and a core. It is easy to separate the stem into bark and core, either by chemicals and/or by enzymatic retting. The bark constitutes 25 to 40% of the stem dry weight and shows a dense structure. On the other hand, the core is wood-like and makes up the remaining 60 to 75% of the stem. The core exhibits an isotropic and almost amorphous pattern (Aji et al., 2009). However, the bark shows an orientated, highly crystalline fibre pattern. Kenaf bast and core are quite different with respect to their chemical compositions. Research indicates that kenaf core fibres are higher in holocellulose and lignin, while kenaf bast fibres are higher in cellulose, extractive, and ash content (Abdul Khalil et al., 2010).

1.1.2 MDF manufacture

A recent research trend has been aimed at improving the characteristics of MDF. Most important factors that influence the strength of the MDF as a composite material are the inherent strength of its components; fibres and the bond strength between fibres and binder. Recently, the focus of research was shifted to increase the yield of pulping processes without affecting the quality of the product. Furthermore, attention is being given to increasing the use of mechanical and thermo-mechanical pulp in fibre productions. In the MDF production, thermo-mechanical pulping (TMP) is commonly used to refine raw material into fibres (Li et al., 2011). In the refining step, the raw materials are ground to fibres. During grinding hot steam is used to heat the fibres and thus soften the lignin. This saves electrical energy and yields longer and stronger fibres. The crucial unit in fibreboard production is the refiner, since it controls the basic properties of the fibres. Bad refining cannot be compensated in the subsequent processing steps. Schematically, refiner mechanical pulping (RMP) and thermo-mechanical pulping (TMP) essentially consist of three phases: fibrization of chips/fibre bundles to convert them to intact single or broken fibres, the fibrillation of single or broken fibres to develop bonding ability, and finally, the creation of fines (Xing, Deng, et al., 2006). The refining process can be augmented by pressure and temperature using processes known as the pressurized refiner mechanical pulping and thermo-mechanical pulping. Some researchers have shown that thermal–mechanical refining conditions have obvious effects on final MDF panel properties. However, Labosky (Labosky et al., 1993) reported that an increase in refining pressure did not significantly affect MDF strength or dimensional stability properties. Krug (2001) suggested that increasing the steam pressure resulted in shorter fibre lengths, lower strength, and elastic properties, but improved long-term swelling properties. Roffael et al. found that high pulping temperature resulted in lower thickness swelling (TS) and water absorption (WA) of MDF panels (Roffael et al., 2007).

Groom et al. (2004) studied the effects of varying refining pressure on the properties of refined fibres. They found that steam pressure caused some changes in the chemical composition of refined fibres. The percentages of extractives and glucose increased, while the xylose, galactose, and mannose decreased with increasing refining pressure. Xing (2006) reported that both preheating retention time and steam pressure directly affected MDF panel properties.

1.2 Problem statement

Within the past few years, there has been a dramatic increase in the use of natural fibres such as kenaf for making a new type of environmentally–friendly composites. Recent advances in natural fibre development, genetic engineering, and composite's science offer significant opportunities for improving materials from renewable resources with enhanced support for global sustainability.

Out of the variety of non-woods available (eg. bagasse, hemp, cornstalk, wheat straw. kenaf), kenaf was chosen as the raw material to work with because it is widely grown in tropical and subtropical regions and its high fibre quality. It is regarded as the most promising and suitable raw material for both developing and developed countries. Given the supply of wood, particularly in developing countries, it was decided to study kenaf for the production of MDF manufacturing. An extensive literature review revealed the few efforts have been made in the mechanical pulping of kenaf and MDF production.

Kenaf can be used as the form of whole stem, bast or core. Using the whole stem is a more attractive method as it bears significant practical and economic advantages: it is a simple process and free of the additional separation costs. Some researchers have reported that newsprint paper of excellent quality can be made from whole kenaf stalks and that kenaf pulp can be mixed with conventional softwood pulps to produce a wide range of paper grades (Li et al., 2011). By properly selecting the right pulp and refining condition kenaf can be a suitable material for fibre production in MDF manufacturing.

The evidence outlined above suggests potential usage of kenaf as whole raw materials for MDF. It is critical to obtain the right refining condition to product good fibre properties that will result in good MDF performance. In this study, whole kenaf stems were refined using TMP refiner under different refining parameters. The fibre properties were analyzed for each refining condition. The fibres then were used in the MDF production and MDF performances were evaluated. The main purpose of the study was to determine the optimum refining conditions for whole stem to produce MDF panels with high-strength properties and good dimensional stability. This research will have an impact on economic development in Malaysia and other southern states by providing agriculturists with alternatives in crop choices and adding value to the kenaf crop.

1.3 Objectives

The main object of this study was to evaluate and optimized the refining/pulping variables for kenaf stem for the production of MDF of acceptance strength. In this thesis, MDF was prepared from kenaf, rubberwood and their mixture. The parameters of preparation and the resulting intermediate products as well as the final MDF, were characterized and compared at each experimental step. The present research concentrated on the conditions of preparation (pulping, fibre processing) and the factors that influenced the main properties of MDF boards such as mechanical and physical properties.

The objectives of this study were to:

1. Determine the effect of temperature and time on the morphological properties of Kenaf, bast and core such as length of fibre, fibre diameter, wall thickness of fibre, Runkel ratio (RR), Aspect ratio (AR) and Compression ratio (CR).
2. Determine the effect of temperature and time on the pH and buffering capacity of kenaf bast and core fibres.
3. Evaluate the effect of thermo mechanical pulping (TMP) refining variables on the basic properties of MDF manufactured from kenaf and rubber wood fibres.
4. Optimize of the processing variables in kenaf-rubber wood MDF panel manufacturing technology.

Several analyses are employed to characterize the physical properties of fibres and the subsequent changes of fibre structure as a result of refining. The analyses include the following aspects:

1) Morphological modification: fibre separation modes, changes in fibre length, width, pit structure, lumen area and cell wall thickness, Aspect ratio, Runkel ratio and Compression ratio, etc.
2) MDF board characteristics: density profile, bonding ability, tensile strength, modulus of rupture and modulus of elasticity, thickness swelling, water absorption, etc.

The results from these analyses were compared and analyzed against those of the microscopic micrographs to establish possible interrelations between the fibre characteristics and MDF properties. Correlation regressions were established between fibre morphology and board properties.

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