UNIVERSITI PUTRA MALAYSIA

CHARACTERISATIONS OF KRAFT PULP AND PAPER PROPERTIES FROM ACACIA AURICULIFORMIS A. CUNN. EX BENTH.- A CONFOCAL MICROSCOPY ANALYSIS

LIEW KANG CHIANG

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By

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Thesis Submitted in Fulfilment of the Requirement for the Degree of Doctor of Philosophy in the Faculty of Forestry Universiti Putra Malaysia

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Chairman : Dr. Jalaluddin Harun

Faculty : Forestry

This study was carried out to characterise the transverse dimensions of mechanically treated (beaten) kraft *Acacia auriculiformis* pulp (AAP) and mixed tropical hardwood commercial pulp (MTHCP) fibres using the fast and non-destructive method of optical sectioning ability of confocal laser scanning microscopy. Also included in the study are the determination of chemical constituents, fibre morphologies using the image analyser and the optimum pulping conditions. Laboratory handsheets were produced using pulps beaten at varying beating degrees using the PFI mill, and evaluated for their physical and mechanical properties. Established standards were followed throughout the study.

Results from the chemical constituents and fibre morphology determinations for *A. auriculiformis* sample were within the comparable range of previous studies. Optimum kraft pulping conditions was achieved at 19% active alkali for *A. auriculiformis* wood chips with a 51.9% screened yield, 0.085% reject and Kappa number 19.1. Laboratory handsheets were produced from AAP and MTHCP fibres

that were beaten using the PFI mill, at 3 beating degrees; 0, 5000 and 10000 revolutions. Generally, the AAP fibres exhibited comparable, if not better, physical and mechanical properties than MTHCP. As beating progressed, pulp freeness decreased with increasing drainage time. This has resulted the tensile strength, bursting strength, tearing resistance, and folding endurance to increase but an inverse for bulk and air permeance.

Beaten and unbeaten fibre cross-sectional images were generated under epi-fluorescent mode, and different fibre cross-sectional images can be observed, with unbeaten fibres usually uncollapsed to partial and fully collapsed fibres of beaten fibres. Generally, transverse dimensions determined from image analysis were found to decrease in centreline perimeter, lumen area, fibre area, lumen perimeter, fibre thickness, fibre wall thickness, and aspect ratio except for fibre width and collapse index, due to the beating process. In general, it was found that AAP has lower fibre transverse dimensions than MTHCP.

Regression equations were built using the Maximum R Improvement method involving pulp and paper properties and calculated fibre transverse dimensions. Predictions were done at a beating range of 0-10000 rev. with CI dominantly influenced the pulp and paper properties in the 1-variable equations, followed by the inclusion of AR in most of the 2-variable equations for AAP and MTHCP. The correlation coefficients for fibre transverse dimensions indicated positive relationships with freeness, bulk, tear index and air permeance whilst negative with drainage time, tensile index, breaking length, burst index and fold.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia
sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

PENCIRIAN SIFAT PULPA KRAFT DAN KERTAS DARI *ACACIA AURICULIFORMIS* A. CUNN. EX BENTH. – ANALISIS KONFOKAL MIKROSKOPI

Oleh

LIEW KANG CHIANG

Januari 2002

Pengerusi : Dr. Jalaluddin Harun

Fakulti : Perhutanan

Penyelidikan ini dijalankan untuk mencirikan beberapa dimensi merentas gentian pulpa kraft *Acacia auriculiformis* (AAP) dan pulpa komersial kayu keras tropika campuran (MTHCP) secara mekanikal menggunakan keupayaan pemotongan optikal mikroskopi konfokal imbasan laser yang merupakan kaedah yang cepat dan tidak memusnahkan. Kajian di dalam penyelidikan ini merangkumi penentuan kandungan kimia, morfologi gentian menggunakan alat penganalisa imej dan keadaan pempulpaan optimum. Kertas makmal dibuat menggunakan pulpa yang dipukul pada pelbagai takat menggunakan pemukul PFI, dan beberapa ujian dijalankan untuk mengkaji sifat-sifat fizikal dan mekanikal mereka. Piawaian digunakan sepanjang penyelidikan dijalankan.

Keputusan daripada penentuan kandungan kimia dan morfologi gentian pada sampel *A. auriculiformis* adalah tergolong dalam julat perbandingan dengan penyelidikan dahulu. Pempulpaan optimum didapati pada kegunaan 19% aktif alkali dengan hasil bertapis 51.9%, hasil terbuang 0.085% dan nombor Kappa 19.1. Kertas makmal

dihasilkan daripada gentian AAP dan MTHCP, dipukul menggunakan PFI, pada 3 takat pukulan, 0, 5000 dan 10000 revolusi. Pada amnya, gentian AAP menunjukkan sifat-sifat fizik dan mekanik yang setanding atau lebih baik berbanding dengan MTHCP. Apabila pukulan diteruskan, kebebasan pulpa menurun dengan peningkatan masa pengaliran. Ini mengakibatkan kekuatan regangan, pecahan, koyakan, dan lipatan kertas meningkat tetapi keputusan sebaliknya bagi sifat pukal dan penembusan angin.

Beberapa imej keratan-rentas gentian yang tidak dipukul dan dipukul dijana di bawah mod "epi-fluorescent", dan imej keratan-rentas gentian boleh dilihat; dengan gentian yang tidak dipukul biasanya tidak runtuh kepada runtuhan sebahagian dan runtuh sepenuhnya bagi gentian yang dipukul. Pada amnya, dimensi merentas ditentukan daripada analisa imej didapati menurun dalam lilitan garisan tengah, ruangan lumen, ruangan gentian, lilitan lumen, ketebalan gentian, ketebalan dinding gentian, nisbah aspek kecuali bagi kelebaran gentian dan indek keruntuhan, disebabkan oleh proses pukulan. Pada amnya, didapati AAP mempunyai dimensi-dimensi merentas gentian yang lebih rendah daripada MTHCP.

Formula regresi dibuat menggunakan kaedah "Maximum R Improvement" yang melibatkan sifat-sifat pulpa dan kertas dan dimensi merentas gentian yang dikira. Ramalan dilakukan pada julat pukulan 0-10000 rev. dengan CI mempengaruhi kukuh sifat-sifat pulpa dan kertas dalam persamaan 1-pembolehubah, dengan diikuti oleh AR dalam kebanyakan persamaan 2-pembolehubah untuk AAP dan MTHCP. Perkaitan koeffisien untuk dimensi merentas gentian menunjukkan hubungan positif dengan kebebasan, sifat pukal, indek koyakan dan penembusan angin sementara negatif dengan masa pengaliran, indek regangan, kepanjangan memutus, indek pecahan dan lipatan.

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I certify that an Examination Committee met on 11th January 2002 to conduct the final examination of Liew Kang Chiang on his Doctor of Philosophy thesis entitled "Characterisations of Kraft Pulp and Paper Properties from *Acacia auriculiformis* A. Cunn. ex Benth. – A Confocal Microscopy Analysis" in accordance with Universiti Pertanian Malaysia (Higher Degree) Act 1980 and Universiti Pertanian Malaysia (Higher Degree) Regulations 1981. The Committee recommends that the candidate be awarded the relevant degree. Members of the Examination Committee are as follows:

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Date: 14 MAR 2002

DECLARATION

I hereby declare that the thesis is based on my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at Universiti Putra Malaysia or other institutions.

LIEW KANG CHIANG

Date: 16 JAN 2002

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LIST OF ABBREVIATIONS

AAP	19% *Acacia auriculiformis* pulp
MTHCP	Mixed tropical hardwood commercial pulp
rev.	Revolutions
a.a.	Active alkali
CLSM	Confocal laser scanning microscopy
CP	Centreline perimeter
LA	Lumen area
FA	Fibre area
LP	Lumen perimeter
D_{max}	Fibre width
D_{min}	Fibre thickness
PW	Projected width
WT	Fibre wall thickness
AR	Aspect ratio
CI	Collapse index

CHAPTER 1

INTRODUCTION

In recent years, forest-based supply of the world has been declining. As the demand for wood increases with the increasing populations and incomes, forests are rapidly cleared and degraded, withdrawn from production for conservation purposes or other reasons, leaving the saddle to the remaining production forests (Brown and Ball 2000). The decline became evident in the last 30 years and prompted the intensification of reforestation with natural or exotic trees i.e. *Pinus* spp., *Eucalyptus* spp., *Picea* spp., *Abies* spp., *Larix* spp., *Tectona* spp., *Araucaria* spp. and *Acacia* spp.

In Malaysia, the Compensatory Forest Plantation Project was launched in 1982, aimed at establishing 188,000 ha of fast-growing species of trees within a 15 year-period. The objective of this project was to supplement the projected shortage of timber to meet domestic consumption in the mid 1990's. Among the species planted were *Acacia mangium*, *Gmelina arborea* and *Paraserianthes falcataria* (Mohd. Nor and Salleh 1992).

Another *Acacia* spp., *Acacia auriculiformis* was also introduced and planted at degraded sites such as tin tailing areas in Peninsular Malaysia (Ang 1986, Ang and Yusuf 1989, Ang 1991) and in reforestation tree project in Sarawak (Joseph 1992). *A. auriculiformis* A. Cunn. ex Benth. is a multipurpose, leguminous tree in the subfamily Mimosoideae, and grow naturally in Australia, Papua New Guinea and Indonesia. It is a vigorous, nitrogen-fixing tree with an outstanding ability to grow well on harsh sites and on different types of soil in the tropics, including very infertile, clayey, saline and seasonally-waterlogged soils (Turnbull 1986). Doran and Turnbull (1997) add that the trees could also tolerate acidic, alkaline and moderate dry seasons making them a very useful species for the rehabilitation of degraded lands. This species is also better known outside its natural range of distribution as the most adaptable species of all kinds of tree planting programmes in tropical humid and subhumid lowland regions (Pinyopusarerk 1990). It is widely cultivated as an exotic plant in Asia, Africa and South America and increasingly used for reforestation in new areas. In southeast Asia, *A. auriculiformis* is planted for fuelwood purposes, erosion control, ornament or shade and a promising species for pulpwood production (Turnbull 1986). Doran and Turnbull (1997) added that with significant improvement on their stem form by selection and breeding would provide outstanding prospects for industrial plantations to produce paper pulp and other timber by-products.

In Malaysia, there are currently 18 paper mills utilising mainly waste paper and only one integrated pulp and paper mill in Sabah, namely the Sabah Forest Industries Sdn. Bhd. (Lai 1997), that is utilising fibres from their plantation grown *Acacia* spp., *A. mangium*. *A. mangium* also has proven to produce excellent particleboard – a type of wood composite (Tham 1976). Therefore, there is a need to evaluate the suitability of planted *A. auriculiformis* as a source of wood, especially pulpwood. In pulp and paper, much work was done in evaluating its pulping feasibility and paper properties (Guha and Pant 1966, Phillips and Logan 1976, Phillips et al. 1979, Logan and Balodis 1982, Mohd. Nor et al. 1986). Logan (1987) found that the plantation-grown trees had the potential for the production of unbleached kraft pulp (for bags, wrapping paper, linerboard) and high quality neutral sulfite semichemical pulp (for corrugating, medium and higher-grade packaging-type products). The sulfate process with 13% alkali yields up to 55% of screened pulp and is therefore less suitable for high-yield mechanical pulps (Phillips et al. 1979).

Different pulping processes and conditions, pulp refining and other pulp processing treatments, and product furnish compositions were used to modify and enhance certain papermaking and product properties and behaviours. Pulp fibres, which are tubular in shape, can be changed. In papermaking, mechanical treatment of beating process is used to collapse the fibres' shapes. It introduces external forces to the fibres, which subsequently changes their native cross-sectional shape and collapsed (Jang and Seth 1998, Jang et al. 1996). Jang and Seth (1998) stated that these fibre cross-sectional properties could be measured accurately with the help of image analysis, therefore enabling the examination of the nature of changes in fibres with mechanical treatment. In addition, the development of automated computer based image analysis systems over the last 15 years (Ilic and Hillis 1983, Lee and Rosen 1985, Schnell and Sell 1989, Peachey and Osborne 1990, Evans 1994, Russ 1995, Van der Heijden et al. 1995, Diao et al. 1996) has greatly improved the speed and accuracy with which wood cell dimensions can be measured, either for wood anatomical descriptions or for wood quality assessment. With this information on fibre morphology and fibre transverse dimensions could facilitate the determination of the ultimate product qualities, and whether or not certain products can be produced from certain fibres or fibre furnishes (Kibblewhite and Bailey 1988).

The conventional light microscopy is a time consuming and expensive sample preparation, often involving embedding, sectioning and staining of the specimen. Such technique generally allows the measurement of large numbers of cells but at poor resolution or with limited accuracy (Donaldson and Lausberg 1998). These limitations were overcome with by the introduction of the current technology of confocal laser scanning microscopy (CLSM) that has found wide applications in the biological sciences as well as material sciences. It is a relatively new light microscopial imaging technique introduced around 1980 by M. Petran and A. Boyde (Pawley 1990, Boyde 1994). The primary value of the CLSM to the scientists is its ability to produce optical sections through a 3-dimensional (3-D) specimen – e.g., living materials such as an entire cell or a piece of tissue, non-living materials such as circuit boards or electronic chips. This ability could produce information from only one focal plane to a good approximation and able to solve 3-D biological or material problems where information from regions distant from the plane of focus can obscure the image of thick objects. Digital confocal images generated from this computer-controlled CLSM are amendable to image processing and analysis (Anonymous 2001). These advantages could be utilised in this study to generate information on the fibres' transverse dimensions for the plantation-grown *A. auriculiformis* hardwood. Currently, the information on their fibre cross-sectional properties for use in pulp and paper research, whether as individual fibre or within a population of fibres, is not available. Therefore, in this study, kraft pulp fibres of *A. auriculiformis* were prepared, treated mechanically and viewed under epifluorescence mode, and compared with mixed tropical hardwood commercial pulp (MTHCP). Cross-sectional images are generated, using the optical sectioning ability from the CLSM. Later, the images were processed and analysed using image analysis for their transverse/cross-sectional dimensions. This study specifically aims:

(a) To determine the chemical constituents of *A. auriculiformis*, and their fibre morphology using the image analyser.

(b) To determine the optimum pulping conditions for *A. auriculiformis* wood chips, prepare laboratory handsheets from the pulp produced at various beating degrees and evaluate their physical and mechanical properties.

(c) To generate cross-sectional images of *A. auriculiformis* kraft pulp fibres beaten at various beating degrees using the CLSM and determine their cross-sectional dimensions using the image analysis software.

(d) To evaluate the relationships of laboratory handsheet properties with different calculated fibre transverse dimensions, and the correlations between the fibre transverse dimensions and pulp and paper properties with the beating.

CHAPTER 2

LITERATURE REVIEW

2.1 General Overview

This chapter presents a review of literature regarding *A. auriculiformis*, chemical constituents of its wood, fibre morphology, kraft pulping and paper properties, and CLSM.

2.2 *Acacia auriculiformis* A. Cunn. ex Benth.

The genus *Acacia* comprised about 1,100 species where 850 occur in Australia, Papua New Guinea and Indonesia. It is an exotic tree to Malaysia where nine species have been introduced; *A. mangium, A. auriculiformis, A. crassicarpa, A. aulacocarpa, A. holosericea, A. cincinnata, A. farnesiana, A. podalyriaefolia* and *A. richii* (Boland et al. 1984).

Acacia auriculiformis is a nitrogen-fixing member of the family Leguminoseae, sub-family Mimosoideae. Its natural occurrence is in Papua New Guinea, islands of the Torres Straits, Northern Australia and Queensland to 15^{o}S (Jackson 1994). Its botanical name of *Acacia auriculiformis* A. Cunn. ex Benth. came from the Latin name *auricula* - external ear of animals, *forma* - form, figure or shape in allusion to