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UNIVERSITI PUTRA MALAYSIA

# MORPHOLOGY OF MECHANICALLY REFINED AND CHEMICALLY-TREATED OIL PALM FRUIT BUNCH FIBRES

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Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Master of Science

**MORPHOLOGY OF MECHANICALLY REFINED AND CHEMICALLY-TREATED OIL PALM FRUIT BUNCH FIBRES**

By

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**September 2007**

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

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The study of surface characterisation of oil palm empty fruit bunch (EFB) fibre before and after physical and chemical treatments was carried out by using light microscopy, Scanning Electron Microscopy (SEM) and Fourier Transform Infrared (FTIR) techniques. One of the objectives of the study was to evaluate fibre microphological characteristics of different parts of oil palm empty fruit bunch. This study was also conducted to examine the effects of chemical pre-treatment and mechanical refining on the morphological characteristics of EFB fibres and to evaluate the surface characteristics of chemically modified EFB fibre after being treated with succinic anhydride. For comparison, samples from fresh fruit bunches were also examined. The mechanical refining process employs pre-treatment with water and sodium hydroxide (1%). Chemical modification on EFB fibres with succinic anhydride was carried out for 1,2,3,4,5 and 6 hour each at 80$^{o}$C, 100$^{o}$C and 120$^{o}$C. The objective of this study was to examine the effects of succinic anhydride modification on the morphology of EFB fibres, as well as the extent of cell wall bulking.

Oil palm EFB differs from wood fibres since it does not posses structures such as cambium, ray cells, sapwood or heartwood portion. These differences are highlighted in this study. EFB is formed from structures of vascular bundles which consist of xylem cells, phloem cells, vessels and fibrous sheath and embedded within soft ground parenchymatous tissue. The examination on the surface morphology of the fibre vascular bundle suggested that this material may require pretreatment and chemical modification to enhance surface quality within a fibre/matrix composite due to its waxy surface nature. Light and SEM micrographs revealed on the presence of cell inclusions such as stegmata which marked it's presence through the globular protrusion on the surface of fibre. Silica bodies are found abundantly embedded within the stegmata cells which are detachable upon processing.

This study also clarified on the differences in properties of fibres according to different parts of a single fruit bunch. The study on the properties of individual fibre from different section of EFB (core, stalk and spikelets) concluded that fibre properties within the fruit bunch are not homogenous. The stalk portion of the fruit bunch has short fibre lengths (μm) and high tendency to collapse. Compare to the core of the fruit bunch, the fibre length from this part is longer. Being compared to wood fibre properties, the core of EFB exhibit quiet a comparable properties especially referring to its Runkel ratio, coefficient of suppleness and felting power.

The results also indicate that mechanical refining significantly changed the morphology of EFB fibre structure. The untreated and unrefined EFB fibre is still in it's original cylindrical shape suggesting the presence of lignin which holds up the cell wall. The

Special thanks to the lecturers and staffs of Faculty of Forestry especially to fellow colleagues: Sapari Mat, Frisco Nobilly, Norzanalia, Mastura, Rasmina, Nurulhuda and Dr. Manohar for being such a good listener and source of encouragements. Heartfelt gratitude to her fellow postgraduates, especially to FORGRAD members, and to her other numerous friends for their inspirations and guidance. Much appreciation also goes to Hazeline Aasyiqeen, Rohaida, Ann and friends for continuous support and helping hands.

Last but not least, the author wishes her deepest appreciation to her dearest parents and brother for their encouragement, understanding, love and sacrifices along the completion of this study.

surface of the fibres with is covered mainly by thick waxy cuticle layer with occurrences of globular protrusion indicating the presence of silica bodies. The effect of water soaking and sodium hydroxide has extensively removed the waxy cuticle layer and most silicate structures from the fibre surface. Refined fibres appeared defiberized and fibrillated especially on sodium hydroxide (NaOH) pre-treated fibres. Internal and external fibrillation which relates to shearing in fibre wall and peeling of super-facial layer was extensively observed. Longitudinal compression of disintegrated fibre was observed extensively especially on sodium hydroxide treated fibres. Most of the silica bodies have been detached from fibre surface, creating a porous look. Swelling of cellulose, hemicellulose and lignin contributes to the efficiency of the refining process. This occurrence also attributed to the delignification and dissolution of non-cellulosic substance (i.e hemicellulose).

The chemical modification treatment succinic anhydried on EFB fibres has resulted for morphological changes such as swelling and surface smoothing. Through SEM examination, swelling of cell wall was clearly observed on individual fibre cell wall, areas surrounding the voids and also stegmata cells although it was less obvious on metaxylem vessel, protoxylem, protophloem, pits and perforation plates. Surface cracks and degradation due to the cell wall bulking and swelling effect was not visible on any the cells. Introduction of foreign moieties on to the surface hydroxyl group has made the appearance smoother due to removal of waxy layer which also reveals the stegmata cells and voids area. The extent of esterification was examined through the Weight Percent Gain (WPG) value. WPG subsequently increases as reaction time prolonged and with higher reaction temperature. The swelling of fibre, measured through cell diameter to

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

**MORFOLOGI GENTIAN TANDAN KOSONG KELAPA SAWIT SETELAH RAWATAN FISIKAL DAN KIMIA**

Oleh

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Kajian keatas sifat-sifat permukaan gentian tandan kosong kelapa sawit setelah menjalani rawatan fizikal dan kimia telah dijalankan dengan menggunakan teknik mikroskopi cahaya, Scanning Electron Microscopy (SEM) dan Fourier Transform Infrared (FTIR). Objektif kajian ini adalah untuk meneliti siftat-sifat morfologi gentian tandan kelapa sawit dari bahagian-bahagian yang berbeza. Kajian ini juga dijalankan untuk meneliti kesan proses rawatan fizikal dan kimia keatas morfologi gentian serta menilai sifat-sifat permukaan gentian selepas rawatan suksinik anhidrida. Untuk perbandingan, sampel-sampel yang diperoleh dari tandan kelapa sawit yang masih segar turut diteliti. Proses pengisaran secara mekanikal dijalankan dengan rawatan rendaman air dan sodium hidroksida (1%). Rawatan modifikasi kimia dengan menggunakan suksinik anhidrida telah dijalankan pada tempoh 1, 2,3,4,5 dan 6 jam pada suhu 80$^{o}$C, 100$^{o}$C dan 120$^{o}$C. Objektif kajian ini adalah untuk menyelidik kesan modifikasi kimia dengan menggunakan suksinik anhidrida keatas morfologi gentian tandan kosong kelapa sawit dan juga tahap pengembangan dinding sel.

Gentian tandan kosong kelapa sawit amat berbeza dengan gentian kayu memandangkan ia tidak mengandungi struktur-struktur tertentu seperti kambium, sel-sel ruji, bahagian gubal mahupun teras. Perbezaan ini diberikan penekanan lagi di dalam kajian ini. Gentian kelapa sawit adalah terbentuk dalam struktur berkas vaskular yang terdiri dari sel-sel xilem, sel-sel floem, sel salur dan selaput gentian yang dikelilingi oleh tisu-tisu parenkima yang lembut. Penelitian terhadap morfologi permukaan berkas vaskular telah menunjukkan bahawa permukaannya yang diselaputi lapisan berlilin yang tebal ini memerlukan rawatan dan modifikasi kimia bagi mempertingkatkann kualiti permukaan di dalam komposit gentian/matrik. Mikrograf dari mikroskop cahaya dan SEM telah menunjukkan kehadiran sel stegmata yang kelihatan seperti bonjolan di permukaan gentian. Di dalam sel stegmata terdapat jasad-jasad silika yang mudah terlerai semasa gentian diproses.

Kajian ini juga memperjelaskan perbezaan sifat gentian berdasarkan bahagian-bahagian yang berbeza di didalam sesebuah tandan. Kajian terhadap sifat-sifat gentian individu yang diperoleh dari bahagian-bahagian tandan yang berbeza (empulur, tangkai dan spikelet) telah menunjukkan bahawa ia tidak ia tidak bersifat homogenus. Bahagian tangkai tandan adalah terdiri dari gentian yang terpendek (μm) dan kebolehan untuk meruntuh yang tinggi. Jika dibandingkan dengan bahagian empulur tandan, gentian di bahagian ini adalah lebih panjang. Dari segi sifat gentian, empulur tandan kosong mempunyai gentian yang hampir menyamai sifat-sifat gentian kayu dari segi nisbah Runkel, koefisyen kelenturan dan kemampuan gentian untuk terjalin.

Keputusan kajian juga menunjukkan pengisaran secara mekanikal telah mengubah struktur morfologi gentian tandan kosong. Gentian yang tidak dirawat dan dikisar kelihatan masih berbentuk silinder yang menandakan kehadiran lignin yang berfungsi untuk memegang dinding sel. Permukaan gentian secara umumnya diselaputi lapisan berlilin yang tebal dengan bonjolan-bonjolan oleh taburan jasad silika. Rawatan rendaman air dan sodium hidroksida telah menghilangkan lapisan berlilin yang tebal dan kebanyakan struktur-strukutur jasad silika yang terdapat di permukaan gentian. Gentian yang telah dikisar kelihatan lebih terurai dan bercabang terutamanya pada gentian yang telah dirawat dengan sodium hidroksida. Fibrilasi dalaman dan luaran yang disebabkan oleh koyakan dinding gentian dan pengelupasan lapisan luaran amat nyata kelihatan pada gentian tersebut. Permukaan gentian kelihatan lebih poros tanpa kehadiran jasad-jasad silika. Pengembangan unsur selulosa, hemiselulosa dan lignin telah membantu dalam menghasilkan proses pengisaran yang lebih efisyen. Ini juga disebabkan oleh proses nyah-lignin dan terlarutnya unsur-unsur bukan selulosa seperti hemiselulosa.

Rawatan modifikasi kimia dengan menggunakan suksinik anhidrida keatas gentian tandan kosong kelapa sawit telah menghasilkan perubahan morfologi seperti pembengkakan dan pembersihan permukaan gentian. Melalui penelitian SEM, pengembangan pada dinding sel amat jelas kelihatan pada dinding gentian individu, di sekeliling kawasan lompang dan juga sel stegmata. Kesan ini sebaliknya tidak kelihatan jelas pada sel salur metaxilem, protoxilem, protofloem, pit dan plat perforasi. Rekahan permukaan gentian dan degradasi akibat dari pengembangan dinding sel tidak kelihatan pada gentian yang dirawat. Pengenalan unsur-unsur asing pada kumpulan hidroksil telah menghasilkan permukaan gentian yang lebih licin. Ini disebabkan oleh kehilangan

permukaan kutikel berlilin yang mendedahkan sel-sel stegmata dan lompangan kosong. Tahap esterifikasi telah dinilai melalui Peratus Berat Perolehan (PBP). Nilai PBP meningkat dengan pertambahan masa tindakbalas dan kenaikan suhu tindakbalas. Tahap pengembangan gentian yang dinilai menerusi nisbah diameter sel kepada diameter lumen sel juga bertambah secara langsung dengan nilai PBP. Corak spectrum yang diperoleh menerusi ujian FTIR telah menunjukkan beberapa kawasan yang aktif seperti di 1705 – 1750 $cm^{-1}$ 1230 – 1282 $cm^{-1}$ dan 3200 – 3600 $cm^{-1}$. Puncak-puncak spectrum ini dipengaruhi oleh kumpulan berfungsi dan juga tahap esterifikasi (nilai PBP).

## ACKNOWLEDGEMENTS

The author wishes to express her sincere gratitude to her supervisor Assoc. Prof. Dr. Paridah Md. Tahir for her constructive comments, assistance and advice throughout this study. Without her contribution of ideas, guidance, moral support and encouragement, the completion of this study would have not been possible. Special gratitude is due to committee member Prof. Mohd Hamami Sahri and Assoc. Prof. Dr. Jalaluddin Harun for their constructive comments, valuable suggestion and encouragement.

Grateful acknowledgements are extended to the staffs of Wood Anatomy Laboratory Faculty of Forestry, especially to Pn. Halimah Hussein and Mr. Mohamad Talib for their assistance, guidance and support. The author is also indebted to the staff of Chemistry Laboratory, Ms. Siti Fazelin Mahamad for her help and support. Acknowledgments are due to the Institute of Bioscience (IBS) Universiti Putra Malaysia, particularly to the staffs of Microscopy Unit Pn. Nooraini Mohd. Ain, Mr. Rafiuz Zaman Haroun and Mr. Ho Oi Kuan for their assistance and guidance that has made this study a success. Much appreciation goes to the Malaysian Institute of Nuclear Technology (MINT) Research for granting permission in using testing facilities. Sincere thanks to Mr. Kamrolzaman Hussein for his assistance during the conduction of sample testing.

The author is greatly indebted to Universiti Putra Malaysia for the much needed financial support and to the Faculty of Forestry for granting her study leave and providing facilities.

cell lumen diameter ratio, also increase proportionally with the value of WPG. The spectrum obtained through examination with FT-IR has depicted several active regions such as 1705 – 1750 $cm^{-1}$ 1230 – 1282 $cm^{-1}$ and 3200 – 3600 $cm^{-1}$ which corresponds to the concentration of a functional group that strongly affects the intensity of the peaks which also corresponds to the extent of esterification (WPG values).

I certify that the Examination Committee met on 12th of September 2007 to conduct the final examination of Adlin Sabrina Muhammad Roseley on her Master of Science thesis entitled "Morphology of Mechanically Refined and Chemically-Treated Oil Palm Empty Fruit Bunch Fibres" in accordance with Universiti Pertanian Malaysia (Higher Degree) Act 1980 and Universiti Pertanian Malaysia (Higher Degree) Regulations 1981. The Committee recommends that the student be awarded the degree of Master of Science.

Members of the Examination Committee were as follows:

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Date : 21 February 2008

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

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## 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 UPM or other institutions.

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**ADLIN SABRINA MUHAMMAD ROSELEY**

Date : 1 February 2008

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