



UNIVERSITI PUTRA MALAYSIA

***CHARACTERISATION OF NANO WHISKERS FROM KENAF BAST AND
EFFECTS ON BIONANO COMPOSITES VIA SOLUTION CASTING***

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FROM KENAF BAST AND EFFECTS ON
BIONANOCOMPOSITES VIA SOLUTION
CASTING**

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By

LUKMANUL HAKIM ZAINI

**Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia,
in Fulfilment of the Requirements for the Degree of Master of Science**

March 2013

DEDICATION

My Beloved Mother, Jasni

My Beloved Father, R. Much. Zainal Abidin, S.pd

My Beloved sister, Khairunnisa



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

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March 2013

Chairman : Y. Bhg. Professor Paridah Md. Tahir, Phd

Faculty : Institute Tropical Forestry and Forest Product

There are limited studies of isolated cellulose whiskers from kenaf bast. Kenaf bast contains less than 30% of total weight, it needs small usage but high performance – high value. Cellulose nano whiskers (CNW) can be isolated by sulphuric acid hydrolysis from kenaf bast fibres and potentially have higher strength, increase efficiency and added value compared to normal fibre. Moreover, CNW has good properties as reinforcing material in any type of composites. In this study, cellulose whiskers were isolated from kenaf (*Hibiscus cannabinus* L.) bast fibres via sulphuric acid hydrolysis. Raw bast kenaf, NaOH treated and bleached fibres morphology was analyzed by Field Emission Scanning Electron Microscopy (FE-SEM) while the whiskers were analyzed by Transmission Electron Microscopy (TEM). Fourier Transform Infrared Spectroscopy (FTIR), X-ray diffractometry (X-RD), and thermogravimetric analysis (TGA) used to characterize all samples. Nanocomposites were produced by incorporating various amounts (2.5 – 10%) of resulted kenaf whiskers with Cellulose Acetate Butyrate (CAB). Resulted nanocomposites characterized by FTIR, TGA, DSC, and DMA.

The TEM images showed that the sulphuric acid whiskers have average diameters and length range of 3 nm and 305 nm, respectively. The FTIR study indicated that during the conversion process, most of the hemicellulose and almost all the lignin were removed by the NaOH and subsequent bleaching treatments. The crystallinity of kenaf whiskers was 72%. It is suggested that the lower crystallinity value compared to NaOH and bleached fibres because of the acid attacked the crystalline region. Thermogravimetric analysis (TGA) indicated that a two-stage decomposition behaviour was revealed in the kenaf whiskers due to incorporation of the sulphate group with the cellulose crystals.

FTIR analysis showed no intermolecular hydrogen bonding between CAB and whiskers. Thermal analysis found that whiskers reinforcement did not affect the decomposition temperature of resulted nanocomposites. However, good miscibility detected by the single decomposition temperature of nanocomposites. The addition of kenaf whiskers nanofillers did not have any effect on matrix melting temperature, recrystallization during first and second heating and cooling and glass transition temperature. It was concluded that kenaf whiskers addition did not affected the onset of translational and rotational backbone motions in the composite matrix. The dynamic mechanical improvement showed by the increment of kenaf whiskers as filler content into the CAB matrix is believed due to filler-filler interactions through hydrogen bonding.

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

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Oleh

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Pengerusi : Y. Bhg. Profesor Paridah Md Tahir, Phd

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Terdapat kajian terhadap selulosa *whiskers* terencil dari kulit kenaf. Kulit kenaf mengandungi kurang daripada 30% daripada jumlah berat, ia memerlukan penggunaan kecil tetapi berprestasi tinggi - nilai yang tinggi. Selulosa nano *whiskers* (CNW) boleh diasingkan oleh hidrolisis asid sulfurik dari gentian kulit kenaf dan berpotensi mempunyai kekuatan yang lebih tinggi, meningkatkan kecekapan dan nilai tambah berbanding serat biasa. Selain itu, CNW mempunyai ciri-ciri yang baik sebagai mengukuhkan bahan dalam mana-mana jenis komposit. Dalam kajian ini, selulosa *whiskers* telah diasingkan daripada kenaf (*Hibiscus cannabinus* L.) gentian kulit melalui hidrolisis asid sulfurik. Morfologi kulit kenaf, serat yang dirawat NaOH dan serat terluntur telah dianalisis mengikut Field Emission Scanning Electron Microscopy (FE-SEM) manakala *whiskers* dianalisis oleh Transmission Electron Microscopy (TEM). Fourier Transform Infrared Spectroscopy (FTIR), X-ray diffractometry (X-RD), dan thermogravimetric analysis (TGA) digunakan untuk mencirikan semua sampel. *Nanocomposites* telah dihasilkan dengan menggabungkan pelbagai jumlah (2.5 - 10%) kenaf *whiskers* dengan Butyrate Asetat selulosa (CAB).

Komposit nano yang dihasilkan kemudian dicirikan oleh FTIR, TGA, DSC, dan DMA.

Imej-imej TEM menunjukkan bahawa kenaf *whiskers* mempunyai diameter purata dan julat panjang masing-masing 3 nm dan 305 nm. Kajian FTIR menunjukkan bahawa semasa proses penukaran, kebanyakan hemiselulosa dan hampir semua lignin telah dikeluarkan oleh NaOH dan seterusnya rawatan pelunturan. Penghabluran kenaf *whiskers* adalah 72%. Ini mencadangkan bahawa nilai penghabluran yang lebih rendah berbanding dengan NaOH dan gentian terluntur kerana asid telah diserang rantau kristal. Termogravimetri analisis (TGA) menunjukkan bahawa tingkah laku dua peringkat penguraian telah didedahkan oleh TGA dalam kenaf *whiskers* kerana penubuhan kumpulan sulfat dengan kristal selulosa.

FTIR analisis menunjukkan tiada ikatan hidrogen molekul antara CAB dan *whiskers*. Analisis terma didapati bahawa tetulang *whiskers* tidak menjejaskan suhu penguraian nanokomposit yang dihasilkan. Walau bagaimanapun, Kebolehcampuran boleh dikesan oleh suhu penguraian tunggal nano komposit. Tambahan pula, kenaf *whiskers* nanofillers tidak mempunyai apa-apa kesan ke atas suhu lebur matriks, penghabluran semula semasa pemanasan pertama dan kedua dan penyejukan dan glass transition temperature. Ia menyimpulkan bahawa tambahan kenaf *whiskers* tidak menjejaskan permulaan gerakan translasi dan putaran tulang belakang dalam matriks komposit. Peningkatan mekanikal dinamik ditunjukkan oleh kenaikan kenaf *whiskers* sebagai kandungan pengisi ke dalam matriks CAB dipercayai disebabkan interaksi pengisi-pengisi melalui ikatan hidrogen.

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I certify that an Examination Committee has met on 14 March 2013 to conduct the final examination of Lukmanul Hakim Zaini on his Master of Science thesis entitled “Characterisation of cellulose nano whiskers from kenaf (*Hibiscus cannabinus* L.) bast and effect of filler loading to bionanocomposite properties”. In accordance with Universities and University College Act 1971 and the Constitution of the Universiti Putra Malaysia and [P.U. (A) 106] 15 March 1998. The committee recommends that the candidate be awarded the Master of Science.

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DECLARATION

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

LUKMANUL HAKIM ZAINI

Date: 14 March 2013



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