ISOLATION OF NANOFIBRILLATED CELLULOSE DERIVED FROM N36 *ANANAS COMOSUS* LEAF VIA CHEMO-MECHANICAL TREATMENT AND REINFORCEMENT IN CHITOSAN-BASED COMPOSITE FILMS.

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MASTER OF SCIENCE

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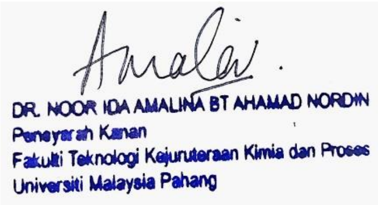
SUPERVISOR'S DECLARATION

We hereby declare that We have checked this thesis and, in our opinion, this thesis is adequate in terms of scope and quality for the award of the degree of Master of Science



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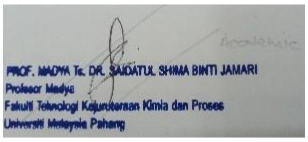

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ABSTRAK

Pengeluaran bahan berharga dari biojisim bersaiz nano menjadi fokus utama pembangunan industri untuk menghasilkan produk komposit berasaskan mesra alam, untuk pelbagai aplikasi. Kajian ini pada dasarnya, difokuskan pada penemuan kaedah mesra alam untuk penggunaan serat daun N36 *Ananas comosus* kepada bahan yang berpotensi. Sellulosa nanofibrilasi berjaya diasingkan dari serat daun nanas (NFC) dengan pengisaran bola dengan kehadiran alkohol isopropyl (IPA). Kesan alkohol isopropil dan masa pengisaran untuk menghasilkan fibril bersaiz nano dianalisis melalui pencirian sellulosa nanofibrilasi termasuk mikroskop pengimbasan pelepasan medan, X-ray pembelauan, termogravimetrik analisis, Potensi Zeta, Inframerah jelmaan Fourier, Analisis saiz zarah. Secara perbandingan, sampel NFC 1:4─15min memiliki ciri-ciri yang diingini dalam aspek morfologi, ukuran serat, hasil, peratus penghabluran, kestabilan terma, dan keseragaman pada penyebaran serat PALF. Suhu degradasi terma yang tinggi 323 ℃ dan indeks penghabluran 67%, menunjukkan potensi penguatan untuk meningkatkan kestabilan terma dan sifat mekanik komposit. Lebih-lebih lagi, ia menunjukkan morfologi permukaan yang sangat baik, kestabilan dengan penggumpalan fiber yang rendah, dan keseragaman fibril dengan lilitan serat berukuran 25.84 ± 8.30 nm, potensi zeta -32.31 ± 2.51 dan polidispersi indeks (PDI) 0.103. Oleh itu, penambahan IPA memberi impak yang besar terhadap penghasilan fibril dengan melemahkan ikatan hidrogen antara molekul, sehingga mengurangkan masa pengisaran NFC tanpa menyebabkan kerosakan melampau pada sifat lain. Sample 1:4─15min NFC ditambahkan dengan komposisi yang berbeza (1-5 wt.%) dalam matriks kitosan untuk meningkat dan memperbaiki biofilm kitosan yang asal. Pengaruh kandungan NFC dalam matriks kitosan dianalisis melalui SEM, FTIR, transmisi optik, XRD, TGA, Kelarutan, kebolehtelapan wap air (WVP), kebolehan basah, pencirian mekanikal, dan penguburan tanah. Secara adil, biofilm CS + 3% menyatakan ciri pilihan dalam masalah penyebaran, sifat penghalang air, peningkatan tingkah laku mekanikal 33.6%, penurunan 58.81% dan 70.63% penurunan indeks kembangan air, kelarutan, pengurangan biodegradasi menunjukkan interaksi yang lebih baik melalui pembentukan ikatan hidrogen dengan kumpulan amina dari kumpulan kitosan dan hidroksil NFC dan peningkatan tahap kristaliniti (12.1%) menghalang matriks merosot. Lebih-lebih lagi, ia menunjukkan sifat penghalang wap mekanikal dan wap air dengan kekuatan tegangan 83.6 MPa dan WVP 6.40×10^{-9} $gm^{-1}h^{-1}Pa^{-1}$.

ABSTRACT

This study principally focused on discovering a green method for the utilization of N36 Pineapple leaves fibre (PALF) to highly potential material by chemo-mechanical method and reinforcement of NFC in chitosan matrix. Mechanical methods are simple and do not require chemical reagents; this type of method is outstanding due to their high production capacity and low cost. However, some disadvantages include the possibility of contamination and amorphization, the formation of irregular shapes with poor homogeneity on nanofibrillation, and high energy consumption with extended treatment time. As a preliminary study, the cellulose was isolated from PALF using steam-alkaline coupled treatment (SAC). In that order, the concentration of sodium hydroxide (NaOH) (0.5 wt.%, 1.0 wt.%) and retention of steam treatment (30 min, 60 min) were varied for pulping and subsequently bleached with sodium chlorite ($NaClO_2$). The PALF and treated fibre were characterized using Scanning Electron Microscopy, Thermal Gravimetric Analysis (TGA), Fourier Transform Infrared Spectroscopy (FTIR) and Moisture analysis to study surface morphology, thermal stability, functional group and hydrophobicity respectively. Based on results, S60 A1 with 1 wt.% NaOH and 60 min steam treatment with 3 wt.% of $NaClO_2$ exhibit excellent thermal stability and surface morphology, where the maximum degradation occurs at 333.50 ℃ which is 9% improvement compared to untreated PALF and loosen structure of fibre bundle with reduced diameter (4.720 mm). This cellulose was utilized for the second objective to produce Nanofibrillated cellulose (NFC). NFC was successfully defibrillated by ball milling with the presence of isopropyl alcohol. The effect of isopropyl alcohol (ratio of cellulose to IPA) and the milling time (15 and 30 min) on nano fibrillation was analyzed through characterization of NFCs including Field Emission Scanning electron microscopy (FESEM), X-ray diffraction (XRD), TGA, Zeta-potential (ZP), FTIR and Particle size analysis (PSA). Comparatively, NFC 1:4─15min which milled for 1:4 cellulose to IPA ratio for 15 min expresses desirable features in the concern of morphology, fibre size, yield, crystallinity, thermal stability, and homogeneity on the disintegration of PALF fibre. High thermal degradation temperature of 323 ℃ and crystallinity index of 67%. Moreover, it exhibits high yield (92.45%), excellent surface morphology, stability with low self-agglomeration, and uniformity in defibrillation with fibre diameter of 25.84±8.30 nm, zeta-potential of -32.31±2.51 and PDI of 0.103. Hence, addition of IPA gives significant impact on defibrillation by disrupt the intermolecular hydrogen bond, so that less milling time is convenient on production of NFC without causes severe damage on other properties. Sample of 1:4─15min NFC reinforced with different composition (1-5 wt.%) in chitosan (CS) matrix to refine and improve the neat chitosan biofilm. The effect of NFC content in chitosan matrix were analyzed through SEM, FTIR, optical transmittance, XRD, TGA, Solubility, Water vapor permeability (WVP), wettability, mechanical characterization, and soil burial. Fairly, the CS+3% biofilm express preferred feature in the concern of dispersion, water barrier properties, mechanical behavior enhancement. 33.6%, 58.81% and 70.63% decrement of swelling index, solubility, biodegradability indicates the better interaction through formation of hydrogen bond with amine groups from chitosan and hydroxyl groups of NFCs and the elevation of crystallinity level (11%) prevent the matrix to degrade. Moreover, it exhibits well refined mechanical and water vapor barrier properties with tensile strength of 83.6 MPa and WVP of $6.40 \times 10^{-9} gm^{-1}h^{-1}Pa^{-1}$. As conclusion, reinforcement of 3 wt.% of 1:4─15min NFC with CS improved the mechanical and barrier properties remarkably.

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