FABRICATION AND CHARACTERIZATION OF MICROCRYSTALLINE CELLULOSE FROM OIL PALM EMPTY FRUIT BUNCH FIBER REINFORCED POLYLACTIC ACID COMPOSITES

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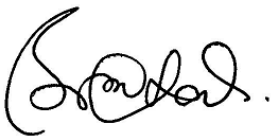
I hereby declare that I have checked this thesis and in my opinion, this thesis is adequate in terms of scope and quality for the award of the degree of Doctor of Philosophy (Chemical Engineering).



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Thesis submitted in fulfillment of the requirements
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I would like to dedicate my thesis
to my beloved parents and siblings,
with your love, sacrifices, supports and prayers,
make me able to get such success and honor.

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ABSTRAK

Di dalam kajian ini, komposit dihasilkan daripada mikrokristal selulosa (MCC) yang diekstrak daripada gentian tandan kosong kelapa sawit (EFB) bersama asid poli laktik (PLA). Proses pengasingan MCC daripada EFB ini melalui proses mekanik kimia, dimana ia melibatkan proses hidrolisis alkali dan proses ultrasonik. Tiga langkah utama telah digunakan untuk mengestrak MCC, iaitu penyingkiran lignin, penyingkiran hemiselulosa dan akhirnya pengekstrakan MCC. MCC yang terhasil kemudiannya telah dianalisis menggunakan spektroskopi inframerah jelmaan Fourier (FTIR), mikroskopi imbasan electron (SEM), analisis termogravimetri (TGA) dan teknik pembelauan sinar-X (XRD). Indeks kristal di dalam MCC didapati sebanyak 81% iaitu lebih tinggi daripada gentian EFB dan selulosa. Suhu degradasi, dan kandungan baki menunjukkan MCC yang diekstraks menggunakan kaedah ini mempunyai kestabilan terma yang baik. Pengoptimuman dilakukan dengan menggunakan kaedah gerak balas permukaan (RSM) bagi menentukan keadaan perawatan optimum kepekatan alkali, suhu ultrasonik serta masa pendedahan ultrasonik bagi mencapai indeks kristal MCC yang tertinggi. Penghasilan komposit MCC bersama asid poli laktik (PLA) dilakukan melalui kaedah larutan serta acuan dengan kandungan MCC yang berbeza (1, 3, 5 & 7 wt %) sebagai pemboleh ubah. Ujian mekanikal menunjukkan bahawa 5% kandungan MCC di dalam PLA mempunyai sifat mekanikal tertinggi dan peratusan ini dipilih sebagai kandungan MCC yag optimum di dalam komposit MCC/PLA. Analisis morfologi permukaan komposit MCC/PLA oleh SEM menunjukkan hubungan lekatan MCC dan PLA yang baik. Bagi meningkatkan sifat mekanikal PLA dan memberikan interaksi permukaan yang lebih baik antara gentian dan matrik polimer, pengubahsuaian dilakukan ke atas gentian dan matrik polimer (iaitu MCC dan PLA) secara berasingan. Pengubahsuaian struktur melalui mekanisma pemanjangan rantaian dilakukan bagi meningkatkan sifat mekanikal PLA sementara rawatan permukaan gentian dilakukan bagi meningkatkan keserasian dan kelekatan gentian MCC. Bagi kajian pengubahsuaian PLA, Joncryl yang bertindak sebagai penyambung rantaian dimasukkan ke dalam PLA filem dengan kandungan yang berbeza (1, 3, dan 5 wt %) dan PLA yang telah diubahsuai (Mod-PLA) kemudian dicirikan. Kandungan Joncryl sebanyak 1% di dalam PLA mempamerkan nilai kekuatan tegangan (TS) dan modulus kekuatan (TM) yang tertinggi tetapi ia juga mengurangkan pemanjangan pada takat putus. Peratusan ini dipilih sebagai kandungan optimum Joncryl di dalam PLA bagi meneruskan kajian selanjutnya. Bagi pengubahsuaian MCC, permukaan MCC dirawat dengan menggunakan *polyhedral oligomeric silsesquioxane* (POSS) dengan kandungan yang berbeza (5, 10 dan 15 wt %). MCC yang telah diubahsuai (POSS-MCC) kemudian dicirikan melalui analisis struktur, interaksi kimia, morfologi serta analisis haba. Analisis morfologi permukaan MCC yang telah diubahsuai (POSS-MCC) mendedahkan bahawa terdapat zarah POSS pada permukaannya. Berdasarkan pencirian lain, POSS-MCC menunjukkan peningkatan sifat hidrofobik mikrokristal selulosa (MCC) dan mempamerkan sifat terma yang lebih rendah berbanding MCC. Penghasilan komposit yang terakhir dilakukan dengan menggabungkan MCC yang telah diubahsuai (POSS-MCC) bersama dengan PLA yang telah diubahsuai (Mod-MCC/Mod-PLA), dan sifatnya dianalisis. Sebagai kesimpulan daripada perbandingan kajian semua komposit, komposit Mod-MCC/Mod-PLA telah menunjukkan peningkatan sifat terma dan sifat mekanik yang mempunyai nilai TS, TM dan pemanjangan pada takat putus (EB) yang tertinggi berbanding dengan komposit lain yang telah dihasilkan sepanjang kajian dijalankan.

ABSTRACT

In this research, composites were prepared from microcrystalline cellulose (MCC) extracted from oil palm empty fruit bunch (EFB) fibre and poly (lactic) acid (PLA) polymer. Mechanochemical process which involves simultaneous alkali treatment assisted with ultrasonic process was implemented to isolate MCC from raw oil palm EFB fibre. Three steps have been used to prepare the MCC, namely removal of lignin, removal of hemicellulose and finally production of MCC. The MCC produced was further characterized by fourier transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), thermogravimetric analysis (TGA) and also X-ray diffraction (XRD) measurement. The crystallinity index in MCC is found to be 81% which is higher than that of EFB fiber and cellulose. The degradation temperature, and the residue content revealed good thermal stability of MCC extracted through this mechanochemical technique. The optimization was done by using response surface methodology (RSM) in order to determine the optimum treatment condition of alkali concentration, ultrasonic temperature and ultrasonic exposure time towards the highest crystallinity of MCC produced. Composites were fabricated from MCC extracted reinforced poly lactic acid (PLA) polymer with different loading of MCC content (1, 3, 5 & 7 wt %) via a solution and casting method. Mechanical testing revealed that 5 wt % of MCC content in PLA exhibited the highest mechanical properties and this percentage was selected as the optimum fiber content for MCC/PLA based composite. Morphological analysis of MCC/PLA composites fractured surface by SEM revealed good adhesion of MCC with PLA. In order to improve mechanical properties of PLA and give better surface interaction between fibre and matrix, separate modifications were done to both fibre and matrix (i.e. MCC and PLA) respectively. Structural modification through chain extension and branching mechanism was done to improve the mechanical properties of PLA polymer while fiber surface treatment was done to improve the compatibility and the adhesion of MCC fibre. In PLA modification, Joncryl acts as a chain extender was incorporated into the PLA film with different loading (1, 3, and 5 wt%) and the characteristics of the modified PLA (Mod-PLA) films were characterized. 1 wt% of Joncryl content in PLA exhibited highest tensile strength (TS) and tensile modulus (TM) values but also led reduction in elongation at break. This percentage was selected as the optimum Joncryl content in modified PLA-based for further composite development study. In case of MCC modification, the MCC surface was incorporated by polyhedral oligomeric silsesquioxane (POSS) material with different percentage of POSS (5, 10 and 15 wt%). The modified MCC (POSS-MCC) were then characterized through structural, chemical interaction, morphological as well as thermal analysis. Morphological analysis of the modified MCC surface by FESEM revealed that POSS particles has been successfully grafted on it. Based on other characterization, it was found that the POSS-MCC improve the hydrophobicity of microcrystalline cellulose (MCC) but exhibit lower thermal properties in comparison with MCC. Fabrication of final composite was done by reinforced the modified MCC (POSS-MCC) into modified PLA matrix (Mod-MCC/Mod-PLA) and was further characterized. As conclusion from the comparative study of all the composites fabricated, Mod-MCC/Mod-PLA composite film exhibits improvement in both thermal properties and mechanical properties with highest TS, TM and elongation at break (EB) in comparison with other composites prepared along with the study development.

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β	Heating Rate (°C/min)
T_g	Glass Transition Temperature (°C)
T_m	Melting Temperature (°C)
T_c	Crystallization Temperature (°C)
F_{max}	Maximum peak load (N)
σt	Tensile Strength (MPa)
T_{max}	Temperature of maximum reaction rate (°C)
Z	Frequency factor
R	Gas constant
I_{DSC}	Degree of crystallinity (%)
L_o	Initial measured length (m)
L	Length at breaking point (m)
d	Interplaner spacing
θ	Bragg angle
λ	X-ray wavelength
β	Full width at half maximum (FWHM)
K	Scherrer‘s constant

LIST OF ABBREVIATIONS

ASTM	American Standard Testing Method
DSC	Differential scanning calorimetry
DTG	Differential thermo gravimetry
EB	Elongation at break
EFB	Empty fruit bunch
FTIR	Fourier transforms infrared spectroscopy
FWHM	Full width at half maximum
GPa	Giga Pascals
JC	Joncryl
LODP	Level-off degree of polymerization
MCC	Microcrystalline cellulose
PLA	Poly lactic acid
POSS	Polyhedral oligomeric silsequioxane
SEM	Scanning electron microscopy
TAPPI	Technical Association of the Pulp and Paper Industry
TGA	Thermo gravimetric analysis
TM	Tensile modulus
TS	Tensile strength
UTS	Universal tensile strength
XRD	X-ray diffraction

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