SYNTHESIS AND CHARACTERIZATION OF NANOPARTICLE INCORPORATED POLYMER COMPOSITE MATERIALS

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ABSTRACT

This thesis is composed with the aim of the synthesis of nanoparticle impregnated oil palm fibre reinforced composites. This aim is initiated with the synthesis of copper nanoparticles (CuNPs) at ambient condition in aqueous medium using copper chloride precursor, sodium borohydride (SBH), ascorbic acid and polyvinyl alcohol (PVA) of two different molecular weights $(M_w s)$. The formation of characterization of the nano copper sols are studied. The physical appearance of prepared sol has been found to be stable for a couple of weeks when kept in ambient atmospheric condition, as confirmed by ultraviolet-visible absorption spectroscopy. Transmission electron microscopy exhibits spherical morphology of CuNPs with an average size of 3.5±1.1 nm.The effect of SBH amount and PVA (wt.% and M_ws) on the size of CuNPs are also delineated. As a potential application of CuNPs, theywere impregnatedon/into oil palm empty fruit bunch (EFB) fibres via cationization process. Conventionally, same EFBfibres were also treated with alkali solutions. Treated fibres were characterized by Fourier transformed infrared (FTIR) spectroscopy, X-ray diffraction (XRD) study, field emission scanning electron microscopy (FESEM), energy dispersive X-ray (EDX) study and thermogravimetric analysis (TGA). Numerous physical and mechanical properties of different single fibres were measured and especially the mechanical properties were analyzed by the Griffith model and Weibull statistical distribution. The weak link scaling of Weibull analysis has provided valuable information to scale the strength of one EFB fibre to predict the strength of other one. A significant increase in mechanical property of CuNPs modified fibres with respect to the control ones has been observed. CuNPs impregnated EFB fibres show increases of tensile strength (~34%) and antifungal activity (24%) with respect to control fibres. These findings strongly suggest that CuNPs can be used as an effective agent in natural fibres to improve their mechanical property and durability. Therefore, different doses CuNPs impregnated strong and durable fibres were used to develop theCuNPs impregnated fibre reinforced unsaturated polyester resin nanocomposite. The composite behaviour, mechanical property and biodegradability have been investigated systematically by using the commonly used techniques such as FTIR, XRD, FESEM, TGA, differential scanning calorimetry (DSC), universal tensile testing tester, etc. The developed nanocomposites mechanical properties and biodegradability were optimized by response surface methodology (RSM) also. The biodegradability of the developed composites was inversely proportional to the CuNPs loadings. The change of weight gain (due to moisture absorption) for all samples is highly related with a typical Fickian diffusion behaviour. Moreover, higher mechanical (tensile and flexural) performances were obtained for CuNPs treated fibre (NF) composites compared to those of untreated fibre based composites. Among all of the fibre reinforced composites tested, NF reinforced unsaturated polyester resin composites (30% fibre) registered for the highest mechanical properties. It was found that standard micromechanical models (Rule of Mixture, Inverse Rule of Mixture and Halpin-Tsai Model) which are commonly used to predict the strength of traditional synthetic fibre composites can be applied to such natural fibre systems with mixed success. Throughout the work, three schemes were proposed. The obtained properties of the developed nanocomposites indicate that they can be considered for both indoor-outdoorapplications.

ABSTRAK

Tesis ini di jalankan bertujuan untuk memperbangunkan komposit yang di perkukuhkan dengan gentian minyak sawit bersaiz nano. Matlamat ini dimulakan dengan mengsintesis nano-zarah kuprum (CuNPs) pada keadaan ambien di dalam medium akueus menggunakan kuprum pelopor klorida, natrium borohydride (SBH), asid askorbik dan alkohol polyvinyl (PVA) pada dua berat molekul yang berbeza (MWS). Pembentukan dan pencirian sols kuprum nano telah dikaji. Penampilan fizikal sol didapati stabil selama beberapa minggu apabila disimpan dalam keadaan atmosfera ambien setelah disahkan spektroskopi penyerapan ultraviolet. Transmission electron microscopy oleh mempamerkan morfologi sfera untuk CuNPs dengan saiz purata bernilai 3.5 ± 1.1 nm. Kesan daripada jumlah SBH dan PVA (wt. % dan MWS) pada saiz CuNPs juga ditandakan. Sebagai satu aplikasi CuNPs yang berpotensi, sampel tersebut telah di terapkan pada minyak gentian tandan kelapa sawit (EFB) melalui proses cationisasi. Secara formalnya, gentian EFB tersebut dirawat dengan rawatan alkali. Gentian yang telah dirawat dianaliskan oleh Fourier transformed infrared (FTIR) spectroscopy, X-ray diffraction (XRD) study, field emission scanning electron microscopy (FESEM), energy dispersive X-ray (EDX) study dan thermogravimetric analysis (TGA). Sifat-sifat fizikal dan mekanikal gentian tunggal yang berbeza telah diukur terutamanya menggunakan sifat-sifat mekanikal yang dianalisis oleh model Griffith dan Weibull pengagihan statistik. Pautan bersisik lemah analisis Weibull telah memberikan maklumat yang penting kepada skala kekuatan satu gentian EFB untuk meramalkan kekuatan gential EFB yang lain. Peningkatan yang ketara dalam sifat mekanikal gentian CuNPs diubahsuai mengikut sample kawalan. Penerapan CuNPs kepada gentian EFB menunjukkan peningkatan kekuatan tegangan sebanyak (~ 34%) dan aktiviti antikulat sebanyak (24%) berkenaan dengan mengawal gentian. Penemuan ini mencadangkan bahawa CuNPs boleh digunakan sebagai agen berkesan dalam gentian asli untuk meningkatkan sifat mekanik dan ketahanan. Oleh itu, dos CuNPs yang berlainan menghasilkan gentian kuat dan tahan lama telah digunakan untuk menghasilkan damar nanokomposit poliester tidak tepu yang diperkukuhkan dengan gentian yang diterap dengan CuNPs. Sifat-sifat komposit seperti, sifat mekanikal dan biodegradasi telah disiasat secara sistematik dengan menggunakan teknik-teknik yang biasa digunakan seperti FTIR, XRD, FESEM, TGA, kalorimetri imbasan pembeza (DSC), mesin ujian universal, dan lain-lain lagi. Sifat mekanik dan biodegradasi nanokomposit adalah dioptimumkan dengan mengunakan response surface methodology (RSM). Sifat biodegradasi komposit adalah berkadar songsang dengan bebanan CuNPs. Perubahan jisim (disebabkan oleh penyerapan kelembapan) bagi semua sampel berkait rapat dengan tingkah laku penyebaran Fickian . Selain itu , prestasi mekanikal yang lebih tinggi (tegangan dan lenturan) telah diperolehi untuk serat yang dirawat oleh CuNPs (NF) komposit berbanding dengan komposit berasaskan gentian yang tidak dirawat. Antara semua komposit yang diperkukuhjakn oleh gentian diuji, komposit resin poliester yang diperkukuhkan oleh NF (30% serat) dikenalpasti memperoleh sifat-sifat mekanik tertinggi. Ia telah didapati bahawa piawaian model mikro mekenikal (Rule of Mixture, Inverse Rule of Mixture and Halpin-Tsai Model) yang biasa digunakan untuk meramalkan kekuatan komposit gentian sintetik tradisional boleh digunakan untuk sistem gentian semula jadi dengan jayanya. Sepanjang kerja ini, tiga skim telah dicadangkan. Sifat-sifat yang diperolehi daripada kemajuan nano-komposit menunjukkan bahawa ia boleh dipertimbangkan untuk kedua-dua aplikasi dalaman-luaran.

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LIST OF ABBREVIATIONS

AAS	Atomic absorption spectroscopy
AATCC	American Association of Textile Chemists and Colourists
AB	After burial
AF	Alkali treated EFB fibre
AFM	Atomic force microscopy
AMA	Allyl methacrylate
ANOVA	Analysis of variance
ASTM	American Standard Testing Method
BB	Before burial
BD	Biodegradability
С	Degree of cationization
CF	Cationized EFB fibre
CF-C	CF reinforced composite
CHPTAC	3-Chloro-2-hydroxypropyl)trimethylammonium chloride
CRP	Chemical reduction process
CRP	Chemical reduction process
CTAB	Cetyltrimethylammonium bromide
Cu	Copper
CuNPs	Copper nanoparticles

D	Coefficient of diffusion,
D _{hkl}	Average size of the cellulose crystallites
d	Diameter
DEG	Di-ethylene glycol
DI	Deionized water
DMSO	Dimethyl sulfoxide
DSC	Differential scanning calorimetry
DTG	Differential thermo gravimetry
E	Moment of inertia of the composite beam
E. coli	Escherichia coli
EB	Elongation at break
EDX	Energy dispersive X-ray
E _F	Elastic modulus of fibre
EFB	Empty fruit bunch
E _M	Elastic modulus of matrix
F	Faraday's constant
FESEM	Field emission scanning electron microscopy
FTIR	Fourier transformed infrared
FWHM	Full width at half-maximum
G	Modulus of rigidity

- GDI Gasoline Direct Injection
- GHz Gigahertz
- GPa Giga Pascals
- HDEHP Bis(ethylhexyl)hydrogen phosphate
- HMP Hexameta phosphate
- IROM Inverse rule of mixing
- K Equilibrium constant
- kHz kilohertz
- 1 Span of the composite beam
- L Length
- MC Moisture content
- MDF Medium density fibre
- MHz Megahertz
- M_m Moisture content at saturation point
- MNP Metal nanoparticles
- MOP Molded oil palm
- MPa Mega Pascal
- MRSA Methicillin-resistant Staphylococcus aureus
- M_w Molecular weight
- m_w Weibull modulus

- NF Nanosol treated EFB fibre
- NF-C NF reinforced composite
- NFRT Natural fibre reinforced thermoplastic
- nm Nanometer
- NP Nanoparticle
- NPs Nanoparticles
- NR Natural rubber
- OPA Oil palm ash
- OPF Oil palm fibre
- OPFr Oil Palm Fronds
- OPT Oil Palm Trunk
- P Load within the elastic range
- PAA Na Polyacrylic acid sodium salt
- PCS Photon correlationspectroscopy
- PEG Polyethylene glycol
- PEI Polyethylene imine
- PF Phenol formaldehyde
- P_f Probability of failure
- PLA Poly(lactic acid)
- PP Polypropylene
- pp Particles

- ppm Parts per million
- PU Polyurethane
- PVA Polyvinyl alcohol
- PVC Polyvinyl chloride
- PVP Polyvinylpyrrolidone
- PWD Pulsed wire discharge
- PZC Zero point charge
- R Gas constant (J/mol K)
- RC Regenerated Cellulose
- ROM Rule of mixing
- RSM Response surface methodology
- S. aures Staphylococcus aureus
- SBH Sodium borohydride
- SDS Sodium dodecyl sulfate
- SEM Scanning electron microscope
- SPR Surface plasmon resonance
- SS Sodium succinate
- T Temperature (in Kelvin)
- t Treatment time
- T₀ Onset temperature
- Tc Crystallization Temperature (⁰C)

TCF	Total chlorine-free
T _d	Degradation temperature
TEM	Transmission electron microscopy
TEPA	Tetraethylenepentamine
Tg	Glass Transition Temperature (⁰ C)
TGA	Thermo gravimetric analysis
T _m	Melting endotherm
Tm	Melting Temperature (⁰ C)
TM	Tensile modulus
TOAB	Tetraoctylammonium bromide
-	-
TS	Tensile strength
TX-100	Triton X-100
UF	Untreated EFB Fibre
UF-C	UF reinforced composite
USA	United States of America
UV-vis	Ultra-violet visible
V	Volt
$V_{\rm F}$	Volume fraction of fibre
V_{M}	Volume fraction of matrix
VR	Unsaturated polyester resin
W _f	Weight of fibre
WL	Weight loss

W_{m}	Weight of matrix
XRD	X-ray diffraction
Y	Young's modulus
β	FWHM (in radians)
ΔΕ	Redox potential
ΔH	Heat of fusion (J/g)
θ	Diffraction angle
λ	Wavelength of X-ray (1.541 Å)
$ ho_{f}$	Density of fibre
$ ho_m$	Density of matrix
$\sigma_{\scriptscriptstyle ave}$	Average tensile strength
∂	Total deflection
ΔG^0	Gibb's free energy
μg	Microgram
μm	Micrometer
20	Scattering angle