DEVELOPMENT OF SUGAR PALM FIBRE REINFORCED THERMOPLASTIC POLYURETHANE COMPOSITES

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The Author

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LIST OF SYMBOLS

OH	Hydroxyl
$KMnO_4$	Potassium Permanganate
NaOH	Sodium Hydroxide
T_g	Glass Temperature
N_2	Nitrogen
O_2	Oxygen
°C	Degree Centigrade
MPa	Mega Pascal
Hz	Hertz
min	Minutes
T_m	Melting Temperature
C-O-C	Carbon-Oxygen-Carbon
µm	Micron
mm	Miley Meters
rpm	Revolution Per Minutes
GPa	Giga Pascal
Cl	Chlorine
PO_4	Phosphate
SO_4	Sulphide
SO_2	Sulphur Dioxide
Pb	Lead
Al	Aluminium
Ca	Calcium
Cu	Copper
Fe	Iron

Mn	Manganese
Hg	Silver
Ni	Nickel
K	Potassium
Zn	Zinc
C_3H_6O	Acetone
θ	The Angle of Degree for the Reflection Ray
C %	Crystallinity Percent
ΔH	Energy of Fusion
H^+	Hydrogen Bond

LIST OF ABBREVIATIONS

SPF	Sugar Palm Fibre
TPU	Thermoplastic Polyurethane
TGA	Thermogravimetric Analysis
DSC	Differential Scanning Calorimetry
SEM	Scanning Electron Microscope
FTIR	Fourier Transform Infrared Spectroscopy
EDX	Energy Dispersive X-ray
XRD	X-ray diffraction
MMCs	Metal Matrix Composites
CMCs	Ceramic Matrix Composites
PMCs	Polymer Matrix Composite
PP	Polypropylene
PE	Polyethylene
PA	Poly Amide
PPS	Poly Phenylene Sulphide
PEEK	Poly Ether Ketone
IC	Integrated-Circuits
PUR-foam	Polyurethanes Building Foam
ATR	Adiabatic Temperature Rises
PU	Polyurethane Thermoset
WPC	Wood Plastic Composite
TS	Tensile Strength
YM	Young’s Modulus
h	Hour
KOH	Potassium Hydroxide

SPB	Sugar Palm Bunch
SPT	Sugar Palm Trunk
HIPS	High Impact Polystyrene
GMT	Glass-Mat-Thermoplastic
FRP	Fibre Reinforced Polymer
NMT	Natural Fibre-Mat-Reinforced Thermoplastic
GMT	Glass Fibre Based Materials
PVC	Poly (Vinyl Chloride)
SEBS	Styrene-Ethylene-Butylene-Styrene
TMPTA	Trimethylolpropane tri-acrylate
TPGDA	Tripropylene Glycol Diacrylate
RTM	Resin Transfer Moulding
CM	Compression Moulding
MAPP	Maleated Polypropylene (coupling agent)
SLS	Sodium Laulryl Sulfate
PHBV	(P/ poly H / hydroxyl B/ butyrate V/ valerate)
PLA	Poly (Lactic) Acid
HDPE	High Density Polyethylene
TMA	Thermal Mechanical Analysis
TG	Thermogravimetric
PBS	Polybutylene Succinate
DTG	Derivative Thermogravimetric
DMTA	Dynamic Mechanical Thermal Analysis
RHF	Rice Husk Flour
WF	Wood Flour
PS	Polystyrene
AEX	Antioxidant Effectiveness

DMA	Dynamic mechanical Analysis
RH	Re-Heat
TPS	Thermoplastic System
LDPE	Low Density Polyethylene
CTDIC	Cardanol Derivative of Toluene Diisocyanate
RF	Radio Frequency
LF	Low Frequency
KF	Kenaf Fibre
LNR	Liquid Natural Rubber
O-HBDS	O-Hydroxybenzene Diazonium Salt
SCFs	Sansevieria Cylindrical Fibres
SCFP	Sansevieria Cylindrical Fibres Polyester
DC	Different Conditions
ASTM	American Society for Testing and Materials
LTC	Low Temperature Compounding
CCD	Central Composites Design
ANOVA	Analysis of Variance
DT	Decomposition Temperature
MSD	Mean Square Deviation
TPU/SPF	Composites materials come from combining the sugar palm fibre with thermoplastic polyurethane
10 wt. % TPU/SPF composites	10 % of sugar palm fibre combined with 90 % of thermoplastic polyurethane composites
20 wt. % TPU/SPF composites	20 % of sugar palm fibre combined with 80 % of thermoplastic polyurethane composites
30 wt. % TPU/SPF composites	30 % of sugar palm fibre combined with 70 % of thermoplastic polyurethane composites (untreated fibre)

170 °C	The transition temperature in the extruder and it is representing the set temperature such as; 160-170-200 °C in the zones of the extruder
180 °C	The transition temperature in the extruder and it is representing the set temperature such as; 170-180-200 °C in the zones of the extruder
190 °C	The transition temperature in the extruder and it is representing the set temperature such as; 180-190-200 °C in the zones of the extruder
160 µm	Sugar palm fibre size 160 µm reinforced thermoplastic polyurethane composites with fibre loading 10 wt. %
250 µm	Sugar palm fibre size 250 µm reinforced thermoplastic polyurethane composites with fibre loading 10 wt. %
425 µm	Sugar palm fibre size 425 µm reinforced thermoplastic polyurethane composites with fibre loading 10 wt. %
0 % NaOH TPU/SPF	Untreated 30 wt. % sugar palm fibre with size 250 µm reinforced thermoplastic polyurethane composites
2 % NaOH TPU/SPF	2 % concentration of NaOH dissolve in distilled water treated of 30 wt. % sugar palm fibre with size 250 µm reinforced thermoplastic polyurethane composites
4 % NaOH TPU/SPF	4 % concentration of NaOH dissolve in distilled water treated of 30 wt. % sugar palm fibre with size 250 µm reinforced thermoplastic polyurethane composites
6 % NaOH TPU/SPF	6 % concentration of NaOH dissolve in distilled water treated of 30 wt. % sugar palm fibre with size 250 µm reinforced thermoplastic polyurethane composites
0.033 % $KMnO_4$ TPU/SPF	0.033 % concentration of $KMnO_4$ solvent in acetone treated of 30 wt. % of sugar palm fibre with size 250 µm reinforced thermoplastic polyurethane composites
0.066 % $KMnO_4$ TPU/SPF	0.066 % concentration of $KMnO_4$ solvent in acetone treated of 30 wt. % of sugar palm fibre with size 250 µm reinforced thermoplastic polyurethane composites
0.125 % $KMnO_4$ TPU/SPF	0.125 % concentration of $KMnO_4$ solvent in acetone treated of 30 wt. % of sugar palm fibre with size 250 µm reinforced thermoplastic polyurethane composites
70 °C Microwave TPU/SPF	Setting temperature of the microwave was 70 °C to treat 30 wt. % of sugar palm fibre with size 250 µm reinforced thermoplastic polyurethane composites

80 °C Microwave TPU/SPF	Setting temperature of the microwave was 80 °C to treat 30 wt. % of sugar palm fibre with size 250 μm reinforced thermoplastic polyurethane composites
90 °C Microwave TPU/SPF	Setting temperature of the microwave was 90 °C to treat 30 wt. % of sugar palm fibre with size 250 μm reinforced thermoplastic polyurethane composites

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ABSTRAK

Gentian buah Kabung (SPF) mempunyai sifat tegangan yang baik.Ianya sangat sesuai sebagai agen tetulang di dalam bahan komposit. Termoplastik poliuretana (TPU) adalah polimer yang fleksibel dan mahal. TPU mempunyai masa pemprosesan yang pendek, rendah tenaga dan boleh di kitar semula. Ia mempunyai sifat-sifat mekanikal dan rintangan kekasaran terbaik. Secara berlawanan, gentian semulajadi adalah bersifat hidrofilik, sedangkan kebanyakan polimer-polimer adalah bersifat hidrofobik. Sangat sedikit penyeldikan mengenai termoplastik poliuretana bertetulangkan gentian semulajadi yang telah di kaji.

Objektif kajian ini adalah untuk membangunkan satu gentian semulajadi SPF baharu sebagai penggunaan yang nobel keatas struktur komposit termoplastik poliuretana. Penyentisis gentian SPF dan TPU dilakukan dengan menggunakan kaedah penyemperitan dan kemudian bahan yang dihasilkan telah dimampatkan dalam acuan menggunakan pengacuan mampatan pada suhu 190 °C. Kerja eksperimen dibahagikan kepada tiga bahagian. Pertama, penyediaan struktur komposit TPU/SPF baharu tanpa sebarang perubahan kemudian dioptimumkan keadaan operasi seperti (halaju putaran, suhu penyemperitan, saiz gentian, dan kandungan gentian) pada sifat-sifat akhir mekanikal dan haba. Kedua, percamtuman baharu dengan menggunakan tiga kaedah rawatan lanjutan kimia NaOH, kimia $KMnO_4$ dan gelombang mikro fizikal. Diakhir proses, kajian terhadap kesan ke atas sifat kekuatan tegangan, juga dikaji kesan kaedah rawatan ke atas tingkah laku percamtuman akhir dengan menggunakan pelbagai pencirian seperti, Analisis Termogravimetri (TGA), Perbezaan Pengimbasan Kalorimeter (DSC) Pengimbasan Mikroskop Elektron (SEM) , Tenaga Serakan Sinar-X (EDX), Spektrokopi Fourier dijelmakan inframerah (FTIR) dan Pembelauan Sinar-X (XRD).

Hasil keputusan menunjukkan bahawa keadaan operasi yang optimum diperolehi secara eksperimen pada 40 rpm minimum halaju putaran, suhu 190 °C, 250 saiz mikron gentian, dan 10 % kandungan gentian. Nilai kekuatan tegangan, modulus dan terikan pada keadaan ini masing-masing adalah 14.01 MPa, 23.34 MPa, dan 253.186 %. Selain itu kekuatan lenturan dan nilai modulusnya adalah 11.162 MPa dan 145.252 MPa. Di samping itu, kekuatan hentaman adalah 100.2 kJ/m^2. Kekuatan tegangan yang lebih tinggi diperolehi pada kaedah rawatan NaOH dengan 6% kepekatan berkesan di ikuti oleh kekuatan hentaman yang lebih tinggi dan nilai terikan masing-masing sebanyak 5.488 MPa, 103.4 kJ/m^2 dan 48.012 %. Juga, modulus dan tegangan lenturan dengan nilai 6 % NaOH TPU/SPF masing-masing ialah 293.936 MPa, 30 MPa dan 11.1632 MPa. Walaubagaimanapun, kekuatan lenturan tertinggi direkodkan pada nilai komposit TPU/SPF yang dirawat 2 % NaOH pada 12.606 MPa, manakala kekuatan hentaman tertinggi dalam kaedah ini adalah 103.4 kJ/m^2 dengan 6 % komposit NaOH TPU/SPF. Kestabilan haba tertinggi adalah 17.56 % dan penguraian suhu pada 372 °C dicapai dengan 4 % komposit NaOH TPU/SPF selepas membandingkan mereka dengan semua jenis rawatan untuk komposit TPU/SPF. Pengaktifan itu kemudian ditambahbaik dengan menggunakan larutan kimia kalium permanganat, untuk meningkatkan kekuatan tegangan, modulus, dan nilai terikan sebanyak 8.986 MPa, 876.436 MPa, dan 1.442 %

masing-masing, pada 0.125 $KMnO_4$ dan 6 % NaOH pra-rawatan TPU/SPF. Pada kesan yang sama terdapat pengurangan dalam kekuatan lenturan, modulus dan nilai kekuatan hentaman sebanyak 4.988 MPa, 180.744 MPa dan 53.9914 kJ/m^2 masing-masing, untuk kaedah rawatan $KMnO_4$ pada komposit TPU/SPF di akibatkan oleh degradasi gentian. Untuk analisis TGA SPF dirawat dengan 0.033 $KMnO_4$ mencatatkan kestabilan haba yang lebih baik sebanyak 15.23 % dan suhu penguraian 374 °C apabila dibandingkan dengan 6 % komposit NaOH TPU/SPF yang dirawat. Di samping itu, kaedah rawatan gelombang fizikal meningkatkan kekuatan tegangan, lenturan, kekuatan dan modulus tegangan kepada 18.42 MPa, 13.66 MPa dan 1307.562 MPa masing-masing. Walaubagaimanapun, kekuatan hentaman bagi kaedah gelombang mikro pada suhu 70 °C telah menurun kepada 71.527 kJ/m^2. Juga, komposit ini mencatatkan kestabilan haba tertinggi 13.47 % dan suhu penguraian 389 °C daripada analisis TGA jika dibandingkan dengan suhu operasi lain rawatan gelombang mikro. Di dalam analisis SEM menunjukkan terdapat kesan yang jelas terhadap struktur komposit TPU/SPF sebelum dan selepas kaedah rawatan pada gentian tersebut. Pada masa yang sama, EDX mengenalpasti bahan sisa yang disimpan di atas permukaan komposit selepas rawatan. Sementara itu, analisis DSC menunjukkan keserasian sifat mekanikal. Keputusan XRD dan FTIR menunjukkan pematuhan yang baik pada ciri-ciri mekanikal pada sampel masing-masing, di mana sampel XRD daripada gelombang mikro 70 °C yang gentian dirawat memberi hasil yang lebih baik bagi kesemua sampel yang diuji. Komposit TPU/SPF yang mempunyai SPF telah dirawat dengan rawatan gelombang 70 °C boleh dipertimbangkan sebagai bahan komposit yang sesuai kerana ia mempunyai sifat mekanikal dan haba yang baik. Komposit ini dijangka boleh digunakan untuk bahagian dalaman automotif.

ABSTRACT

Sugar palm fibre (SPF) has good tensile properties. It is suitable to be used as a reinforcing agent in composite materials. Thermoplastic polyurethane (TPU) is a flexible and expensive polymer, which has a shorter processing time and is lower in energy, tougher, and recyclable. It has excellent abrasion resistance and has good mechanical properties. In contrast, the natural fibre is hydrophilic in nature, whereas most polymers are hydrophobic in nature. To date, very little study about the thermoplastic polyurethane reinforced natural fibre has been investigated.

The objective of this study is to develop a new natural fibre (SPF) as a novel utilisation composite thermoplastic polyurethane structure. The synthesis of SPF fibre and TPU was carried out using an extruder and the resulting material was compressed in the mould using compression moulding at 190 °C. The experimental procedure was divided into three parts. First is the preparation of new TPU/SPF composite structures without any changes, then optimising the operation conditions (rotation velocity, the temperature of extrusion, fibre size, and fibre contents) on the final mechanical and the thermal properties. Next, the new assemblies were developed by applying three treatment methods on SPF: chemical NaOH, chemical $KMnO_4$, and physical microwave. Finally, the effects of these methods were studied using tensile strength property and the effect of the treatment methods testing over the behaviour of the final assemblies using characterizations such as thermogravimetric analysis (TGA), differential scanning calorimetry (DSC), scanning electron microscope (SEM), energy dispersive X-ray (EDX), Fourier transform infrared spectroscopy (FTIR) and X-ray diffraction (XRD).

The results indicated that optimum operating conditions obtained experimentally were at 40 rpm min rotation velocity, 190 °C temperature, 250 µm fibre size, and 10 wt. % fibre content. The values of tensile strength, modulus and strain at these conditions were 14.01 MPa, 23.34 MPa, and 253.186 % respectively. The flexural strength and modulus values were at 11.162 MPa and 145.252 MPa. In addition, the impact strength was at 100.2 kJ/m^2. Higher tensile strength was obtained for NaOH treatment method with the effective concentration of 6 %, followed by higher impact strength and tensile strain values as 5.488 MPa, 103.4 kJ/m^2, and 48.012 % respectively. Also, the flexural and tensile modulus and flexural strength for 6 % NaOH TPU/SPF values were 293.936 MPa, 30 MPa, and 11.1632 MPa respectively. However, the highest flexural strength was recorded at 2 % NaOH treated TPU/SPF composite value such as 12.606 MPa, while the biggest impact strength of this method was 103.4 kJ/m^2 for 6 % NaOH TPU/SPF composites. Higher thermal stability of 17.56 % and decomposition temperature of 372 °C were achieved from 4 % NaOH TPU/SPF treated fibre composite. The activation was then improved by applying a potassium permanganate chemical solution, with enhanced tensile strength, modulus, and strain values at 8.986 MPa, 876.436 MPa, and 1.442 % respectively, for 0.125 $KMnO_4$ and 6 % NaOH pre-treatment TPU/SPF. The same deteriorated effect was observed in the flexural strength, modulus and impact strength values at 4.988 MPa, 180.744 MPa and 53.9914 kJ/m^2 respectively, for the $KMnO_4$ treatment method on the TPU/SPF composites due to fibre degradation. TGA analysis SPF treated with 0.033 $KMnO_4$ recorded better thermal stability of 15.23 % and decomposition temperature of 374 °C when compared with 6 % NaOH treated fibre TPU/SPF composites. On the other hand, the physical microwave treatment method

enhanced further tensile, and flexural, strength and tensile modulus as 18.42 MPa, 13.66 MPa and 1307.562 MPa respectively. However, the impact strength for the microwave method at 70 °C deteriorated at 71.527 kJ/m^2. Also, these composites recorded highest thermal stability at 13.47 % and decomposition temperature of 389 °C by TGA analysis when compared with other operating temperatures of microwave treatment. SEM analysis showed that there was a clear effect on the structure of TPU/SPF composites before and after the treatment methods on the fibre. At the same time, the EDX identified residual trace materials deposited on the surface of the composites after the treatment. Meanwhile, the DSC analysis was compatible with the mechanical properties. XRD and FTIR results showed a good agreement with the mechanical characterization of the samples respectively, where the XRD sample of the 70 °C microwave treated fibre gave better results in the tested samples. The TPU/SPF composite which has SPF treated at 70 °C microwave treatment can be considered as a suitable composite material because of it has good mechanical and thermal properties. This is expected for the use of the interior parts in the automotive.

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