SYNTHESIS AND CHARACTERIZATION OF PAN NANOFIBER REINFORCED WITH FUNCTIONALIZED MIXTURE CNT/CNF BY ELECTROSPINNING TECHNIQUE

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ABSTRACT

In this study, functionalization of CNT and CNF was done using oxidative acid treatment with concentrated HCl and HNO_3 solution mixture at 3:1 ratio. Various process variables such as duration, mode of treatment, agitation and temperature are controlled, and consequently the functional groups growth on the surfaces of both CNT and CNF was determined extensively by the aid of Fourier Transform Infrared Spectroscopy (FTIR). The study proceeds by synthesizing the PAN/CNT-CNF polymer nanocomposite fiber using electrospinning technique. The study used PAN polymer solution containing 10 wt % mixture of CNT: CNF at various ratios of 8:2, 2:8, 6:4, and 4:6. Some critical criterions of operational parameters such as applied voltage (12 kV, 12.5 kV, 13 kV) and working distance (13 cm, 14 cm, 15 cm) were also being optimized beforehand. The available evidences supported that, the optimal operational parameter set was found to be at 15 cm, 13 kV set division as validated by series of scrutinized characterization assessments; where the set up successfully produced the thinnest average nanofiber diameter at 318 nm with the range of 288 to 340 nm. The graphitized nanocomposite fiber was then assessed and characterized comprehensively based on its morphological and elementary study using FESEM-EDX, thermal stability behaviour using TGA and DSC, crystallography assessment by XRD analysis, and conductivity study via EIS. Based on the overall assessment of various field of characterizations, overwhelming evidences has led to the confirmatory conclusion, corroborating PB15Z as the best electrospun PAN/CNT-CNF nanocomposite fiber with an exceptionally thin fiber diameter of 280 nm with 232 nm to 304 nm in range. It possessing the highest carbon content approximately 80 % in total and a good conductivity value recorded at 2.3555 x 10^{-4} S/m. The fiber also showed the most stable behaviour under intense heat exposure, withstanding the thermal introduction with gradual decrease of weight without any sudden and major fluctuation of weight.

ABSTRAK

SINTESIS DAN PENCIRIAN SERAT NANO PAN DIPERKASA DENGAN CNT/CNF YANG TERFUNGSIONALISASI MENGGUNAKAN KAEDAH PEMINTALAN ELEKTRO

Di dalam kajian ini, kaedah rawatan secara pengoksidaan menggunakan asid dilakukan dengan larutan pekat HCl dan HNO_3 pada nisbah 3:1. Pelbagai pembolehubah seperti masa, mod rawatan, agitasi dan suhu adalah terkawal di dalam kajian ini, dan kemudiannya kehadiran kumpulan berfungsi diatas permukaan kedua-dua bahan karbon nano dicirikan dengan bantuan spektroskopi penjelmaan Fourier infra-merah (FTIR). Langkah berikutnya diteruskan dengan menghasilkan serat polimer komposit nano berasaskan PAN/CNT-CNF menggunakan kaedah pemintalan elektro. Kajian ini menggunakan larutan polimer PAN yang mengandungi 10 % berat campuran CNT: CNF dalam pelbagai nisbah, iaitu 8:2, 2:8, 6:4, dan 4:6. Parameter operasi kritikal seperti voltan gunaan (12 kV, 12.5 kV, 13 kV) dan jarak kerja (13 sm, 14 sm, 15 sm) dioptimumkan terlebih dahulu. Hasil kajian menunjukkan bahawa set parameter operasi optimum adalah 15 sm dan 13 kV; dimana ia berjaya menghasilkan purata diameter serat nano terkecil pada 318 nm dengan julat 288 nm hingga 340 nm. Serat komposit nano yang digrafitkan kemudiannya dicirikan secara menyeluruh dari aspek analisis morfologi dan kajian elemen menggunakan FESEM-EDX, kestabilan terma menggunakan TGA dan DSC, penilaian kristalografi menerusi analisis XRD, dan keupayaan kekonduksian elektrik melalui EIS. Secara keseluruhannya, berdasarkan variasi penilaian dan pencirian, bukti-bukti substansial telah membawa kepada kesimpulan bahawa PB15Z merupakan serat polimer komposit nano yang terbaik, dimana ia mempunyai diameter serat yang kecil iaitu 280 nm dengan julat pada 232 nm hingga 304 nm. Ia turut mempunyai kadar kandungan karbon yang tertinggi pada 80 % kandungan karbon, serta kekonduksian yang baik pada 2.3555 x 10^{-4} S/m. Ia juga turut menunjukkan sifat tahan haba kerana tidak menunjukkan kehilangan berat secara drastik pada suhu tinggi.

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LIST OF ABBREVIATIONS

1D	-	One dimension
2D	-	Two dimension
3D	-	Three dimension
AC	-	Alternating current
C_{60}	-	Fullerene
CF	-	Carbon fiber
CNF	-	Carbon nanofiber
CNT	-	Carbon nanotube
ConA	-	Concanavalin
DMAC	-	Dimethylacetamide
DMF	-	Dimethylformamide
DMSO	-	Dimethlysulfoxide
DNA	-	Deoxyribonucleic acid
DSC	-	Differential scanning calorimetry
DTG	-	Differential thermogravimetric
EDX	-	Energy dispersive x-ray
EIS	-	Electrochemical impedance spectroscopy
FESEM	-	Field emission scanning electron microscopy
FTIR	-	Fourier transform infrared spectroscopy
MWNT	-	Multiwall carbon nanotube
NMR	-	Nuclear magnetic resonance
PAN	-	Polyacrylonitrile
PEEK	-	Polyether ether ketone
PMMA	-	Polymethyl methacrylate
PP	-	Polypropylene
PS	-	Polystyrene
Pt	-	Platinum
SEM	-	Scanning electron microscope
SWNT	-	Single wall carbon nanotube
TEM	-	Transmission electron microscopy
Tg	-	Glass transition temperature

TG	-	Thermogravimetry
TGA	-	Thermogravimetric analysis
Tm	-	Melting temperature
VGCNT	-	Vapour grown carbon nanotube
XRD	-	X-ray diffraction

LIST OF SYMBOLS

°	-	Degree
E	-	Voltage
I	-	Current
Z′	-	Real impedance
Z″	-	Imaginary impedance
θ	-	Theta
π	-	Pi
σ	-	Conductivity
Ω	-	Ohm

LIST OF UNITS

cm	-	Centimeter
cm^{-1}	-	Reciprocal wavelength
cm^3	-	Centimeter cubic
cP	-	Centipoise
Ea	-	Activation energy
eV	-	Electron volt
g	-	Gram
g/cm^3	-	Density
GPa	-	Gigapascal
hr	-	Hour
K	-	Kilo
KV	-	Kilovolt
m	-	Meter
mA	-	Milliampere
mg	-	Milligram
min	-	Minute
ml	-	Milliliter
mm	-	Millimeter
mW	-	Milliwatts
nm	-	Nanometer
S/m	-	Electrical conductivity
TPa	-	Terapascal
V	-	Volt
W/mK^{-1}	-	Thermal conductivity
wt	-	Weight
μm	-	Micrometer
Ω.cm	-	Electrical resistivity

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