PROCESSING AND CHARACTERIZATIONS OF HDPE/PLASTICIZED UHMWPE/MWCNT NANOCOMPOSITES AND THEIR FOAMS FOR EMI SHIELDING EFFECTIVENESS

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by

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Submitted

in fulfillment of the requirements of the degree of
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to the

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Dedicated To My Father

Late Mr. Ramkrishna Rao Bakshi

&

My Mother

Mrs. Tarulata Rao Bakshi

CERTIFICATE

*This is to certify that the thesis entitled, "**Processing and Characterizations of HDPE/Plasticized UHMWPE/MWCNT Nanocomposites and Their Foams for EMI Shielding Effectiveness**" submitted by **Mr. Ashok Kumar Bakshi** to the **Indian Institute of Technology Delhi**, for the fulfilment of award of the degree, **Doctor of Philosophy**, is a record of bonafide research work carried out by him under my supervision and guidance. This thesis has been prepared in conformity with the rules and regulations of the Indian Institute of Technology Delhi, New Delhi.*

*The thesis, in my opinion, is worthy of consideration for award of the degree of **Doctor of Philosophy** in accordance with the regulations of the Institute. To the best of my knowledge, the results embodied in the thesis have not been submitted to any other University or Institute for the award of any other Degree or Diploma.*

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ABSTRACT

This work emphasises primarily the hypothesis that after foaming, a minimum percolation threshold of multiwall carbon nanotubes (MWCNTs) volume fraction (%) is required for the enhancement of the total electromagnetic interference (EMI) shielding effectiveness of nanocomposites (NCs), based on MWCNTs incorporated in the dispersed phase [plasticized ultrahigh molecular weight polyethylene (p-UHMWPE)] and high density polyethylene (HDPE) as a matrix. Several processing and characterization techniques were utilised to conduct the investigation. In the beginning of this study, the effect of plasticization on the processability of UHMWPE with a viscosity average molecular weight ($\bar{M}_v$) of 3.2 million g/mole was investigated by using two grades (H5 and 210P) of low molecular weight olefin-based wax and utilising the HAAKE internal cord mixing device. H5 wax was basically a Fischer-Tropsch process-based hard wax having the $\bar{M}_v$ of 1000 g/mole, and 210P was a polyethylene wax having the $\bar{M}_v$ of 2000 g/mole. It was found that H5 wax was more effective in plasticizing UHMWPE in terms of peak torque (M, Nm) and processing temperature window (ΔT, °C), which were obtained from a Gaussian model applied to mixing data to get the torque versus temperature plots.Quasi static, dynamic and nano mechanical, thermal and morphological characterizations were carried out to support this observation which was obtained from torque vs. temperature plots. It was revealed that p-UHMWPE having 10 phr of H5 wax loading had an effective characteristic properties as compared to that of other compositions. Due to comparatively low molecular weight and short chains, H5 had an advantage of penetrations to uncoil entanglements among the amorphous phase of the microstructure. In the second step of this study, 10 phr of H5 wax loading was utilized to plasticize the UHMWPE and then melt blended with HDPE matrix at 5, 10, 20 and 30 wt. % using HAAKE batch mixture. To investigate the impact of p-UHMWPE on microstructural attributes, neat UHMWPE was also melt blended with HDPE at same series of compositions

for the comparison of quasi static mechanical, melt as well as solid state rheological, thermal and morphological properties. An improved interfacial adhesion was seen with the blends having p-UHMWPE throughout the compositions. Blends having 20-30 wt. % of p-UHMWPE showed improved tensile and impact properties compared that of the neat UHMWPE based blends. Phase immiscibility was observed in both of the cases. However, p-UHMWPE based blends showed improved melt elasticity as well as shear thinning behaviour. Interestingly, it was observed that after foaming at multiple levels of temperature (125-140, °C), pressure (90-120, bar) and saturation time (2-30, min), an effective cell size distribution (45-135, micron) and cell density (1.69×10^4 /cc) was found to be at temperature of 135°C and foaming pressure of 120 bar kept for saturation time of 15 minutes with the blend having 20 wt. % of plasticized UHMWPE. This was confirmed that foam processability was improved of the blend due to plasticization of UHMWPE. Further, 20 wt. % of p-UHMWPE was maintained to prepare HDPE/p-UHMWPE/MWCNT ternary NCs. MWCNTs was incorporated at 0.5,1,2,4 and 8 phr levels. NCs were characterised in terms of quasi mechanical, thermal, melt and solid state rheological, electrical and surface as well as crystalline morphological aspects. It was observed that rheological and electrical percolation thresholds were found to be at 1.5 phr and 0.69 phr of MWCNTs, respectively. SEM and TEM results confirmed that MWCNTs were aligned in the interface of dispersed phase (p-UHMWPE) and matrix phase (HDPE). Post foaming of NCs at 135°C and 120bar for saturation time of 15 min, EMI shielding effectiveness were experimented using vector network analyser (VNA). Totals shielding effectiveness was improved progressively with the MWCNTs loadings upto 8 phr. interestingly, maximum total EMI shielding effectiveness was observed in the range of 0.5-1 phr of MWCNTs content nanocomposite foam. Dielectric permittivity was also enhanced in the both NCs as well as their foams. Cell density was improved progressively upto 2 phr of MWCNTs content, after it slightly reduced till 8 phr.

सार

यह कार्य मुख्य रूप से इस परिकल्पना पर जोर देता है कि फोमिंग के बाद, मल्टीवॉल कार्बन नैनोट्यूब (MWCNTs) वॉल्यूम अंश (%) की न्यूनतम अंतःस्रावी सीमा MWCNTs पर आधारित नैनोकंपोजिट्स (NCs) की परिरक्षण प्रभावशीलता कुल विद्युत चुम्बकीय हस्तक्षेप (EMI) की वृद्धि के लिए आवश्यक छितरे हुए चरण [प्लास्टिसाइज्ड अल्ट्राहाई आणविक भार पॉलीथीन (p-UHMWPE)] और मैट्रिक्स के रूप में उच्च घनत्व पॉलीथीन (HDPE) में शामिल है। जांच करने के लिए कई प्रसंस्करण और लक्षण वर्णन तकनीकों का उपयोग किया गया था। इस अध्ययन की शुरुआत में, 3.2 मिलियन g/mole की चिपचिपाहट औसत आणविक भार ($\bar{M}_v$) के साथ UHMWPE की प्रक्रियात्मकता पर प्लास्टिककरण के प्रभाव की जांच कम आणविक भार ओलेफ़िन-आधारित दो ग्रेड (H5 और 210P) का उपयोग करके की गई थी। मोम और HAAKE आंतरिक कॉर्ड मिक्सिंग डिवाइस का उपयोग करना। H5 मोम मूल रूप से एक फिशर-ट्रॉप्स प्रक्रिया-आधारित हार्ड मोम था जिसमें 1000 g/mole का $\bar{M}_v$ था, और 210P एक पॉलीथीन मोम था जिसमें 2000 g/mole का $\bar{M}_v$ था। यह पाया गया कि पीक टॉर्क (M, Nm) और प्रोसेसिंग टेम्परेचर विंडो (ΔT, °C) के संदर्भ में UHMWPE को प्लास्टिक बनाने में H5 मोम अधिक प्रभावी था, जो टॉर्क बनाम प्राप्त करने के लिए डेटा को मिलाने के लिए लागू गॉसियन मॉडल से प्राप्त किया गया था। तापमान भूखंड। इस अवलोकन का समर्थन करने के लिए अर्ध स्थिर, गतिशील और नैनो यांत्रिक, थर्मल और रूपात्मक लक्षण वर्णन किया गया था जो टोक़ बनाम तापमान भूखंडों से प्राप्त किया गया था। यह पता चला कि एच5 वैक्स लोडिंग के 10 phr वाले p-UHMWPE में अन्य रचनाओं की तुलना में एक प्रभावी विशेषता गुण थे। तुलनात्मक रूप से कम आणविक भार और छोटी श्रृंखलाओं के कारण, H5 को माइक्रोस्ट्रक्चर के अनाकार चरण के बीच उलझावों को खोलने के लिए भेदन का लाभ मिला। इस अध्ययन के दूसरे चरण में, H5 मोम लोडिंग के 10 phr का उपयोग UHMWPE को प्लास्टिक बनाने के लिए किया गया और फिर 5, 10, 20 और 30 wt.% पर HDPE मैट्रिक्स के साथ मिश्रित किया गया, HAAKE बैच मिश्रण का उपयोग करते हुए। माइक्रोस्ट्रक्चरल विशेषताओं पर p-UHMWPE के प्रभाव की जांच करने के लिए, अर्ध स्थिर यांत्रिक, पिघल के साथ-साथ ठोस राज्य रियोलॉजिकल, थर्मल और रूपात्मक गुणों की तुलना के लिए रचनाओं की एक ही श्रृंखला में स्वच्छ UHMWPE को HDPE के साथ मिश्रित किया गया था। संपूर्ण रचनाओं में p-UHMWPE वाले मिश्रणों के साथ एक बेहतर इंटरफेशियल

आसंजन देखा गया। 20-30 wt.% वाले मिश्रण। p-UHMWPE के wt. % ने स्वच्छ UHMWPE आधारित मिश्रणों की तुलना में बेहतर तन्यता और प्रभाव गुणों को दिखाया। दोनों ही मामलों में चरण की असमानता देखी गई। हालांकि, p-UHMWPE आधारित मिश्रणों ने बेहतर पिघल लोच के साथ-साथ कतरनी पतला व्यवहार दिखाया। दिलचस्प रूप से, यह देखा गया कि तापमान (125-140, °C), दबाव (90-120, Bar) और संतृप्ति समय (2-30, min) के कई स्तरों पर फोमिंग के बाद, एक प्रभावी सेल आकार वितरण (45-135 , μm) और सेल घनत्व (1.69 $\times 10^4$ /cc) 135 °C के तापमान और 120 Bar के फोमिंग दबाव को 15 min. के संतृप्ति समय के लिए रखा गया 20 wt. % p-UHMWPE वाले मिश्रण के साथ पर पाया गया । यह पुष्टि की गई थी कि UHMWPE के प्लास्टिककरण के कारण मिश्रण की फोम प्रक्रिया क्षमता में सुधार हुआ था। इसके अलावा, HDPE/p-UHMWPE/MWCNT टर्नरी NCs तैयार करने के लिए p-UHMWPE का 20 wt. % बनाए रखा गया था। MWCNTs को 0.5,1,2,4 और 8 phr स्तरों पर शामिल किया गया था। एनसी को अर्ध-यांत्रिक, थर्मल, मेल्ट और सॉलिड स्टेट रियोलॉजिकल, इलेक्ट्रिकल और सतह के साथ-साथ क्रिस्टलीय रूपात्मक पहलुओं के संदर्भ में चित्रित किया गया था। यह देखा गया कि रियोलॉजिकल और इलेक्ट्रिकल परकोलेशन थ्रेसहोल्ड क्रमशः MWCNTs के 1.5 phr और 0.69 phr पर पाए गए। SEM और TEM परिणामों ने पुष्टि की कि MWCNTs को परिक्षिप्त चरण (p-UHMWPE) और मैट्रिक्स चरण (HDPE) के इंटरफ़ेस में संरेखित किया गया था। 15 मिनट के संतृप्ति समय के लिए 135 °C और 120 Bar पर NCs के फोमिंग के बाद, वेक्टर नेटवर्क विश्लेषक (VNA) का उपयोग करके EMI परिरक्षण प्रभावशीलता का परीक्षण किया गया। MWCNTs लोडिंग के साथ 8 phr तक कुल परिरक्षण प्रभावशीलता में उत्तरोत्तर सुधार हुआ। दिलचस्प बात यह है कि MWCNTs सामग्री नैनोकम्पोजिट फोम के 0.5-1 phr की सीमा में अधिकतम कुल EMI परिरक्षण प्रभावशीलता देखी गई। NCs के साथ-साथ उनके फोम दोनों में ढांकता हुआ पारगम्यता भी बढ़ाया गया था। MWCNTs सामग्री के 2 phr तक सेल घनत्व में उत्तरोत्तर सुधार किया गया था, इसे 8 phr तक थोड़ा कम करने के बाद।

TABLE OF CONTENTS

LIST OF FIGURES

Chapter 1: Introduction and literature survey

Chapter-2: Materials and Experimental Methods

Chapter-3: Studies on the effect of plasticization in processability of UHMWPE using low molecular paraffin and olefin wax

Chapter-4: Effect of plasticized UHMWPE on melt processability and super critical CO_2-assisted foamabiity processability of HDPE/UHMWPE binary blends

Chapter-5: Processing and characterizations of HDPE/p-UHMWPE/MWCNT nanocomposites and their scCO$_2$-assisted foams

LIST OF TABLES

LIST OF EQUATIONS

LIST OF ABBREVIATIONS

AM: Amplitude modulation

AFM: Atomic Force Microscopy

$CaCO_3$: Calcium Carbonate

CB: Carbon Black

CNF: Carbon nanofiber

CNTs: Carbon nanotubes

CQDs: Carbon quantum dots

CPNCs: Carbon-based polymer nanocomposites

CPCs: Conducting poymer composites

CCS: Complex cellular structure

CSM: Continues stiffness measurement

CR: Chloroprene Rubber

DCP: Dicumyl Peroxide

DC: Direct Current

DUHMWPE: Disentangled Ultrahigh Molecular Weight Polyethylene

DSC: Differential Scanning Calorimetry

DTGA: Differential Thermogravimetry Analysis

EM: Electromagnetic

EMI: Electromagnetic Interference

SE: Shielding Effectiveness

EPS: Expandable Polystyrene

EPE: Expanded Polyethylene

EPP: Expanded Polypropylene

FM: Frequency modulation

Fe_3O_4: Ferric Oxide

h- Fe_3O_4: Hollow ferric Oxide

FeCLPU: Flexible Lignin-Based Electromagnetic Shielding Polyurethane

FWHM: Full width half maximima

GnP: Graphene Nanoplatelets

GO: Graphene Oxide

HDPE: High density polyethylene: HDPE

HF: High Frequency: HF

HTM: High Temperature Melting

ICP: Intrinsically Conductive Polymers

LLDPE: Linear Low-Density Polyethylene

LF: Low Frequency

LDPE: Low-Density Polyethylene

MAH: Maleic Anhydride

MDPE: Medium Density Polyethylene

MF: Medium Frequency

μcell: Microcellular Foaming

MIR: Multiple Internal Reflections

MWCNTs: Multiwall Carbon Nanotubes

NC: Nanocomposite

NBR: Acrylonitrile Butadiene Rubber

NRL: Naval Research Laboratory

NA: Network Analyzers

PBA: Physical Blowing Agent

PBAT: Poly (butylene adipate-co-terephthalate)

PET: Poly (ethylene terephthalate)

PE-*g*-G: Polyethylene grafted on the surface of graphene

IIR: Poly (isobutylene-co-isoprene) rubber

PCL: Poly (ε-caprolactone)

PLA: Poly(lactic acid)

PMMA: Poly(methyl meth- acrylate)

PVA: Poly(vinyl alcohol)

PEMA: Poly(ethyl methacrylates)

PA: Polyamide

PANI/PAn: Polyaniline

PC: Polycarbonate

PE: Polyethylene

PFT: Polymerization filling technique

PNC: Polyolefin nanocomposites

POs: Polyolefins

PP: Polypropylene

PPy: Polypyrrole

PS: Polystyrene

PVF: Polyvinyl fluoride

RFI: Radio Frequency Interference

rGO: Reduced Graphene Oxide

SNA: Scalar network analyzers

SWCNTs: Single Carbon Nanotubes

SSSP: Solid-State Shear Pulverization

SBR: Styrene Butadiene Rubber: SBR

SCF: Supercritical Fluid

TPE: Thermoplastic Elastomers

TPU: Thermoplastics polyurethanes

TGA: Thermogravimetry Analysis

TEM: Transmission Electron Microscopy

UHF: Ultra High Frequency

UHMWPE: Ultrahigh Molecular Weight Polyethene

UTM: Universal Testing Machine

VGCNHs: Vapor Grown Carbon Nanofibers

VNA: Vector Network Analyzers

VHF: Very High Frequency

VR: Viscosity Ratio

vGP plot: van Gurp-Palmen plot

WAXD: Wide Angle X-ray Duffraction

ZN: Ziegler–Natta catalyst

LIST OF SYMBOLS

E	: Electric field of electromagnetic radiations
H	: Magnetic field of electromagnetic radiations
P	: Power of electromagnetic radiations
Z_0	: Intrinsic wave impedance of free space
Z	: Wave impedance
L	: Wavelength of incident wave
ε_r^*	: Complex permittivity
ε'_r	: Real (storage cycle) permittivity
ε''_r	: Imaginary (loss cycle) permittivity
μ_r^*	: Complex permeability
μ'_r	: Real (storage cycle) permeability
μ''_r	: Imaginary (loss cycle) permeability
θ	: Electrical phase angle
$(tan\theta)e$	: Electrical dissipation factor
$(tan\theta)m$	: Magnetic loss tangent
A	: Absorption shielding coefficient
R	: Reflection shielding coefficients
T	: Transmission shielding coefficients
A_{eff}	: Effective absorption coefficient
SE_R	: Reflection Shielding Effectiveness
SE_A	: Absorbance Shielding Effectiveness
SE_M	: Shielding Effectiveness due to MIR
SE_T	: Total Shielding Effectiveness
$SE_{T_{SP}}$	: Specific EMI shielding
δ	: Skin depth
d	: Thickness of the nanocomposite sample
f	: Frequency of EM wave
σ_{total}	: Total conductivity
σ_{DC}	: DC conductivity
σ_{PNC}	: DC conductivity of nanocomposites

σ_0 : Characteristics electrical conductivity

dB : decibel

$\beta_{E,\sigma}$: Electrical critical exponent

v_E : Volume fraction at electrical percolating threshold

h : Characteristic tunnelling distance

g : Gap between the two conducting fillers

ΔG_{mix} : Gibbs free energy of mixing

ΔH_{mix} : Enthalpy of mixing

ΔS_{mix} : Entropy of mixing

γ : Total surface tension

γ^d : Surface tension of dispersive components

γ^p : Surface tension of polar components

w_{fg} : Weight of rubber filler gel

w_r : Weight of rubber sample

m_{fg} : Weight fraction of the filler

m_r : Weight fraction of rubber

χ : Viscosity ratio

η_D : Viscosity of the dispersed phase

η_M : Viscosity of matrix phase

$T_{c\ onset}$: Onset crystallization temperature

T_c : Peak crystallization temperature

T_m : Peak melting temperature

$T_{m\ onset}$: Onset melting temperature

T_g : Glass transition temperature

$T_{d\ onset}$: Onset degradation temperature

T_d : Peak degradation temperature

ΔH_0 : Change in enthalpy of fusion of 100% crystalline HDPE and/or UHMWPE

ΔH_m : Change in enthalpy of fusion/melting

X_C : Percentage crystallinity

2θ : Diffraction angle

A_a : Total area under amorphous regions

A_c : Total area under crystalline peaks

K : Scherrer's constant

L : Crystallites size

$\dot{\gamma}_{app}$: Apparent shear rate

τ_{app} : Apparent shear stress

η_{app} : Apparent viscosity

Q_v : Volumetric flow rate

Ω : Screw rotation speed (rpm)

λ : Relaxation time

K : Flow consistency index

α : Flow behaviour index

η_0 : Zero shear rate viscosity

η^* : Complex viscosity

ω : Angular frequency

G^* : Melt rheological complex modulus

G' : Melt rheological storage modulus

G'' : Melt rheological loss modulus

G_0 : Characteristics storage modulus

$\beta_{R,G'}$: Rheological critical exponent

v_R : Volume fraction at rheological percolating threshold

η''/η_0 : Reduced imaginary viscosity

η'/η_0 : Reduced dynamic viscosity

δ : Rheological phase angle

$tan\delta$: Rheological loss tangent

E^* : Solid state dynamic complex modulus

E' : Solid state dynamic storage modulus

E'' : Solid state dynamic loss modulus

$E_{r'}$: Storage modulus in the rubbery plateau

α^* : Crystal- crystal slip

R : Universal gas constant

T : Absolute temperature in Kelvin scale

$\langle M_e \rangle$: Average molecular weight in entanglements

V_e : Entanglement density

ρ : Density of the sample

P_{max} : Maximum indentation load

h_{max} : Maximum penetration depth

h_C : Contact depth

ε : Geometrical constant

H_i : Hardness of the indented sample

E_{eff} : Effective elastic modulus contact

E_S : Elastic moduli of the indented sample

ν_S : Poisson's ratio of the indented sample

A_C : Projected contact area at maximum load

E_i : Elastic Modulus of Berkovich indenter

ν_i : Poisson's ratio of Berkovich indenter

N_c : Cell density

D : Average cell size

σ_y : Yield strength

E_Y : Young's modulus

σ_u : Ultimate strength

σ_b : Breaking strength

ε_b : % Elongation at break