FOAMABILITY OF POLYPROPYLENE AND ITS BLENDS WITH ETHYLENE ACRYLIC ELASTOMER MODIFIED BY GAMMA RADIATION

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by

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"If an egg is broken by an outside force, life ends.

If broken by an inside force, life begins.

Great things always begin from the inside"

– Jim Kwik.

Dedicated

To My

Mother "**Late Reba Dutta**"

&

Wife "**Saheli Bhattacharyya**"

CERTIFICATE

*This is to certify that the thesis entitled, "**Foamability of Polypropylene and Its Blends with Ethylene Acrylic Elastomer Modified by Gamma Radiation**" submitted by **Mr. Anindya Dutta** to the **Indian Institute of Technology Delhi**, for the fulfillment 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

The present study dealt with the introduction of crosslinks in ethylene acrylic elastomer and further checking of foamability of polypropylene (PP)/γ-irradiated ethylene acrylic (AEM) elastomer blends and finally the mechanical performances of the foams were evaluated. In present study, foaming of blends was done with supercritical CO_2 in a batch mode. Initially, optimization of microcellular foam processing of PP/unirradiated ethylene acrylic elastomer blends was carried out through design of experiment approach using Taguchi technique and the correlation among foamability, morphological and rheological parameters of blends was established. In this section, blends with low elastomer content (10 wt.%) were optimized for better foamability. Furthermore, γ-irradiation was done of PP/unirradiated AEM elastomer blends at several radiation doses not only to optimize the γ-irradiation dose but also to optimize the elastomer concentration of blend in terms of degradability during melt processing of blends. Radiation dose of 25 kGy was determined to be optimum in terms of degradation of PP/unirradiated elastomer blend. After the optimization of elastomer concentration, in order to identify the effect of extent of crosslinking of elastomer and the effect of blend morphology on the foamability of PP/γ-irradiated elastomer blends, crosslinking of ethylene acrylic elastomer was carried out using γ-radiation with several doses prior to melt blending and subsequently 10 wt.% of the irradiated elastomers (optimized previously) were mixed with PP in a micro-compounder at three different screw speeds to attain distinct morphological differences among blends. The microcellular morphology development of blends with foaming was analyzed with the screw speed, extent of crosslinking and foaming temperature. Preferences of homogenous and heterogeneous cell nucleation were explained through the surface tension and interfacial

energy of neat polymers [in presence of air and liquid e.g. water and poly(ethylene glycol) 200 (PEG200)]. Additionally, the mechanical responses of PP/γ-irradiated ethylene acrylic elastomer blends were compared with the results of PP/unirradiated ethylene acrylic elastomer blends. Gamma radiation of AEM elastomer was done at 25 kGy optimized irradiation dose. Moreover, thorough correlations was drawn among the mechanical characteristics of blends with their morphological (SEM, WAXD), thermal (DSC), thermo-mechanical (DMA), rheological (frequency sweep) and fractured surface morphological aspects. From several theoretical models, fitted with mechanical parameters of blends, it was determined that the γ-irradiated elastomer had lesser interfacial adhesion with PP than the unirradiated elastomer. Finally, the mechanical performances of foams of PP/γ-irradiated elastomer blends were evaluated and made a comparative study with the mechanical data of foams of PP/unirradiated elastomer blends. Microcellular foam morphology was observed for blends; whereas, for PP submicron and irregular foam morphology was noticed. The yielding behaviuor of blends was marginalized and the yielding of blends with γ-irradiated elastomer was decimated. Most interestingly, necking phenomenon and strain induced crystallization could be observed for foams of blends. Tensile modulus of foam sample was significantly increased by 3 times. From the impact fractured surface morphologies of foams, the enhanced impact strength of foams could be perceived.

सार

वर्तमान अध्ययन ने एथिलीन ऐक्रेलिक इलास्टोमेर में क्रॉसलिंक की शुरूआत और पॉलीप्रोपाइलीन (पीपी) / γ-विकिरणित एथिलीन ऐक्रेलिक (एईएम) इलास्टोमेर मिश्रणों की फोमिंग की जांच की और आखिरकार फोम के यांत्रिक प्रदर्शन का मूल्यांकन किया गया। वर्तमान अध्ययन में, एक बैच मोड में सुपरक्रिटिकल कार्बन डाइऑक्साइड के साथ मिश्रणों की फोमिंग की गई थी। प्रारंभ में, पीपी / अविकिरणित इथाइलीन एक्रिलिक इलास्टोमेर मिश्रणों के माइक्रोसेलुलर फोम प्रसंस्करण का अनुकूलन टैगुची तकनीक का उपयोग करते हुए प्रयोग दृष्टिकोण के डिजाइन के माध्यम से किया गया था और मिश्रणों के फोमेबिलिटी, रूपात्मक और रियोलॉजिकल मापदंडों के बीच संबंध स्थापित किया गया था। इस खंड में, कम एलास्टोमेर सामग्री (10 wt%) के साथ मिश्रण बेहतर फोमेबिलिटी के लिए अनुकूलित किया गया था। इसके अलावा, γ-विकिरण पीपी / अविकिरणित एईएम इलास्टोमेर मिश्रणों पर किया गया था, जो कई विकिरण खुराक पर न केवल विकिरण खुराक को अनुकूलित करने के लिए, बल्कि मिश्रणों के पिघल प्रसंस्करण के दौरान क्षरण की दृष्टि से इलास्टोमेर एकाग्रता का अनुकूलन करने के लिए भी किया गया था। 25 kGy की विकिरण खुराक पीपी / अविकिरणित इलास्टोमेर मिश्रण के क्षरण के संदर्भ में इष्टतम होने के लिए निर्धारित की गई थी। इलास्टोमेर एकाग्रता के अनुकूलन के बाद, इलास्टोमेर के क्रॉसलिंकिंग की सीमा के प्रभाव की पहचान करने और पीपी / γ -विकिरणित इलास्टोमेर मिश्रणों की फेनिलबिलिटी पर मिश्रण आकारिकी के प्रभाव की पहचान करने के लिए, एथिलीन एक्रिलिक इलास्टोमेर के क्रॉसलिंकिंग को कई खुराकें γ-विकिरण के साथ उपयोग किया गया मिश्रणों को पिघलाने से पहले, और बाद में 10 wt% विकिरणित इलास्टोमर्स का (पहले से अनुकूलित) मिश्रणों के बीच अलग-अलग रूपात्मक मतभेदों को प्राप्त करने के लिए तीन अलग-अलग पेंच गति पर एक माइक्रो-कंपाउंडर में पीपी के साथ मिलाया गया था। फोमिंग के साथ मिश्रणों के सूक्ष्म कोशिकीय विकास का विश्लेषण पेंच गति, क्रॉसलिंकिंग की सीमा और झाग तापमान के साथ किया गया था। सजातीय और विषम सेल न्यूक्लियेशन की वरीयताएँ सतह के तनाव और स्वच्छ पोलीमर की अंतरापर्णीय ऊर्जा के माध्यम से समझाई गईं [वायु और

तरल की उपस्थिति में उदा। पानी और पाली (इथाइलीन ग्लाइकॉल) 200 (PEG200)]। इसके अतिरिक्त, पीपी / γ-विकिरणित एथिलीन ऐक्रेलिक इलास्टोमेर मिश्रणों के यांत्रिक प्रतिक्रियाओं की तुलना पीपी / अविकिरणित एथिलीन एक्रिलिक इलास्टोमेर मिश्रणों के परिणामों के साथ की गई थी। एईएम इलास्टोमेर का γ-विकिरण 25 kGy अनुकूलित विकिरण खुराक पर किया गया था। इसके अलावा, उनके रूपात्मक (SEM, WAXD), थर्मल (DSC), थर्मो-मैकेनिकल (DMA), रियोलॉजिकल (Frequency sweep) और खंडित सतह आकारिकी पहलुओं के साथ मिश्रणों की यांत्रिक विशेषताओं के बीच पूरी तरह से सहसंबंध तैयार किए गए थे। कई सैद्धांतिक मॉडलों से, मिश्रणों के यांत्रिक मापदंडों से सुसज्जित, यह निर्धारित किया गया था कि γ-विकिरणित इलास्टोमेर में पीपी के साथ अविकिरणित इलास्टोमेर की तुलना में कम इंटरफेशियल आसंजन था। अंत में, पीपी / γ-विकिरणित इलास्टोमेर मिश्रणों के झागों के यांत्रिक प्रदर्शन का मूल्यांकन किया गया और पीपी / अविकिरणित इलास्टोमेर मिश्रणों के यांत्रिक दत्त-सामग्री के साथ एक तुलनात्मक अध्ययन किया गया। मिश्रणों के लिए माइक्रोकेलुलर फोम आकृति विज्ञान देखा गया था; जबकि, पीपी के लिए उप माइक्रोन और अनियमित फोम आकारिकी देखा गया था। मिश्रणों के उपज व्यवहार को हाशिए पर रखा गया था और γ-विकिरणित इलास्टोमेर के साथ मिश्रणों की उपज को हटा दिया गया था। सबसे दिलचस्प बात, नेकिंग की घटना और तनाव प्रेरित क्रिस्टलीकरण मिश्रणों के फोम के लिए मनाया जा सकता है। फोम के नमूने के तन्यता मापांक में 3 गुना की वृद्धि हुई थी। फोम के खंडित सतह आकारिकी के प्रभाव से, फोम की बढ़ी हुई प्रभाव शक्ति को माना जा सकता है।

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LIST OF EQUATIONS

LIST OF ABBREVIATIONS

Abbreviation		Description
AEM	:	Ethylene acrylic elastomer
ANOVA	:	Analysis of variance
BSE	:	Backscattered electron
CBA	:	Chemical blowing agent
CD	:	Cell density
CFC	:	Chlorofluorocarbon
CS	:	Cell size
DCP	:	Dicumyl peroxide
DF	:	Degrees of freedom
DMA	:	Dynamic mechanical analysis
DOE	:	Design of experiment
DSC	:	Differential scanning calorimetry
DTGA	:	Differential thermogravimetric analysis
E-b-P	:	Ethylene propylene block copolymer
EMA	:	Ethylene methyl acrylate elastomer
EOC	:	Ethylene octane copolymer
EPDM	:	Ethylene propylene di-ene rubber
EPE	:	Expanded polyethylene
EPP	:	Expanded polypropylene
EPR	:	Ethylene propylene rubber
EPS	:	Expanded polystyrene
E-r-P	:	Ethylene propylene random copolymer
EVA	:	Ethylene vinyl acetate
FTIR	:	Fourier transformed infra-red spectroscopy
$FWHM_c$	:	Full width at half maxima for crystallization
$FWHM_m$	:	Full width at half maxima for fusion

HCFC	:	Hydrochlorofluorocarbon
HDPE	:	High density polyethylene
HFC	:	Hydrofluorocarbon
HMSPP	:	High melt strength polypropylene
IPD	:	Interparticle distance
iPP	:	Isotactic polypropylene
LDPE	:	Low density polyethylene
LLDPE	:	Linear low density polyethylene
MFI	:	Melt flow index
NG	:	No gel
NMR	:	Nuclear magnetic resonance spectroscopy
OA	:	Orthogonal array
PBA	:	Physical blowing agent
PE	:	Polyethylene
PEG200	:	Polyethylene glycol 200
PET	:	Polyethylene terephthalate
POE	:	Polyethylene-octene elastomer
PP	:	Polypropylene
SBS	:	Styrene butadiene styrene rubber
$scCO_2$	:	Supercritical carbon dioxide
SE	:	Secondary electron
SEM	:	Scanning electron microscope
SS	:	Sum of squares
TAIC	:	Triallylisocyanurate
TGA	:	Thermogravimetric analysis
TPO	:	Thermoplastic olefin
TPS	:	Thermoplastic polystyrene elastomer
TPV	:	Thermoplastic vulcanizate
TSE	:	Twin screw extruder
UF	:	Unfoamed material
UHMWPE	:	Ultra high molecular weight polyethylene

UTM	:	Universal testing machine
V.R.	:	Viscosity ratio
VER	:	Volume expansion ratio
vGP plot	:	van Grup-Palmen plot
WAXD	:	Wide angle X-ray diffraction
WGRT	:	Waste ground rubber tire
XRD	:	X-ray diffraction

LIST OF SYMBOLS

Symbol		Description
2θ	:	X-ray scattering angle
A	:	Area of the SEM micrograph (centimeters squared)
C	:	Gibson and Ashby parameter
d	:	Number average cell size
D_n	:	Number average particle size
D_w	:	Weight average particle size
E	:	Tensile modulus / Tensile moduli of foam
E'	:	Storage modulus
E''	:	Loss modulus
E_0	:	Tensile moduli of non-porous material
E_a	:	Activation energy for degradation
$E_{a(m)}$	:	Activation energy of matrix
$E_{a(r)}$	:	Activation energy of elastomer
$E_{a(theo)}$	:	Activation energy of blend evaluated theoretically
E_b	:	Tensile modulus of blend
E_b/E_m	:	Relative tensile moduli
E_d	:	Tensile modulus of elastomer
E_m	:	Tensile modulus of matrix
F	:	Corrected factor of heterogeneous nucleation/ geometrical factor
G^*	:	Complex modulus
G'	:	Elastic modulus
G''	:	Viscous modulus
I	:	Impact strength
I_b	:	Impact strength of blend
I_b/I_m	:	Relative impact strength
I_m	:	Impact strength of matrix
K_b	:	Interphase interaction parameter in Nicolais-Narkis model

m_1	:	Weight of sample in air
m_2	:	Weight of sample along with sinker in fluid
m_s	:	Weight of sinker in fluid
n	:	Order of degradation
n	:	Gibson and Ashby parameter
N_c	:	Cell density
R	:	Universal gas constant
R^2	:	Coefficient of determination
S	:	Neilsen parameters for first power-law model
S/N	:	Signal to noise ratio
T	:	Absolute temperature
tanδ	:	Tan delta
T_c	:	Crystallization temperature
T_g	:	Glass transition temperature
T_i	:	Initiation degradation temperature
T_m	:	Melting temperature
T_m	:	Maximum degradation temperature
W_{el}	:	Weight fraction of elastomer present in blends
W_f	:	Final weight of sample
W_{het}	:	Free energy barrier for initiating the heterogeneous nucleation
W_{het}	:	Surface geometries of the nucleating agents
W_i	:	Initial weight of sample
wt.%	:	Weight percentage
A	:	Degree of conversion at time t
α	:	Porosity model parameter
γ	:	Surface tension
γ_{12}	:	Interfacial tension between materials 1 and 2
γ^d	:	Dispersive component of the surface tension
γ^p	:	Polar component of the surface tension
γ-radiation	:	Gamma radiation

δ	:	Phase angle
ΔH_c	:	Enthalpy of melting
$\Delta H_{experimental}$	:	Experimentally achieved enthalpy of fusion of PP
$\Delta H_{\text{theoretical}}$	:	Theoretically determined enthalpy of fusion of 100% crystalline PP
ΔP	:	Pressure exerted by the $scCO_2$ on the cell walls
ΔT	:	Temperature range
ε	:	Strain at break
ϵ_b	:	Strain at break of blend
ϵ_b/ϵ_m	:	Relative strains at break
ϵ_m	:	Strain at break of matrix
η^*	:	Complex viscosity
θ / θ_c	:	Contact angle
ρ	:	Density
ρ	:	Density/ Density of foam
ρ_f	:	Density of the foamed sample
ρ_{fl}	:	Specific gravity of the fluid
ρ_o	:	Density of non-porous material
ρ_u	:	Density of the unfoamed sample
σ	:	Tensile strength
σ_b	:	Tensile strength of blend
σ_b/σ_m	:	Relative tensile strength
σ_m	:	Tensile strength of matrix
τ	:	Shear stress
φ	:	Volume expansion ratio
φ_r	:	Volume fraction
χ	:	Crystallinity percentage/Total crystallinity percentage
ω	:	Frequency
ΔG^*_{homo}	:	Gibb's free energy for homogeneous nucleation
ΔG^*_{het}	:	Gibb's free energy for heterogeneous nucleation
$\bar{d}$	:	Average cell size of foam
$\overline{N}_c$	:	Average cell density of foams

$\bar{\eta}$	:	Predicted average of signal to noise ratio
$\bar{T}$	:	Overall experimental average of signal to noise ratio
$\dot{\gamma}$	:	Shear rate