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PROPERTIES OF EPOXY/UHWPE BLENDS



INVESTIGATION ON THERMAL, MECHANICAL AND MORPHOLOGICAL

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ABSTRACT

Epoxy/ultrahigh molecular weight polyethylene (UHMWPE) blends have been prepared by in-situ polymerization method. The effects of UHMWPE from 2.5 wt % to 10 wt % on the tensile properties of blends have been studied. Tensile properties of blends are characterized and were shown that content 10% UHMWPE was effected on performance of polymer blends significantly. Thermal characteristics of the polymer blends were determined by using differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA). TGA thermogram indicated that increased thermal stability of blends after incorporation of UHMWPE. The DSC was shown that T_g of polymer blends increased. The enthalpy of melting (ΔH_m) obtained from DSC curves was increased with increase in UHMWPE content in epoxy matrix, due to depend on UHMWPE content in blend systems. The rheology of polymer was envisaged by DMA is affected on the polymer blend. The incorporation of UHMWPE increased mechanical properties of blends. Degradation kinetic parameters have been calculated for thermal degradation processes using two mathematical models namely, Coats-Redfern and Broido's methods. Morphological behaviour of fractured blends has been studied using scanning electron microscopy (SEM).

Keywords: epoxy, UHMWPE, blend, mechanical performance, thermal characterization, DMA, kinetic parameters.

1. INTRODUCTION

Polymer mixtures or blends are widely used materials in modern industry. Polymer blends represent one of the most rapidly growing areas in polymer material science. This field got its intensive development during recent years and the literature on this topic is really immense. Application of polymer blends in numerous fields such as adhesion, colloidal stability, and design of blends and biocompatible materials requires a fundamental understanding of the structure, phase state and composition of blends in the vicinity of interacting surfaces. The thermodynamic, mechanical and rheological properties of polymer blends are the object of numerous investigations. The fundamentals of the physical chemistry of polymer blends and interpenetrating polymer networks (IPNs) are presented in some monographs [1-2]. It is also known that polymer materials are hardly ever used in their pure form and are usually filled with additives that improve their processability and properties. For these purposes, particulate disperse fillers and fibrous fillers are widely used.

Blending of polymers is an effective way in producing advance multicomponent polymeric material with new property profiles [3-4]. Polymer blending is a convenient route for the development of new polymeric materials, able to yield materials with property profiles superior to those of the individual components. This method is usually cheaper and less time-consuming for the creation of polymeric materials with new properties than the development of new monomers and/or new polymerization routes. An additional advantage of polymer blends is that the properties of the materials can be tailored by combining component polymers and changing the blend composition. They can be compatibilized. Ultra-high molecular weight polyethylene (UHMWPE) has long linear chains and high crystallinity, which provide chemical inertness and good mechanical strength [5]. Owing to its excellent bulk physical and chemical properties and low cost in comparison with perfluorinated polymer, UHMWPE appears to be a promising base material for the development of alternative hydrocarbon-based PEMs.

2. MATERIALS AND METHODS

2.1 Materials

The diglycidyl ether bisphenol A (DGEBA) based epoxy resin Araldite A 31, is a clear liquid with a viscosity of 30000 to 50000 mPa at 25 °C and Hardener K 31 (aromatic) amine type, is a clear liquid with a viscosity of 30000 to 50000 mPa at 25 °C, were obtained from Huntsman Advanced Materials, Bangalore, India. Ultrahigh molecular weight polyethylene (UHMWPE) of XM-220 grade, whose average particle size is 30-50 μ m, density is 0.94 g/cc and T_m is 135 °C were obtained from Honam Petrochemical Co. As a gift sample.

2.2 Sample preparation

A series of epoxy/UHMWPE were fabricate with varying amounts of UHMWPE viz., 0, 2.5, 5, 7.5 and 10 wt. %, by in- situ polymerization method. The blend was mixed 20 min to achieve a reasonably uniform dispersion. The liquid mixture was then cast in preheated cleaned, releasing agent smeared moulds and cured at room temperature for 24 h. To ensure complete curing, the blend sheets were post cured at 120 °C for 4 h. The post cured blend plates were left in the oven and allowed to cool gradually to ambient temperature before removal from the

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moulds. The cured blend laminates were machined into different specimens for mechanical and other tests.

2.3 Techniques

2.3.1 Physico - mechanical properties

The prepared epoxy/UHMWPE blends were characterized for physical properties such as density and surface hardness according to ASTM D 785 and ASTM D 2240 methods respectively. The tensile behaviour of the blends were measured using JJ Lloyds Universal Testing Machine, model Z20, 20 KN, USA as per ASTM D-638 test method at a crosshead speed of 50 mm/min and a gauge length of 50 mm. Minimum five samples were tested at room temperature for each formulation and an average values are reported. Charpy impact strength (unnotched) was measured in a Win PEN CEAST S. p. A., Italy according to ISO 179.

2.3.2 Thermal behaviours

The transition temperatures of the samples were examined by using differential scanning calorimeter (DSC) model DSC-Q 200, Dupont TA instrument, USA. All samples were sealed in hermetic aluminum pans. The thermal behaviours of the samples were investigated at the heating rate of 10 °C/min from ambient to 300 °C under nitrogen gas flow of 60 mL/min.

The thermal degradation parameter of epoxy blends were evaluated using Dupont TA Instrument with TGA-Q 50 module. The instrument was calibrated using pure calcium oxalate sample before analysis. About 8-10 mg of sample was subjected to dynamic TGA scans at a heating rate of 20 °C/min in the temperature range of ambient to 700 °C in N₂ gas atmosphere. The TGA curves were analysed as percentage weight loss as a function of temperature. The oxidation index (OI) was calculated based on the weight of carbonaceous char (CR) as related by the empirical equation;

$$OI \times 100 = 17.4 \times 0.4CR$$
(1)

The thermal degradation kinetic parameters were determined for epoxy/UHMWPE blends using Coats-Redfern [6] and Broido's [7] methods which provide overall kinetic data. For the sake of calculations and to know the nature of the decomposition, the complete thermo gram was divided into distinct sections according to their degradation processes.

The kinetic parameters of thermal degradation process have been calculated using two models. Coats-Redfern (CR) [6] relation is as follows;

$$\log (-\log (1 - \alpha)/T^2) = (\log (AR/\beta E_a)) - (E_a/2.303RT)$$
(2)

Where, α is the fraction of sample decomposed at temperature T, T is the derivative peak temperature, A is the frequency factor, β is the heating rate, E_a is the activation energy, and R is the gas constant.

A plot of log $\{-\log (1-\alpha)/T^2\}$ versus 1/T gives the slope for evaluation of the activation energy most appropriately. Mathematical expression of Broido's (BR) [7] method is as follows;

$$Log (-log (1-\alpha)) = - (E_a/2.303R) ((1/T) + K)$$
(3)

where, $(1-\alpha)$ is the fraction of number of initial molecules not yet decomposed, T is the peak temperature of derivative curve of TGA, R is the gas constant and E_a is the activation energy can be calculated from the plot of log $(-\log(1-\alpha))$ versus 1/T.

The storage modulus (E) and the mechanical loss factor (Tan $\delta = E'/E'$) as a function of temperature (T), were assessed by dynamic mechanical thermal analysis (DMA) using a Dupont TA instrument, USA, with model 2980-DMA. DMA thermo grams were recorded in tension mode at 5 Hz frequency at a heating rate of 3°C/min in the temperature range 30 - 160 °C.

2.3.3 Chemical resistance

The chemical resistance of the blends was studied as per ASTM D 543.06 method. For this purpose different chemical reagents such as 10 % HCl, 10 % NaOH, 10 % H_2SO_4 , 10 % Na_2CO_3 , 10 % NH_4OH , benzene and water were used.

2.3.4 Morphological behaviours

The scanning electron microscope (SEM) was used to study the micro phase separation and morphology of the blends. Using a SEM of Hitachi S-2150, morphological studies on the Cryofractured surface of epoxy/UHMWPE blends were performed. The tensile or impact fractured samples were used for SEM studies. The fracture samples were glued on the aluminum sample holders and gold coated before analysis.

3. RESULTS AND DISCUSSIONS

The measured physico-mechanical properties such as density, surface hardness and tensile behaviours of epoxy/UHMWPE blends are nominated in Table-1. From Table-1 it was observed that epoxy laminate had an average density of 1.02 g/cc. After incorporating UHMWPE, decrease in the density of the blends was observed. This decrease in density of the blends is attributed to the incorporation of low dense UHMWPE (0.94 g/cc) in high dense epoxy matrix. The blends under this investigation consists of two components namely thermoset, epoxy matrix, and thermoplastic UHMWPE. Density of a composite depends on the relative proportion of matrix and reinforcing materials and this is one of the most important factors determining the properties of the composites. The theoretical density of blends in terms of weight fraction can easily be obtained as per the following equation [16];

$$P_{ct} = \frac{1}{\left(\frac{Wm}{Pm}\right) + \left(\frac{Wp}{Pp}\right)}$$
(4)

Where, W and P represent the weight fraction and density respectively. The suffix p, m and ct stand for the matrix and the composite materials respectively. The suffix 'p'



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indicates the UHMWPE. The actual density (P_{ce}) of the composite can be determined experimentally by simple water immersion technique. The void content is the cause for the difference between the values of true density and the theoretically calculated one. The volume fraction of voids (V_v) in the composites is calculated using the following equation:

$$V_{v} = \frac{(Pct - Pce)}{Pct} \times 100$$
(5)

The theoretical and measured densities of the blends along with the corresponding volume fraction of voids are presented in Table-1. It may be noted that the blend density values calculated theoretically from weight fractions using Eq. (4) are not equal to the experimentally measured values. This difference is a measure of voids and pores present in the composites. It is clearly seen that with the addition of UHMWPE as the filler material, high voids are found in the blends. The calculated void content of the blends lies in the range 0.78-13 %. The voids significantly affect some of the mechanical properties and even the performance of composites in the workplace. Higher void contents usually mean lower fatigue resistance, greater susceptibility to water penetration and weathering [16]. The knowledge of void content is desirable for estimation of the quality of the blends. It is understandable that a good composite should have fewer voids. However, presence of void is avoidable in composite making particularly through hand-lay-up route.

Surface hardness is a measure of resistance to indentation. Surface hardness indicates the degree of compatibility and crosslink density. A slight increase in surface hardness from 61 to 73 shore D with increase in UHMWPE content was noticed.

Mechanical properties of blends generally depend on factors such as UHMWPE content, particle size and shape, the degree of adhesion between UHMWPE, polymer matrix and degree of dispersion the UHMWPE within the matrix. From Table-1 it was noticed that much lower values of tensile modulus, tensile strength and tensile elongation were obtained than those of epoxy matrix but in 10 % it is same. Tensile modulus UHMWPE is 0.8 GPa and pristine epoxy is about 2.15 GPa that indicates reduction in tensile modulus. Tensile strength and percentage of elongation at break of epoxy/UHMWPE blends markedly reduced from 9.5 MPa to 8.9 MPa and from 4.9 to 3.6 respectively with increase in UHMWPE content.

The impact strength evaluation is an important parameter to study the toughening behaviour of polymer blends. The influence of on the impact strength of epoxy/UHMWPE blends is shown in Figure-1. The impact strength of the blends is higher than pristine epoxy (12 kJ/m²). From Figure-1 it was noticed that a noticeable improvement in impact strength of the blends as increase in UHMWPE content.

3.1 Thermal behaviour

Figure-2 shows the dynamic DSC thermograms for neat epoxy and their blends with different contents of UHMWPE. The DSC has been shown that higher T_g obtained from DSC thermograms is given in Table-1 and it lies in the range 68 - 76 °C. There was no significant change in melting temperature (T_m) for pristine epoxy and its composites. The enthalpy of melting $(\triangle H_m)$ loaded blend was increase from 2.84 to 13.65 J/g as increase is UHMWPE from 2.5 to 10 wt. %. The TGA thermograms of epoxy and blends are shown in Figures-3, (a)-(e) along with derivative thermograms. TGA thermograms of all epoxy blends are shown in Figure-4. The temperature range of thermal degradation was analyzed from the TGA thermograms and is given in Table-3. TGA thermogrmas of pristine epoxy and its blends indicate one stage thermal degradation process. The thermal degradation of neat epoxy occurred in the temperature range 282-540°C with the major weight loss of around 96.1 %. The decomposition temperature of blends was started at 205 °C and takes place up to 463 °C, which corresponds to the weight loss ranging from 96.1 to 99.37 %. It was found that the pure epoxy degrades slightly faster than the blends. The onset degradation of epoxy/UHMWPE blends is slightly higher than that of neat epoxy (Table-5). This can be attributed to the presence of UHMWPE, which has relatively higher thermal stability than epoxy. The obtained percentage ash content is low in blends as compared to neat epoxy (3.9 %). The ash content of epoxy blends lies in the range 0.63-3.9. This is due to the TGA studies conducted in an inert gas media.

Some characteristics TGA data related to the temperature corresponding to weight loss such as T_0 (temperature of onset decomposition), T_{10} (temperature for 10% weight loss), T_{20} (temperature for 20% weight loss), T_{50} (temperature for 50% weight loss) and T_{max} (temperature for maximum weight loss) are the main criteria to indicate the thermal stability of the blends. The relative thermal stability of epoxy blends have been evaluated by comparing the decomposition temperatures at different percentage weight loss (Table-5). Higher the values of T_{10} , T_{20} , T_{50} and T_{max} higher will be the thermal stability of the blends [9].

Figure-3 and Table-5 data reveals that the initial stage thermal degradation process pattern is almost same for both unfilled and UHMWPE filled epoxy blends. But after T_{50} a marked improvement in thermal stability for blends than epoxy was noticed. This can be attributed to the synergistic effect of UHMWPE and matrix present in the blend. Higher the values of oxidation index (OI), higher will be the thermal stability [9-10]. From the Table it was observed that very low oxidation index values and it lies in the range 0.04-0.27. Annakutty *et al* reported that, the char yield is directly correlated to the potency of flame retardation [11]. From the aforesaid investigation, it can be concluded that the flame resistance behaviour of blends was very low.



3.2 Kinetic analysis of thermal degradation

Thermal degradation kinetic parameters was evaluated from TGA curves using Coats-Redfern and Broido's methods. The plots of $\ln \left[-\ln (1-\alpha)\right]/T^2$ versus 1/T (CR) and ln [-ln (1- α)] versus 1/T (BR) for epoxy/ UHMWPE blends are shown in Figures 5 and 6, respectively. The regression analysis gives the slopes, constants and R² for degradation process. The linear plot with concurrency value (R^2) closer to one was chosen for both methods. The R^2 values and calculated activation energy (E_a) for thermal degradation process and for each method are tabulated in Table-6. The E_a values lies in the range 67.2 - 131.2 kJ/mol for Coats-Redfern and 78.5 -142.3 kJ/mol for Broido's. Lower Ea values were observed for UHMWPE loaded epoxy blends. This is due to after incorporation of UHMWPE, formation of low cross link density of epoxy between interlayers of UHMWPE, for which less energy is sufficient for degradation.

3.3 Dynamic mechanical analysis (DMA)

The effect of UHMWPE on storage modulus and Tan δ values of epoxy/ UHMWPE blends is given in Table-7. In all the cases, the values of E' decreased with increasing temperatures. Also, at any given temperature, the storage modulus decreased with increasing thermoplastic loading which might be due to lowering of the stiffness of matrix. The E' of pristine epoxy measured at 40 °C was 1.899 GPa and it decreases, for blends. The maximum E' was measured to be 1.969 GPa with 10 wt. % UHMWPE, which is about 7 % higher than that of pristine epoxy. Table-7 also lists the average values of T_g for different wt. % of UHMWPE. The measured Tg of pristine epoxy was 108 °C. The absence of entanglement surrounding the UHMWPE and a lower cross-link density have been attributed to the decrease in Tg. Table-7 lists the average values of Tan δ for different percentage weight of UHMWPE. It can be seen from Table-7, that Tan δ show no significant change by the addition of UHMWPE. As temperature is increased, it is observed that the damping goes through a maximum in the transition region and then

decreases in the rubbery region. The damping is low below T_g as the chain segment in that region is frozen. Below T_g , the deformations are thus primarily elastic and the molecular slips resulting in viscous flow is low. Also, above T_g , in the rubbery region, the damping is low because the molecular segments are free to move, and consequently, there is little resistance to flow. In the transition region, on the other hand, the damping is high because of the initiation of micro-processing. The shift of relaxation peak towards low temperature as a result of low crosslink density and poor interaction between epoxy and UHMWPE can be attributed to the presence of UHMWPE. The samples displayed a slightly different dynamic mechanical behaviour. The values of cross linking density may be calculated by using (7) as given by Kaji [12];

$$\rho = E/3RT \tag{7}$$

Where, E is the storage modulus at $T_g + 30^{\circ}$ C, R is the gas constant and T is the absolute temperature at $T_g + 30^{\circ}$ C. A gradual drop of storage modulus with addition of UHMWPE revealed increased flexibility of the blend samples. The stiffness of the resultant material was also temperature sensitive [13]. The storage modulus dropped with increasing temperature which indicated that all formulations gradually passed from stiff hard solid-to-soft and flexible material which agreed well with previous reasoning (Manzione and Gillham 1981; Verchere *et al* 1991; Chikki *et al* 1997)

3.4 Scanning electron microscopic (SEM) analysis

The morphological behaviour strongly influences the properties of polymer blends [14]. SEM images for all blends are shown in Figure-10 (a) - (e). These Figures exhibited two phases morphology for epoxy/UHMWPE blends. SEM images of blends clearly indicated two phase morphologies because the presence of both hydrophobic (UHMWPE) and hydrophilic (epoxy) phases. The number of domains depends on the UHMWPE content in the methods. SEM images also reveal poor interaction between epoxy and UHMWPE.

UHMWPE content in	Density (g/cc)		Void content	Tensile modulus	Tensile	Elongation	Surface hardness
epoxy (wt %)	Expt	Theor	(%)		at break (%)	(Shore D) ±2	
0	1.02	-	-	2.15	9.5	4.9	61
2.5	1.01	1.018	0.78	1.52	6.2	2.3	56
5	0.97	1.016	4.5	1.56	5.3	2.3	55
7.5	0.92	1.014	9.2	1.7	7.3	3.8	68
10	0.88	1.012	13	2.1	8.9	3.6	73

Table-1. Mechanical properties of epoxy/ UHMWPE blends.



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UHMWPE content in epoxy (wt. %)	10 % HCl	10 % H ₂ SO ₄	10 % NaOH	10 % Na ₂ CO ₃	10 % NH4OH	C ₆ H ₆	H ₂ O
0	1.78	2.68	1.15	0.85	1.45	21.69	1.24
2.5	3.16	6.33	1.32	1.03	1.34	32.44	1.11
5.0	4.85	9.03	1.65	1.45	1.53	33.83	1.57
7.5	1.06	1.36	1.02	0.85	1.21	49.63	0.92
10	1.79	3.22	0.86	1.38	1.03	30.44	1.21

Table-2. Chemical resistance of pristine epoxy and epoxy/ UHWPE blend polymer.

Table-3. Data obtained of epoxy/UHMWPE blends.

UHMWPE content in epoxy (wt %)	T _g (°C)	T _m (°C)	$\Delta \mathbf{H}$ (J/g)
0	74.7	-	-
2.5	68.8	136	2.84
5	76.0	135	4.65
7.5	69.3	135	10.64
10	74.9	136	13.65

 Table-4. Thermal degradation temperature range obtained from derivative TGA curves of epoxy/ UHMWPE blends.

UHMWPE content in epoxy (wt %)	Temperature range (°C) ±2			IDDT (°C)	Oxidation	Ash content
	T ₀	T _p	T _c	IDPT (°C) ±4	index (OI)	(%)
0	282	377	540	372	0.27	3.9
2.5	233	378	528	398	0.15	2.2
5	227	383	530	382	0.20	3.0
7.5	243	380	522	391	0.04	0.63
10	264	390	529	385	0.20	3.0

Table-5. Thermal data obtained from TGA thermograms of epoxy/ UHMWPE blends.

UHMWPE	Temperature at different weight loss (± 2 °C)					
content in epoxy (wt %)	T ₀	T ₁₀	T ₂₀	T ₅₀	T max	
0	205	353	365	387	463	
2.5	221	351	371	406	515	
5	224	355	372	410	507	
7.5	230	361	373	410	502	
10	230	364	377	412	508	

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UHMWPE content	Activation energy (E _a) (kJ/mol) <u>+</u> 4 %						
in epoxy (wt %)	CR	R ²	BR	R ²			
0	131.2	0.978	142.3	0.981			
2.5	67.2	0.989	78.5	0.992			
5	78.5	0.985	89.9	0.989			
7.5	74.8	0.988	86.3	0.991			
10	81.4	0.976	92.8	0.982			

Table-6. Activation energies calculated by coats-Redfern (CR) and Broido's (BR) methods with the respective concurrency value (R^2) for epoxy/ UHMWPE blends.

Table-7. Results of DMA analysis of epoxy/UHMWPE blends.

UHMWPE	E' (MPa)	E ["] max	Tan _{max} δ	Tempera	n (10 ⁻³	
content in EP (wt. %)	E' (MPa) at 40 ⁰ C	(MPa)		E" max	Tan _{max} δ	$\rho (10^{-3} \text{ mol/cm}^3)$
0	1899	205	0.70	95	108	1.65
2.5	1338	179	0.72	74	85	1.39
5	893	89	0.58	91	104	0.8
7.5	1704	242	0.84	74	83	1.81
10	1969	140	0.46	86	114	1.64

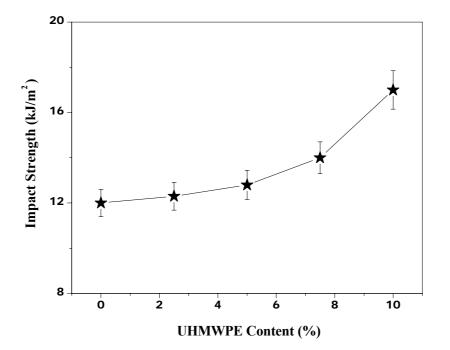


Figure-1. Influence of UHMWPE on impact strength of epoxy/ UHMWPE blends.

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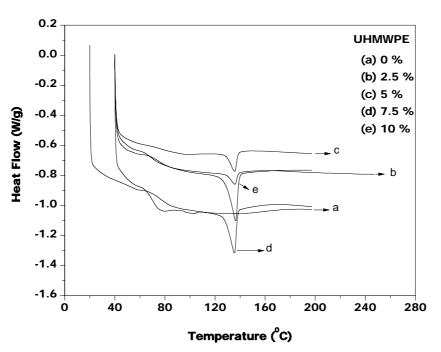


Figure-2. DSC thermograms of epoxy and epoxy/UHMWPE blends.

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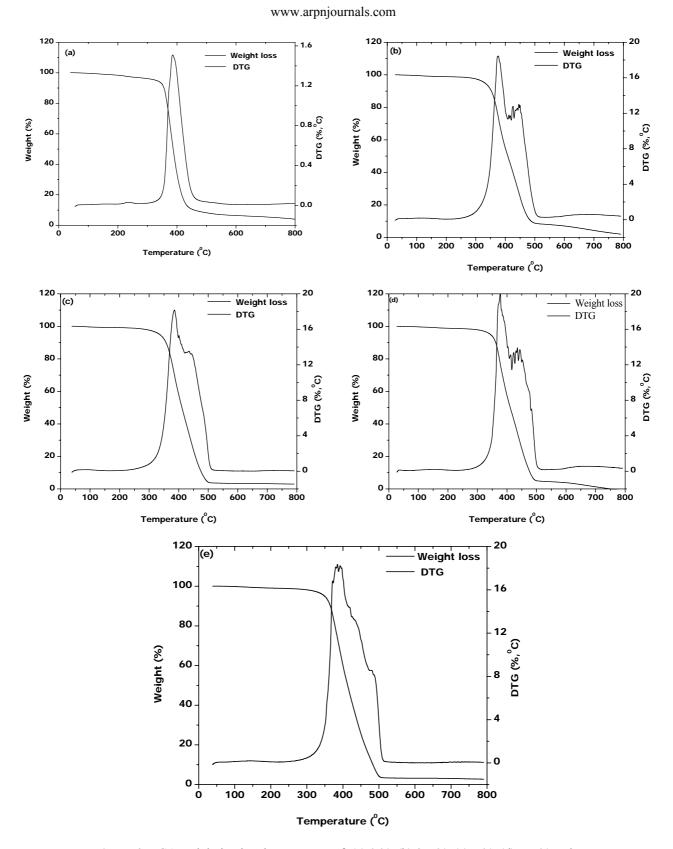


Figure-3. TGA and derivative thermograms of, (a) 0 %, (b) 2.5 %, (c) 5 %, (d) 7.5 % and (e) 10 % of UHMWPE loaded blends.

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120 UHMWPE (a) 0 % 100 (b) 2.5 % (c) 5 % (d) 7.5 % 80 (e) 10 % Weight (%) 60 40 20 0 500 600 ò 100 200 300 400 700 800 Temperature (°C)

Figure-4. TGA thermograms of epoxy and its blends.

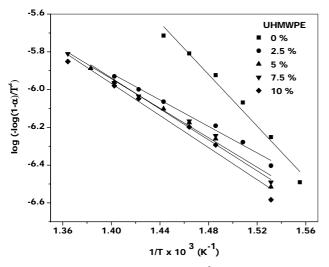


Figure-5. Plots of $\log\{-\log(1-\alpha)/T^2\}$ versus 1/T using coats-Redfern method for epoxy/UHMWPE blends.

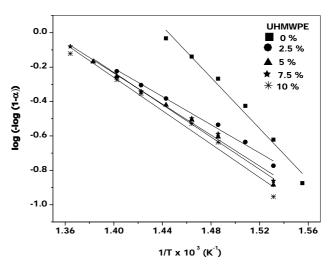


Figure-6. Plots of ln[-ln(1-α)] versus 1/T using broido's method for epoxy/ UHMWPE blends.

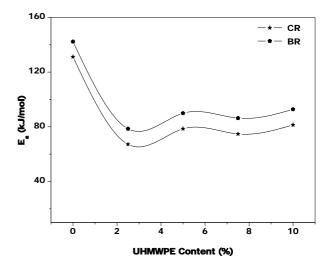


Figure-7. Activation energy as a function of weight percent of UHMWPE in EP/UHMWPE for both methods.

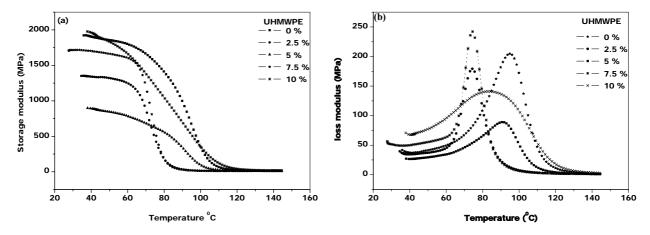


Figure-8. Plot of (a) storage modulus and (b) loss modulus as a functional of thermal of EP/UHMWPE blend.

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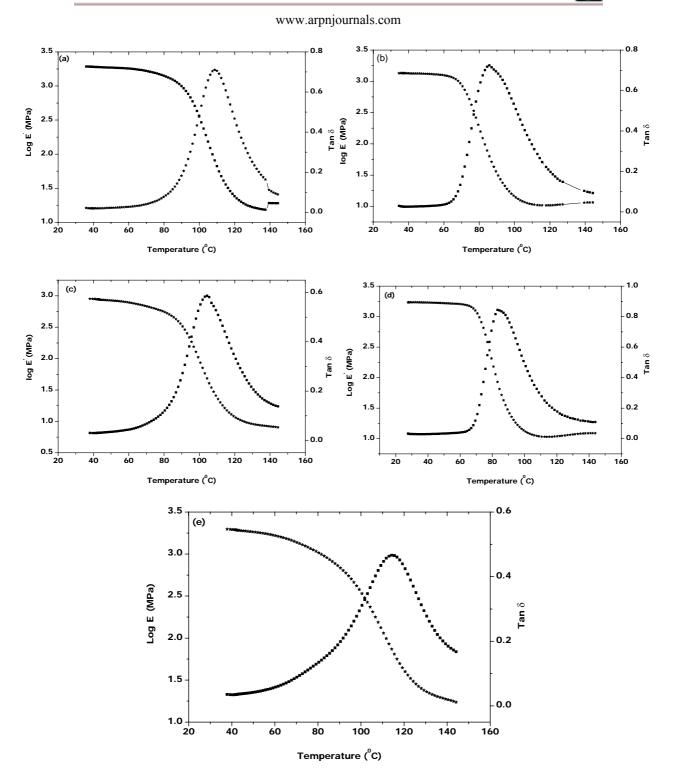


Figure-9. The variation in elastic modulus and Tan δ with temperature for (a) 0 %, (b) 2.5 %, (c) 5 %, (d) 7.5 % and (e) 10 % UHMWPE content in epoxy blend.

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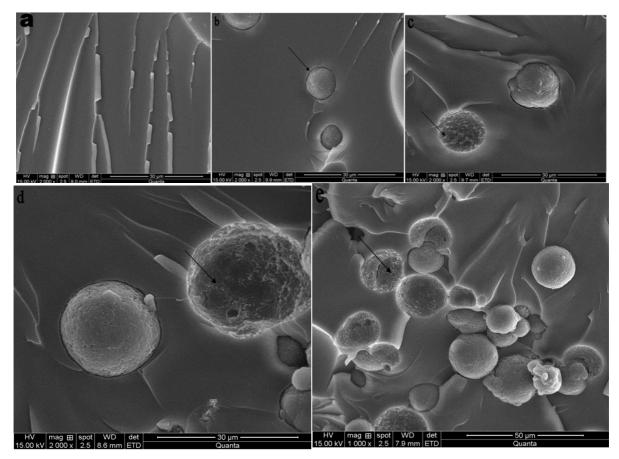


Figure-10. SEM images of (a) 0 %, (b) 2.5 %, (c) 5%, (d) 7.5% and (e) 10% UHMWPE loaded epoxy blends.

4. CONCLUSIONS

Epoxy/UHMWPE blends have been prepared by method. in-situ polymerization The prepared epoxy/UHMWPE blends show significant improvement in impact strength and chemical resistance. The impact strength of epoxy increased from 12 to 17.4 kJ/m² with increase in UHMWPE content from 2.5 to 10 %. The tensile strength of blends continually decreased with increasing of UHMWPE content due to no epoxy -UHMWPE tethering. However, improvement in tensile modulus and surface hardness with increase in UHMWPE content in epoxy matrix was noticed. TGA thermograms indicates that all epoxy blends are stable upto 233 °C and undergo one step thermal degradation in the temperature range 233-528°C. Kinetic parameters of thermal degradation were evaluated by using Coats-Redfern and Broido methods. Kinetic studies reveal that the activation energy calculated by two methods is comparable. Lowest activation energy values were observed for blends as compared to neat polymer. The DSC and DMA data exhibited that the dispersed UHMWPE in the polymer matrix led to a slight increase of T_g. The results indicate that UHMWPE concentration affects the state of dispersion, which has a dominating influence on the thermal and mechanical properties.

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