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Kinetic of Thermal Degradation of RTV Silicon Rubber Blends Reinforced With Nanoparticles by TGA and DSC Analysis Techniques

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Abstract

Binary and ternary polymeric blends from epoxy (EP), unsaturated polyester (UPE), with silicone rubber (room temperature Vulcanizes (RTV)) were prepared. Silicone rubber (SR) was mixed well with sufficient amount of ethanol before its adding to resins. These blends were reinforced by nano Al with 1% wt, and Al_2O_3 with 1% wt to develop the properties of blends. TGA and DSC were obtained from the thermal degradation computed by using Coast-Redfern technique. Kinetic and thermodynamic parameters were studied for all specimens were presented a good linear correlation coefficient close to unity using Minitab 16. DSC profiles for all composites shows there are a single endothermic peak which represents the glass transition temperature. Activation energy of binary and ternary epoxy blends decreases with increasing the percentage of SR while the addition of Al and Al_2O_3 nanoparticles shows an increase in the activation energy when added to binary and ternary blends for epoxy.

Keywords: Thermal decomposition; Al_2O_3 nanoparticles; binary blend; RTV silicone rubber.

1. Introduction

It is obvious that silicone rubber has a very good thermal stability toward higher temperature and it found its way in using as electrical Insulation, medical application. It has a good resistance toward chemicals and lubricating oils as well as water [1-2]. Silicon rubber considered as a highly cross linking polysiloxane with network of three dimensional which prepared by polycondensation polymerization. However, it can be used for long period of time below 250 C°, however this is because of the energy bond of Silica-Oxygen is 460.5 kJ/mol which is much higher than energy bond of Carbon-Oxygen 358 kJ/mol and Carbon- Carbon bond 191 kJ/mol [3], for this reason it considered as a high duty in using as high temperature adhesives also as a protective material used as a seal in internal combustion engines [4-5]. Epoxy resin also has a great resistance to chemicals and excellent electrical and mechanical property. Epoxy blended with nano carbon to improve its property in order it can be used in airplanes, building and automobiles [6-8]. Unsaturated polyester it is a material of a wide use for produce of polymer composite because of easy to manufacture and competitive in properties [7], and used in different area including automotive filed, construction, and electrical application and coating [8-9], because it has good mechanical property, good corrosion resistance, and low weight [10]. Degradation and thermal stability of polymers is a very important from the point of view of scientific and industrial field therefore thermal degradation must be well understood which in turn give guarantees of a long service life of the product and leads to good development of materials that increases thermal stability [11].

Development of polymers having a superior thermal property is the aim of many researchers, therefore seeking a suitable technique using either enforcements of organic and inorganic nano particles or modifying by introducing functional groups. Both of them had used to alter the performance of the polymer completely by enhancing it at high temperature environment [12]. However, enhancing

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performance promoted many investigators to use nano particles that should be uniformly dispersed in the polymer avoiding the agglomeration that effects adversely on improvement. More recently nano silica particles [13], nano alumina particles [14] and nano poly methyl methacrylate particles with natural particles [15], more recently a modification by introducing functional groups of trifluorovinyl ether which contributed in the enhancement of thermal stability under nitrogen and air environments. poly(dimethylsiloxane) has thermal stability up 300 C this stability significantly improved with addition methyl phenyl or diphenylsiloxane up to 400C. Thermal degradation at elevated temperature under nitrogen environment was mainly back biting reaction because of the residual Silicon-Hydroxyl groups while after the second stage the structure significantly damaged [16-20]. According to a recent study, hydroxyl-terminated PDMS depolymerization mainly occurs from the end of chains at moderate temperatures with low activation energy adding POSS which react with the hydroxyl-terminal which eliminate back biting depolymerization [21-24]. Stability is protecting of polymer from decomposition when it faces higher temperature.

2. Kinetic Theory

The degradation kinetic of was achieved by using the following equation [23]:

$$d\alpha/dT = k(T)\, f(\alpha) \quad \text{.....(1)}$$

Where α is the fractional disappearance of the polymer and measured continuously by TGA. k(T) is rate constant and f (α) represent the chosen reaction model which describe the decomposition reaction. k(T) is the Arrhenius equation.

$$d\alpha/dT = A \exp(-E/RT)\, f(\alpha) \quad \text{.....(2)}$$

E represent the activation energy. However, in the present work Coats-Redfern reaction model has been employed to study the kinetics of the extent of decomposition. This model is considered a multi-heating rate [25] employed in determination of kinetic paramerters of activation energy and frequency factors under diffusion control and so called Janders:

$$\ln [g(x)/T^2] = \ln [AR/\beta E] [1-(2RT/E)] - (E/RT) \quad \text{..(3)}$$

$$g(x)=[1-(1-\alpha)1/3]^2 \quad \text{.....(4)}$$

Where (1-α) represent the fraction of remain, β is the used heating rate, A is frequency factor, R is gas constant, T is decomposition temperature and E is activation energy.

Plotting ln $g(x)/T^2$ against 1/T for each heating rate produce a straight line of slope of (–E/R). Frequency factor directly determined from intercept at Y axis:

$$\text{Intercept}=\ln[AR/\beta E][1-(2RT/E)] \quad \text{.....(5)}$$

The entropy of activation is defined as the difference between the entropy of the transition state and the sum of entropies of the reactants. Free energy of change of decomposition is the driving force of a chemical reaction and hence is the difference between the enthalpy of the transition state and the sum of enthalpies of the reactants in the ground state. The Thermodynamic parameters: entropy of activation (ΔS), enthalpy of activation (ΔH) and Gibbs free energy (ΔG) were calculated using Equations (6)-(8).

$$A = (kT/h)\, e^{(\Delta S/R)} \quad \text{.....(6)}$$

where A is frequency factor, h and k are Planck's constant and Boltzmann constant respectively.[26]

$$\Delta H = E - RT \quad \text{.....(7)}$$

$$\Delta G = \Delta H - T\,\Delta S \quad \text{.....(8)}$$

3. Experimental

3.1. Materials

Epoxy (EP) resin (Sikadur-52 from Sika Australia Pty Limited Company) was used in this research. The mixing ratio for resin and hardener is A: B (2: 1), Unsaturated polyester resin (UPE) is a liquid with moderate viscosity which can be cured to the solid state by adding (Methyle Ethyle Keton Peroxide, MEKP) as a hardener, while cobalt octoate acts as a catalyst to accelerate the solidification process. The percentage of the hardener to the resin is (2%), while it is (0.5%) for accelerator, SUPERMAX universal silicone sealant RTV made in U.A.E. was used in this research, it is a liquid form of adhesive. Typically, it looks, feels, and acts like a gel. Aluminum Oxide (Al_2O_3) nano pawder (Alpha-Al_2O_3, 99.0%) purchased from Skyspring Nano materials, Inc. USA, with average diameter of 40 nm, appearance: White Powder, a specific surface area of 60m^2/g. Aluminum (Al) nano powder (Purity: 99.0%) purchased from Hongwu International Group Ltd USA, with average diameter of 40 nm, appearance: Black Powder, a specific surface area of ~60m2/g.

3.2. Instruments

The Differential Scanning Calorimetry (DSC) test was performed according to (ASTM D3418) using thermal analysis instrument (DSC), thermal decomposition carried out at constant heating rate of 10 Co/min in inert atmosphere between 35-650 Co.

3.3. Methods

3.3.1. Preparation of Polymer Blends with Ethanol

(EP 93% + SR 7%) and (EP 80% + SR 20%) for EP binary blends were selected for investigation as shown in **Table 1**. The silicone rubber was mixed

well with sufficient amount of ethanol till it looks like gel form and convert to the white color, in order to ensure a homogeneous mixture because both silicone rubber and epoxy are sticky. Epoxy resin was added with different weight ratios 7 and 20% of silicone rubber. The hardener ratio for all samples has been fixed when the weight ratios of the silicone rubber were added. The samples were cast with hand lay-up technique in Teflon mold. The mixture was vacuumed from bubbles for 30 mins and cured at 50˚C for 3hrs, then left at room temperature for 24 hours in the molds. After that the blends were removed from the Teflon molds and cured further at temperature 50°C for (4) hours in an oven for heat treatment.

(UPE 97% + SR 3%) and (UPE 93% + SR 7%) for UPE binary blends were prepared by the same manner. Also (EP 94% + UPE 3% + SR 3%) and (EP 60% + UPE 20% + SR 20%) for EP ternary blends were prepared by the same method of binary blends preparation.

3.3.2. Preparation of Binary and Ternary Blends Nanocomposites

The nanoparticles were preheated at 100 ºC for 2-3 hours to remove moisture content. EP or/and UPE resins according to ratios chosen of blends with a weight ratio (1%) of Al or Al_2O_3 nanoparticles mixed together in a suitable glass beaker heated up to ٧0 ºC by an oil bath with the help of proper mechanical stirring 1h, at this stage resulted better dispersion of nanoparticles. Then, the mixture was cooled to room temperature and the sealant silicone rubber was added with the fixed different ratio for all samples. Before that, the silicone rubber was mixed well with a sufficient amount of ethanol till it looks like a gel form and converts to the white colour, in order to ensure a homogeneous mixture. Then the total mixture was placed in a high intensity Ultra-sonicator for about 30 mins. To avoid temperature, rise during the sonication process, external cooling system i.e cool water was circulated around the beaker. After that the hardener was added to modified epoxy mixture (100%) and mixed thoroughly by mechanical stirring (10 minutes), then using vacuum system (10-2 bar) to remove the bubble before molding and finally left at room temperature for 24 hours for curing, and was cast in an oven for 1 hour at 50 °C for post curing, it was left for 48 hours before pulling out from molds and left for 7 days before any test to get better curing conditions and kept then in vacuum chambers.

4. Results and Discussion

4.1. Thermogravimetric Analysis

TGA and DSC is an important tool to study the kinetic of thermal decomposition, to describe the way where the polymer degrades and to determine the melting point and glass transition temperature and to study the thermodynamics of thermal and to demonstrate the stability.

Thermal decomposition trends are shown in **Figures (1, 2 and 3)** for silicon rubber, epoxy and unsaturated polyester respectively. In case of silicon rubber, its clearly that the weight loss of reaction occurs in two stages, however the first the weight loss 46.8183% and in the second 40.2179%, which is clearly shown in **Figure 1**. It can be seen that the first stage hydration occurs and caused a weight loss. Water is detected at 117 Cº and begin to evaporates with volatiles and this process ends at 365.217 Cº, the second weight loss occurs due to thermal decomposition which leads to chains scission of the elastomeric material which ends at 593.029 Cº. DSC profile shows single endothermic peak which represents the melting temperature equal to 142.9 Cº.

In case of epoxy, the weight loss in two stages [22]. In first stage water detected at 129.6 Cº end at 320.84 Cº, all volatiles and water vapor are eliminated at this stage then followed the second stage which start at 320.84 °C and ends at 594.85 °C, however this stage considered the main stage which losses occurs mainly in it about 70.5464% due to polymer backbone scission occurs randomly converting the mass to the char then convert to gas.

DSC profiles shows four peaks, first endothermic represent the glass transition temperature and equal to 119 °C and three exothermic peaks first crystallization at 338.1 Cº and the two others represent the thermal decomposition of epoxy at 466.5 Cº and 570.7 Cº respectively as shown in **Figure 2.**

Table 1. The Mixing Ratios (MR) of SR in EP and UPE.

Sample No.	Blends	Mixing Ratios (MR)
4A	EP/SR	(EP 93% + SR 7%)
6A	EP/SR	(EP 80% + SR 20%)
2B	UPE/SR	(UPE 97% + SR 3%)
4B	UPE/SR	(UPE 93% + SR 7%)
2C	EP/UPE/SR	(EP 94% + UPE 3% + SR 3%)
6C	EP/UPE/SR	(EP 60% + UPE 20% + SR 20%)

Unsaturated polyester thermal decomposition mainly in single stage [23]. Almost at 59.9C° starts to weight loss and gradually ends at 445.825C° with weight loss of 93.866%. The residual solid and covered to gaseous products is at 594.63C°. DSC profiles four peaks, first endothermic glass transition temperature equal to 103C°, second crystallization was at almost 275C° followed two clear peaks which represent the thermally decomposition of the polymer at 407.8C° and 546.8C° respectively as shown in **Figure 3**.

By using Coats-Redfern equation (3) to determine the kinetic parameters of activation energy and frequency factor by plotting $\ln(x)/T^2$ against 1/T which gives a linear relationship with a high correlation R^2 close to 100%, as shown in **Figure 4** to **Figure 13.**

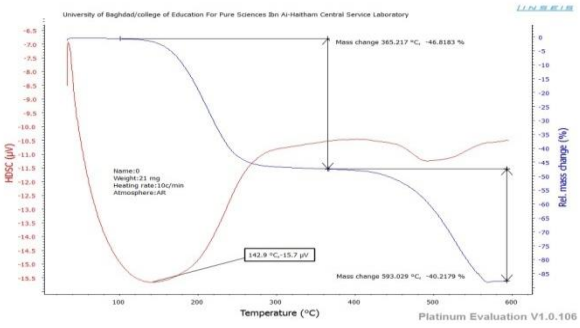

Figure 1. TGA and DSC profiles for silicon Rubber (SR) at heating rate 10 C°/min.

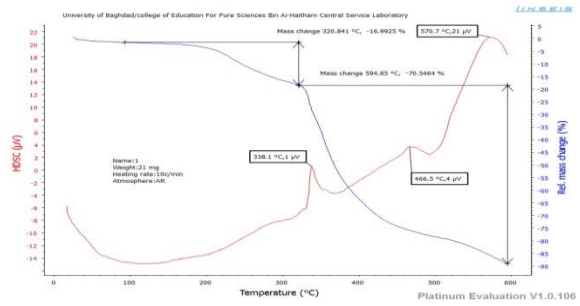

Figure 2. TGA and DSC profiles for epoxy (EP) at heating rate 10 C°/min

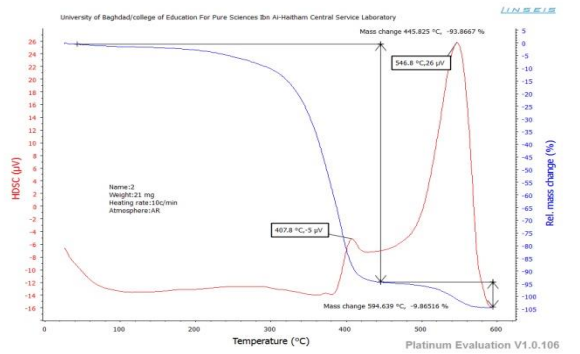

Figure 3. TGA and DSC profiles for unsaturated polyester (UPE) at heating rate 10 C°/min.

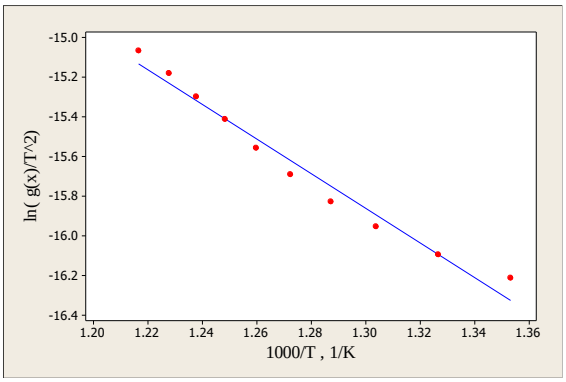

Figure 4. A plot of thermal decomposition of SR utilizing coats-redfern method.

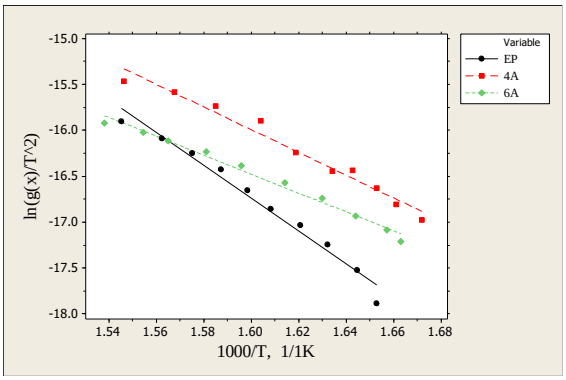

Figure 5. A plot of thermal decomposition of EP, 4A, 6Autilizing coats -redfern method.

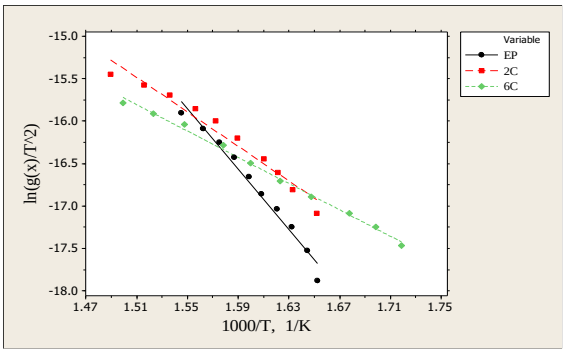

Figure 6. A plot of thermal decomposition of EP, 2C, 6C utilizing coats -redfern method

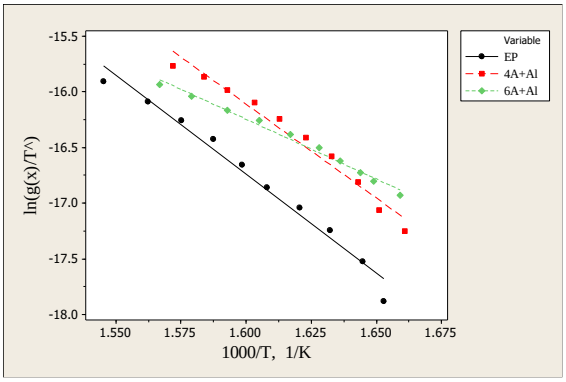

Figure 7. A plot of thermal decomposition of EP, 4A+Al, 6A+Al utilizing coats -redfern method.

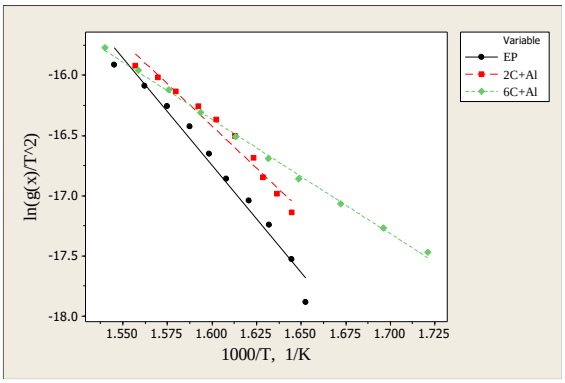

Figure 8. A plot of thermal decomposition of EP, 2C+Al, 6C+Alutilizing coats -redfern method.

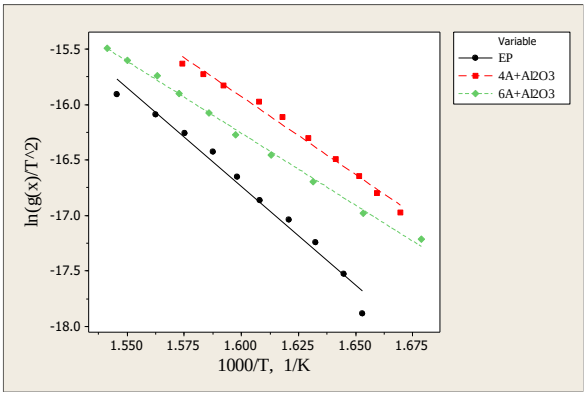

Figure 9. A plot of thermal decomposition of EP,4A +Al_2O_3, 6A+Al_2O_3 utilizing coats –redfern method

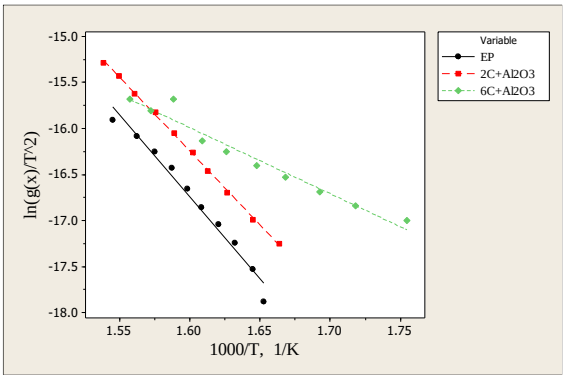

Figure 10. A plot of thermal decomposition of EP, 2C +Al_2O_3, 6C+Al_2O_3 utilizing coats -redfern method

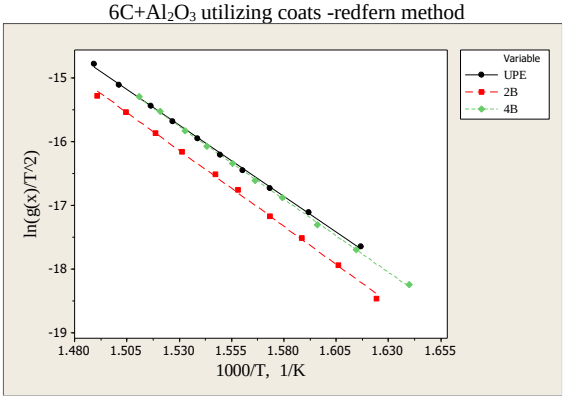

Figure 11. A plot of thermal decomposition of UPE, 2B, 4Butilizing coats -redfern method

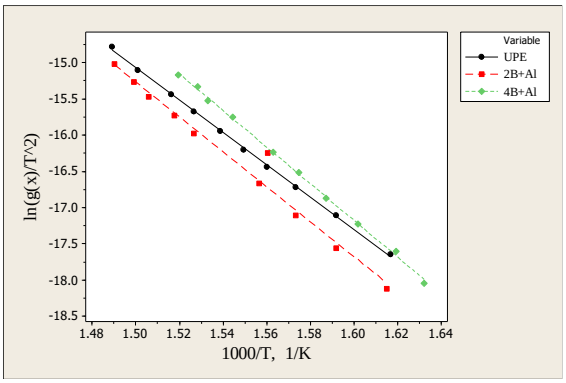

Figure 12. A plot of thermal decomposition of UPE, 2B+Al, 4B+Al utilizing coats -redfern method

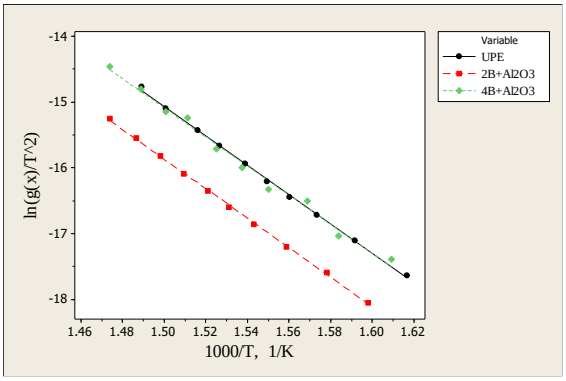

Figure 13. A plot of thermal decomposition of UPE, 2B+Al_2O_3, 4B+Al_2O_3 utilizing coats -redfern method

4.2. Kinetics Data and Thermodynamic Parameters

The experimental results of kinetics data and thermodynamic properties for the polymers and its composites are determining by using the equations (6-8) as shown in **Table 2** and **Table 3** respectively.

It can be seen the activation energy of blends 4A and 6A decreases with increasing the percentage of SR, this is because of silicon rubber acts as softening media which contribute in decreasing the rigidity of epoxy chain network, therefore glass transition temperature decreases.

The polymers are completely miscible since a single glass temperature obtained for all blends used in the study. The activation energy of 2C and 6C shows a decline, since the addition of UPE was on account of a decrease of EP content and SR content was invariant.

The effect of Al addition in 4A+Al and 6A+Al composites shows a decrease since the stability increase in the first and in the second seem the catalyzing action occurs besides to higher percentage of SR in the second, while, in comparison with 4A and 6A the activation energy is much higher since thermal stability increased by addition of Al particles which mixed homogeneously with others so increasing the rigidity as well and the glass temperature also increases.

In case of 2C+Al and 6C+Al composites, the activation energy decreases with increasing the

percentages of UPE from 3% to 20%, it seems effecting on epoxy network contributing in a decrease the attraction force between network epoxy chains, therefore activation energy decreases at higher temperature, while this effect seems to be absent at lower temperature because the glass temperature increases with increasing the percentages of UPE in the composite.

The effect of addition Al_2O_3 seems it enhances the values of activation energy when a comparison was made between 4A with 4A+Al_2O_3 and 6A with 6A+Al_2O_3 since alumina nanoparticles were considered a refractory material which resists higher temperature therefore contributes in increasing activation energy. The same attitude observed between 2C with 2C+Al_2O_3 and 6C with 6C+Al_2O_3 composites. For composites 2C+Al_2O_3 and 6C+Al_2O_3 comparison with 4A+Al_2O_3 and 6A+Al_2O_3 values of glass transition temperature increases with addition of UPE to the composite it seems contributing in enhancement of rigidity. Besides it retains heat more since alumina considers ceramic material and its thermal conductivity much lower than aluminum.

In case of UPE and its blends 2B and 4B, it seems that the addition of SR enhances the thermal stability which leads to increase the activation energy and glass transition temperature.

The same attitude observed in 2B+Al and 4B+Al composites.

The highest increasing in thermal stability and glass transition temperature observed when alumina were added as it can be observed in composites 2B+Al_2O_3 and 4B+Al_2O_3, since alumina contribute in rigidity by increasing the attraction force in the network chain of composites.

Total enthalpy of decomposition increases with addition Al nanoparticles as compared between 4A with 4A+Al and 6A with 6A+Al, however these nano particles seem to be retained heat evolved since there is no enough time to dissipating heat, therefore the magnitude of entropy increases.

The same attitude observed when comparison was made with 4A+Al_2O_3 and 6A+Al_2O_3. Also, comparison of 2B with 2B+Al and 4B with 4B+Al shows that the value of enthalpy and entropy increases for the same reason mentioned.

Table 2: The kinetics data for thermal decomposition process

Groups	Sample No.	Glass Transition Temp., C°	Activation Energy, E,(KJ/mol)	Frequency Factor, A (S^{-1})	R^2
Pure	**SR**	-	72.3318	$5.9480x10^{2}$	97.3%
	EP	119.0	148.6543	$3.969x10^{8}$	97.7%
	UPE	102.4	186.3998	$2.0379x10^{13}$	99.9%
Blends	**4A**	116.0	103.3430	$1.0179x10^{5}$	97.2%
	6A	101.4	85.88361	$1.8193x10^{3}$	98.8%
	2B	102.8	190.9725	$1.0088x10^{12}$	100%
	4B	104.0	198.4551	$2.8496x10^{12}$	99.8%
	2C	118.0	84.63650	$1.8781x10^{3}$	96.3%
	6C	120.2	64.54151	$2.1822x10^{1}$	99.6%
Blends +Al1%	**4A+Al**	121.0	141.0885	$1.7795x10^{8}$	97.1%
	6A+Al	123.5	89.79121	$5.7378x10^{3}$	98.9%
	2B+Al	120.2	200.8662	$5.0250x10^{12}$	97.2%
	4B+Al	123.5	210.6767	$5.8965x10^{13}$	99.9%
	2C+Al	125.6	116.3128	$6.9027x10^{5}$	97.1%
	6C+Al	169.5	79.24075	$6.0304x10^{2}$	99.8%
Blends+$Al_2O_3$1%	**4A+ Al_2O_3**	125.1	117.4768	$1.8783x10^{6}$	98.9%
	6A+ Al_2O_3	127.0	108.3314	$2.1346x10^{5}$	99.6%
	2B+ Al_2O_3	143.5	186.6493	$2.9047x10^{11}$	99.9%
	4B+ Al_2O_3	146.4	187.1481	$2.2058x10^{11}$	99.1%
	2C+ Al_2O_3	126.1	134.1048	$3.8542x10^{7}$	97.7%
	6C+Al_2O_3	130.3	110.8774	$1.3803x10^{6}$	95.9%

Table 3. Thermodynamics parameters of thermal decomposition

Groups	Sample No.	ΔH, KJ/mol	-ΔS,J/mol	ΔG,KJ/mol
Pure	**SR**	65.7376	203.9209	227.4469
	EP	143.3543	86.6108	225.7283
	UPE	181.0774	4.8993	184.2132
Blends	**4A**	98.1498	163.5294	200.2885
	6A	80.6783	196.9760	203.9813
	2B	193.1069	21.1785	206.7293
	4B	185.6174	29.8249	204.8247
	2C	79.3905	178.0560	171.6348
	6C	59.2679	234.2521	207.8307
Blends +Al1%	**4A+Al**	135.9270	101.3741	198.8519
	6A+Al	84.5275	187.5241	203.2396
	2B+Al	195.4283	16.5836	206.2740
	4B+Al	205.2235	4.4287	208.1278
	2C+Al	111.0406	147.7110	204.6894
	6C+Al	73.9436	206.2931	205.3523
Blends+$Al_2O_3$1%	**4A+ Al_2O_3**	112.2877	139.2567	199.1839
	6A+ Al_2O_3	103.0755	157.4440	159.6121
	2B+ Al_2O_3	181.6438	42.6937	209.9079
	4B+ Al_2O_3	181.2196	40.2914	207.5299
	2C+ Al_2O_3	128.8417	113.5753	200.7236
	6C+ Al_2O_3	105.5194	237.3492	207.4578

5. Conclusions

Addition of SR to EP tend to decline of glass transition temperature and activation energy as in 4A and 6A. UPE tend to enhance glass transition temperature and some decline activation energy as in 2C and 6C. Addition of Al particles as in 4A+Al, 6A+Al improved glass transition temperature and increase the activation energy as compared with 4A and 6A.UPE blends and all its composites showed improvement in activation energy and the glass transition temperature. DSC profiles for all composites showed there is a single endothermic peak

which represents the glass transition temperature; therefore, it will be concluded the all ingredients of composite are completely miscible and this due to effective blending.

6. Conflicts of interest

"There are no conflicts to declare".

7. Formatting of funding sources

Self

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