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Epoxy-Al₂O₃ nanocomposites with enhanced T_g and thermal stability

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ABSTRACT

An extensive range of nanoparticle loadings, between 0.5 wt% to 20 wt%, were investigated in detail for assessing the thermal stability of epoxy-Al₂O₃ nanocomposites. Alumina nanoparticles were distributed uniformly throughout the viscous epoxy system with the assistance of an optimized ultrasonic dual-mixing method. The influence of nanoparticle cluster size and interparticle distance on the glass transition temperature (T_g) and thermal stability of the nanocomposites was examined in this study. Nanoparticle dispersion in epoxy was characterized using atomic force microscopy (AFM). Thermogravimetric analysis was utilized to investigate the thermal stability of nanocomposites, while differential thermal analysis was employed to estimate T_g . It was discovered that a key threshold loading concentration of nanoparticles such as 3 wt% in epoxy was found responsible for significant increase in T_g as well as the thermal stability. This type of improvements is ascribed to the advancement of a strong epoxy-nanoparticle interface, which is facilitated by dispersion of nanoparticles such as thermal stability. This type of heat through the matrix.

KEYWORDS: Epoxy; Al₂O₃; T_g; Ultrasonic dual mixing; Thermal stability.

1. Introduction

Modern society has a strong need for improved materials, which is crucial for the development of new products. To

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address this difficulty, new materials are being developed daily. The potential use of nanoparticles as fillers in the polymeric composites has been the subject of numerous investigations in recent years [1–5]. Many various forms, shapes, and features of nanofillers have been studied, such as graphenes [6], MXenes [7], carbon nanotubes [8,9], spherical nanoparticles [1,10–16], and nanoclays [17]. Owing to their large aspect ratio, one- and two-dimensional materials such as MXenes, carbon nanotubes, and graphene have become popular fillers for several polymers [6,7,9,18–21]. But these materials with high conductivity prevents them from being used in situations where electrical insulation is needed. Furthermore, it is difficult to disperse such high aspect ratio nanostructures uniformly in viscous polymers like epoxy. In such cases, functional materials are also needed, which adds complexity and expense to the process and makes it challenging to implement at an industrial level. Another significant problem that limits these materials use in a variety of industries is their inherent toxicity. But when it comes to ceramic nanoparticles like titania, alumina, zirconia, etc., these problems are essentially non-existent. Because of aspect ratio (~ 1) , these nanoparticles are inexpensive, easily obtainable, and less difficult to disperse in viscous polymers. Still, Al₂O₃ nanoparticles are thought to be the best material to use for reinforcing epoxy-like polymers [22–26]. Agglomeration is frequently caused by strong van der Waals interactions among nanoparticles. So, ultrasonic dual mixing is the best technique for guaranteeing uniform dispersion of nanoparticles in viscous polymers such as epoxy [27].

Al₂O₃ nanofillers are added to epoxy nanocomposites to improve their thermal stability as well as glass transition temperature (T_g) [19,23,28]. However, a comprehensive analysis of epoxy-Al₂O₃ nanocomposites' thermal stability has not yet been investigated. The current project's goal is to assess the thermal stability of epoxy-Al₂O₃ nanocomposites by a systematic study involving a wide range of nanoparticle loading into epoxy, from 0.5 wt% to 20 wt%. An efficient ultrasonic dual mixing was applied for dispersing nanoparticles in viscous epoxy.

2. Experimental

Based on Loctite E60-HP, a two-component epoxy, was obtained from Loctite Hysol, USA, and consisted of an aliphatic polyamine-derived hardener and epoxy resin made from diglycidyl ether of bisphenol-A. We purchased methyl ethyl ketone (MEK) from Sigma Aldrich in India. The Al_2O_3 nanoparticles' average size was about ~ 10 nm, supplied from American Elements, United States.

Alumina nanoparticles containing 0.5–20 weight percent were incorporated into epoxy by employing an ultrasonic dual mixing (UDM) [27]. The detailed information about UDM process is reported in another published work [27]. MEK was mixed into 3-fold volume of epoxy resin to cool down the mixture and help in reducing viscosity of the epoxy resin. The epoxy resin and MEK mixture were thoroughly mixed with the maximum concentration of Al₂O₃ nanoparticles (20 wt%) using an impeller with four blades running at a high-speed of 2000 rpm for 30 min. After that blend was processed for 1 h using UDM that included an impeller and an ultrasonic vibrator (Vibracell ultrasonic processor, Sonics & Materials, Inc.). Afterwards, the MEK was extracted from the mixture by heating it to 70 °C while maintaining a high vacuum [29]. In accordance with the technical data sheet of the material supplier, an aliphatic polyamine-based curing agent (or hardener) was mixed with the epoxy resin consisting of alumina nanoparticles. The mixture was then blended using an impeller at a very low speed of 300 rpm for a short duration of 10 min and the trapped air was removed using high vacuum. After that, the substance was cast into a petri dish coated with Teflon and allowed to cure for 24 h at room temperature.

The phases of alumina nanopowder and epoxy nanocomposites were identified by X-ray diffractometer (XRD) utilizing a Bruker D8 Advance Diffractometer that was run at 40 kV of voltage and 40 mA of current under 1.54 Å wavelength Cu-Kα radiation. There was a 2 s dwell period for each step. Before they were analyzed, the nanocomposites were ground into a fine powder. Fourier transform infrared (FTIR) spectroscopy was used to evaluate the samples' molecular structure properties. Specimens were originally ground into a fine powder. The powder was then combined with potassium bromide (KBr, Aldrich 99.9%) and compressed in a die with a load of 10 tons to produce a thin film for infrared examination. Using an FTIR spectrometer (Thermo Nicolet Nexus 1600), the infrared spectra were captured under ambient circumstances. Atomic force microscopy (AFM) in tapping mode was used to analyze the microstructure of cured epoxy nanocomposites (NT-MDT, Ntegra). Using AFM pictures of the nanocomposites, the distribution of nanoparticles as well as their cluster sizes in epoxy matrix were investigated with the help of Image] software [18]. The nanocomposites from the bulk samples were chopped into ~ 3 mm pieces and characterized using a thermogravimetric analyser (TGA) and differential thermal analyser (DTA) (Perkin Elmer, Pyris Diamond). For the thermal measurements, a highly pure alumina powder (20 mg) was utilized as the reference material. TGA thermograms were used to measure the samples' thermal stability. For TGA analysis, each sample, which weighed around 20 mg, was heated at a rate of 10 °C per min between 30 and 700 °C using a N₂ purging gas flow rate of 200 ml/min. The glass transition temperature (T_g) from DTA analysis was determined in accordance with ASTM E1356 standard [18]. A heating rate of 10 °C/min and an operational temperature range of 30 to 300 °C with N₂ purging gas were employed for the DTA measurements. At least three repetition tests were performed for each composition, and the mean values and standard deviation are reported.

3. Results and Discussion

3.1. Structural analysis

XRD pattern of Al₂O₃ nanoparticles (**Fig. 1**) shows a broad hump around 22° instead of any sharp peaks, which indicates no crystallinity, only their amorphous nature. The XRD pattern of the neat epoxy and epoxy-Al₂O₃ nanocomposites is exhibited in **Fig. 2**. The XRD pattern of the neat epoxy also exhibits amorphous nature as revealed by the broad hump around 19° instead of any sharp peaks. The loading of nanoparticles from 0.5 wt% to 20 wt% into the epoxy does not reveal any notable change in XRD pattern which makes it difficult to detect the presence of nanoparticles. Even with the large loading of the nanoparticles, the pattern shows no new peaks, indicating that the epoxy's structure has not changed.



Fig. 1. XRD pattern of Al_2O_3 nanoparticles.

To detect any possibility of chemical interaction between alumina nanoparticle and epoxy that may create new chemical bonds is characterized by FTIR. An FTIR spectrum of alumina nanoparticles is depicted in Fig. 3. The broad band between 630 and 755 cm^{-1} in the Al₂O₃ nanoparticle spectrum represents a distinctive absorption band of transition alumina. The stretching vibration of the Al-O-Al bond is truly responsible for this band's expansion, and the distribution of vacancies among the octahedral and tetrahedral sites causes the Al-O vibrational frequencies to spread out [30,31]. There are bands at the surface caused by chemisorbed and adsorbed substances in addition to alumina bands. The superposition of vibration bands of isolated OH groups, bound hydroxyl groups, and stretching vibrations of adsorbed water molecules results in the very large band with a centred at 3456 cm⁻¹ [30, 32]. All these bands are overlaid under normal circumstances, making resolution challenging.

The bending of molecular water is the cause of the band at 1632 cm⁻¹. The existence of peaks at 3456 and 1632 cm⁻¹ [31] indicates that the nanoparticle's surface unquestionably contains OH groups. The OH group can be found on the particle's surface as free OH groups or absorbed water (H₂O) molecules as OH groups, or on both [32, 33]. The peak assignment of FTIR spectra of neat epoxy is represented by alphabets 'A' to 'P'. The peaks in FTIR spectra at 3418, 3060, 2924, 2360, 1727, 1608, 1510, 1461, 1360, 1290, 1250, 1182, 1107, 1036, 828, 730 and 698 cm⁻¹, respectively [30,32,34] are corresponding to O-H stretching of hydroxyl group, C-H vibration of epoxy ring, CH3 methyl group vibration, atmospheric CO₂ vibration, aromatic C=O stretching, aromatic C=C stretching, aromatic ring stretching, CH₃ asymmetrical bending, CH₃ symmetrical bending, epoxy ring mode: the C–C, C–O and C–O bonds all stretching in phase, CO vibration of the epoxy ring, C-N stretching, C-O-C stretching, aromatic C–O stretching, aromatic ring bent out of plane and doublet due to aromatic ring vibration, respectively.

Fig. 4 depicted the FTIR spectra of epoxy-Al₂O₃ nanocomposites. In contrast to the neat epoxy, no new peak forms when 0.5–2 weight percent of Al₂O₃nanoparticles are added to the epoxy. This suggests that no new chemical bond between the nanoparticle and epoxy is formed. In the FTIR spectra of epoxy-Al₂O₃ nanocomposites, the band centered at 3418 cm⁻¹ which is because of the O-H stretching of hydroxyl group present in molecular structure of epoxy displays shifting towards lower wavelength in the spectra while there is no change in the rest of the spectra. This shifting results from a bonding between the neat epoxy and the hydroxyl group found on the surface of Al₂O₃ nanoparticles (**Fig. 3**).



Fig. 2. XRD pattern of epoxy-Al₂O₃ nanocomposites.







Fig. 4. FTIR spectra of epoxy-Al₂O₃ nanocomposites.

3.2. Morphology of epoxy-Al₂O₃ nanocomposites

Fig. 5 depicts the AFM images of epoxy-Al₂O₃ nanocomposites having 3, 5, 10, and 20 wt% of nanoparticle content. It has been noted that many of the nanoparticles are uniformly distributed as individual particles in the case of nanocomposite containing 3 wt% nanoparticle with nominal clustering (**Fig. 5a**). While for the nanocomposite containing 5 wt% nanoparticle content, only limited particles are appearing as individual particles whereas most of the

particles are in clustered form (**Fig. 5b**). The clustered nanoparticles are indicated by dotted circles while the individual nanoparticles are designated by the solid circles. However, further increase in nanoparticle content in epoxy up to 10 wt% and 20 wt% leads to significant clustering (**Fig. 5c and 5d**). Thus, for Al₂O₃ nanoparticles (~ 10 nm), the threshold limit for uniform dispersion into epoxy is 3 wt%. However, beyond this limit, an optimized UDM does not seems effective enough to break the cluster of particles into epoxy.



Fig. 5. AFM images of epoxy-Al₂O₃ nanocomposites consisting nanoparticle content of (a) 3, (b) 5, (c) 10 and (d) 20 wt%.

concentration nanoparticles The of versus average cluster size is shown in **Fig 6.** When epoxy encompasses nanoparticles at concentrations between 0.5 to 3 wt%, there is a slight increase in cluster size equivalent to a twofold rise in cluster size. However, with the further increase in the concentration of nanoparticles, the cluster's size grows almost exponentially. Based on both theoretical calculations and experimental data, Fig. 7 shows the nanoparticle concentration vs average inter-particle distance. The theoretical inter-particle distance was calculated using a theoretical equation based on cubic distribution of particles in a polymer [35]. The detail about the theoretical equation is available in earlier published work [36]. The theoretical value for inter-particle distance is marginally smaller than the one that is measured experimentally due to presence of some form of clusters of nanoparticles for every concentration. As the concentration of nanoparticles in epoxy grows, it is clear that the space between particles decreases.

The experimental and theoretical average inter-particle distance for threshold limit of nanoparticle (3 wt%) dispersion into epoxy are \sim 35 nm and \sim 30 nm respectively, which can limit the polymer chain's mobility effectively and can affect the thermal properties of the nanocomposites in a significant manner.



Fig. 6. Nanoparticle content versus average cluster size of epoxy-Al_2O_3 nanocomposites.



Fig. 7. Nanoparticle content versus average interparticle distance of epoxy-Al_2O_3 nanocomposites.

3.2. T_{g} of epoxy-Al₂O₃ nanocomposites

DTA curves representing Tg of epoxy-Al₂O₃ nanocomposites for raising the nanoparticle content from 0.5 to 20 wt% are displayed in Fig. 8. However, Fig. 9 shows the Tg trend (with standard deviation). As the nanoparticle loading increases from 0.5 to 3 weight percent, the nanocomposite's T_g continues to rise but beyond this limit it shows monotonic decrease in the Tg value up to 20 wt% of particle content. The maximum increase in Tg of the epoxy-Al₂O₃ nanocomposite with threshold limit of 3 wt% particle content is about $\sim 28\%$ in comparison to that of the neat epoxy. Fig. 10 depicted the effect of cluster size on Tg of the epoxy-Al2O3 nanocomposites. When cluster sizes rise from 12 to 29 nm, the T_g of the nanocomposite is seen to continue rising; however, at larger cluster sizes (48 to 150 nm), a decrease in Tg has been noted. However, a second order polynomial relation is found as $T_q = -1.391 \times 10^{-4} x^2 - 0.170 x +$ 82.503 with correlation factor, $R^2 = 0.799$, indicating a good relation between Tg and cluster size. Additionally, it has been observed that the behaviour of the Tg of the epoxy-Al₂O₃ nanocomposites is not only dependent on the nanoparticle content but also on the size of cluster. Thus, the increment in nanoparticle content up to 3 wt% having cluster size of 29 nm (containing ~3 nanoparticles) results in a significant increase in the Tg of the epoxy-Al2O3 nanocomposite compared to that of the neat epoxy. But further increase in nanoparticle content up to 20 wt% with a cluster size of 150 nm (containing ~15 nanoparticles) results in a significant decrease in the T_g of the nanocomposite.

The rise in T_g indicates that the influence of matrix/particle interfacial interactions must be constraining the polymer chains [37]. Particles thereby limit the polymer chain's mobility and strengthen the cross-linking process. Alumina nanoparticles can increase the apparent crosslink density and function as physical cross-linkers [38]. The production of a significant amount of immobilized polymer at the interface between epoxy and well-dispersed Al₂O₃ may be the cause of the increase in T_g [38]. Because the increase in T_g is anticipated to be proportionate to the nanoparticle's surface area, the T_g should only rise in tandem with an increase in nanoparticle content if the particles are evenly distributed throughout the base matrix. In the case of epoxy-Al₂O₃ nanocomposites (containing 3 wt% of nanoparticle content), it has observed that practically individual nanoparticles are homogeneously distributed in the base matrix with nominal clustering of \sim 3 nanoparticles. But the increase in nanoparticle content beyond 3 wt% to 20 wt% results in significant increase in the cluster size from 48 to 150 nm (containing \sim 5 to 15 nanoparticles) that may decrease the extent of cross-linking and therefore resulting the decrease in the Tg. In the current study, the size of Al₂O₃ nanoparticles is quite small (~ 10 nm). Therefore, the Al₂O₃ nanoparticles are more prone to clustering because of their very high specific surface area and dominance of strong van der Walls forces due to their very small size. That makes the breaking of clusters of such small nanoparticles much more difficult and thereby restricts their advantage regarding

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improving the $T_{\rm g}$ of the resulting nanocomposite for high nanoparticle content.



Fig. 8. DTA curves representing $T_{\rm g}$ of epoxy-Al_2O_3 nanocomposites.



Fig. 9. Nanoparticle content versus $T_{\rm g}$ of epoxy-Al_2O_3 nanocomposites.



Fig. 10. Effect of cluster size (x) on $T_{\rm g}$ of epoxy-Al_2O_3 nanocomposites.

3.3. Thermal stability of epoxy-Al₂O₃ nanocomposites

TGA graphs of epoxy and epoxy- Al_2O_3 nanocomposites for weight losses up to 50% are shown in **Fig. 11**. The thermal decomposition temperature (Td) values for nanocomposites at different weight loss percentages are displayed in Table 1.



Fig. 11. TGA curves displaying the weight remaining percentage of epoxy-Al₂O₃ nanocomposites versus temperature.

Weight Remaining (%)	Thermal decomposition temperature (T _d) $^{\circ}$ C									
	Neat	Epoxy-Al ₂ O ₃ nanocomposites containing nanoparticle (wt%)								
	Ероху	0.5	1	2	з	5	7	10	15	20
90	307	320	324	336	341	332	331	336	323	315
80	333	341	344	355	358	353	350	354	346	336
70	347	355	359	367	372	365	363	367	360	350
50 Char yield % at 700 °C	375	382	390	397	401	395	396	396	391	384
	9.75	9.85	10.48	10.99	11.44	12.07	13.65	14.34	15.63	17.29

Table 1. TGA results for epoxy-Al₂O₃ nanocomposites and neat epoxy.

Because most researchers view 50% weight loss as a sign of structural instability, this percentage of total weight loss is used in this work to determine the nanocomposites' structural destabilization point [39]. Fig. 12 displays the variance in weight loss percentage versus Td for neat epoxy and epoxy-Al₂O₃ nanocomposites. The T_d of the epoxy nanocomposite, which contains 3 weight percent Al₂O₃ nanoparticles, is determined to be 341 °C for a 10% weight loss, while the neat epoxy's Td is 307 °C for the same percentage of weight loss. The Td of the neat epoxy at 50% weight loss of the material is found to be 375 °C, while the Td of the epoxy nanocomposite (which includes 3 wt% Al₂O₃ nanoparticles) is 401 °C. Consequently, compared to plain epoxy, the thermal stability of epoxy nanocomposite increases as the nanoparticle concentration rises from 0.5 to 3 wt%. However, the thermal stability of the nanocomposite drastically decreases when the loading of nanoparticles in epoxy is increased to 20 wt%.



Fig. 12. Weight loss % versus T_d of neat epoxy and epoxy-Al₂O₃ nanocomposites.

Table 1 shows that at 700 °C, the neat epoxy's TGA curve produced a char yield percentage of around 9.75. The TGA curves of epoxy nanocomposites with 20 wt% of particles indicate that 17.29 is the maximum char yield percent at 700 °C. Because ceramic nanoparticles (Al₂O₃) are present, it is evident that as the nanoparticle concentration increases from 0.5 to 20 wt%, the char yield percentage increases continuously.

Degradation of aliphatic polyamine-cured epoxy network usually involved two steps in the breakdown process. The evaporation of residual solvent and adsorbed humidity causes the first stage, which happens between 35 and 310 °C. [40]. The heat breakdown of the cured epoxy network causes the second step to occur between 310 and 450 °C. Dehydration leads to the aliphatic polyamine-cured epoxy network's degradation because it removes water molecules from the oxypropylene group. This happens while the epoxy network breaks down and double bonds are formed [40]. Additionally, the (O-CH₂) and (CH-CH₂) bond scission processes run concurrently. Unsaturated phenolic compounds are the result of the degradation and are either emitted as gaseous products or polymerize to produce char [40].

TGA thermograms (**Fig. 11**) thus show that the degradation of epoxy nanocomposites likewise follows a twostage process. Since Al₂O₃ nanoparticles are ceramic, they prevent heat from passing through the epoxy when they dispersed evenly [39,41,42]. Because more nanoparticles form more obstacles. Thus, the resulting nanocomposites' capacity to endure heat is enhanced. But when the particle content increased up to 3 wt%, the particle-to-polymer interface becomes more dominant than the particle-to-particle interface, which leads to increased thermal stability. However, further increase in particle concentration results in a noticeable clustering which reduces the cross-linking density. Thermosetting polymers have high breakdown temperatures due to their high cross-linking density [43]. The cross-linking density is greatest when the entire stoichiometry of the epoxy matrix is maintained. The stoichiometry of the epoxy matrix will fail if foreign particles (Al₂O₃) are added over a certain limit [43]. This will drop the decomposition temperature and diminish the crosslink density. Therefore, a decrease in the decomposition temperature is observed when the nanoparticle content surpasses the threshold limit. Epoxy nanocomposites with nanoparticle quantities over their threshold limit (3 wt%) show less heat retardant action, leading to decreased thermal stability.

3.4. Thermal degradation kinetics of epoxy- Al_2O_3 nanocomposites

The kinetic analysis provides information about the mechanism of polymer breakdown together with indications about the energy barriers of the process. The recognized Coats Redfern model (CR) [44] and integral model of Horowitz and Metzger (HM) [45] have been employed in this work to collect kinetic data on the thermal degradation of epoxy composites. **Fig. 13** shows the CR plots of epoxy nanocomposite and epoxy treated with one order reaction. **Fig. 14** shows the Horowitz and Metzger plots of neat epoxy and epoxy composite. **Fig. 15** shows the (activation energy) E_a of neat epoxy and epoxy composites, which were calculated using HM and CR plots.





For epoxy-Al₂O₃ nanocomposites, the activation energy of decomposition increases with the increase in nanoparticle content from 0.5 to 3 wt% and decreases for further increase in nanoparticle content up 20 wt% for both the CR and HM methods (**Fig. 15**). The activation energy determined using

CR method reaches a maximum value of ~ 118 kJ/mol for 3 wt% epoxy-Al₂O₃ nanocomposite. But the activation energy calculated using HM method for the same nanocomposite reaches a maximum value of ~ 132 kJ/mol. Therefore, the addition of Al₂O₃ nanoparticles to epoxy leads to a significant increase in the resulting nanocomposite's E_a of decomposition, that is responsible for the increased thermal stability of the nanocomposite.



Fig. 14. Horowitz–Metzger method-based heat degradation kinetics of neat epoxy and epoxy-Al₂O₃ (3%) nanocomposite.



Fig. 15. CR and HM methods-based E_a of decomposition of the neat epoxy and epoxy-Al₂O₃ nanocomposites.

For epoxy nanocomposites, ease of decomposition likewise exhibits a pattern like thermal stability. When comparing the

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epoxy-Al₂O₃ (3 weight percent) nanocomposite to neat epoxy, the greatest improvement in the E_a of decomposition is approximately 51% when using the CR method and 42% when using the HM method. Because different kinetic equations are used for HM and CR techniques of decomposition, the activation energy of decomposition for a given nanocomposite varies. The observed rise in activation energy during the breakdown of nanocomposites may be ascribed to effective structural evolution, especially when considering the dispersal of homogenous nanoparticles within the epoxy matrix. The ceramic nature of the nanoparticles may be the cause of the increased activation energy. This is because the nanoparticles restrict the molecular motion of polymer chain segments, which enhances T_g. By limiting the heat flow in the epoxy resin, they can also stop the formation of random scission epoxy networks. The path of constraint to random scission of molecules is enhanced in the case of uniformly distributed nanoparticles throughout the matrix. Because there are more barriers preventing heat from passing through the matrix. When the quantity of particles in epoxy is increased beyond 3 wt%, however, the accumulation of nanoparticles and their unequal diffusion within the epoxy have a negative effect on the activation energy of breakdown, making them less effective in blocking heat.

3.5. Integral procedural decomposition temperature (IPDT)

The thermal stability of the neat epoxy and epoxy-Al₂O₃ nanocomposite was further evaluated using IPDT, which is based on Doyle's method [46]. The literature contains comprehensive guidelines for calculating IPDT [29]. Using TGA thermograms, IPDT which includes the entire stability spectrum is computed in three stages: first disintegration, rapid disintegration, and the final step of char production. **Fig. 16** displays the IPDT for neat epoxy and epoxy composites. Using TGA curves, for neat epoxy the IPDT is calculated around 398.48 °C. However, the IPDT continues to rise when the nanoparticle concentration rises from 0.5 to 20 weight percent. The largest concentration of nanoparticles (20 wt%) yields the highest IPDT value. For epoxy nanocomposite, the highest IPDT value is 525.15

As a result of the oxide nanoparticle addition, the epoxy has a notably greater thermal resistance than neat epoxy. The pattern of IPDT alteration with increasing particle concentration is shown to be different from the pattern observed for E_a . According to [47], the physical differences between the IPDT and the E_a of decomposition are most likely the root reason. The specimen's overall stability is reflected by the IPDT throughout the duration of its three stages. In contrast, the E_a of decomposition only considers fast decomposition and ignores the other two components. The reason behind the rise in IPDT as particle content increases is the corresponding rise in char content. The rise in char

content that corresponds with an increase in particle content is what causes the IPDT to rise.



Fig. 16. Nanoparticle content versus IPDT of epoxy- Al_2O_3 nanocomposite.

4. Conclusions

UDM process has been optimized to prepare epoxy-Al₂O₃ nanocomposites. A threshold limit of roughly 3wt% of Al₂O₃ nanoparticles was determined with a cluster size of 29 nm (containing approximately three nanoparticles) and an interparticle spacing of about 35 nm. At the nanoparticle threshold limit, the T_g value greatly increased by around 28% and similarly thermal stability of the nanocomposite was noticeably increased. A strong matrix-particle interaction was found responsible for the nanocomposite's improved T_g value and thermal stability because of even dispersion of the nanoparticles. It was demonstrated that the rise in Tg was caused by the chain segment mobility of the epoxy polymer decreasing because of the interaction between the nanoparticle and matrix. Because nanoparticles can function as physical cross-linkers by raising the apparent crosslink density, their inclusion limits the free motion of polymer chains and strengthens the cross-linking action. The alumina nanoparticles serve as insulators to heat transfer through the matrix, may be responsible for the increase in thermal stability. The even distribution of the nanoparticles produces a wide range of obstacles to the heat transfer in the epoxy matrix.

Disclosure statement

The authors declare no relevant financial or non-financial interests.

Data availability

Raw data of the research article is available with the authors and will be provided as per a request from the journal.

Ethical approval

Not applicable.

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