

Studies on Thermo-Mechanical Properties of Novolac Epoxy Resin Nano Composites

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ABSTRACT: The Novolac epoxy/CaCO₃ nano composites were prepared by high speed mechanical mixing of resin matrix with varying amount of nano CaCO₃ (0, 1, 3, 5 and 7 wt%) for 30 min and subsequent curing at 110° C for 1 hr and post curing at 70° C for 5 hrs. The prepared nano composites were characterized for mechanical properties (viz., tensile strength, elongation at break impact resistance and toughness) and thermal stability. It was observed that 5 wt% nano CaCO₃ loaded epoxy matrix showed remarkable improvement in the tensile strength, elongation at break, impact resistance and toughness as compared to the neat epoxy matrix. The thermal stability of nano composite system was also found maximum for same composition. The morphological studies of nano composites were also investigated and found higher exfoliation and rougher surface in 5 wt% nano filler added matrix as observed by SEM.

KEYWORDS: Epoxy resin matrix, nano composites, novolac epoxy.

I. INTRODUCTION

Epoxy resins are most commonly used thermosetting polymer for variety of applications such as adhesives, coatings, flooring, electrical / electronic applications, aerospace and marine products due to their excellent mechanical and thermal properties. Among the epoxy resins the novolac resin showed the excellent chemical resistance, flame retardancy, electrical insulation and thermal stability. The resin performance is always depend upon the degree of cross linking. It is obvious, as the curing percent increases the brittle character in the matrix system increases, which restrict the matrix for number of applications [1-3]. These shortfall can be bridged by addition of nano fillers in the resin matrix uniformly before cure. Many studies showed the incorporation of inorganic nano fillers such as SiO₂, TiO₂, ZnO, CaCO₃, Al₂O₃ and clay improve the mechanical and thermal properties of resultant nano composites greatly when the dispersion of nano filler is uniform [4-6]. One basic problem associated with the nano filler is the tendency to agglomerate during the mixing with the matrix [7]. To avoid the agglomeration, the prolong mixing of nano filler is required. The reduction of agglomeration of nano filler leads to high degree of exfoliation within the matrix system. The preparation methods and the particle size of nano fillers also have the major impact on properties of nano composites. The smaller the particles of nano filler larger will be the surface area and have the better dispersion within the chains of macro molecules [8-9].

In the present study nano composites were prepared by high speed mechanical mixing of novolac epoxy resin with varying amount of nano CaCO₃ for 30 min and subsequent curing with suitable curing agent. Further cured nano composite samples were subjected to post curing operation in an air oven. The prepared nano composites were tested for mechanical and thermal properties. The morphological properties were also studied during the investigation.

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II. EXPERIMENTAL

Materials

The novolac epoxy resin matrix (Lapox L238) of epoxide equivalent weight of 176-181 gm/eq and viscosity range 20000-50000 (m.Pa.S) at 27 °C was procured from Atul Ltd, Gujarat, India and the Triethylene Tetraamine (TETA)(71H) with amine value 1400-1600mg KOH/gm and density value 0.978 gm/cm³ procured from Parikh Chemicals, Kanpur, India was selected as curing agent. Nano CaCO₃ was purchased from Reinste Nano Ventures Pvt. Ltd. Delhi, the average particle size and the specific surface area of nano CaCO₃ were 40-90 nm and 15-40 m²/g respectively. These materials were used as received.

Preparation of nano composites

The nano filler particles were dried in an oven at 70° C for several hours. The epoxy resin was preheated in the same way to lower the viscosity and to enable a better wetting of the particles. Now estimated quantity of resin and nano filler were mixed thoroughly followed by mechanical mixing using electrical stirrer for 30 min. Thereafter stoichiometric amount of curing agent were mixed and poured the mixer into a teflon mold and subjected to cure in an air oven at 110° C for 1 hr and post cure at 70° C for 5 hrs. The samples were designated as EPC-0, EPC-1, EPC-3, EPC-5 and EPC-7 respectively, corresponding to the CaCO₃ concentration ie 0, 1, 3, 5 and 7 wt% in the epoxy matrix.

III. CHARACTERISATION OF NANO COMPOSITES

Mechanical Properties

Determination of tensile strength and elongation-at-break

The Prepared Dumb bell shaped nano composite samples were used to determine the tensile strength and elongation at break as per ASTM D638 by using Universal Testing Machine (UTM) (Zwick Roell Pvt. Ltd. Germany, model Z010 M) with cross head speed of 25 mm/min. Tensile strength and elongation at break values were automatically recorded on computer system.

Determination of Impact Strength

The test specimens were prepared according to ASTM standard D-256. The size of the specimens are 2.5 x 0.5 x (1/8) inch with a recommended notch depth of 0.100 inch. The test specimen was clamped on to the impact testing machine (m/s CEAST[®] Resil Impactor, Model 6957, Germany) in the position so that notched end of the specimen is facing the striking edge of the pendulum. Now, the pendulum hammer is released, allowed to strike the specimen and swing through. If the specimen does not break, more weights are attached to the hammer and the test is repeated until failure was observed. The impact strength was read directly from the dial directly attached to the tester.

Determination of Thermal Stability

The thermal stability of all Nano Composite samples were studied by using Thermo gravimetric Analyzer (TGA)(T A instrument, Inc USA, Model TGA Q 500 V20.10 build 36) at a heating rate of 10°Cmin⁻¹ in nitrogen atmosphere with a water flow rate of 10 mLmin⁻¹. A pre weighed sample was put on the pan which was then placed on the thermal balance of the instrument. The dynamic thermograms between temperature and weight loss were recorded. The initial degradation temperature (IDT), i.e. the measure of thermal stability of the system, was to be considered after 5% weight loss as in the initial stages, impurities and moisture present in the system decompose first. Percent char yield at 800°C was noted from TGA traces.

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Morphological Properties

The scanning electron microscope (SEM) images of the fractured surface of samples were obtained by using high resolution and low vacuum SEM equipment, Carl Zeiss EVO 50X VP, England with accelerating voltages 1-40 KeV to investigate the microstructure and fractured surfaces of nano composites. The samples were mounted on aluminium stubs using carbon tape. The samples were coated with a thin layer of gold to prevent charging before the observation by SEM.

IV. RESULTS AND DISCUSSIONS

Studies of Tensile Strength and elongation at break

The stress strain curve obtained during tensile test revealed the mechanical nature of the material. The effect of nano CaCO_3 concentration on tensile strength and elongation-at-break on nano composites have been given in Figure 1 and the experimental data are shown in Table 1. It is clear from the table that as the concentration of nano CaCO_3 increased the tensile strength and elongation both increased respectively and found maximum for the sample EPC-5. The remarkable change of about 20.66 % and 10 % in tensile strength and elongation respectively, have been observed as compare to neat epoxy matrix. This revealed that the nano particles start filling in to amorphous region of polymer via uniform dispersion up to 5 wt% of matrix. When the amorphous region of epoxy resin has been completely filled by the nano particles, the nano composite could achieve maximum tensile strength and elongation due to the load sharing and toughening of matrix. The further loading of nano CaCO_3 in the epoxy matrix led to the agglomeration of nano particles in the nano composite, results the decrease in tensile strength and elongation respectively[10].

Table 1: Mechanical properties of nano composite samples (Experimental data)

Sample Code	Tensile Strength (Kg/cm ²)	Impact Strength (kJ/m ²)	Elongation at break	Toughness (Joule)
EPC-0	83.39	1.940	2.70	0.117
EPC-1	86.40	2.109	2.91	0.120
EPC-3	90.12	2.5	2.93	0.20
EPC-5	100.62	3.0	2.97	0.25
EPC-7	88.16	2.5	2.40	0.21

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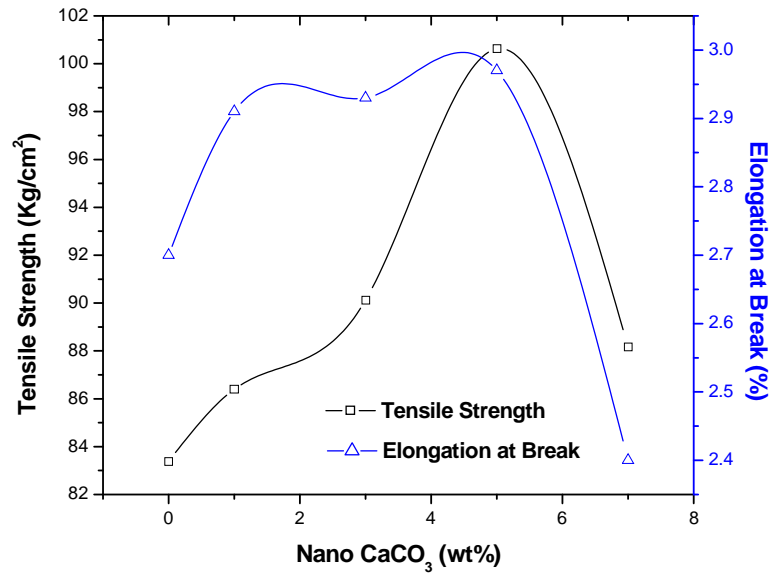


Figure 1: Tensile strength and elongation of nano composites

Studies on Impact strength and toughness

The effect of nano calcium carbonate on the impact strength and toughness on various composite samples have been shown in Figure 2. Which clearly indicates that impact strength and toughness were increased with increased in nano CaCO₃ volume in the epoxy matrix up to 5 wt%. Beyond 5 wt% nano CaCO₃ addition, the impact strength and toughness decreased. Impact strength and toughness of pure novolac epoxy sample EPC-0 were found to be 1.94 kJ/m² and 0.117 Joule respectively, which increased upto 3.0 kJ/m² and 0.25 Joule and decreased thereafter. The increase of impact strength of nano composite systems could be explained on the basis that too many nano CaCO₃ particles results in aggregation in the matrix and deteriorate the toughness, where as nano CaCO₃ exhibits reinforcing capacities when added in small amounts. From these results, it can be concluded that the maximum improvement in the both properties of epoxy matrix was observed in EPC-5 sample. However, The addition of nano particles led to decrease the free space between polymer chains. Since nano CaCO₃ are polar in nature, which led to attract resin molecules during the curing process of matrix creating more complicated network and formed the hydrogen bonding between the polymer chain and nano particles, led to enhance the impact strength and toughness of nano composites[11].

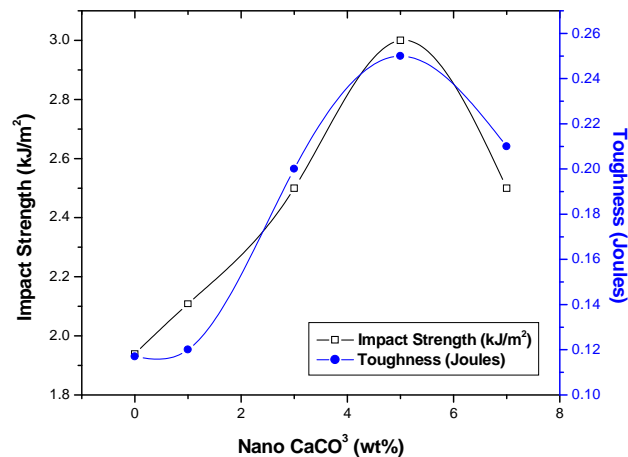


Figure 2: Impact strength and toughness of nano composites

Thermogravimetric Analysis

Studies on the thermal stability nano composites

The dynamic thermograms of composite samples EPC-0 and EPC-5, obtained at programmed heating rate of 10°C/min are shown in Figure 4. The TGA experiments were run to a maximum temperature upto 800°C, well below the decomposition temperature of nano CaCO₃. The thermograms obtained from the TGA analysis showed that the values of initial degradation temperature (IDT) and final degradation temperature (FDT) increased as the addition of nano CaCO₃ in epoxy increased and found maximum for 5wt% loading and decreased thereafter. The lower weight loss at high temperature suggest that oxidative crosslinking may contribute to cure at high temperatures. The percent char yield was found to be maximum i.e. around 40% with 5 wt% of nano CaCO₃ added matrix. On comparing the results, it could be concluded that thermal stability of composite samples were found to be increased with increased nano calcium carbonate concentration in the matrix, this might be due to the post curing and toughening of epoxy matrix by nano particles.

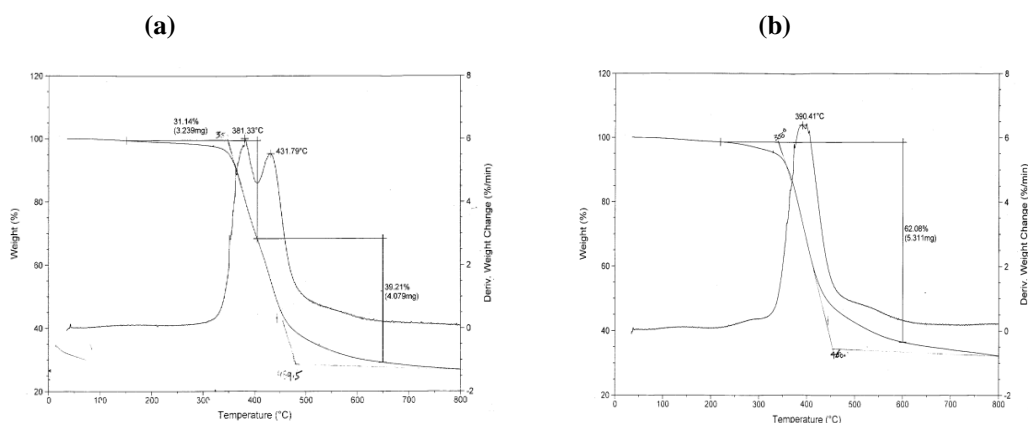


Figure 4: TGA thermograms of (a) EPC-0 sample (b) EPC-5 Sample

Morphological studies

All nano composite samples showed different degree of surface roughness. The surface of neat epoxy resin showed (refer **Figure 5a**) less roughness than the other nano composites samples. The maximum surface roughness was observed in EPC-5 sample as shown in **Figure 5b**. The lower surface roughness indicates more possibility of crack initiation and propagation. However, it is clear that as the loading of nano CaCO₃ increased in the epoxy matrix the

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roughness of the fractured surface also increased. The increase in the surface roughness increases the surface area to deflect the crack propagation in the samples, which also increases the absorbed energy of fracture by the crack length during the deformation. The higher energy absorption also enhances the fracture toughness,. Hence, it could be concluded that 5wt% of CaCO₃ loaded nano composite have the uniform exfoliation of nano filler and maximum surface roughness as compared to other nano composite samples and neat matrix.

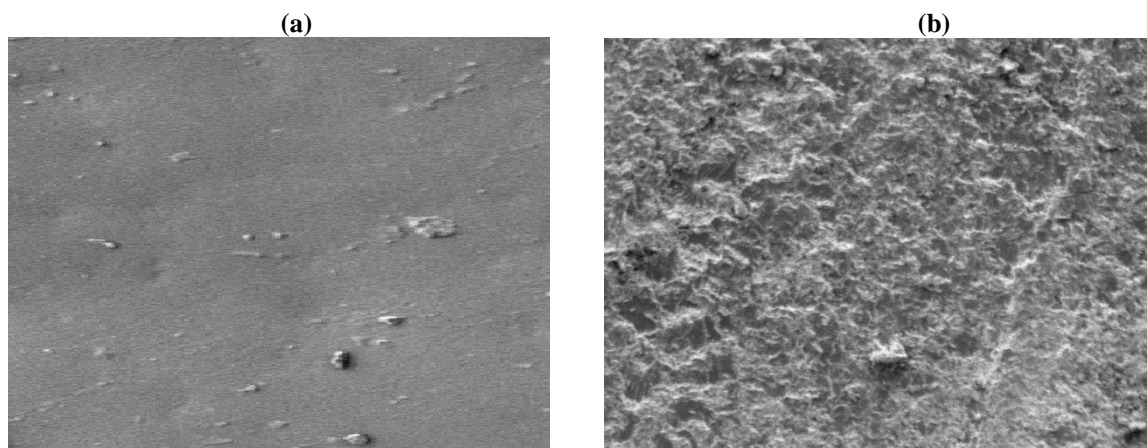


Figure 5: SEM image of (a) EPC-0 sample (b) EPC-5 sample

V. CONCLUSIONS

In the view of above results and discussions the following conclusion could be drawn—

1. As the concentration of nano calcium carbonate in the novolac epoxy matrix increased the tensile strength , elongation, impact strength and toughness increased respectively and found maximum in EPC-5 sample and thereafter decreased due to the agglomeration of nano particles in the matrix. The improvement in the mechanical properties were due to load sharing by nano particles when subjected under load.
2. The thermal stability was also found maximum in case of EPC-5 sample as compared to the other composition of nano composite and the neat epoxy matrix due to the thermal conduction by nano filler.
3. The high degree of exfoliation was observed in EPC-5 sample than other nano composites, which showed the complete filling of free space available in between the chains of polymer by the nano particles.

REFERENCES

- [1] Lee H., Neville K. Hand book of epoxy resins. New York: Mc Graw Hill; (1967).
- [2] Weatherhead RG. FRP Technology: Fibre reinforced resin systems. London: Applied science publisher ltd; (1980).
- [3] Pan G , Du Z, Zhang C , Li C , Yang X , Li H; Synthesis, characterization, and properties of novel novolac epoxy resin containing naphthalene moiety; Polymer (2007), 36: 3686-3693
- [4] Wetzel F, Hauptert MQ, Zhang. Epoxy nanocomposites with high mechanical and tribological performance. Compos Sci Technol 2003; 63: 2055
- [5] Quan shi, Wang Li, Haojie Yu, Song Jiang, Zhao, Dong X, A novel epoxy/ CaCO₃ nano composite and its mechanism of toughness improvement. Macro Mater eng 2006; 291: 53-58.
- [6] He H, Li K, Wang J, Sun G.; Study of thermal and mechanical properties of nano CaCO₃/epoxy nano composite; mater. And Desgn.(2001), 32: 4521-4527.
- [7] Dihayati Y., Aziz AR, Leong YC, Harcharan S; Properties of stearic acid coated nano calcium carbonate particles; Um-repository proceeding, (2004) 629-01.
- [8] Zhang K, Wang L, Wang F, Li Z. Preparation and characterization of modified-Clay-reinforced and Toughened Epoxy-Resin Nanocomposites. J Appl Polym Sci 2004; 91:2649-2652
- [9] Shao-Yun Fu, Xi-Qiao Feng, Lauke B, Mai YW. The particle Size, particle/matrix interface adhesion and particle loading on mechanical properties of particulate-polymer composite. Compo Part B 2008; 39:933-961.



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- [10] K.S. Harishanand , H. Nagabhushana , B.M. Nagabhushana , Parimesh Panda , Richeek Gupta , M.S. Muruli , N. Raghavendra , K.R. Vishnu Mahesh; Comparative Study on Mechanical Properties of ZnO, ZrO₂ and CeO₂ Nanometal Oxides Reinforced Epoxy Composites; Adv. Polym Sci and Tech.: An Inter J 2013; 3(1): 7-13.
- [11] Fan-Long Jin, Soo-Jin Park; Interfacial toughness properties of trifunctional epoxy resins/calcium carbonate nanocomposites; Mater. Sci Engg A 475 (2008) 190–193.