Tensile behavior of environmental pollutant crumb rubber filled epoxy composites

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Abstract

Tensile test is conducted on environmentally pollutant crumb rubber/epoxy composite. Crumb rubber particles with different volume fractions (10, 20 and 30%) are reinforced with epoxy matrix. Stress-strain curves reveal brittle fail for all the samples. Crumb rubber filled epoxy composites reveal higher modulus (13%–28%) and strength (28%–44%) than neat epoxy sample. Epoxy with 30 volume % of crumb rubber depicts higher modulus and strength compared with all other compositions, exemplifying use of crumb rubber for utilitarian applications. Crumb rubber/epoxy composites register higher specific modulus and strength for all compositions in comparison with neat epoxy. Scanning electron micrographs of test samples are used to analyze property - composition correlations. Finally, property map is plotted to compare the results of present study with existing ones to highlight the efficacy of the proposed composites.

1. Introduction

Particulate polymer composites are realized by mixing particles in resinous matrix. Weight sensitive composites have gained a lot of attention due to their use in variety of applications in manufacturing, marine, automobile and aerospace sectors. Low cost particulate fillers have generated ample attention in reducing the density and composite cost without compromising the intended properties [1]. Particulate fillers are incorporated into polymers mainly to improve specific properties [2]. Synergic effect of good mechanical properties coupled with lower material cost can be attained by reinforcing particulate fillers into polymers [3, 4]. Non-disposable waste tire derived crumb rubber is one such filler. Each year across the globe a lot of tires are disposed due to cycle end. Disposal of waste tires has turn into an exceedingly complex challenge owing to their long life and non-biodegradability [5]. Furthermore, cross link structure of tires and presence of stabilizers pose additional problems in decaying of waste tires [6]. Therefore, crumb rubber use in utilitarian composites can minimize disposal and landfill burden issues of waste tires and help in manufacturing composites with good performance. Epoxy resins are widely used thermo-set polymers due to their wide use in automotive, aerospace and marine industries attributed to low cost, excellent mechanical properties, high specific strength and very good adhesion [7–11]. Development of polymer matrix composites with crumb rubber particles serves twin purpose of beneficial use of environmental pollutant and decrease in the cost of components. Interest to exploit the advantage of low cost crumb rubber particles has made it essential to investigate the composites for tensile loading. In the current investigation crumb rubber is used for preparation of epoxy composites. Reports on crumb rubber reinforced thermosetting composites are very scarce and study of tensile response is not reported in literature and is the scope of the present study. Open mold casting technique is used to synthesis the composites. Four types of epoxy composites with different volume fractions of crumb rubber (0, 10, 20 and 30) are prepared. Prepared composites are tested for normal strain rate (5 mm min \(^{-1}\)) conforming ASTM D638 standards. Scanning electron microscope is utilized to study structure-property correlations. Finally, property map is plotted for normal strain rate values to compare the results of present study with existing ones to highlight the efficacy of the proposed composites.
2. Materials and methods

2.1. Constituents
In the present study, LAPOX L-12 Epoxy and K6 hardener are used as matrix resin and hardener, respectively, supplied by Atul, Valsad, Gujarat, India. Crumb rubber particles are used reinforcement procured from Arihant Chemicals, Delhi, India.

2.2. Sample preparation
Composites in the current study are fabricated by open mold casting method. Composite are realized by mixing desired quantity of crumb rubber in epoxy resin to achieve uniform and homogeneous slurry. Mixing of constituents is carried very slowly so as to minimize the formation of voids. Prepared slurry is degassed for a period of 15 min prior to adding the hardener. Slurry is thoroughly mixed to start the polymerization process. Finally, the mixture is poured in the aluminum molds coated with silicon releasing agent to enable easy removal of casting. Curing is done for a period of 24 h. Four types of composites are prepared with different volume fractions of crumb rubber (0, 10, 20 and 30 vol.%). EC-VV nomenclature is used to represent the composition of samples, E depicts epoxy matrix, C denotes crumb rubber particles and VV represents volume fraction of crumb rubber particles. Samples are trimmed from the cast slabs confirming ASTM D638 [12]. Density of the samples is measured using ASTM D792-13 [13].

2.3. Tensile tests
Specimens are tested under ambient conditions by means of Zwick (Zwick Roell Z020, ZHU) universal test setup for tensile tests conforming ASTM D638 [12]. A continuous cross-head movement of 5 mm min$^{-1}$ is applied on the specimens. A minimum of three samples are tested for each composition.

2.4. Imaging
Energy dispersive spectrum of crumb rubber particles is carried out to determine the elemental composition (AMETEK EDAX). Scanning electron microscope is used to study the tensile tested samples (JEOL JSM 6380 LA). Sputtering is done on the samples to enhance the conductivity.

3. Result and discussions

3.1. Material processing
The mechanical properties of composites are highly dependent on the fabrication technique, filler dispersion and presence of void content. Open mold casting technique is used to prepare the composites and care was taken to mix the constituents (epoxy and crumb rubber) properly. Theoretical density of E, EC-10, EC-20 and EC-30 are estimated to be 1192.00, 1207.50, 1232.70 and 1254.40 kg m$^{-3}$ respectively calculated using rule of mixture whereas experimental density are measured to be 1192.00, 1217.90 ± 24.15, 1243.80 ± 30.81 and 1269.70 ± 31.36 kg m$^{-3}$ respectively [14]. Further, the void content approximations reveal EC-10, EC-20 and EC-30 composites have void content of 0.85, 0.89 and 1.20, respectively. Although the void content increases with increasing filler content, observed values are small and within the limits. Lower void content also depicts the consistency attained in fabrication of composites. Additionally to assure complete curing of composites, differential scanning calorimetric tests were conducted and reported in [15]. Curing curves of neat epoxy were narrow whereas EC-10, EC-20 and EC-30 were wider revealing that crump rubber reinforcement in epoxy matrix releases more quantity of heat implying complete polymerization [15]. Particle size examination of crumb rubber reveals average size as 182.24 μm (Figure 1) [14]. Physical properties of crumb rubber and epoxy matrix are available in [14].

Energy dispersive spectrum and quantitative analysis of crumb rubber particles is shown in figure 2. Quantitative analysis reveals carbon (56.16%), oxygen (18.48%), sulfur (7.55%) and calcium (6.43%) as main elemental composition while trace quantities of aluminium, silica, titanium, iron and zinc. X-Ray Diffraction analysis reveals that crumb rubber epoxy composites have main peaks at 2θ value of 31.82 and 77.26 for carbon and zinc aluminum sulfide respectively while smaller peaks of quartz and zinc oxide that are primarily metal oxides are also seen [15].

3.2. Tensile stress strain curves, modulus and strength
Representative tensile stress–strain curves for neat epoxy and crumb rubber filled epoxy composites are presented in figure 3(a). Stress–strain profiles of all the samples demonstrate similar curves up until maximum stress consisting linear elastic area followed by abrupt brittle failure. Similar trends are observed in earlier studies on epoxy based composites [16, 17]. Stress-strain profiles observed with tensile tests are different as compared
with compression stress-strain curves wherein higher plateau region is observed revealing good energy absorption capabilities [14]. Although the stress strain profiles are similar but reinforcing crumb rubber in epoxy matrix enhancing the stress notably is evident from figure 3. Epoxy resins can be toughened by reinforcing secondary phase particles like rubber, silica beads, titania and alumina [18].

Experimentally measured tensile modulus is presented in figure 4(b). Tensile modulus of EC-10, EC-20 and EC-30 samples increase as compared with E0 (figure 4(b)). Tensile modulus of all the composites increases with increase in filler content and are in the range of 13%–28% higher as compared with E0. Rubber particles dispersed in epoxy matrix assist in the load transfer effectively and contribute in enhancing the modulus of the composites. Reinforcement of rubber fillers increases the stiffness of epoxy based composites [19–21]. Uniformly dispersed crumb rubber particles in epoxy matrix assist in bridging the cracks generated in the matrix by stretching to large strains before final rupture [19]. As the crumb rubber content increases, the ability of composites further increases to bridge the cracks. Further, it must also be noted that chemical composition and cure schedule of the epoxy rubber composites play a vital role in increasing the toughness of the samples with rubber particles [22]. These changes are mainly attributed to the use of hardener to cross link the resin and increase the compatibility between the constituents. K-6 hardener used in the present study is a diamine that

Figure 1. Particle size analysis of crumb rubber [14]. John Wiley & Sons. [original copyright notice].

Figure 2. EDS spectrum and quantitative analysis of crumb rubber particles [14]. John Wiley & Sons. [original copyright notice].
Figure 3. Representative tensile stress-strain curves of crumb rubber epoxy composites.

Figure 4. (a) Modulus, (b) Strength, (c) Specific modulus and (d) Specific strength of crumb rubber epoxy composites.
reacts with and opens up the epoxide rings to form a 3D network polymer [23]. Further, specific modulus of the samples are also presented in figure 4(d). Specific modulus of EC-10, EC-20 and EC-30 depict an increase in the range of 11%–22% compared with neat epoxy exemplifying the use of crumb rubber particles in tensile conditions.

Experimentally measured strength of the samples are presented in figure 4(c). EC-10, EC-20 and EC-30 samples show an increase in strength as compared with neat epoxy sample. Increase is in the range of 28%–44% and maximum increase is observed with EC-30. Reinforcing crumb rubber in load bearing matrix increases the filler-matrix bonding and thereby increases the strength. Reinforcing rigid or ductile fillers in brittle matrix acts as bridging constituent and applies compression traction on the crack while ductile particles in particular provide crack shielding by plastically deforming around the material nearby the crack tip. However crack shielding by yielding of rubber particles is negligible and toughness is mainly attributed to particle bridging [24]. In addition to crack shielding and deformation of crumb rubber particles to loading, uniform dispersion of particles at high filler loading becomes very important. Dispersion of crumb rubber particles at EC-30 is depicted in figure 5. It can be clearly seen that crumb rubber particles are uniformly dispersed through the specimen and reveal good bonding with epoxy matrix. These instances facilitate the enhancement in strength. Specific strength plots for the samples are also shown in figure 4(e). In line with trends specific strength of EC-10, EC-20 and EC-30 samples reveal an increase in specific strength in the range of 27%–37% as compared with neat epoxy sample. EC-30 sample reveals the highest modulus and strength as compared with all the compositions tested.

3.3. Scanning electron micrographs
Fracture surfaces of specimens as observed in scanning electron microscopy are presented in figure 6. Fracture features in neat epoxy sample can be seen in a wherein striation marks and brittle failure marks can be clearly observed (figure 6(a)). Micrographs of EC-10, EC-20 and EC-30 are presented in figures 6(b)–(d) respectively. It can be clearly observed that striation and deformation marks are less in number for crumb rubber/epoxy composites compared with neat epoxy indicating. Good bonding between the crumb rubber and epoxy matrix can also be seen. In line with the trends observed for modulus and strength higher filler loadings reveal low deformation marks and contribute significantly in enhancing the properties of composites.

Property map is presented in figure 7 to compare the tensile strength of different composites with the present results to highlight the effectiveness of investigated samples. Modulus of E0, EC-10, EC-20 and EC-30 are compared with existing studies and mapped with density of samples. Crumb rubber/epoxy composites outperform composites reinforced with cenospheres, hollow glass microballoons, nanoclay and eggshell particulates exemplifying the use of crumb rubber in most widely used epoxy resin. Although the density of crumb rubber/epoxy composites is higher in comparison than all the composites compared, significance of using waste non-disposal tire derived crumb rubber is clearly obvious.
4. Conclusion

In the present investigation, tensile response of environmental pollutant crumb rubber filled epoxy composites is carried out. Density of composites increases with increase in filler content while lower void content reveals the consistency attained in fabricating the samples. All the specimens depict lower strains to failure and nature of failure is brittle. Crumb rubber/epoxy composites reveal higher modulus (13%–28%) as compared to neat epoxy owing to the enhanced ability of crumb rubber particles to effectively transfer load from the matrix. Similarly, strength of all the composites reveals increase in the range of 28%–44% with increasing filler content in comparison to neat epoxy attributed to the crack shielding and deformation of crumb rubber particles to tensile loading. Specific modulus and specific strength of crumb rubber/epoxy composites also present higher values as compared with neat epoxy revealing the usefulness of using crumb rubber. EC-30 reveals highest modulus and strength among all the compositions studied. Micrographs of samples reveal good bonding between constituents and lower deformation for increase in the modulus and strength of crumb rubber composites. Property map depicts the aptness of using crumb rubber in widely used matrix resin.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).
Statement of opposing interest

Author affirms there is no conflict of interest of this article.

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References

[1] Gupta N, Singh Brar B and Woldesenbet E 2001 Effect of filler addition on the compressive and impact properties of glass fibre reinforced epoxy Bull. Mater. Sci. 24 219–23
[2] Shahapurkar K, Garcia C D, Doddamani M, Kumar G M and Prabhakar P 2018 Compensive behavior of cenosphere/epoxy syntactic foams in arctic conditions Composites Part B: Engineering 135 253–62
[3] Acosta J L, Morales E, Ojeda M C and Linares A 1986 Effect of addition of sepiolite on the mechanical properties of glass fiber reinforced polypropylene Die Angewandte Makromolekulare Chemie 138 103–10
[4] Shahapurkar K, Doddamani M, Mohan Kumar G C and Gupta N 2019 Effect of cenosphere filler surface treatment on the erosion behavior of epoxy matrix syntactic foams Polym. Compos. 40 2109–18
[5] Bulei C, Todor M P, Heput T and Kiss I 2018 Directions for material recovery of used tires and their use in the production of new products intended for the industry of civil construction and pavements IOP Conf. Series: Materials Science and Engineering 294 012064
[6] Wu B and Zhou M 2009 Recycling of waste tyre rubber into oil absorbent Waste Manage. (Oxford) 29 1553–9
[7] Pearson R A and Yee A F 1989 Toughening mechanisms in elastomer-modified epoxies J. Mater. Sci. 24 2571–80
[8] Tesoro G 1988 Epoxy resins–chemistry and technology 2nd Edition, ed Clayton A. May Price: $195.00. Journal of Polymer Science Part C: Polymer Letters (New York: Marcel Dekker) 1, 288 1988. 26(12): p. 539–539
[9] Shahapurkar K, Chavan V B, Doddamani M and Kumar G C M 2018 Influence of surface modification on wear behavior of fly ash cenosphere/epoxy syntactic foam Wear 414-415 327–40
[10] Garcia C D, Shahapurkar K, Doddamani M, Kumar G M and Prabhakar P 2018 Effect of arctic environment on flexural behavior of fly ash cenosphere reinforced epoxy syntactic foams Composites Part B: Engineering 151 265–73
[11] Shahapurkar K, Darekar V, Banjan R, Nidasosi N and Soudagar M E M 2020 Factors affecting the solid particle erosion of environment pollutant and natural particulate filled polymer composites—A review Polym. Polym. Compos. 0967391120971411
[12] D638-14, A. 2014 Standard Test Method for Tensile Properties of Plastics, in ASTM. (USA: ASTM International PA)
[13] D792-13, A. 2013 Standard Test Methods for Density and Specific Gravity ( Relative Density) of Plastics by Displacement, in ASTM. (USA: ASTM International PA)
[14] Shahapurkar K 2020 Compressive behavior of crump rubber reinforced epoxy composites Polym. Compos. 42 329–41

Figure 7. Mapping of tensile strength against density of composites [16, 17, 25, 26].
[15] Shahapurkar K, Soudagar M E M, Shahapurkar P, Mathapathi M, Khan T M Y, Mujtaba M A, Ali M D I, Thanaiah K, Siddiqui M I H and Ali M A 2021 Effect of crumb rubber on the solid particle erosion response of epoxy composites *J. Appl. Polym. Sci.* n/a 51470

[16] Gupta N and Nagorny R 2006 Tensile properties of glass microballoon—epoxy resin syntactic foams *J. Appl. Polym. Sci.* 102 1254–61

[17] Shahapurkar K, Doddamani M and Kumar G C M 2018 Tensile behavior of cenosphere/epoxy syntactic foams *AIP Conf. Proc.* 1943 020100

[18] Lee J and Yee A 2001 Inorganic particle toughening I: micro-mechanical deformations in the fracture of glass bead filled epoxies *Polymer* 42 577–88

[19] Kunz-Douglass S, Beaumont P W R and Ashby M F 1980 A model for the toughness of epoxy–rubber particulate composites *J. Mater. Sci.* 15 1109–23

[20] Zhao Q and Hoa S V 2006 Toughening mechanism of epoxy resins with micro/nano particles *J. Compos. Mater.* 41 201–19

[21] Yee A F and Pearson R A 1986 Toughening mechanisms in elastomer-modified epoxies *J. Mater. Sci.* 21 2462–74

[22] Meeks A C 1974 Fracture and mechanical properties of epoxy resins and rubber-modified epoxy resins *Polymer* 15 675–81

[23] Verma A, Negi P and Singh V K 2018 Experimental investigation of chicken feather fiber and crumb rubber reformed epoxy resin hybrid composite: mechanical and microstructural characterization *Journal of the Mechanical Behavior of Materials* 27 3–4

[24] Sigl I S, Mataga P A, Dalgleish B J, McMeeking R M and Evans A G 1988 On the toughness of brittle materials reinforced with a ductile phase *Acta Metall.* 36 945–55

[25] Panchal M, Raghavendra G, Reddy A R, Omprakash M and Ojha S 2020 Experimental investigation of mechanical and erosion behavior of eggshell nanoparticulate epoxy biocomposite *Polym. Polym. Compos.* 0967391120943454

[26] Maharsia R R and Jerro H D 2007 Enhancing tensile strength and toughness in syntactic foams through nanoclay reinforcement *Mater. Sci. Eng. A* 454 416–22