Visual Abstract

Mechanical and fractured surface characterization of epoxy/red mud/fly ash/aluminium powder filled hybrid composites for automotive applications

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ABSTRACT. In recent decades, one can observe a great increase in the replacement of traditional materials with polymer composites in high-strength and lightweight applications. High fuel consumption by automobile and aerospace vehicles built from legacy alloys has been a great challenge to material engineers. This has called for researches into lighter material development of the same or even superior mechanical properties to the existing materials in this area of applications. In the present study, epoxy based simple and hybrid composites were prepared with the incorporation of industrial waste as fillers at different weight percentages. Effect of filler type, combination and its concentration on mechanical properties such as tensile, impact and flexural strength were investigated. SEM analysis was carried out for fractured surfaces of composites, wherein minor voids, crack initiations and filler pullouts were seen indicating the necessity of coupling agent addition for still better performance. Among hybrid composites, epoxy/fly ash/red mud/aluminium powder (91/6/1.5/1.5 wt%) has showed the highest ultimate tensile modulus, flexural strength and hardness value compared to other composites under study.

KEYWORDS. Epoxy, Red mud, Fly ash, Aluminium powder, Hybrid composite.

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Introduction

Polymer composites usage has increased in engineering applications in recent years as a result of the need to overcome the limitations of traditional materials. The use of waste/low-cost fillers in polymer composites is widely researched to give the cost benefits and also to complement the value addition to industrial waste/byproducts [1]. In the present study, fly ash which is a byproduct of coal burning is used as filler along with red mud and aluminium powder. A composite is a synergistic mixture of two or more constituents that are insoluble in each other and differ in physical form and chemical makeup [2, 3]. The goal is to take advantage of both materials' exceptional features while minimizing the weaknesses of either [4]. Among the common matrices used for the development of advanced composites, epoxy resin has attained dominance among its counterparts because of its excellent properties like chemical, thermal and electrical resistance, mechanical strength and dimensional stability [5, 6].

Advantages of polymer composites include high specific strength, durability, corrosion resistance, lightweight, design flexibility, fatigue resistance and easy processability [3]. Composites are more suitable for making aircraft parts, boat hulls, interiors of many mass rapid transit vehicles, body frames of bicycles, marine and military vehicles [7, 8]. Among thermoset based polymer composites, epoxy composites are preferred for many advanced applications due to its versatile properties and tailor-made processing [9]. Both fibre reinforced and particle filled epoxy -composites can be used in automobile industry [10, 11]. The micro/nanofiller composites are widely researched in recent times to customise the performance of polymer composites for intended applications [12]. In the present study, epoxy based simple and hybrid composites were prepared using industrial waste/by-products as fillers. Red mud (a waste produced during the processing of bauxite into alumina in Bayer cycle), fly ash (the residue of the coal burning plant) and aluminium powder (a byproduct of bauxite ore processing) is selected as fillers. The fly ash is very low density filler, red mud is inexpensive filler and aluminium powder is hard filler which can enhance the dimensional stability of composites. Hence their combination can be a better formulation to develop lightweight components with cost advantages for automotive applications [13, 14]. The most literature reported until now focuses more on simple composites of epoxy or with dual filler combination. In the present work three filler combination was tried to take advantages of their overall presence and also the amalgamations of fillers selected for the study stands to be unique/rare. All the three fillers selected were industrial byproducts hence their reutilization is sort of value addition situation.

Materials and methods

Materials

Epoxy resin (Araldite AW 106) and hardener (HV 953) were used as matrix material. The epoxy grade and hardener was selected based on the mechanical strength desired and also based on its ability to cure at room temperature. Red mud (about 100 micron particle size), fly ash (an average particle size of 25 μm) and aluminium powder (particle size is in the range of 70-200 μm) is used as fillers. When sieve analysis was carried out with the procured aluminium powder, it was found that about 95% weight of powder passed through 70 mesh, about 65% weight passed through 100 mesh and finally 12% retained on mesh number 200. Indicating average particle size of about 150 μm. The composition of each filler was varied at 3, 6 and 9 wt% for simple composites preparation.

(a) (b)

Figure 1: (a) Molds used for composite sheet preparation (b) Prepared epoxy sheet

Method

The fillers selected were characterised to determine the particle size and its distribution by SEM and sieve analysis. The epoxy and harderner was mixed in calculated proportion using lab scale stirrer and then degassed and casted on rectangular mold of dimension 250*250*5 mm as shown in Fig. 1(a). Initially, the mold preparation was done by cleaning, followed by application of mold release agent and gel coat.

ASTM Code	Type of tests	Specimen Dimension (mm)	Cross head speed
ASTM D 3039	Tensile	250 * 25 * 5	1.5 mm/min
ASTM D 790-03	Flexural	140 * 18 * 5	1.5 mm/min
ASTM D 256	Impact (Izod)	64 * 12.7 * 5	-
ASTM D 2240	Hardness (shore D)	-	-

Table 1: ASTM Standards followed for mechanical properties evaluation

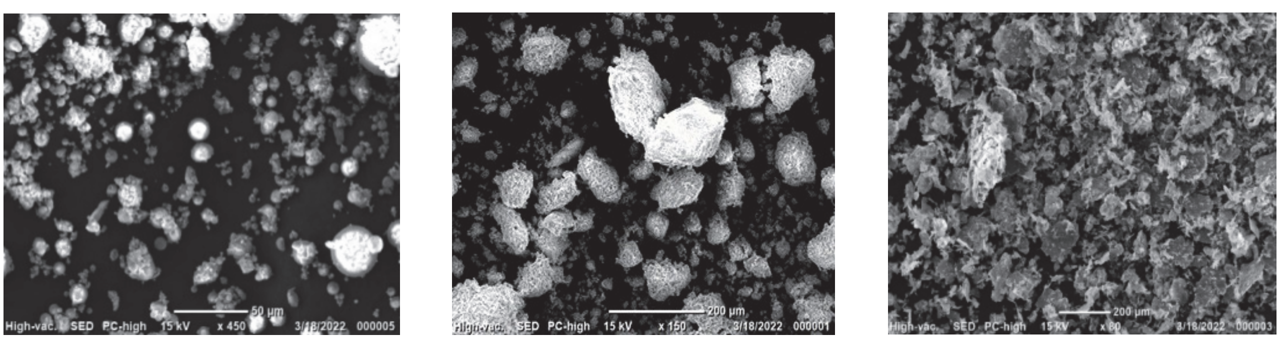
The premixed resin, hardener and filler was slowly poured onto the mold and spread evenly and left undisturbed until it is cured. Then the composite sheet was removed (Fig. 1b) and used for specimen cutting according to the ASTM standard as shown in Tab. 1 for further testing.

RESULTS AND DISCUSSION

The fillers, red mud, fly ash and aluminium powder were characterized by SEM and sieve analysis. The prepared composites were tested for mechanical properties like hardness, tensile strength and modulus, impact strength, flexural strength and modulus, the average readings of 5 specimens were reported. The fractured surfaces were characterized by SEM analysis to explain the mode and reason for the failure, also to understand the interfacial behavior of the filled composites.

Fillers characterization

The red mud is made out of different oxide compounds, including the iron oxides which give its red tone [15]. The SEM micrograph of red mud, fly ash and aluminium powder is shown in Fig. 2.



(a) (b) (c)

Figure 2: SEM micrograph of fillers (a) Fly ash (b) Red mud (c) Aluminium powder

Red mud shows an average particle size of about 100 µm, fly ash display an average particle size of 25 µm and aluminium powder particle size is in the range of 70-200 µm. The red mud and alumnium powder shows rough surface morphology, which is desired for better mechanical anchorage of filler with matrix.

Epoxy to hardener ratio optimization

For better composite properties, constituent material selection and maintaining its weight proportion is very crucial. Epoxy resin used in present study is *Araldite AW 106* and hardener is *HV-953 U*. It was observed that 10:8 (wt%) epoxy: hardener ratio is better with respect to strength and toughness as shown in Tab. 2. Beyond this ratio brittleness of the composite increased and hence the 10:8 ratio is maintained throughout the study.

Epoxy:Hardener ratio (wt%)	Tensile strength (MPa)	Impact strength (J/m)	Flexural strength (MPa)	Hardness (Shore D)
10:1	14.45 ±1.1	97.82 ± 2.3	46.3 ± 1.0	64 ± 2
10:5	25.94 ± 2.1	130.43 ± 3.1	48.6 ± 0.9	65 ± 1
10:8	30.07± 1.4	206.52 ± 3.7	50.7 ± 1.3	67 ± 2

Table 2: Mechanical properties of composites at different epoxy: hardener ratio

Hardness of simple composites

The inorganic filler addition mainly improves the hardness of polymer composites. The composites prepared were tested for Shore D hardness and the values were shown in Fig. 3 for 3 wt% filler. As expected, the hardness of all the composites were found to be higher, which indicates the improved resistance of composites against deformation/indentation. Red mud filled composite has shown highest hardness of 65 Shore D because of its rigid nature whereas, fly ash addition marginally enhanced the hardness due to its soft texture. As the red mud contains more mineral fillers which are harder and rigid, this might have resulted in increased hardness and impact strength of composites [16-18].

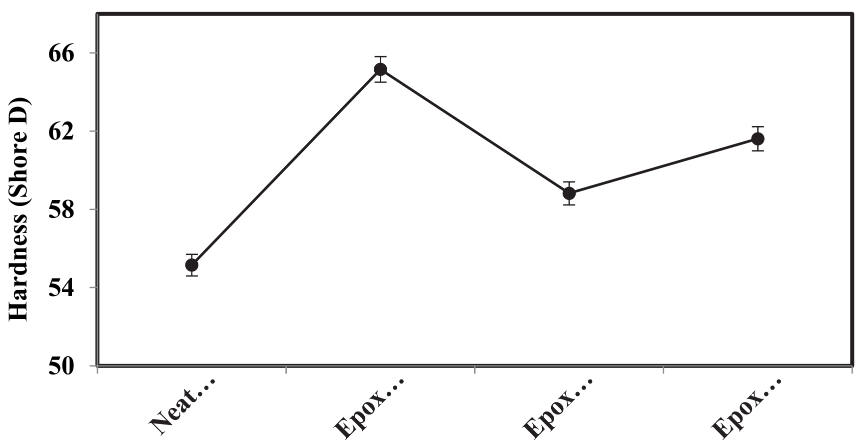


Figure 3: Hardness values of composites with different types of fillers.

Tensile testing of simple composites

The prepared composites were tensile tested using UTM machine according to ASTM D3039 at a grip separation speed of 1.5 mm/min and the results are shown in Tab. 3. Upon entering the dimensions of the test specimen, the inbuilt software calculates the tensile strength and modulus.

Composition	Peak load (kg)	Tensile strength at peak load (N/mm^2)	Ultimate modulus (MPa)
Neat epoxy	316.9 ± 3.2	23.2 ± 1.1	375.6 ± 3.2
Epoxy/Red mud	307.2 ± 2.1	29.3 ± 0.8	551.7 ± 4.1
Epoxy/Fly ash	332.2 ± 3.9	26.3 ± 0.6	364.2 ± 2.8
Epoxy/Al. Powder	330.6 ± 4.4	23.9 ± 1.5	428.2 ± 4.0

Table 3: Tensile test results of composites at 3 wt% of different filler loading.

The red mud filled composite shown highest ultimate modulus of 551.75 MPa because of presence of cementite particles and oxides in red mud and better interfacial adhesion between red mud and epoxy due to rough surface morphology [19]. Fly ash has shown the lowest ultimate modulus of 364.27 MPa because of its soft and smooth surface texture. Tensile strength of composites is not much changed with filler type and is more dominated by matrix property. The fly ash filled composites are more resistant to load applied.

The tensile tests were performed for different weight percent of red mud and the effect of same on tensile properties were recorded as shown in Tab. 4. As red mud concentration increases above 3 wt%, the tensile strength decreases, this shows that red mud can be used only to enhance the bulkiness and reduce the cost beyond 3%. But there is an improvement in the modulus upto 6 wt% of loading and marginal decrease at 9 wt%. So about 3 wt% of red mud can be used without much sacrifice in the properties. To improve the properties further, it requires addtional coupling agent or interfacial adhesion promoter at higher loadings. Similarly, effect of different weight percent of fly ash loading on tensile properties were studied. From the results it is apparent that as concentration of fly ash increases the tensile strength increased due to effective bonding at the interface and effecient load transfer to the filler at the interface, which is also evident from smooth interfacial morphology observed in fractured surface micrographs. Hence fly ash can be a better filler for cost reduction without much compromise in the properties. Effect of different weight percent of alumnium powder loading on tensile properties were also shown in Tab. 4. The minor inconsistency in tensile strength values with respect to peak load recorded can be attributed to filler agglomeration, micro voids/porosity presence and small variations in geometrical dimensions of the cut specimen edges. Modulus values are encouraging upto 6% loading level. So, alumnium powder also requires effective interface adhesion promoter to use as a successful filler with epoxy [5].

Composition	Peak load (kg)	Tensile strength at peak load (N/mm^2)	Ultimate modulus (MPa)
Tensile properties of Red mud filled composites			
Neat epoxy	316.9	23.22	375.62
Epoxy: Red mud (97:3 wt%)	307.2	29.36	551.72
Epoxy: Red mud (94:6 wt%)	283.8	23.69	417.28
Epoxy: Red mud (91:9 wt%)	264.2	21.72	369.93
Tensile properties of fly ash filled composites			
Epoxy: Fly ash (97:3 wt%)	332.2	26.31	364.25
Epoxy: Fly ash (94:6 wt%)	332.2	26.37	514.32
Epoxy: Fly ash (91:9 wt%)	345.9	26.09	376.26
Tensile properties of aluminium powder filled composites			
Epoxy: Al. Powder (97:3 wt%)	330.6	23.93	428.21
Epoxy: Al. Powder (94:6 wt%)	236.3	19.09	403.97
Epoxy: Al. Powder (91:9 wt%)	218.4	19.18	351.52

Table 4: Tensile properties of composites with different types of fillers.

Flexural testing of simple composites

The neat epoxy and its composites were subjected to three point bending test to evaluate their flexural properties and the results are tabulated in Tab. 5 for 3 wt% of different filler types. The result shows that red mud and fly ash filled composites were better than aluminium powder filled composites. This again shows poor interfacial load transfer ability of aluminium powder filled composites.

Composition	Peak load (kg)	Flexural strength at peak load (N/mm^2)	Ultimate modulus (MPa)
Neat epoxy	16.38 ± 0.7	45.32 ± 1.1	2751.42 ± 12.3
Epoxy : Red mud (97:3 wt%)	19.98 ± 1.2	63.96 ± 2.3	4170.13 ± 10.7
Epoxy : Fly ash (97:3 wt%)	18.04 ± 1.1	67.53 ± 2.6	4226.82 ± 11.6
Epoxy : Al. powder (97:3 wt%)	17.12 ± 0.9	40.22 ± 1.4	2505.72 ± 9.4

Table 5: Flexural test results of composites at 3 wt% of filler loading.

The flexural properties of composites were also evaluated at different weight percent of different fillers as shown in Tab. 6. The results shows that upto 6 wt% loading level there is an improvement in flexural strength and modulus for red mud and fly ash filled composites. May be beyond this concentration there is an agglomeration, poor wettability and no proper load transfer. So, 6 wt% is the maximum permissible loading to improve the flexural strength. The flexural test results at different concentration of aluminium powder filled composites shows that the flexural strength was not so encouraging but modulus was improved due to higher stiffness of the composites. The good flexural properties are much needed for dynamic applications of polymer composites under vibrations in automobile industry [10, 20]. The inorganic fillers addition also helps in improving the fire retardancy of polymer composites, which can be fulfilled by the fillers selected in the present study [11].

Composition	Peak load (kg)	Flexural strength at peak load (N/mm^2)	Ultimate modulus (MPa)
Neat epoxy	16.38	45.32	2751.42
Epoxy: Red mud (97:3 wt%)	19.98	63.96	4170.13
Epoxy: Red mud (94:6 wt%)	22.12	64.25	4688.47
Epoxy: Red mud (91:9 wt%)	15.85	39.22	2874.46
Flexural test results of fly ash filled composites			
Epoxy: Fly ash (97:3 wt%)	18.04	67.53	4226.82
Epoxy: Fly ash (94:6 wt%)	20.10	59.68	4519.37
Epoxy: Fly ash (91:9 wt%)	17.02	40.23	2736.04
Flexural test results of aluminium powder filled composites			
Epoxy: Al. Powder (97:3 wt%)	17.12	40.22	2505.72
Epoxy: Al. Powder (94:6 wt%)	14.13	43.30	3886.15
Epoxy: Al. Powder (91:9 wt%)	15.14	31.55	3886.08

Table 6: Flexural test results of composites at different percent filler loading.

Impact testing of simple composites

Epoxy resins tend to be brittle if not properly cured and resin to hardener ratio is not optimized. In the present study epoxy:hardener ratio of 10:8 wt% was optimized to get better impact strength. Toughness of composites is very important to use them for load bearing applications in automobiles [9]. The composites containing different fillers at different loading levels were impact tested and the result is shown in Fig. 4. From the results it can be seen that, in red mud filled composites, impact strength increased with increasing in filler concentration whereas it decreased with aluminium filler loading. Maximum impact strength of 139 J/m was recorded for 3% fly ash filled composite.

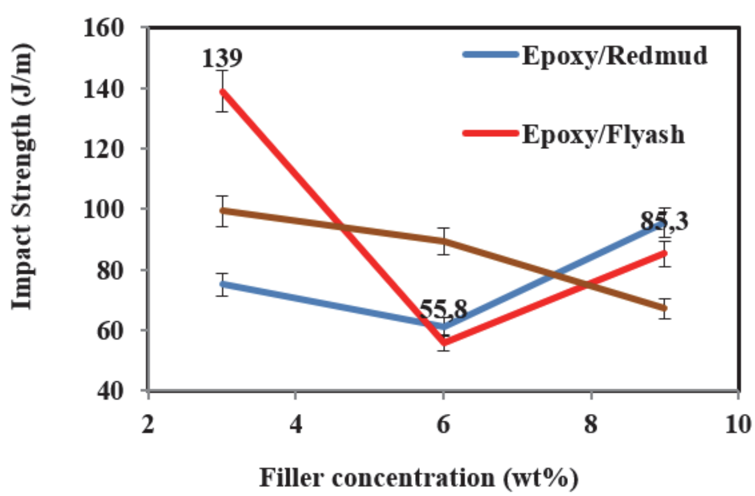
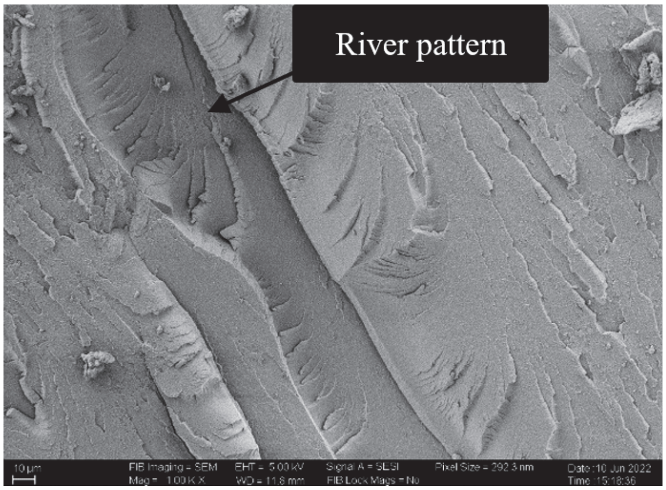


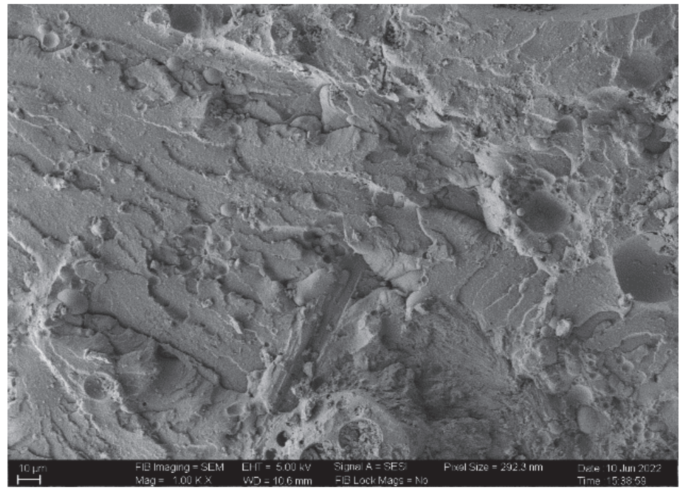
Figure 4: Impact strength of composites at different filler loading.

Morphological analysis of composite fractured surface

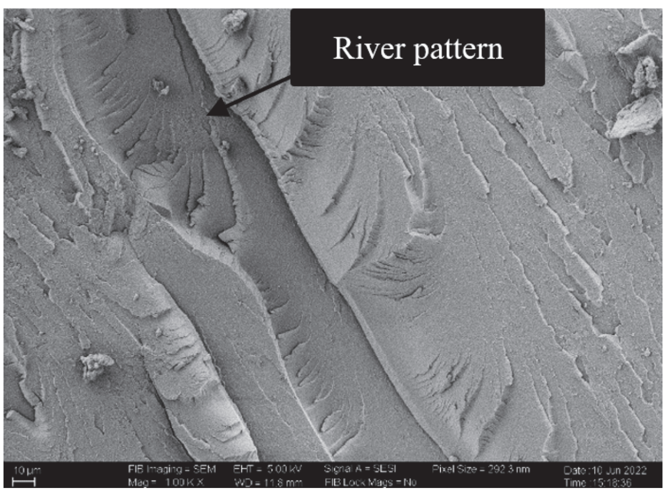
The morphological analysis of tensile fractured surfaces of plain epoxy resin, 9 wt% filled red mud, fly ash and aluminium powder filled composites were investigated using scanning electron microscope. The SEM images of tensile fractured surfaces are shown in Fig. 5. Micrograph shows that fly ash being finer in size (around 25 μ) has intimately mixed with the matrix without much separation at the interface and bonded well with the epoxy due to its hydrophilic nature.



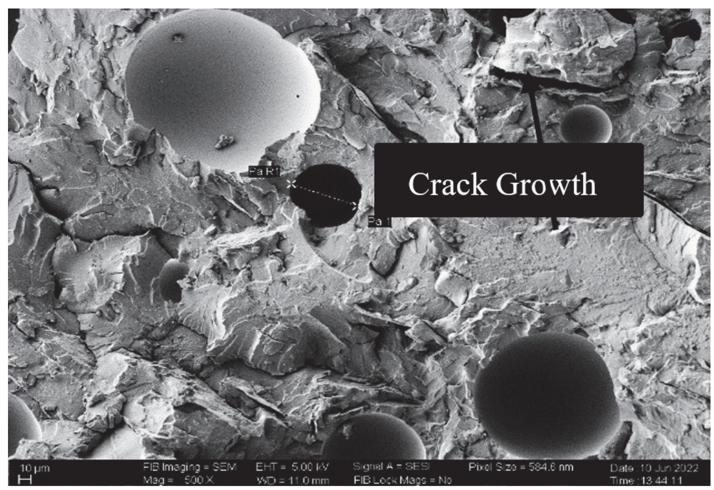
(a) Neat epoxy.



(b) Fly ash filled composite.

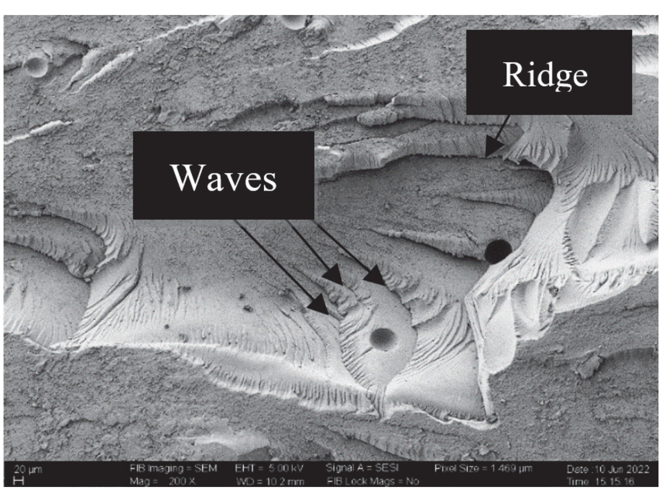


(c) Red mud filled composite.



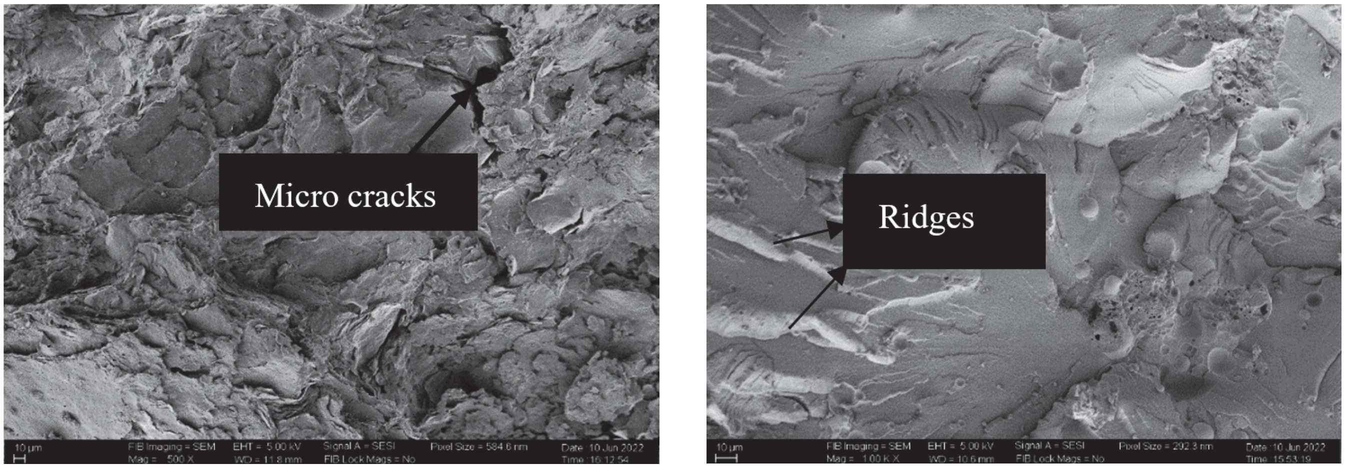
(d) Alumnium powder filled composite.

Figure 5: SEM images of tensile fractured surfaces of neat epoxy and its composites.



(a) Neat epoxy.

(b) Red mud filled.



(c) Aluminium powder filled. (d) Fly ash filled.

Figure 6: SEM images of impact test fractured surfaces of neat epoxy and its composites.

Neat epoxy shows river pattern morphology at the fracture surface. In case of red mud and aluminium filled composites micro voids can be seen on fractured surfaces, also filler pullouts because of weak matrix/particles bonding. This may be the possible reason for lower peak load recorded with these composites [8]. Red mud filled composites shows crack pinning effect whereas the alumnum powder filled composites shows the crack deflection effect. Both the mechanism of crack propagation arrest is encouraging for filled composites. The impact fractured surfaces of composites were also SEM analysed to probe the causes of failure. The morphological analysis of impact fractured surfaces of neat epoxy, 3 wt% filler loaded composites shows (Fig. 6) minor cracks, river patterns and micro voids in general, this can be attributed to weak matrix/particles bonding. The alumnium filled composites shows very rough morphology and more discontinuity/cracks indicating poor interfacial adhesion, hence lower impact strength.

Mechanical properties of hybrid composites

To study the effect of combination of fillers on composite properties, hybrid composites were prepared using similar procedure as simple composites but with mixture of different types of fillers. For the highest filler loading of 9 wt% in hybrid composites, fly ash was fixed at 6 wt% because of the fine particle size, good interfacial bonding with epoxy, low density and also from simple composite testing analysis it was found that all the mechanical properties tested were superior for 6 wt% loading level. The Tab. 7 shows the composition of hybrid composite prepared and its designations.

Hybrid composite composition	Designation
Epoxy (91 wt%) + Fly ash (6 wt%) + Red mud (3 wt%)	E/F/R
Epoxy (91 wt%) + Fly ash (6 wt%) + Al. Powder (3 wt%)	E/F/A
Epoxy (91 wt%) + Fly ash (6 wt%) + Red mud (1.5 wt%) + Al. Powder (1.5 wt%)	E/F/R/A

Table 7: The hybrid composites designation.

Hardness of hybrid composites

The hardness values of various types of hybrid composites were determined using shore D durometer and results are shown in Fig. 7. The hardness value of epoxy/fly ash/red mud/aluminium powder was higher (75.2) compared to other hybrid composites and neat epoxy, because of closed packing and strong interaction between the hybrid fillers with epoxy matrix. Due to wider range of size distribution (25-200 μm) among the selected fillers, there was an efficient packing observed with hybrid composites, which confine the resistance to dimensional distortion.

Tensile testing of hybrid composites

The specimens of hybrid composites were prepared by cutting the sheet of composite and tensile tested at the grip separation speed of 1.5 mm/min. The results are tabulated in Tab. 8. The modulus of all the hybrid composites were higher than the neat epoxy, which indicates better interfacial adhesion between epoxy/fly ash/red mud/aluminium powder filled hybrid composite.

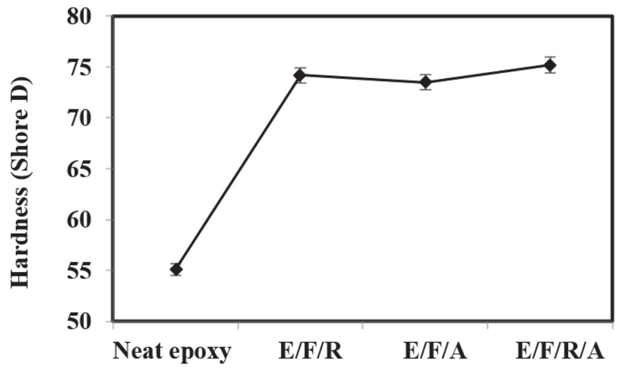


Figure 7: Hardness values of hybrid composites

Composition	Peak load (kg)	Tensile strength at peak load (N/mm²)	Ultimate modulus (MPa)	Hardness (Shore D)
Neat epoxy	316.9 ± 3.2	23.2 ± 1.1	375.6 ± 3.2	55 ± 1
E/F/R	254.2 ± 2.1	20.7 ± 1.4	428.6 ± 4.1	74 ± 3
E/F/A	258.2 ± 2.6	20.4 ± 1.0	444.6 ± 3.8	73 ± 2
E/F/R/A	280.5 ± 4.1	22.1 ± 0.6	466.1 ± 4.4	75 ± 3

Table 8: Tensile test results of hybrid composites.

Flexural testing of hybrid composites

The flexural test was conducted using the same procedure as followed for the simple composites. The E/F/R/A composites showed better flexural strength than other hybrid composites as shown in Tab. 9. The hybrid composite sometime shows the synergistic behavior than simple composites in many properties. This may be due to interactions among the different fillers or change in interaction pattern of hybrid fillers with matrix [6].

Composition	Peak load (kg)	Flexural strength at peak load (N/mm²)	Ultimate modulus (MPa)
Neat epoxy	16.38 ± 0.7	45.32 ± 1.1	2751.42 ± 12.3
E/F/R	15.00 ± 1.1	45.65 ± 0.8	3871.28 ± 14.5
E/F/A	15.12 ± 1.5	41.67 ± 1.3	2188.77 ± 11.6
E/F/R/A	17.55 ± 1.3	47.69 ± 1.8	3030.23 ± 13.6

Table 9: Flexural test results of hybrid composites

Hence it can be concluded that aluminium filler in combination with red mud and fly ash gives superior strength to composite than its only addition to epoxy. So, the proposed composite can be light weight, cost-effective and more robust than plain epoxy matrix for automotive applications.

CONCLUSIONS

In the present study, industrially generated waste/byproducts were used as fillers (fly ash, red mud and aluminium powder) with epoxy matrix for the preparation of simple and hybrid composites. These fillers were mainly selected to decrease the overall cost of composites and to increase the bulkiness without much compromise in the performance or properties, also to provide value addition to the waste. The fillers characterization, composite preparation and mechanical properties evaluation of prepared composites were undertaken. Also, morphological characterizations of fractured surface of the composites were carried out to understand the failure mechanism of filled composites under loading condition.

The following conclusions were drawn from the result analysis:

- The 10:8 epoxy : hardener weight ratio is better with respect to strength and toughness.
- As expected, the hardness of all the composites were found to be higher, which indicates the improved resistance of composites against deformation/indentation.
- Tensile strength of composites is not much changed with filler type and is more dominated by matrix property.
- The results suggested that epoxy with fly ash (3 wt%) showed better flexural strength of 67.53 MPa, highest impact strength of 139 J/m and ultimate tensile modulus of 3690 MPa. Hence fly ash can be a better filler for cost reduction without much compromise in the properties.
- Neat epoxy tensile fractured surface as characterized by SEM shows river type stretched marks. In red mud, fly ash and aluminium powder reinforced composites, crack growth, micro voids were seen on the fractured surface. This again confirms the poor interfacial adhesion, which is responsible for decrease in mechanical strength at higher filler concentration.
- SEM analysis shows that fly ash being finer in size has intimately mixed with the matrix without much separation at the interface and bonded well with the epoxy due to its hydrophilic nature.
- In case of red mud and aluminium filled composites micro voids can be seen on fractured surfaces, also filler pullouts because of weak matrix/particles bonding.
- Alumnium powder filled composites shows more cracks/discontinuity indicating poor adhesion between matrix and filler this may be due to large surface energy difference between epoxy and aluminium.
- Though aluminium alone was not a good filler to improve the mechanical properties of composites, in combination with red mud and fly ash it works better.
- The hybrid composite prepared is a light weight and more robust than plain epoxy matrix for automotive applications.

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