Research Article

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Effect of rice husk (treated/untreated) and rice husk ash on fracture toughness of epoxy bio-composite

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Abstract: Present work studies the effect of particle reinforcement on fracture toughness of bio-composites. The filler used has been taken as rice husk. Epoxy resin has been taken as matrix material. Composites with varying filler loading of 10, 20, 30 and 40 wt.% were fabricated. The fracture toughness was seen to be increasing with increase in filler loading. However beyond 20% there was a decrease in fracture toughness with increase in filler loading. The effect of fibre treatment on toughness was also observed. Rice husk fibres pre-treated with NaOH were used. It was observed that fracture toughness further improved due to treatment. The increase in fracture toughness was significant. Fracture toughness increased from 1.072 to 2.7465 MPa√mm for 20% reinforcement and after treatment it increased to 2.876 MPa√mm. It was observed that concentration of treatment media also affects the fracture toughness. Further the effect of hybridization was observed by addition of rice husk ash as a secondary reinforcement. The fracture toughness of the resulting composites was remarkably higher than that of pure epoxy.

Keywords: rice husk, rice husk ash, fracture toughness, epoxy bio-composite

1 Introduction

Composite materials have been in use for past many years owing to the advantage of obtaining better mechanical properties. In particular glass, carbon, mica and synthetic fibres have a large market share which is ever increasing. The main limitation of these fibres however, is their non-biodegradable nature along with their hazardous effects.

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The environmental awareness in the last decade has shifted the focus to develop eco-friendly composites. As far as applications requiring lower mechanical strengths are concerned a possible alternative can be the natural fibres which are fibres obtained from plants and animals. Natural fibres can be considered as naturally occurring composites consisting mainly of cellulose fibrils embedded in lignin matrix. The cellulose fibrils are aligned along the length of the fibre, which render maximum tensile and flexural strengths, in addition to providing rigidity.

One such fibre which has attained much attention is rice husk. Rice is an agricultural product with an annual production of 759.6 million tons in 2017 [1], of this 22% of this is husk [2] (167.10 million tonnes) an agricultural waste, which give an enormous amount of virtually free of cost raw material. Rice husk has been utilized as a reinforcing material both in raw form and as flour with different matrix materials. Grounded rice husk was seen to impart higher tensile strength compared to raw rice husk [3], polyethylene was used as the matrix material. Rice Husk has been used with most of the polymers like polypropylene [4–6], polyethylene [7–9], polyester resin [10], polyurethane [11] and epoxy [12]. Most of the studies observed that there was a decrease in mechanical strengths due to addition of Rice Husk which can be attributed to the improper adhesion between fibre and matrix. In some of the studies however the strength increased but for low level of filling, which can be attributed to the tendency of rice husk particles to agglomerate [13]. Most of these studies have focused only on the tensile and flexural properties. Studies on the fracture behaviour of bio composites have been few. It can be said that application of fracture concepts to composites is at nascent stage. Most of the standards developed are based on homogeneous materials while the behaviour composites can be isotropic but particle reinforced composites can be idealized to be homogeneous in nature and hence the concepts of fracture mechanics can be applied to the composites. Mode I fracture toughness of bio shell and bio fibre reinforced epoxy bio-composite was studied [14], it was observed that for all the bio fibres used there was a significant improvement in fracture toughness compared to neat epoxy. Sugar palm fibre reinforced epoxy bio-composite
also showed significant improvement in properties compared to neat epoxy [15]. Fracture parameters are essential in accessing the structural integrity and for improvement in the in-service life of the components. The reason behind degradation in mechanical properties due to fibre addition was due to improper adhesion between fibre and matrix and agglomeration of rice husk particles. Certain chemical modifications viz. mercerisation [16, 17], compatibilizer [18], electron beam irradiation [19], plasma treatment [17] etc. have had positive impact on the adhesion between fibre and matrix thereby improving mechanical properties. These processes either change the nature of fibres or create secondary bonds of Vander Waal kind [20].

The effect of chemical treatment on RH reinforced polyethylene composites was done [8]. In the study RH was treated with benzene diazonium salt in presence of alkali, acidic and neutral media. The pH levels were maintained at 10.5, 7 and 6 for basic, neutral and acidic media respectively. The alkali media increased the tensile strength by about 11%, while for acidic media it was 3.5% and for neutral medium there was negligible increase. The results for the Young’s Modulus also showed the same behaviour. The flexural properties were also seen to be increasing after treatment. The increase in flexural strength was similar for acidic and basic media but alkali media was better as far as flexural modulus is concerned. In case of impact strength the results were same with alkali treatment being better than acidic medium followed by neutral media and then raw RH.

Plasma treatment of RH [17] resulted in a significant improvement of mechanical properties of RH composites. Similarly, electron beam irradiation [19] improved the mechanical properties significantly.

Effect of treatment with various concentration of NaOH solution was done [16]. The effect of treatment was observed on wear characteristics and it was observed that wear characteristics improved with higher concentration possibly due to removal of lignin and waxes. The improvement in properties was however limited to 8% concentration. The treatment above this concentration resulted in excessive deterioration resulting in lower wear characteristics.

Hybrid composites for improving the matrix-dominated properties of continuous fibre reinforced composites were perhaps suggested in 1987 [21]. In this technique, a supplementary reinforcement such as particulates, whiskers, or micro fibres is added to the matrix prior to resin impregnation.

Recently Sisal fibre based polymer composites were developed [22]. Their properties were tailored by hybridizing them with natural pine needles and agro waste mustard. A remarkable improvement in mechanical and wear properties was observed.

Specifically rice husk has been used to develop hybrid bio-composites with Bauhinia-Vahlili-weight and Bauhinia-Vahlili-weight/sisal fibres [23]. The mechanical properties like tensile strength and flexural strength improved significantly (34.42 and 33% respectively) and hardness by 7.1% due to hybridization. RH particulates with glass were also used for epoxy matrix [24]. The hardness, tensile modulus and impact energy improved while there was deterioration in tensile and flexural strength.

RHA is 92% silica and can be beneficial in improving the mechanical properties due to the inherent properties of silica [25], in the study they have used RHA as reinforcement in Aluminum alloy (AlSi\textsubscript{10}Mg) Matrix Composites. In another study Rice husk derived silica has been used to develop natural rubber composites and experiments have been conducted for mechanical properties [26]. In another study [27] composite materials were obtained by filling polypropylene with raw and thermally treated rice husks at filler contents from 1 to 20%. The physicochemical properties were determined. A slight increase was observed in the tensile strength of the composites based on white rice husk ash WRHA (and aerosil AR) at degree of filling up to 3%.

Keeping in mind the above work carried out till now the effect of rice husk as filler on fracture toughness is investigated, further the impact of mercerization on the fracture parameter is also considered since fracture parameters can be critical in determining the application of bio-composites this manner.

2 Methods

2.1 Materials and manufacturing

Rice husk obtained from a local mill was thoroughly washed and sun dried to remove moisture. Thereafter it was grounded to sieve size of 125 microns. Moulds were prepared using perpex sheets of 10 mm thickness. Commercially available resin CY-231 was used as the matrix material along with hardener HY-951 as the curing agent. Samples with different concentration of rice husk were prepared. The weight percentage of rice husk was kept as 10, 20, 30 and 40. Sample with alkali treated rice husk was also prepared. For this purpose NaOH was used. Rice husk flour was soaked in 8% volume/volume NaOH solution for 5 hours and then was thoroughly washed with distilled water to remove any traces of NaOH. After this the flour was sun dried to remove moisture.
For casting rice husk flour was mixed with the resin and well stirred and then kept in a furnace for 2 h at 110°C. The mixture was allowed to cool down to 40°C and then hardener was mixed and after stirring for a minute the mixture was poured into the moulds using the hand lay-up technique [28]. The ratio of resin and hardener was kept as 9:1. The casting was allowed to stand for 24 h under gravity after which the moulds were opened and various specimens were prepared. Silicone grease was applied on the inner walls of the mould for easy removal of the casting.

2.2 Preparation of rice husk ash

Different processes have been used by different researchers to obtain silica from rice husk. The process which has been utilised here as proposed [26] can be summarized in following steps:

1. Rice husk was washed with water to remove any foreign material.
2. Hydrochloric acid solution of 0.4 ML⁻¹ was prepared than 100 g cleaned husk was mixed in 1 L of prepared acid solution and boiled at 100–105°C for 30–45 min. After the reaction, the acid was completely removed from the husk by washing with tap water.
3. It was then dried in an oven at 110°C for 3–5 h in oven.
4. The treated husk burned in an electric furnace at 600°C for 6 h; silica was obtained as white ash. The shape of the silica is similar to the shape of the husk but smaller in size.

2.3 Fracture toughness specimen

The estimation of fracture parameters is essential for service life evaluation. Failure of most in service materials starts with crack nucleation and propagation so it becomes important from structural integrity point of view. Fracture toughness of a material is its resistance to crack extension. The fracture toughness test standards ASTM E399-19 ASTM D5045-20 were developed to determine the value of plane strain fracture toughness at or near to the onset of crack initiation. Six types of conventional fracture test specimens are permitted in ASTM fracture test standards. These include compact tension (C(T)) specimen, single edge-notched bend (SEN(B)) specimen in three-point bending, middle-cracked tension (M(T)) panel, disk-shaped compact tension (DC(T)) specimen, arc-shaped tension (A(T)) specimen, and arc-shaped bend (A(B)) specimen.

In this case the SENB specimen has been employed to evaluate fracture toughness of various specimens. The SENB specimen was chosen because of simple design and loading condition, cheap preparation procedure, convenient testing set up and the ability of introducing complete mode mixities. Here we have only focused on the mode I stress intensity factors.

The SENB specimen used for testing is shown in Figure 1 where W = width of the specimen, L = span length, B = thickness and “a” is the crack length. The crack is made normal to the base and at the centre of the specimen. The crack was made using an electrical saw. A sharp pre-crack is introduced in the bend specimen by lightly tapping a sharp new razor blade of thickness 0.2 mm into the tip of the mechanical slit or notch.

![Figure 1: SENB specimen](image1.png)

![Figure 2: SENB specimen](image2.png)
Following equation was used to obtain critical stress intensity factor in mode I
\[ K_I = \frac{PS}{BW^{3/2}} f \left( \frac{a}{W} \right) \] (1)
\[ \infty = \frac{a}{W} \] (2)
\[ f \left( \frac{a}{W} \right) = \frac{3^{-1/2} \left[ 1.99 - \infty (1-\infty)(2.15 - 3.93 \infty + 2.7 \infty^2) \right]}{2(1+\infty)(1-\infty)^{3/2}} \] (3)

Using the fracture load obtained from each specimen, the critical stress intensity factor was calculated. Figure 2(a) & 2(b) represent the sample and fracture specimen.

Fracture toughness tests are conducted at room temperature in a servo hydraulic universal test system ADMET, USA make.

3 Results and discussions

3.1 Effect of RH addition

Figure 3 represents the fracture toughness with varying concentration of rice husk in comparison to pure epoxy. It can be seen that the fracture toughness increases due to addition of rice husk up to 20% filler loading. The fracture toughness for pure epoxy is 1.028 MPa and it increases to 1.275 MPa showing a remarkable increase of around 25% for 10 wt% composite. For 20 wt% composite the increase was even more significant at 159%. This can be attributed to the proper adhesion between the fibre and the matrix and there is a proper transfer of load to the fibres.

However, further increase in rice husk content results in decrease in fracture toughness it however remains greater than pure epoxy. Fracture toughness is basically the resistance by a material to crack growth and when rice husk particles are added to the epoxy resin they form a barrier to crack propagation and hence fracture toughness increases. But at higher level of filler loading the toughness reduces because at higher filler loading there are voids created in the material and also rice husk tends to agglomerate as can be seen in the SEM images. Still the fracture toughness is considerably higher than the pure epoxy up to 40% filler loading. Further to observe the effect of fibre treatment on fracture toughness the filler level was chosen as 20%, value at which fracture toughness was highest.

3.2 Effect of NaOH treatment

To improve the adhesion between fibre and the matrix material, RH was pre-treated with NaOH solution. The concentration of NaOH solution was also varied and its influence on various properties has been observed. In the above section it has been seen that 20 wt% is the optimized filler loading and so all the composites fabricated from now on are based on 20% RH. In a study it was seen that the mechanical strength with alkali treatment improved up to 8% NaOH solution beyond which there was an excessive deterioration of fibres and strength decreased [29]. The tests have therefore been conducted on 8% NaOH solution.

From Figure 4 which shows the comparative fracture toughness for epoxy, 20 wt% reinforced rice husk and 8% NaOH treated rice husk it can be seen that although the fracture toughness improves due to treatment, the increase was not as significant. The increase due to treatment compared to untreated fibre was 3.12% and compared to pure epoxy was 167%. As has been explained in the previous section the reason behind the toughening due to particle addition is the barrier to crack propagation so treatment per se does not have much impact on the toughening mechanism since the reinforcement quantity was same at 20%; however the limited improvement in toughening parameter was due to improved adhesion between the matrix and the particles in which the nature of hydrophilic fibres changes to hydrophobic and hence an improved adhesion occurs.

The effect of RHA addition on the fracture toughness is shown in Figure 5. For studying RHA’s influence on fracture toughness its value has been compared with pure epoxy, 20 wt% untreated RH composite, 20 wt% treated RH composite. The fracture toughness corresponding to 1% Si has been taken. As can be seen RHA addition increases the fracture toughness from 2.746 to 2.952 MPa an increase of around
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Figure 4: Comparison of fracture toughness of pure epoxy, untreated 20 wt% reinforced and 8% alkali treated (20 wt%) reinforced composite

75%. The increase in toughness can be due to the dispersion of second reinforcement in the matrix due to which the barrier to crack extension improves and for the crack to propagate it either has to change the course or more load has to be applied.

Figure 5: Comparison of fracture toughness of pure epoxy, 20 wt% untreated RH composite, 8% treated (20 wt%) and 1% Si reinforced composite

Figure 6 shows the load deflection curve for fracture specimens of composites with different compositions. It can be seen that for 20 and treated 20 wt% reinforcement mode of crack extension is plastic in nature and therefore there is an increase in fracture toughness. For RHA reinforced composite it can be seen that the load bearing capacity of the material has increased due to proper dispersion of small sized silica particles and therefore acts as barrier to crack extension. For 10 wt% reinforcement also the mode of failure is plastic in nature thereby increasing the fracture toughness compared to pure epoxy. While for 30 and 40 wt% it can be seen that mode of failure is purely elastic and so the mode I fracture toughness decreases compared to 10 and 20 wt.% reinforcement of pure RH.

3.3 SEM tests

Scanning electron microscopy was conducted on the different samples at G.B. Pant University of Agriculture and Technology.

Figures 7–9 show the SEM images for 20 wt% untreated RH, 20 wt% 8% NaOH treated and 20 wt% 8% NaOH treated+1%Si composites. From the images it is clear that fibre treatment improves the adhesion between the fibre and the matrix imparting greater strength to the composite. It can also be observed that addition of 1% Si results in a smoother surface of the bio-composite. The little rough-
ness on the surface of second sample is reduced imparting higher fracture toughness to the resulting composite.

3.4 TGA analysis

Thermal analysis of the specimen was conducted at Institute Instrumentation Centre, Indian Institute of Technology, Roorkee. For thermal analysis the samples were prepared in powder form by crushing them and the weight of samples were near 10.5 mg and the base material was alumina powder, which was mixed with composite samples in equal amount and medium in which the tests were conducted was air flowing at 20 ml/min. The rate of change of temperature was 10°C/min and range of temperature was room temperature to 1000°C.

Figure 10 shows thermo gram of neat epoxy with 10 wt% of hardener. Decomposition of this material has been accomplished under two stages ranging from 356°C to 510°C with corresponding rate of decomposition ranging 1.27 mg/min to 0.72 mg/min. Prior to 200°C, the weight loss of 7.63% may be attributed to the expulsion of the moisture, low molecular mass molecules and volatile matter associ-
Figure 11: Thermal analysis of 20 wt% RH reinforced epoxy

Figure 12: Thermal analysis of 20 wt% RH (8% NaOH treated) + 1 wt% RHA reinforced epoxy

ated with the material. The maximum rate of decomposition of 1.27 mg/min was observed at 356°C. Such decomposition has been supported with the heat of fusion of $-2.487 \text{ J/mg}$ centred in the temperature range of 279°C to 511°C with DTA signal of 28.4 $\mu$V to 109.2 $\mu$V. The decomposition of the material had been concluded at 536°C leaving char residue 3% of initial weight.

Figure 11 shows thermo gram of epoxy reinforced with 20 wt% of RH. Decomposition of this material has been accomplished under three stages ranging from 197°C to 301°C and 556°C corresponding rate of decomposition ranging 0.25 mg/min, to 0.54 mg/min and 0.34 mg/min. Prior to 200°C, the weight loss of 11.89% may be attributed to the expulsion of the moisture, low molecular mass molecules and volatile matter associated with the material. The maxi-
mum rate of decomposition of 0.54 mg/min was observed at 301°C. Such decomposition has been supported with the heat of fusion of −1.09 J/mg centred in the temperature range of 302°C to 545°C with DTA signal of 51.3 µV to 44.1 µV. The decomposition of the material had been concluded at 700°C leaving char residue 4.887% of initial weight.

Figure 12 shows thermo gram of epoxy reinforced with 20 wt% of treated RH and 1% RHA. Decomposition of this material has been accomplished under three stages ranging from 192°C to 304°C and 529°C corresponding rate of decomposition ranging 0.17 mg/min, to 0.47 mg/min and 0.30 mg/min. Prior to 200°C, the weight loss of 11.34% may be attributed to the expulsion of the moisture, low molecular mass molecules and volatile matter associated with the material. The maximum rate of decomposition of 0.47 mg/min was observed at 304°C. Such decomposition has been supported with the heat of fusion of −4.52 J/mg centred in the temperature range of 305°C to 522°C with DTA signal of 44.5 µV to 45.1 µV. The decomposition of the material had been concluded at 700°C leaving char residue 9.462% of initial weight.

4 Conclusions

The following conclusions can be made:

1. Fracture toughness of epoxy improves significantly by 157% due to addition of rice husk particles up to 20% filler loading. It was found to be 25% for 10% RH reinforcement. Above 20% the fracture toughness starts decreasing but is still significantly higher than pure epoxy.

2. Treatment of rice husk particles has a profound effect on improving the adhesiveness of rice husk with epoxy thereby increasing its fracture toughness. It increases by 3.12% compared to 20 wt.% RH reinforced epoxy.

3. Hybridization with rice husk ash also improves the fracture toughness by 715% compared to treated sample and as such rice husk ash is a suitable component for developing bio composites. Compared to pure epoxy it is an increase of 167%.

Conflict of Interests: The authors declare no conflict of interest regarding the publication of this paper.

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