RESEARCH ARTICLE OPEN ACCESS

Mechanical properties of rice husk flour reinforced epoxy bio-composite

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ABSTRACT
A bio-composite reinforced with rice husk flour in epoxy resin has been developed. The effect of fibre treatment and weight percentage of rice husk on the mechanical properties was studied and compared with wood dust reinforced epoxy composite. It was observed that addition of rice husk as filler is detrimental to almost all the mechanical properties. About 51% and 26.8% decrease in ultimate strength and Young's Modulus for 40 wt% untreated rice husk reinforcement was observed. The corresponding decrease in flexural strength and flexural modulus was 51%. Similar trend was also observed in hardness and impact strength. However the mechanical properties of rice husk reinforced biocomposites are found to be superior than wood dust reinforced epoxy composite. SEM microscopy was also done to corroborate the results.
Keywords: Rice husk, epoxy, t-test, mechanical properties

I. Introduction

The growing demand in the world for more versatile materials suitable for various industrial applications led to the development of polymeric composites with synthetic fibres as reinforcing agents. However in the recent decade there has been growing concern and protest against the use of these synthetic chemicals which have been proven to have adverse impact on the already stressed state of environmental pollution. This has shifted the focus on utilising natural fibres as replacement of synthetic fibres, capitalising on their advantages of being economical, environment friendly, lower densities, higher filling levels, recyclability, biodegrability and renewable nature [1, 2].

Rice husk (RH) which is a major agricultural waste has been utilized as a filler material extensively. The RH has a huge potential as a possible fibre because rice production worldwide is 700 million tons [3] of which around 22% i.e. 154 million tons is husk [4] and this husk has no potential use and its disposal otherwise is very difficult. It contains cellulose, hemicelluloses, lignin, and ash. According to Marti-Ferrer [5] the lignin and hemi-cellulose contents of rice husk are lower than wood whereas the cellulose content is similar. For this reason rice husk flour can be processed at higher temperatures than wood. Therefore, the use of rice husk in the manufacture of polymer composites is attracting much attention.

Rice husk has mostly been employed as flour however researchers have also utilised chopped rice husk [6]. Different polymers have been used as matrix material. RH was incorporated in polypropylene by Yang et al. [7], Premalal et al. [8], Dimzoski et al. [9], Rosa et al. [5] to name a few. Besides polypropylene rice husk has also been used with other polymeric materials. Ghofrani et al. [10], Atuanya et al. [11], Rahman et al. [12], Nawadon et al. [13] have used polyethylene, Attharangsan et al. [14], Ramasamy et al. [15] have used rubber as the matrix. Besides these matrix materials rice husk has also been used with polyurethane [16], resin [17], poly-lactic acid [18], vinylester [19], tires [20] etc. All studies on rice husk have focused on the effect of filler loading on the mechanical properties. In most of the studies it was observed that with addition of fillers most of the mechanical properties decreased. The tensile strength, flexural strength, impact strength and hardness were seen to decrease, however there was an increase in Young's Modulus with filler loading. The incorporation of filler also made material brittle. However, keeping in mind the advantage of using rice husk as filler material, there still is scope of utilisation of rice husk as reinforcing agent.

II. Experimental Details

2.1 Materials

In the present investigation CY-230 and HY-951 purchased from M/s Excellence Resins Limited, India was used as matrix and hardener respectively. Rice husk obtained from a local rice mill with rice produced in India was used as the reinforcing agent. The rice husk obtained from mill was washed with tap water thoroughly to remove any foreign impurities and sun dried to a moisture level less than 2%. Thereafter it was grounded and rice husk flour is separated on the basis of their sizes with the help of sieve and the rice husk flour obtained with the sieve of ASTM 120 (125 microns) was used for composite fabrication.

2.2 Fabrication

Moulds were prepared using 12 mm thick Perpex sheets. Different weight percentage (wt %) of RH fibre (10, 20, 30 and 40 wt %) and epoxy resin were mixed by mechanical stirring at 3000 rpm in different beakers. The solution obtained by mixing of RHF in resin is kept in the furnace at a temperature of 90 ± 10 °C for two hours. At each interval of 30 minutes the solution is taken out from the electric furnace and remixed by mechanical stirrer at same speed. After two hours the whole solution is taken out and allowed to cool to a temperature of 45°C. When a temperature of 45°C has been attained the hardener HY-951 (9 weight per cent) is mixed immediately [21]. Due to addition of hardener high viscous solution is obtained which is remixed mechanically by the mechanical stirrer. The viscous solution is then poured into different moulds for sample preparation.

2.3 Mechanical testing

Tensile and flexural tests were conducted on 100 kN ADMET make servo controlled universal testing machine according to ASTM D3039 and ASTM D790-10 respectively. The crosshead speed is kept 0.5 mm/min for all tests. Different parameters related to flexural test were calculated using Equations (1) to (3):

$$\sigma_f = \frac{3FL}{2bt^2} \quad (1)$$

$$E_m = \frac{mL^3}{4bt^3} \quad (2)$$

$$\varepsilon_f = \frac{6Dd}{L^2} \quad (3)$$

where, σ_f= flexural stress, E_m= flexural modulus, ϵ_f= flexural strain, where

F= maximum load, L=span length, d= depth of the specimen, b= width of specimen, m= slope of initial straight line of load deflection curve, D= maximum deflection of the centre of the beam.

Izod impact test were conducted according to according to ASTM D256-02$^{\varepsilon 1}$.

Hardness values were measured on the automatic Digital Rockwell Hardness Testing Machine on L-Scale. The results are discussed in chapter 3.

2.4 Morphological study

The scanning electron microscopy was done on fractured surface of tensile specimens using LEO435V6 microscope. The microscope was operated at 10 kV. To obtain the scanning electrons micrographs square samples are cut from the fractured surface and are gold coated to avoid the artefacts associated with sample charging.

III. Results and Discussion

3.1 Tensile properties

Figs. 1-3 show the effect of filler loading on tensile strength, Young's Modulus and % elongation of RH reinforced bio-composite. It is seen that the tensile strength decreases with increase in filler loading. It is observed that the decrease in tensile strength is about 11, 19, 52 and 53 % for 10, 20, 30 and 40 wt% of RH respectively as compared to 42.5 MPa for pure epoxy. It shows that beyond 20 wt% of RH filler loading less than 50% of strength as compared to pure epoxy is obtained. Hence, it can be concluded that more than 20 wt% filer loading is uneconomical from strength point of view. Similar results were also reported by [7-9]. The decrease of tensile strength with increasing filler loading is due to the decrease in the worsening interfacial bonding between the matrix (hydrophobic) and fibre (hydrophilic). It has also been shown that for irregularly shaped fibres the strength of the composites can decrease due to the inability of the filer to support stress transferred from the polymer matrix [22]. The decrease in strength can also be attributed to one of the characteristic of rice husk flour that is agglomeration due to which voids can be generated between filler and matrix [23].

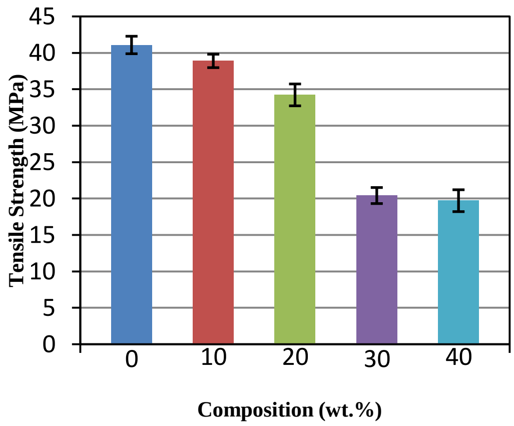

Figure 1 Tensile strength for different filler loading

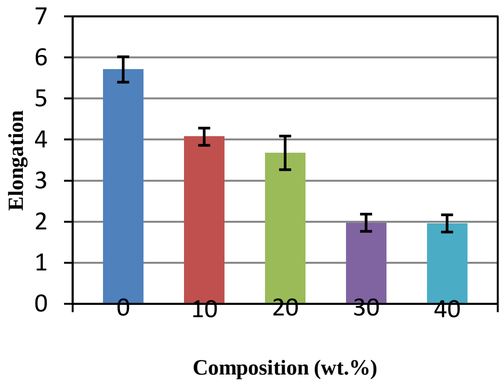

Figure 2 Elongation at break for different filler loading

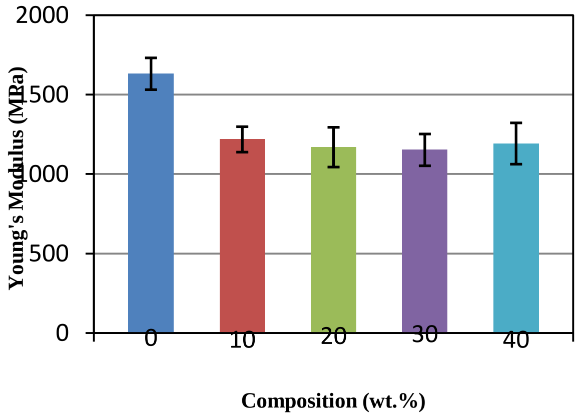

Figure 3 Young's Modulus for different filler loading

Fig. 2 for elongation at break reveals that addition of rice husk as a filler results in inducing brittleness into the composites. This can be adduced to the presence of voids which obstructs stress propagation and induce increased brittleness [7].

The Young's Modulus (Fig. 3) also decreases with increase in filler loading. The Young's Modulus for pure epoxy is 1600 MPa and it reduces to 1200-1350 MPa, a significant reduction of about 25% for 40 wt% filler loading. This behaviour may be understood from the fact that as the filler content increases more load is transferred from the matrix to the filler but since there is no proper adhesion between the filler and matrix there is slippage between the filler and matrix and hence the decrease in the stiffness.

Student's t distribution.

The statistical significance test using t-distribution was carried out to find the significant difference of tensile strength of 10, 20, 30 and 40 wt% reinforced biocomposites. Five samples for each test were taken and there standard deviations and mean were calculated using Equation 5-6

$$\sigma = \sqrt{\frac{n_1 s_1{}^2 + n_2 s_2{}^2}{n_1 + n_2 - 2}} \quad (5)$$

$$t = \frac{\overline{x_1} - \overline{x_2}}{\sigma\sqrt{\frac{1}{n_1} + \frac{1}{n_2}}} \quad (6)$$

where, n is the number of samples, s corresponds to standard deviation and $\bar{x}$ is the mean value of the sample. Subscripts 1 and 2 correspond to the sample group. The significance test results for different groups 10 and 20 wt%, 10 and 30 wt% and 10 and 40 wt% are shown in TABLE 1

Table1. Significance test results

Sample	t_{cal}
10-20%	2.460
10-30%	7.846
10-40%	9.080

The t value corresponding to 8 degree of freedom and 99% confidence level shows that between 10 and 20 wt% the difference is statistically insignificant with 99% confidence level, while for other groups the difference is significant. So keeping in mind the obvious advantage of high fibre replacement 20 wt% filler loading is taken as optimum filler loading.

3.2 Flexural Properties

From the Fig. 4 it can be seen that with the addition of RH as filler the bending strength is seen to be decreasing from around 63 MPa to 31 Mpa for 40% RH composite. Similar observations were made by Dimzoski et al. [9]. This is due to the increase in weak interfacial area between epoxy and rice husk. Flexural strain is also seen to be decreasing as the filler content is increased. The flexural strain (Fig. 5) decreased from 5.8% to 3.6% for increase in filler content from 10 to 40%. Decrease in modulus with respect to pure epoxy is significant; it decreased by 38%, 38.5%, 48% and 50% for 10, 20, 30 and 40% RH respectively (Fig. 6).

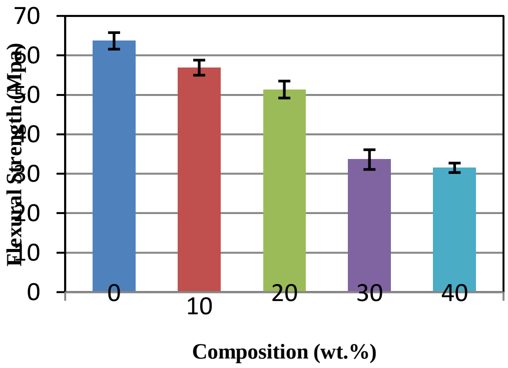

Figure 4 Flexural strength for different filler loading

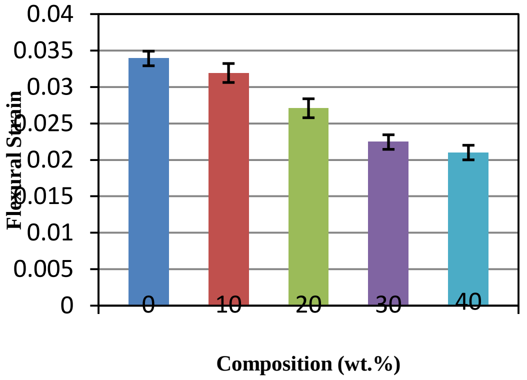

Figure 5 Flexural strain for different filler loading

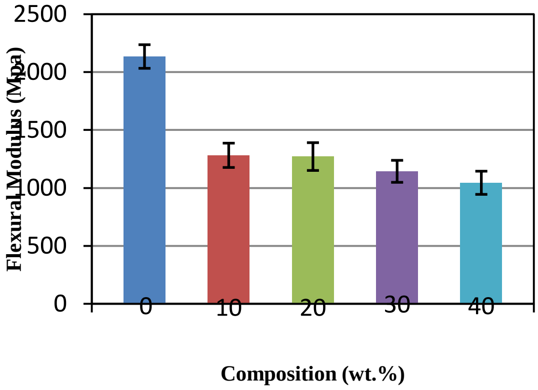

Figure 6 Flexural Modulus for different filler loading

The statistical significance test of flexural strength using t-distribution shows that the t values for 10 and 20 wt.%, 10 and 30 wt.%, 10 and 40 wt.% are 2.454, 8.809 and 9.703 respectively. The tabulated value corresponding to degree of freedom 8 and 99% confidence level is higher compared to 10 and 20 wt. % group and lower for other groups. This shows that there is no significant difference between 10 wt. % reinforced and 20 wt. % reinforced biocomposites. For the other two cases the difference is highly significant as shown in TABLE 2. It is therefore concluded that 20 wt. % is the optimum filler loading with respect to flexural strength also.

Table 2. t values for different groups

Sample	t_{cal}
10-20%	2.4539
10-30%	8.809
10-40%	9.703

3.3 Hardness Test

As known, hardness implies a resistance to indentation, permanent or plastic deformation of material. In a hybrid composite material, filler weight fraction significantly affects the hardness value of the hybrid composite material. Hardness values measured on the Digital Rockwell Hardness Testing Machine L-Scale showing the effect of weight percentage of RH flour on the hardness values of hybrid composite are presented in Fig. 7, it can be seen that the hardness of pure epoxy is reduced with the addition of RH fibres. The reason for this decrease can be attributed to the fact that the fibre is a softer material compared to the matrix and there is improper adhesion between the hydrophobic matrix and hydrophilic filler.

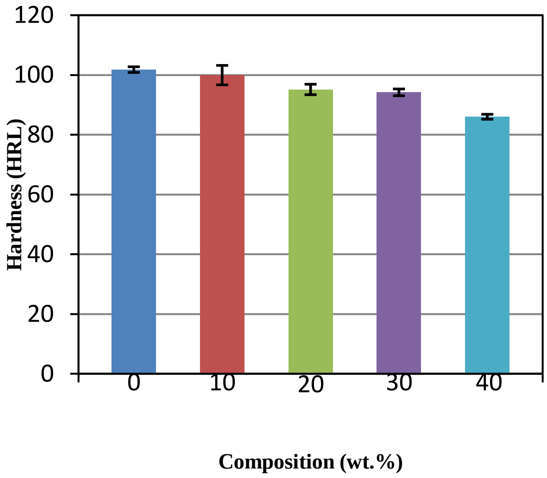

Figure 7 Hardness for different filler loading

3.4 Impact Strength

There is a gradual decrease in impact strength with increase in filler content for RHF composite which is shown in Fig. 8. The similar results are also reported in other works [7, 11]. When a crack is generated due to an impact it propagates towards a poor interfacial region [24]. The probability of poor interfacial region due to increased filler content is high as seen in SEM micrographs. Therefore, as the filler content increases, impact strength tends to reduce gradually. On the other hand, if the filler matrix adhesion is very strong, fillers restrict the mobility of the matrix molecules. In turn, this also results in reduction in impact strength.

The results shown in Fig. 8 can be seen to be confirming to the results as has been shown in literature available i.e. decrease with increase in filler loading. The decrease is quite significant compared to pure epoxy which are about 45%, 54%, 60% and 62% of pure epoxy corresponding to filler amount of 10, 20, 30 and 40 wt.% respectively.

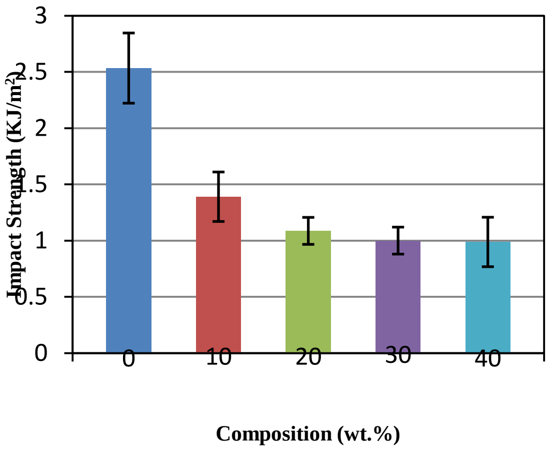

Figure 8 Impact strength for different filler loading

3.5 SEM Morphology

The scanning electron mictoscopy was conducted for 10, 20, 30 and 40 wt.% RH compossites and is shown in Fig. 9(a)-9(d). From Figures 9(a-d) it can be clearly seen that as the filler content increases there is a decrease in the interfacial bonding between the fibres and matrix, also there is agglomeration of RH particles the reasons behind the decrease in tensile and flexural srength. In Figure 9(d) the voids are clearly visible which shows improper adhesion.

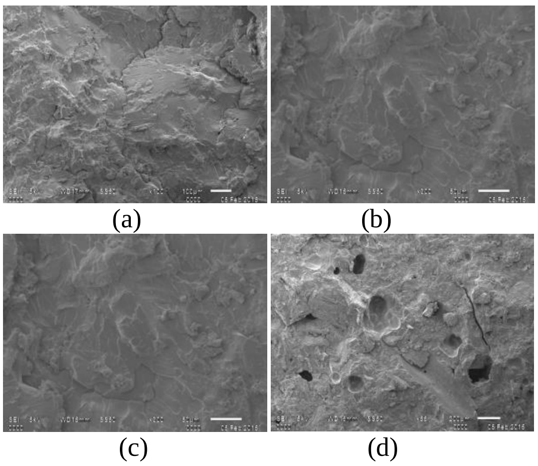

Figure 9 SEM images for fracture specimens for (a) 10% (b) 20% (c) 30% (d) 40% wt. % Rice Husk

IV. Conclusions

From the above results following conclusions can be made:

1. Incorporation of rice husk reduces the mechanical strength of the epoxy. Increase in rice husk content results in decrease in properties.
2. Impact strength and hardness were also seen to be decreasing with increasing filler content.
3. However rice husk as compared to wood dust can be a better reinforcement.
4. The reduction in mechanical properties can be attributed to improper adhesion between fibre and matrix.
5. It was also statistically proven that 20 wt. % rice husk is optimized filler loading.

V. Acknowledgement

The authors acknowledge College of Technology, Pantnagar for providing financial support under TEQIP-II.

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