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Synthesis and characterization of Fly ash /Wooden fiber reinforced Epoxy resin polymer composite ☆

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Abstract

Composite is made from two or more constituent materials with significantly different physical or chemical properties that, when combined, produce a material with characteristics different from the individual components. The mechanical property of a composite depends mainly on the physical and chemical properties of not only the resin, reinforcement used but also the hardener and solvent used. This paper highlights the experimental investigation on the effect of Acetone and Ethyl Alcohol solvent on composite laminates of teak wood powder and fly ash reinforcement with epoxy. The function of the solvent is to reduce the viscosity of the matrix to ensure the uniform distribution of the particulate reinforcement in the matrix upon stirring. Against this background, the present research work has been undertaken to explore the effect of acetone and ethyl alcohol solvent on wood powder and fly ash reinforced with epoxy. Hardener is added to the resin reinforcement mixture to ensure faster and uniform curing of the composite laminate at room temperature. But the hardener used (hy 951) being hydrophilic, absorbs moisture on exposure to atmosphere. The absorbed moisture adversely affects the curing of epoxy resin. This effect is more pronounced when the composite contains wood particles which also hydrophilic and absorbs moisture. Thus the present research also explores the effect of moisture on the curing properties of the composite. The present work thus aims to compare the effect of the ethanol and acetone solvent as well as the hardener properties on the flexibility of wood-fly ash epoxy resin fiber reinforced polymer composites using SEM, XRD, FT-IR test results.

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Keywords: Wood powder; Fly ash; Acetone; Ethyl Alcohol; SEM; XRD; FT-IR;

1. Introduction

The technological advancements, industries are constantly searching for a higher strength, lightweight materials to replace the conventional materials used. Particulate reinforced composites achieve gains in stiffness primarily, but also can achieve increase in strength and toughness [1-4]. Fly ash is one of the residues generated by coal combustion, and is composed of the fine particles that are driven out of the boiler with the flue gases. Since the particles solidify rapidly while suspended in the exhaust gases, fly ash particles are generally spherical in shape and range in size from 0.5 µm to 300 µm.

Wood powder is finely pulverized wood that has a consistency fairly equal to sand or sawdust, but can vary considerably, with particle size. Wood powder is also the main ingredient in wood/plastic composite building products such as decks and roofs [5-7]. Epoxy resins, also known as polyepoxides are a class of reactive prepolymers and polymers which contain epoxide groups. Epoxy has a wide range of applications, including metal coatings, use in electronics / electrical components, high tension electrical insulators, fiber-reinforced plastic materials and structural adhesives. The hardener (hy 951) used has low viscosity and helps in curing the epoxy resin at room temperature. The chemical reaction initiated by mixing resin and hardener results in generation exothermic heat. The hardener has to be stored in dry place at room temperature in sealed containers to avoid exposure to atmospheric moisture.

Acetone is a chemical solvent which has a vapor pressure of 30.6kPa at 25 ºC and has a boiling point of 56.05 °C. Ethyl alcohol is a colorless, flammable liquid whose vapor pressure is 7.86 kPa at 25 °C and has a boiling point of 78.5 °C. Evaporation or low boiling point are due to weak intermolecular attractions. Acetone has no hydrogen bonding whereas ethanol has hydrogen bonding. Since vapor pressure of acetone is high compared to ethanol, consequently acetone evaporates rapidly.

Hardener being hydrophilic in nature easily absorbs atmospheric moisture and gets contaminated. So in order to reduce the contamination of moisture in the composite, hardener is heated to about 40 °C to 45 °C (lukewarm). The effect of moisture absorption by the hardener can be observed in the laminates. The laminates without heated hardener showed rubbery state.

Materials and methods

The composite prepared was a particulate matrix composite. The reinforcement used was teak wood powder and BHS-05 fly ash powder. Both teak wood powder and fly ash were sieved and particles within 150 micron size were collected. To increase the wettability of the reinforcement to epoxy resin LY556, the reinforcement materials were dipped in 1N sodium hydroxide solution for a period of 24 hours. Based on the calculations, the required quantity of wood powder, fly ash and epoxy were weighed and introduced to the epoxy resin in a beaker.

Following cases were considered:

- Use of acetone as the solvent.
- Use of ethanol as solvent.
- Effect of heating of hardener

Acetone of suitable quantity was used to increase wettability of the fly ash- teak wood powder mixture in case 1 and ethyl alcohol was used in case 2. The contents were then mixed thoroughly using a magnetic stirrer. Hardener (HY951) of calculated quantity was introduced either at normal temperature or after heating to a higher temperature (case 3). Hardener is heated to remove the moisture absorbed by HY951 on exposure to atmosphere. The mixture was then poured into a mould cavity. The laminate was allowed to dry for a period of 48 hours, then removed from the mould and inspected for air bubbles and cavities. The specimens were cut according to standards for all the 3 cases and were analyzed using scanning electron microscope for its microscopic structure.

1.1. Table

Table 1. Tabulated standard values of the materials used for synthesizing polymer composites.

Material	Density
Epoxy Resin (LY556)	1.12
Hardener (HY951)	1.19
Wood powder	0.4
Fly ash	0.8

1.2. Polymer composite moulds

The observations were made based on physical, visual perception as well as the images of the microscopic structure of the composites obtained through Scanning Electron Microscope. On physical observation, the laminates prepared using different solvents showed varying degrees of flexibility and surface texture. Even the composite laminates used upon heating and without heating hardener showed slightly different flexibility. The composite laminate prepared using acetone as the solvent (fig .a and b) showed greater resistance to bending as compared to the laminate prepared using ethanol as the solvent(fig. c and d) .In both the cases the hardener was heated to a lukewarm temperature and the moisture absorbed on exposure to atmosphere was removed. The reason for this behavior is due to the traces of residual solvent left in the composite which affects the properties of epoxy matrix. Also ethanol used as solvent gets easily absorbed by the wood powder used as reinforcement, due to which it further alleviates the properties of epoxy, compared to laminate prepared using acetone as solvent.

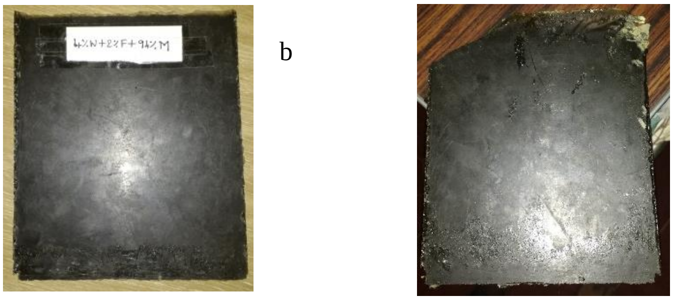
a b 

Fig. 1. (a): Composite Laminate of Acetone with heated Hardener; (b): Composite Laminate of Acetone without heated Hardener;

The composite laminate prepared using hardener exposed to moisture showed incomplete curing irrespective of the solvent used (figure c and figure d).The laminates also possessed a rubber mat like texture. Thus it is evident that moisture hinders the action of the hardener on the matrix.

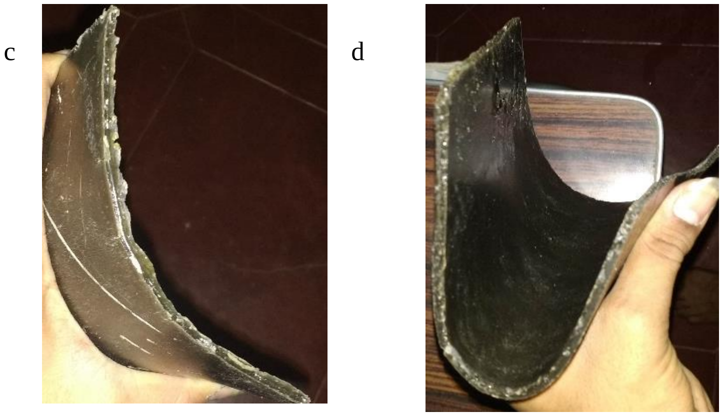


Fig. 1. (c): Composite Laminate of Ethanol with heated Hardener; (d): Composite Laminate of Ethanol without heated Hardener;

2. Results and discussions

2.1. Using acetone solvent with heated hardener

Morphological analysis of the polymer composites were performed with the help of scanning electron microscopy. The SEM images of smooth surfaces of epoxy/Fly ash/ natural fiber tertiary blends are shown in the Figure (g-h). The micrographs confirm the two-phase morphology of the blends. The heterogeneous morphology of the blends is due to the reaction induced phase separation (RIPS). Initially the system was miscible due to the low molecular weight of the epoxy prepolymer but during curing, molecular weight increases and the contribution to the entropy and free energy of mixing lessened resulting in two-phase morphology. In the same composition by increasing the resolution of SEM, the homogenous miscibility of all the composition, marked on SEM images the presence of cenospheres particles, natural fibres in the epoxy resin this is due to heating the hardener during mixing was observed.

2.2. Using acetone solvent without heated hardener

In SEM analysis, it has been found that fly ash particles were uniformly distributed and natural fibers were randomly distributed when observed under lower resolution as indicated in the figure.2. (e) and (f). The higher resolution of SEM image fig. 2. (g) and (h) shows some compatibility of natural fibers/fly ash with the polymer matrix material. SEM images showed broken fractured surface of the natural fiber/fly ash. All these SEM images reveal the homogeneous distribution of natural fiber/ fly ash in the polymer matrix. This is due to preparation of these moulds at room temperature without heating hardener in presence of acetone solvent.

2.3. Using ethanol solvent with heated hardener

The fig.4. SEM images show the microscopic structure of the laminates prepared using ethanol and hardener used upon heating. Careful analysis reveals the presence of micro pores throughout the laminate. On comparison with the laminate prepared using acetone under similar conditions the difference in the surface is evident. The laminates prepared using ethanol has a less smooth surface when compared to acetone. The distribution of natural fibers is more or less similar in both cases, but in the case of acetone solvent, the fractures on the surface are more apparent. The presence of cracks makes the laminate weaker. Increased resolution images show the presence of flakes like structure throughout the laminate surface. This observation is further confirmed by a higher resolution image which reveals the agglomeration of the particles in the polymer composites.

2.4. Using ethanol solvent without heated hardener

The above images reveal the microscopic structure of the composite laminates prepared using ethanol without heating hardener. The image (h) reveals a smooth surface of the composite laminate. The presence of fly ash particles and its distribution is clearly visible. The distribution of natural fiber is more or less uniform. Images of higher resolution shows more fractures visible on the surface as compared to use of hardener upon heating. The higher resolution images reveal agglomeration of all particles in the composition.

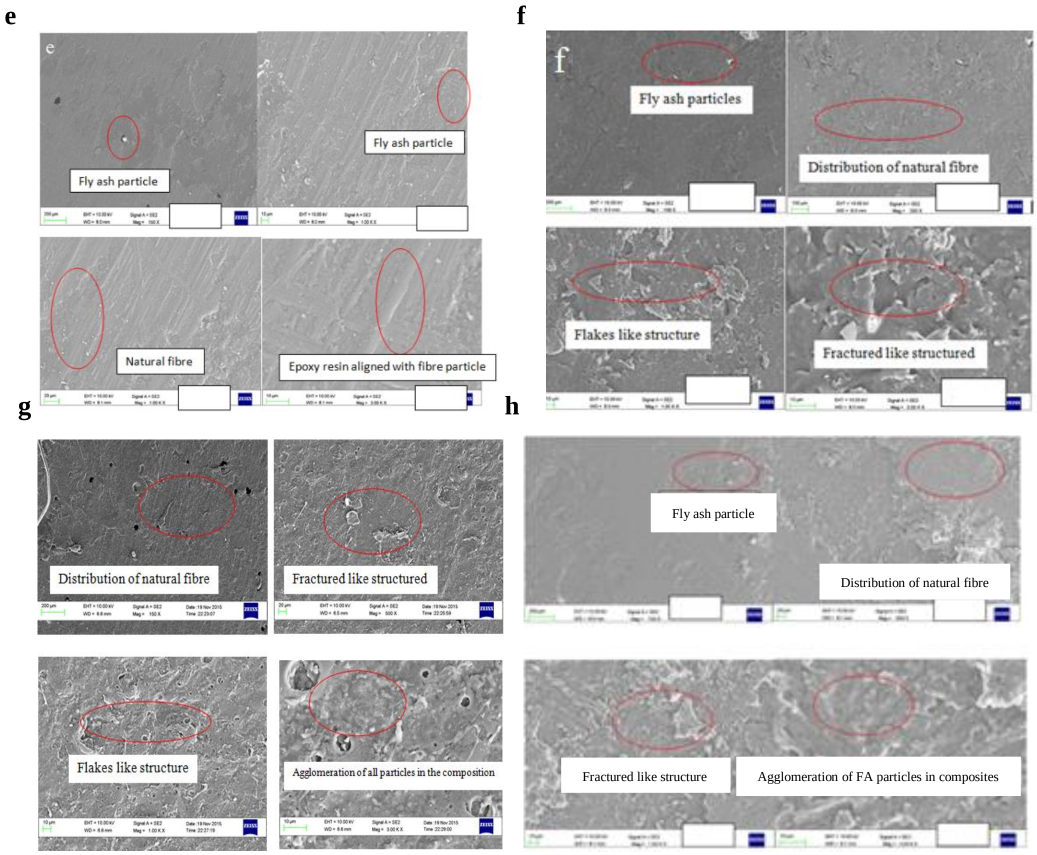


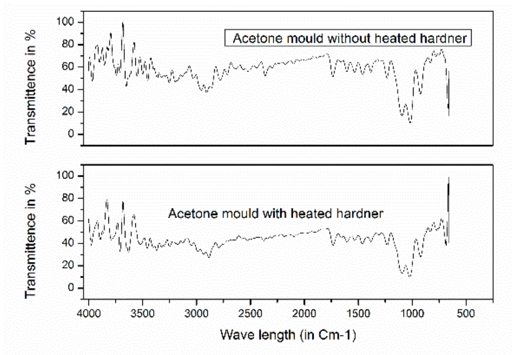
Fig. 2. (e) SEM image of acetone solvent with heated hardener; (f) SEM image of acetone solvent without heated hardener; (g) SEM image of ethanol solvent with heated hardener; (h) SEM image of ethanol solvent without heated hardener;

2.5. FT IR spectra of polymer composites obtained using alcohol/acetone solvent with and without heated hardener

Fig. 3. (i and j) IR spectra of epoxy/fly ash composites have been prepared using acetone and ethyl alcohol solvents at different conditions. Epoxy/fly ash composites have been prepared in ethanol mould with and without heating and hardener as shown in the figure. These composites have absorbed water due to the presence of polar group in the ethanol molecule. So there is small shift in characteristic stretching frequency of epoxy/fly ash composites at 3060-3050, 1275-1255 and 842-820 cm^{-1} of C-H, C-O-C and C-O-O stretching vibrations in ethanol mould with heated hardener. Hence the small shift in the IR peaks reveals the rubber property of epoxy/fly ash composites. Whereas in case of acetone mould using hardener with and without heating shows no shift in the characteristic stretching frequency of epoxy/fly ash composites while mechanical strength increases in these composites, because there is no

absorption of water molecule in presence of hardener.

i



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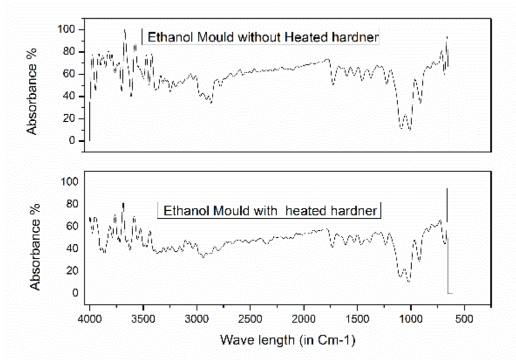
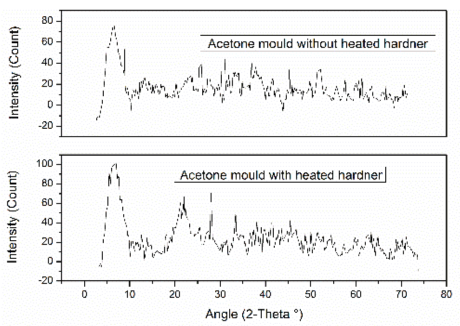


Fig.3.(i) FTIR spectra of acetone solvent with and without heated hardener; (j) FTIR spectra of alcohol solvent with and without heated hardener;

2.6 XRD spectra of polymer composites obtained using alcohol/acetone solvent with and without heated hardener

Fig .4. (k and l) XRD patterns of epoxy/fly ash composites have been obtained in acetone and ethanol solvent at different conditions. In the XRD patterns, clear shift of characteristic peaks of fly ash from 10 to 20, two theta values in the X-ray diffraction patterns which supports the IR peaks and also mechanical properties were observed. Ethanol mould with heated hardener helps in increasing cross linking in the polymeric chain and homogeneous distribution of fly ash. In other graph the characteristic peaks of fly ash has not shifted and clearly confirms the polymerization reaction takes equally in laminates with and without heating hardener. So the flexibility of the laminates is very high in acetone solvent than ethyl alcohol.

k



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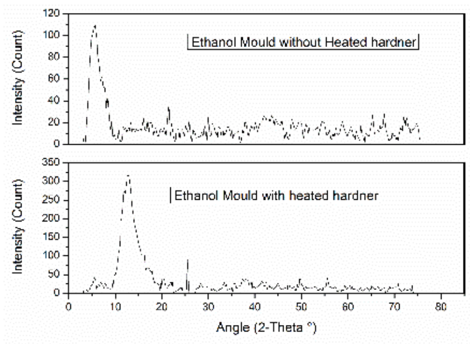

Fig. 4. (k) XRD spectra of acetone solvent with and without heated hardener; (l) XRD spectra of alcohol solvent with and without heated hardener;

Conclusion

Ethanol being a polar solvent, upon mixing with wood powder, the absorption of solvent is more due to porous nature of wood powder and presence of hydrogen bonding in it. The residual solvent is retained in the reinforced polymer composite due to presence of hydrogen bonding and weak Van der Waals forces. Because of this residual solvent in the polymer composites, mechanical properties are altered. Thus, laminates exhibit highly flexible and rubbery state. Ethyl alcohol solvent has higher boiling point and lower vapour pressure than acetone, hence it doesn’t evaporate completely even after drying. The other laminates were prepared using acetone solvent which is low polar, low boiling and highly volatile in nature. Even though wood particles being porous, there is no residual acetone solvent in the reinforced polymer composite, leading to decrease in flexibility of the laminates. FT-IR and XRD spectral data reveals more absorption of moisture in laminates prepared without heating hardener. SEM images show homogenous distribution of wood and fly ash particles, presence of cracks and agglomeration of particles in the laminates. The laminates prepared using ethanol as solvent without heating hardener show rubbery behaviour. The mechanical properties are enhanced when acetone is used as solvent with heated hardener.

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