

## PHYSICAL AND THERMAL CHARACTERIZATION OF RICE HUSK FILLED EPOXY MATRIX COMPOSITES

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### ABSTRACT

This paper presents a study on the physical and thermal characterization of a new class of multi-phase composites consisting of epoxy and rice husk particulates. The effects of incorporation of rice-husk modifies the physical and thermal properties of epoxy composites. Rice-husk particulates of different weight proportions ranging from 0 wt% to 15 wt% are filled in epoxy resin with and without glass-fiber reinforcement. The effective thermal conductivity, coefficient of thermal expansion (CTE) and glass transition temperatures ( $T_g$ ) are measured for the prepared composites. Incorporation of rice husk results in reduction of effective thermal conductivity of epoxy resin about 74.38%. It is observed that the CTE of epoxy decreases of about 22% with 15 wt % of rice husk in the composite. It is also observed that the  $T_g$  of neat epoxy is increased by 14.28% with an increase in rice husk content. A maximum increase of 14°C in  $T_g$  is obtained with 15 wt. % rice-husk.

**Keywords:** Rice Husk, Thermal Properties, Mechanical Properties.

### 1. INTRODUCTION

Environmental safety and ecological sustainability in recent years are some of the major concerns for all. It is thus worth noting that bio byproducts like rice husk are always environment friendly. It is considered as an agricultural waste, largely available from rice milling industries. Because of large production of rice, approximately 600 million tons/year, there is a large amount of rice husk waste which is about 20 wt% of the total rice production [1]. This can be utilized in many useful applications such as light weight concrete[2], an insulating material, fillers in plastics, building materials, panel boards, and activated carbon [3], electricity generation [4], husk-fueled steam engines [5] etc. Rice husk can be used as an alternative biomass energy source against fossil fuels. Fluidized-bed combustion process seems to be a suitable technology for converting rice husk into thermal energy and power generation [6-8]. Various investigators have reported on a variety of applications that involve rice husk. Garcia et al. [9] have used rice husk as a reinforcing material for recovery of used rubber tire in powder form by using sintering method. They anticipated that the use of waste tire and waste rice husk could reduce the environmental problems associated to their accumulation. It is well-known that composites reinforced with natural fibers have many advantages over composites containing inorganic fillers, because of their availability, versatility, recyclability and cost effectiveness [10,11]. Crespo et al. [12] studied the mechanical properties of plasticized PVC composites by incorporating rice husk as fillers. Yang et al. [13] prepared a composite sample with

polypropylene as the matrix and rice husk flour as the reinforcing filler and studied the physical, mechanical and morphological properties. They found that the tensile strength of the composite reduces as the filler weight percentage increases and tensile modulus improves with increasing filler loading. Favaro et al. [14] reported the mechanical and morphological properties of composites prepared with high-density polyethylene (HDPE) matrix and modified/unmodified rice husk fibers as the reinforcing element. Satapathy et al. [15] have investigated the reinforcing potential of SiC particles derived from rice husk through plasma-processing in jute-epoxy composites. Premalal et al. [16] also used rice husk as organic filler in polypropylene and observed that these composites exhibit relatively lower yield strength, Young's modulus, flexural modulus and higher elongation at break as compared to those of talc filled composites. Satapathy et al. [15] have investigated the reinforcing potential of SiC particles derived from rice husk through plasma-processing in jute-epoxy composites.

Considerable work has been reported on the subject of heat conductivity in polymers by Hansen and Ho [17], Peng and Landel [18], Choy and Young [19], Tavman [20], etc. It is well known that thermal transport increases significantly in the direction of orientation and decreases slightly in the direction perpendicular to the orientation. But most of these studies were confined to the thermal behaviour of neat polymers only and not to their composites. Reports are available in the existing literature on experimental as well as numerical and analytical studies on thermal conductivity of some filled

polymer composites [21-33]. Procter and Solc [34] used Nielsen model as a prediction to investigate the thermal conductivity of several types of polymer composites filled with different fillers and confirmed its applicability. Griesinger et. al [35] reported that thermal conductivity of low-density poly-ethylene (LDPE) increased from 0.35 W/mK for an isotropic sample, to the value of 50 W/mK for a sample with an orientation ratio of 50. The thermal and mechanical properties of copper powder filled poly-ethylene composites are found by Tavman [20] while Sofian et al. [32] investigated experimentally on thermal properties such as thermal conductivity, thermal diffusivity and specific heat of metal (copper, zinc, iron, and bronze) powder filled HDPE composites in the range of filler content 0–24% by volume. They observed a moderate increase in thermal conductivity up to 16% of metal powder filler content. Mamunya et. al [36] also reported the improvement in electrical and thermal conductivity of polymers filled with metal powders. In a recent research Weidenfeller et al. [37] studied the effect of the interconnectivity of the filler particles and its important role in the thermal conductivity of the composites. They prepared PP samples with different commercially available fillers by extrusion and injection molding using various volume fractions of filler content to systematically vary density and thermal transport properties of these composites. Surprisingly, they measured that the thermal conductivity of the PP has increased from 0.27 up to 2.5W/mK with 30 vol% talc in the PP matrix, while the same matrix material containing the same volume fraction of copper particles had a thermal conductivity of only 1.25W/mK despite the fact that copper particles have a thermal conductivity approximately 40 times greater than that of talc particles. Tekce et. al [33] noticed the strong influence of the shape factor of fillers on thermal conductivity of the composite. While Kumlutas and Tavman [38] carried out a numerical and experimental study on thermal conductivity of particle filled polymer composites, Patnaik et. al[39] reported the existence of a possible correlation between thermal conductivity and wear resistance of particulate filled composites. Zhao et al.[40] have studied the thermal stability and flammability of rice husk filled high-density polyethylene eco-composites. They observed that the addition of rice husk effectively reduces the thermo-oxidation process of the composite by about 40°C because of silica content in rice husk. Nayak et al.[41] have evaluated the effective thermal conductivity of pine wood dust filled epoxy composites by numerically and experimentally. An exhaustive review of existing literature reveals that although there are quite a number of avenues for utilization of rice husk in various applications. But its reinforcing potential as well as thermal insulation in composites have not been explored adequately. The research reported so far on rice husk filled composites are all on thermoplastic polymer based and no report is available on thermosetting polymers like epoxy reinforced with rice husk particles.

In view of the above, the present work is undertaken to investigate the thermal conductivity of epoxy matrix composites filled with rice husk. The objectives of this

work include fabrication of a new class of low cost composites using rice husk as the reinforcing filler with an objective to improve the insulating properties of epoxy resin and to study its mechanical strength when reinforced with glass fiber. Epoxy is already a known thermal barrier. Epoxy is chosen primarily because it happens to be the most commonly used polymer and because of its insulating nature (low value of thermal conductivity, about 0.363 W/m.K). It also reports the estimation of the equivalent thermal conductivity, coefficient of thermal expansion and glass transition temperature of particulate–polymer composite system.

## 2. EXPERIMENTAL DETAILS

### 2.1 Materials

In the present work, epoxy LY 556 is the resin which is used as the matrix material. The epoxy resin and the hardener are supplied by Ciba Geigy India Ltd. Woven roving E-glass fibers (supplied by Saint Gobain Ltd. India) have been used as the reinforcing material in the composites. Epoxy is chosen primarily because it happens to be the most commonly used polymer and because of its insulating nature (low value of thermal conductivity, about 0.363 W /m.K).

#### 2.1.1 Filler Material (Rice Husk)

Rice husk is chosen as the filler material in this work mostly for its very low thermal conductivity (0.065 W/m.K) and low density (0.12 gm/cc). Moreover, it is renewable, ecofriendly, available at low cost, non-toxic and basically the rice husk is considered as a waste product. The husk is collected from Shiva Shakti Rice Mill, Dhenkanal, Odisha, India, is used as a filler material in the glass fiber reinforced polymer matrix composite.

#### 2.2 Composite Fabrication

Two sets of composite specimens are fabricated for the present work. The low temperature curing epoxy resin (LY 556) and corresponding hardener (HY951) are mixed in a ratio of 10:1 by weight as recommended. Rice husk (RH) particles with average size 100 µm are reinforced in epoxy resin (density 1.1 gm/cc) to prepare the composites. The composites are cast by conventional hand-lay-up technique in glass tubes so as to get cylindrical specimens (dia 20 mm, length 100 mm). Composites R<sub>1</sub>, R<sub>2</sub>, R<sub>3</sub>, R<sub>4</sub>, R<sub>5</sub>, R<sub>6</sub>, and R<sub>7</sub> of seven different compositions (with 0% wt, 2.5% wt, 5% wt, 7.5%wt, 10% wt, 12.5% wt and 15%wt of RH respectively) are made. The castings are left to cure at room temperature for about 24 hours after which the tubes are broken and samples are released. Specimens of suitable dimension are cut using a diamond cutter for further physical and thermal characterization.

Further, cross plied E- glass fibers (360 roving) are reinforced in epoxy resin with 40% weight proportions of the composite and four different weight proportions of rice husk (0% wt, 5%wt, 10%wt, 15%wt) are added as filler material to prepare the composites C<sub>1</sub>, C<sub>2</sub>, C<sub>3</sub>, and C<sub>4</sub> respectively. The composite slabs are made by conventional hand-lay-up technique followed by light compression molding technique. A stainless steel mold

having dimension of  $210 \times 210 \times 40 \text{ mm}^3$  is used. A releasing agent (Silicon spray) is used to facilitate easy removal of the composite from the mold after curing. Care is taken to ensure a uniform sample since particles have a tendency to clump and tangle together when mixed. The cast of each composite is cured under a load of about 25kg for 24 h before it is removed from the mold. Then this cast is post-cured in the air for another 24 h after removing out of the mold. Specimens of suitable dimension are cut using a diamond cutter for physical characterization and mechanical testing. Utmost care has been taken to maintain uniformity and homogeneity of the composite. The designation and detailed composition of the composites are given in Table 1.

Table 1: Designations and detailed compositions of the composites

Designation	Composition
R <sub>1</sub>	Epoxy + 0 wt% RH
R <sub>2</sub>	Epoxy + 2.5 wt% RH
R <sub>3</sub>	Epoxy + 5 wt% RH
R <sub>4</sub>	Epoxy + 7.5 wt % RH
R <sub>5</sub>	Epoxy + 10 wt % RH
R <sub>6</sub>	Epoxy + 12.5 wt % RH
R <sub>7</sub>	Epoxy + 15 wt % RH
C <sub>1</sub>	Epoxy + 40 wt % GF + 0 wt % RH
C <sub>2</sub>	Epoxy + 40 wt % GF + 5 wt % RH
C <sub>3</sub>	Epoxy + 40 wt % GF + 10 wt % RH
C <sub>4</sub>	Epoxy + 40 wt % GF + 15 wt % RH

### 3. MECHANICAL CHARACTERIZATION

#### 3.1 Density and Void Fraction

Each composite under this investigation consists of three components namely matrix, fiber and particulate filler. The theoretical density of composites in terms of weight fraction can easily be obtained as per the following equation [42]:

$$\rho_{ct} = \frac{1}{\left( \frac{V_f}{\rho_f} \right) + \left( \frac{V_m}{\rho_m} \right) + \left( \frac{V_p}{\rho_p} \right)} \quad (1)$$

Where,  $W$  and  $\rho$  represent the weight fraction and density respectively. The suffix  $f$ ,  $m$  and  $ct$  stand for fiber, matrix and the composite materials respectively. The suffix ' $p$ ' indicates the particulate filler materials. The actual density  $\rho_{ce}$  of the composite however can be determined experimentally by simple water-immersion technique. The volume fraction of voids ( $V_v$ ) in the composites is calculated using the following equation:

$$V_v = \frac{(\rho_{ct} - \rho_{ce})}{\rho_{ct}} \quad (2)$$

#### 3.2 Micro-Hardness

Micro-hardness measurement is done using a Leitz micro-hardness tester. A diamond indenter, in the form of a right pyramid with a square base and an angle  $136^\circ$  between opposite faces, is forced into the material under a load of 0.245 N for a loading time of 20 secs.

#### 3.3 Tensile, Flexural and Inter-laminar Shear Strength (ILSS)

The tensile test is generally performed on flat specimens. The commonly used specimens for tensile test are the dog-bone type and the straight-side type with end tabs. During the test, a uniaxial load is applied through both the ends of the specimen. The ASTM standard test method for tensile properties of fiber resin composites has the designation D 3039-76. The length of the test section should be 200 mm. The tensile test is performed in the universal testing machine (UTM) Instron 1195 and the readings are noted to calculate the tensile strength of the composite samples. The test is repeated three times for each sample and the mean value is reported. The flexural strength of a composite is the maximum tensile stress that it can withstand during bending before reaching the breaking point. The three point bend test is conducted on all the composite samples in the same universal testing machine Instron 1195. The dimension of each specimen is 60 mm  $\times$  10 mm  $\times$  4 mm. Span length of 40 mm and the cross head speed of 10 mm/min are maintained. For determination of both flexural strength and ILSS, the test is repeated three times for each composite type and the mean value is reported. The flexural strength of the composite specimen is determined using the following equation.

$$\text{Flexural-Strength} = \frac{3PL}{2bt^2} \quad (3)$$

where,  $L$  is the span length of the sample (mm)

$P$  is maximum load (N)

$b$  the width of specimen (mm)

$t$  the thickness of specimen (mm)

The short-beam shear (SBS) tests are performed on the composite samples at room temperature to evaluate the value of inter-laminar shear strength (ILSS). It is the test, which generally promotes failure by inter-laminar shear. This SBS test is conducted as per ASTM standard (D 2344-84) using the same UTM. Span length of 40mm and the cross-head speed of 5 mm/min are maintained. The ILSS values are calculated as follows:

$$\text{ILSS} = \frac{3P}{4bt} \quad (4)$$

#### 3.4 Scanning Electron Microscopy

The surfaces of the specimens are examined directly by scanning electron microscope JEOL JSM-6480LV. The composite samples are mounted on the stubs with silver paste. To enhance the electrical conductivity of the samples, a thin film of platinum is vacuum-evaporated onto them before photomicrographs are taken.

### 4. THERMAL ANALYSIS

#### 4.1 Experimental Determination of Thermal Conductivity

Unitherm™ Model 2022 is used to measure thermal conductivity of a variety of materials. These include polymers, ceramics, composites, glasses, rubbers, some metals, and other materials of low to medium thermal conductivity. Only a relatively small test sample is required. Non-solids, such as pastes or liquids, can be tested using special containers. Thin films can also be tested accurately using a multi-layer technique. The tests are in accordance with ASTM E-1530 standards. A

sample of the material is held under a uniform compressive load between two polished surfaces, each controlled at a different temperature. The lower surface is part of a calibrated heat flow transducer. The heat flows from the upper surface, through the sample, to the lower surface, establishing an axial temperature gradient in the stack. After reaching thermal equilibrium, the temperature difference across the sample is measured along with the output from the heat flow transducer. These values and the sample thickness are then used to calculate the thermal conductivity. The temperature drop through the sample is measured with temperature sensors in the highly conductive metal surface layers on either side of the sample.

#### 4.2 Thermal Mechanical Analysis

The glass transition temperature ( $T_g$ ) were measured with a Perkin Elmer DSC-7 thermal mechanical analyzer (TMA). The temperature range used was from 30 to 220°C, and the heating rate was 10°C per min. All reported DSC/TMA data are obtained from a second heating cycle.

### 5. RESULTS AND DISCUSSION

#### 5.1 Density and Void Fraction

The theoretical and measured densities of the composites along with the corresponding volume fraction of voids are presented in Table 2. It may be noted that the composite density values calculated theoretically from weight fractions using Equation (2) are not equal to the experimentally measured values. This difference is a measure of voids and pores present in the composites. It is further noticed that with the increase in rice husk content, the void fraction is increasing within the composites. However, the volume percentage of pores and voids in the composites under this investigation are well within the acceptable limit of 10%.

Table 2: Measured and theoretical densities of the composites

Composites	Measured density (gm/cc)	Theoretical density (gm/cc)	Volume Fraction of voids (%)
C <sub>1</sub>	1.412	1.430	1.25
C <sub>2</sub>	0.898	0.934	3.85
C <sub>3</sub>	0.647	0.693	6.63
C <sub>4</sub>	0.502	0.551	8.89

Density of a composite depends on the relative proportion of matrix and reinforcing materials and this is one of the most important factors determining the properties of the composites. The void content is the cause for the difference between the values of true density and the theoretically calculated ones. The voids significantly affect some of the mechanical properties and even the performance of composites in the workplace. Higher void contents usually mean lower fatigue resistance, greater susceptibility to water penetration and weathering [42]. The knowledge of void content is desirable for estimation of the quality of the

composites. It is understandable that a good composite should have fewer voids. However, the presence of void is unavoidable in composite-making particularly through hand-lay-up technique.

#### 5.2 Tensile, Flexural and Inter-laminar Shear Strength

It is well known that the strength properties of composites are mainly determined by the filler content and the fiber strength. So variation in composite strength with varying rice husk content is obvious. These variations in tensile and flexural strengths of the composites C<sub>1</sub>, C<sub>2</sub>, C<sub>3</sub> and C<sub>4</sub> are shown in Figures 1 and 2. The mechanical properties of the composite samples are shown in Table 3. A gradual decrease in tensile strength with increase in rice husk content is noticed. It clearly indicates that the inclusion of rice husk reduces the load carrying capacity of the composite. Similar observations have been reported by Crespo et al. [12] for plasticized PVC composites and also by Yang et al. [13] for polypropylene matrix composites containing rice husk fillers. There can be two reasons for reduction of tensile strength as the rice husk content increases. First reason may be due to the chemical reaction at the interface between the filler particle and the matrix is too weak to transfer the tensile stress, the second reason may be due to a rise of stress concentration at the sharp corner of irregular shaped particulates embedded in the epoxy matrix.

Table 3: Mechanical properties of the composites

Composites	Mean Hardness (Hv)	Tensile Strength (MPa)	FS (MPa)	ILSS (MPa)
C <sub>1</sub>	28.8	362.5	388.8	26.8
C <sub>2</sub>	39.2	278.26	358.8	22.57
C <sub>3</sub>	43.8	228.88	322.17	19.26
C <sub>4</sub>	45.9	144.35	269.52	18.54

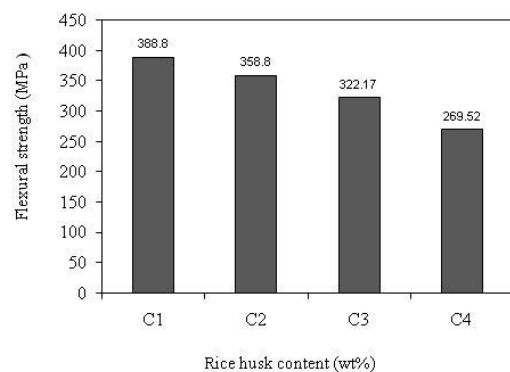


Fig 1. Effect of rice husk content on tensile strength of glass-epoxy composites

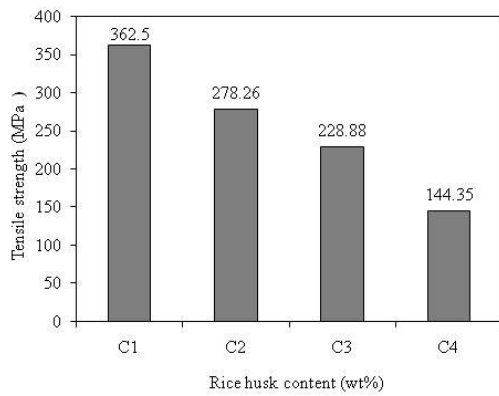


Fig 2. Effect of rice husk content on flexural strength of glass-epoxy composites

The stresses acting on the interface of the two adjacent laminate in a layered composite are called inter-laminar shear stresses. These stresses cause relative deformations between the consecutive laminae and if these are sufficiently high, they may cause failure along the mid-plane between two adjacent laminae. It is therefore required to evaluate the ILSS through tests in which failure of the laminates of the composite initiates in a shear or delamination mode. In this present work, the ILSS values are measured for rice husk filled glass-epoxy composites C<sub>1</sub>, C<sub>2</sub>, C<sub>3</sub> and C<sub>4</sub>. The variation of ILSS values with the rice husk content is shown in Figure 3. It is seen that the inter laminar shear strength reduces with increase in rice husk content.

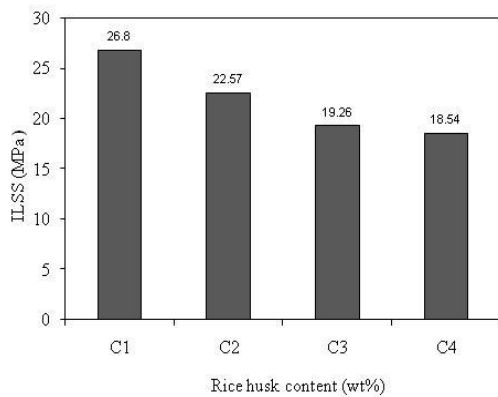


Fig 3. Effect of rice husk content on ILSS of glass- epoxy composites

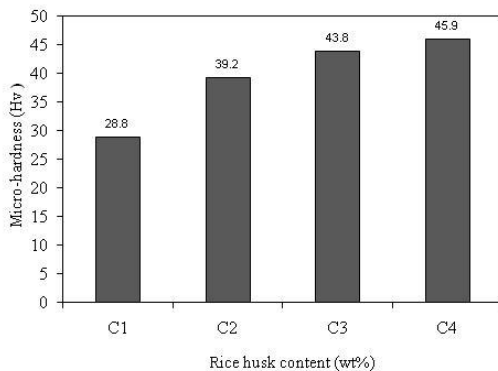


Fig 4. Micro-hardness values of composites with different filler content

In the present work, during flexural test, the span length is very short (40 mm). A large span-to-depth ratio in bending test increases the maximum normal stress without affecting the inter-laminar shear stress and thereby increases the tendency for longitudinal failure. If the span is short enough, failure initiates and propagates by inter-laminar shear failure. The maximum shear stress in a beam occurs at the mid-plane. So in shear test, failure is due to a crack running along the mid-plane of the beam so that the crack plane is parallel to the longitudinal plane.

### 5.3 Micro-Hardness

Hardness is considered as one of the most important factors that govern the wear resistance of any material. In the present work, micro-hardness values of the glass-epoxy composites with rice husk fillers in different proportions have been obtained. The test results (Figure 4) show that with the presence of rice husk particles, micro-hardness of the glass-epoxy composites improved and this improvement is a function of the filler content. Among all the composites under this investigation, the maximum hardness value is recorded for glass-epoxy composite filled with 15 wt% of rice husk. The reduction in tensile strength and improvement in hardness with the incorporation of fillers can be explained as follows: the interfacial bond strength between filler and matrix is less and due to which it breaks when subjected to a tensile force. Where as in hardness test the epoxy matrix phase and solid rice husk filler phase pressed together and touch each other more tightly due to which the interface can transfer pressure more effectively although the interfacial bond strength is less. This might have resulted in an enhancement of hardness.

### 5.4 Effective Thermal Conductivity ( $K_{eff}$ )

The effective thermal conductivity ( $K_{eff}$ ) of the composites are measured with a Unitherm™ Model 2022. Figure 5 presents the variation of  $K_{eff}$  with the rice husk content. It is observed that the  $K_{eff}$  of neat epoxy is about 0.363W/m.K and it gradually decreases to 0.093W/m.K with an increase in rice husk content.

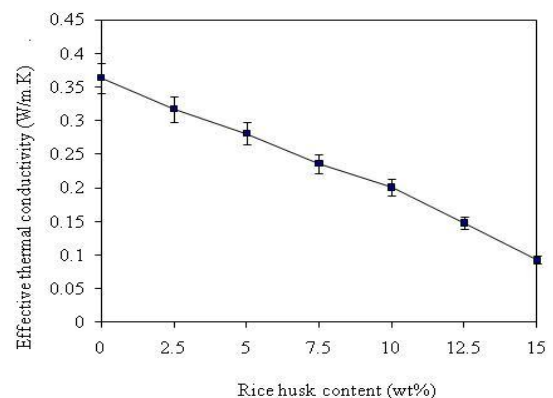


Fig 5. Showing the variation of effective thermal conductivity with wt% rice husk

Table 4: Effective thermal conductivity of composites with different weight proportions of rice husk

Sample	Rice Husk Content (wt%)	Effective thermal conductivity of composites $K_{eff}$ (W/m-K)
R <sub>1</sub>	0	0.363
R <sub>2</sub>	2.5	0.317
R <sub>3</sub>	5	0.281
R <sub>4</sub>	7.5	0.236
R <sub>5</sub>	10	0.201
R <sub>6</sub>	12.5	0.148
R <sub>7</sub>	15	0.093

### 5.5 Coefficient of Thermal Expansion (CTE)

The CTEs of the rice husk-filled epoxy composites were examined with TMA. Figure 6 shows that the CTE of the composite varies with the rice husk content. The CTE of the neat epoxy is about  $63 \times 10^{-6}$  m/m. K and it gradually decreases to  $46 \times 10^{-6}$  m/m.K with the increase in the filler content. A maximum decrease of about 22% in CTE is obtained with 15 wt % of rice husk in the composite. The constraint of mobility of the epoxy chain due to the interaction of rice husk and epoxy are responsible for the reduced CTE of the composites.

### 5.6 Glass Transition Temperature ( $T_g$ )

The glass transition temperature ( $T_g$ ) of the composites are measured with a Perkin Elmer DSC-7 thermal mechanical analyzer (TMA). Figure 7 presents the variation of  $T_g$  with the rice husk content. It is observed that the  $T_g$  of neat epoxy is about  $98^\circ\text{C}$  and it gradually increases to  $112^\circ\text{C}$  with an increase in rice husk content. A maximum increase of  $14^\circ\text{C}$  in  $T_g$  is obtained as the rice husk content increases to 15% wt. The effect of filler in polymer composites on the glass-transition behavior of the polymer matrix has been studied for different polymer-filler composites [43]. Adding micro-sized fillers usually increases the  $T_g$  of composites [44]. In the present study, a shift of  $T_g$  toward a higher temperature can be attributed to the good interaction between rice husk and epoxy, which might be restricting the mobility of the epoxy chain.

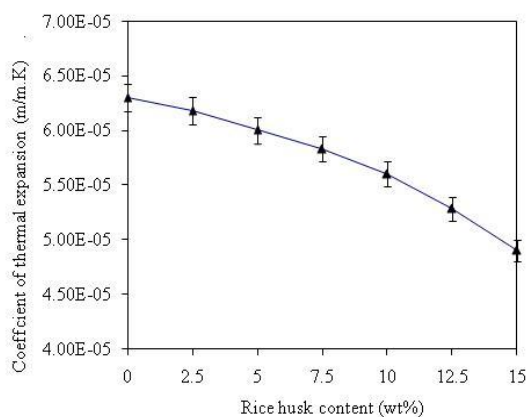


Fig 6. The variation of coefficient of thermal expansion with rice husk content

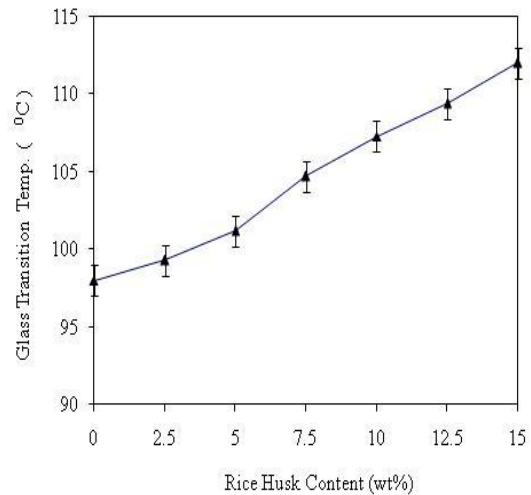


Fig 7. The variation of glass transition temperature ( $T_g$ ) with rice husk content

## 6. CONCLUSIONS

This study yields the following specific conclusions:

- (1) The successful fabrication of glass-epoxy composites with reinforcement of an agricultural waste such as rice husk is possible by hand layup technique.
- (2) Incorporation of rice husk particles as fillers modifies the tensile, flexural and inter-laminar shear strengths of the glass-epoxy composites. A steady decline in the tensile, flexural and inter-laminar shear strength is noticed in the filled composites whereas the presence of this particulate matter has caused improvement in composite micro-hardness.
- (3) The density of the composites is also greatly influenced by the filler content. While designing such composite systems for specific requirements, thus, there is a need for optimizing the rice husk content
- (4) The measured values of effective thermal conductivity are obtained for different weight fractions of rice husk fillers. Incorporation of rice husk results in reduction of thermal conductivity of epoxy resin about 74.38% and thereby improves its thermal insulation capability.
- (5) It is observed that the coefficient of thermal expansion (CTE) of epoxy is greatly influenced by addition of rice husk. A maximum decrease of about 22% in CTE of the epoxy is obtained with 15 wt % of rice husk in the composite. The constraint of mobility of the epoxy chain due to the interaction of rice husk and epoxy are responsible for the reduced CTE of the composites.
- (6) It is also observed that the glass transition temperature ( $T_g$ ) of neat epoxy is increased by 14.28% with an increase in rice husk content. A maximum increase of  $14^\circ\text{C}$  in  $T_g$  is obtained as the rice husk content increases to 15% wt.

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