

## THERMAL AND MECHANICAL BEHAVIOR OF FIBRE-REINFORCED EPOXY CONTAINING LOW CONTENT OF NANO-CuO PARTICLES

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**Abstract-** The current study deals with exploring the effect of nano-copper oxide (CuO) particles and polypropylene fiber on the thermal and mechanical behavior of cured epoxy composite and put them into the same context. The effective thermal conductivity and the mechanical properties (tensile, compressive, flexural and impact strength) of epoxy-CuO, epoxy-fiber and the hybrid system (epoxy-CuO fiber) composites are assessed as a function of filler content. Also, a heat transfer model is developed from first principles and used to evaluate the thermal behavior of the prepared composites. The results showed that CuO improves the thermal conductivity of neat epoxy effectively while polypropylene fiber is not. Further, an enhancement in all the studied mechanical properties is observed in general, with maximum values achieved at 1.5 and 60 (wt.%) of CuO nanoparticles and polypropylene fiber, respectively. Finally, the behavior of hybrid system of nano-CuO and polypropylene fiber does not show appreciable difference comparing to the systems based on a single filler.

**Keywords:** hybrid Epoxy Composite, Nano-CuO, Fiber-Reinforced Epoxy, Composite Thermal Conductivity, Mechanical Behavior.

### 1. INTRODUCTION

Epoxy resins are widely used in various domestic and industrial applications due to their unique properties. They are thermally stable and offer a high strength, good bond properties and excellent chemical resistance. They are also ambient curable polymer with low-shrinkage rate during the curing process. So, they are dimensionally stable and that is a key property to many successful applications. On the downside, however, the fully cured epoxy resins are brittle [1]. Usually, the brittleness issue is avoided by modifying epoxy resins using different reinforcements such as elastomers, fibers, metallic oxides, carbon-based materials and others. While these reinforcements may improve the epoxy brittleness, epoxy composites are still viewed as unattractive alternatives to many traditional materials. The inherent processing complexity of such composites and the associated cost, perhaps, are the major barriers. Nowadays, using nano-sized particles as reinforcements are receiving

a special attention since they can improve epoxy properties and shorten the manufacturing process at lower cost [2, 3]. The role of particulate fillers in enhancing properties of polymeric materials (particularly thermal and electrical properties) is still not fully understood. Perhaps, one of the well-known theories that explains how the particulate fillers interacting inside polymeric composite materials is "percolation theory". The theory is based on a concept that the particles/matrix percolate and hence make pathways, allowing the transmission of stresses, heat and electricity and that in turn may lead to radical changes in properties of the host matrix [4]. Another concept in agreement with percolation theory is that proposed by Maier and Goritz [5] which is based on the role of polymer chains to form bridges among the particles. Far from these concepts, there is another argument based on chain immobilization. It is suggested that the chain cannot mobilize in the vicinity of particles and that forming a network and hence mechanical reinforcement and thermal and electrical enhancement may occur [6]. Recently, these concepts have been modified and used to understand the behavior of nanoparticles polymer composite and fiber reinforced plastic polymer [1, 8].

There are many works focusing on developing new polymer nano-composite systems, especially those systems based on thermoset epoxy. Among these systems and most commonly explored is using clays nanoparticle to reinforce epoxy, achieving a pronounced enhancement in its properties [9]. Using Zirconium phosphate is another route which has been recently investigated to generate multifunctional epoxy [10]. Epoxy resin incorporated with nano-magnetite filler also has been tested, trying to improve both mechanical and magnetic behavior of epoxy-based composite. Core-shell structured iron nanoparticles were used to produce a conductive epoxy [11]. In addition to these fillers, there is a good amount of work using copper and its oxide as nanoparticles to produce an enhanced nano-polymer composite and many of them are reviewed by Sunny et al. [1]. For instance, there is a series of papers done by Wetherhold and Bagwell [12-14] investigated thoroughly the effect of copper on thermal, electrical as well as mechanical properties of vinyl-ester resin.

They found that copper oxide increases the thermal stability and enhanced the strength of this particular resin. Also, it is found that epoxy adhesion strength can be improved using copper oxide. Ying, et al. [16] use copper oxide (CuO) nanoparticles to examine its effect on the tribological characteristics of the epoxy-PTFE composite. Further, Zabihi, et al. found that epoxy resins filled by 5 (wt.%) of nano-CuO produce a composite with optimum density and better thermal stability. Chang, et al. [18], recently, used nanorods of copper oxide (CuO) prepared via hydrothermal reaction to produce epoxy composite suitable for cryogenic applications. Additionally, Eskandari, et al. [19] investigated the effect of CuO and other nano-oxides to develop an acid resistant coating applied on copper substrate and superior corrosion protective properties have been obtained. Jeyanthi, et al. [20] have conducted experiments to study the electromagnetic interference (EMI) shielding effectiveness (SE) of a hybrid epoxy composite formed by hand lay-up technique using copper oxide (CuO) and other fillers (bamboo charcoal and multi-walled carbon nanotubes) as nanofillers. The results showed that 5 (wt.%) of CuO with 1.25 (wt.%) of carbon nanotubes and, and 5.5 (wt.%) of biochar give the maximum EMI SE performance of epoxy composite.

Reviewing the relevant literature reveals clearly that using the native copper or its oxides as a reinforcement in polymeric matrices can enhance their physical characteristics at relatively low loading. While there are many works explored the use of CuO nanoparticle as a reinforcement for epoxy composites, there is a little work, as far as the author knows, put CuO nanoparticle in the same context with other fillers and make a comparative study. Therefore, the present study emphasizes on comparing (in terms of mechanical and thermal properties) between the neat-epoxy resin polymer and the epoxy reinforced only by CuO nanoparticles and those reinforced by polypropylene fibers and, of course, a hybrid composite is included in the current study. Also, a numerical model is developed and used to understand the thermal behavior of particulate filler reinforced polymer. The model allows prediction of composite thermal conductivity and hence a direct comparison with the experimental data is possible. It is believed that such study supported by a numerical model will an important contribution to deepen the understanding of particulate reinforcement (nano-CuO) role as well as fibers (polypropylene).

The paper is presented as follows. In Section 2, the experimental work is described. After that (Section 3), the numerical model including governing equations, boundary conditions and numerical solution is developed and introduced. In Section 4, the results are presented and discussed. In Section 5 (at the end), a discussion is made and conclusions are drawn.

## 2. EXPERIMENT

### 2.1. Materials Used

The epoxy used in all experiments is supplied by DCP company, UK, where diglycidyl ether of bisphenol A,

which is referred here as DGEBA, is the basic resin and the diethylene tetramine is the hardener. The physical characteristics of the polymerized epoxy are: density 1100 kg/m<sup>3</sup>; viscosity 26 mPa at 20 °C. The polypropylene fibers are from Sika for construction chemicals, Egypt. The copper oxide nanoparticles (Nano-CuO, 30-50 nm and purity 99.9%) are supplied by Nano Research Lab, India.

### 2.2. Composite Samples Preparation

The preparation of composite samples is simply by adding the reinforcement directly to the epoxy during the polymerization reaction. The epoxy polymerization is carried out at 45 °C and 1 atm. It is important to note here that the raw chemicals used are purified first by removing any moistures at 40 °C for 4 hours. The epoxy raw materials (Bisphenol A, Epichlorohydrin and tertiary amino compound) are mixed in a 300 ml reactor according to the stoichiometric amount determined by DCP company. The reaction temperature is fixed at 45 °C using a convection water bath. The mixing process is done using a stirrer rotating at 500 rpm and the required amount of filler is added. The mixing process continues till a good dispersion of filler is achieved and the viscosity increases, giving an indication that a polymer with appreciable molecular weight is formed. After that, the product (polymer and fillers) is placed in a container under vacuum to remove any trapped air bubbles for 15 minutes and then poured in a mold, preparing for post-curing process. In this step, the samples are put in a convection oven set at 50 °C for 24 hours. It important to mention here that polypropylene fiber length used in the experiments is between 16-18 mm with 18 μm diameter and CuO particles size is between 30-50 nm.

## 3. THERMAL MODEL

The model proposed here is based on solving the general heat Equation under specific conditions to predict the effective thermal conductivity ( $K_{eff}$ ). The general governing equations of heat transfer in Cartesian coordinate ( $x, y, z$ ) can be written as follows [21-22]:

$$k \frac{\partial^2 T}{\partial x^2} + k \frac{\partial^2 T}{\partial y^2} + k \frac{\partial^2 T}{\partial z^2} + \dot{Q} = \rho C_p \frac{\partial T}{\partial t} \quad (1)$$

where,  $T$  is the temperature,  $k$  the thermal conductivity (matrix of filler),  $\rho$  material density,  $C_p$  material specific heat,  $t$  time and  $x, y, z$  are the coordinates. For a good approximation,  $k$  value is constant and depends on the material type. For epoxy, this value is taken to be 0.19 and for CuO and polypropylene fiber, respectively, is 32 and 0.12 W/m/k. Since the steady-state condition can be attained experimentally, the term  $\partial T / \partial t = 0$  in Equation 1, and hence the above equation reduces to:

$$k \frac{\partial^2 T}{\partial x^2} + k \frac{\partial^2 T}{\partial y^2} + k \frac{\partial^2 T}{\partial z^2} + \dot{Q} = 0 \quad (2)$$

Two cases are considered in the current work, depending on the shape of reinforcements and these are discussed in the subsequent sections.

### 3.1. Particulate Filler Composite

For particulate filler composite, the technique adopted to simulate the heat transfer process in a composite and estimate its effective thermal conductivity include solving Equation (1) in a cell containing a single spherical particle (Figure 1). The volume of spherical particle to the volume of cell is equivalent to that used in the experiments (i.e., volume fraction and hence mass fraction). The governing equation has been solved numerically using Comsol Multiphysics Ver. 5.5. The software is based on finite element method to solve the equation under the proposed boundary conditions. In each numerical experiments, the filler content (CuO) has changed and the effect of this change on the temperature distribution has been observed. Having known the temperature distribution, the composite effective thermal conductivity can be calculated, simply, using Fourier law. This allows investigating the effect of filler content on the thermal conductivity and hence thermal performance of the prepared composite samples. The boundary conditions used are specifying temperature on two opposite faces and imposed.

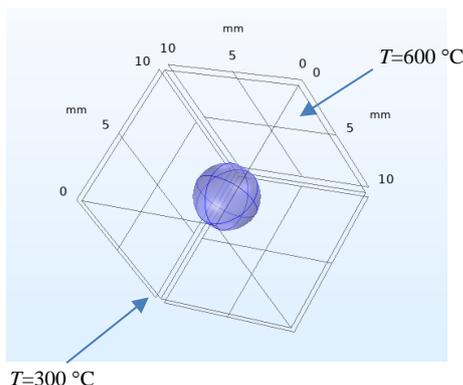


Figure 1. the composite shape used in COMSOL Multiphysics, showing the boundary conditions

$$k_m \frac{\partial T}{\partial n_m} = \frac{T_m - T_p}{R} = k_p \frac{\partial T}{\partial n_p} \quad (3)$$

where,  $m$  and  $p$  denote the matrix and the particle,  $n_i$  is a vector normal to the matrix phase and  $R$  is the interfacial resistance. Applying the above conditions, the numerical solution gives the temperature distribution inside the matrix and the filler as shown in Figure 2.

### 3.2. Fiber Reinforced Composite

In this case the approach is different. Equation 2 reduces to a two-dimensional heat equation and solved numerically. This is better for fiber-composite system better without adding complexities and at the same time obtaining a reasonable solution is possible.

$$k \frac{\partial^2 T}{\partial x^2} + k \frac{\partial^2 T}{\partial y^2} + \dot{Q} = 0 \quad (4)$$

The boundary conditions are selected to mimic the conditions in Lee's apparatus test for thermal conductivity. These conditions are shown in Figure 3.

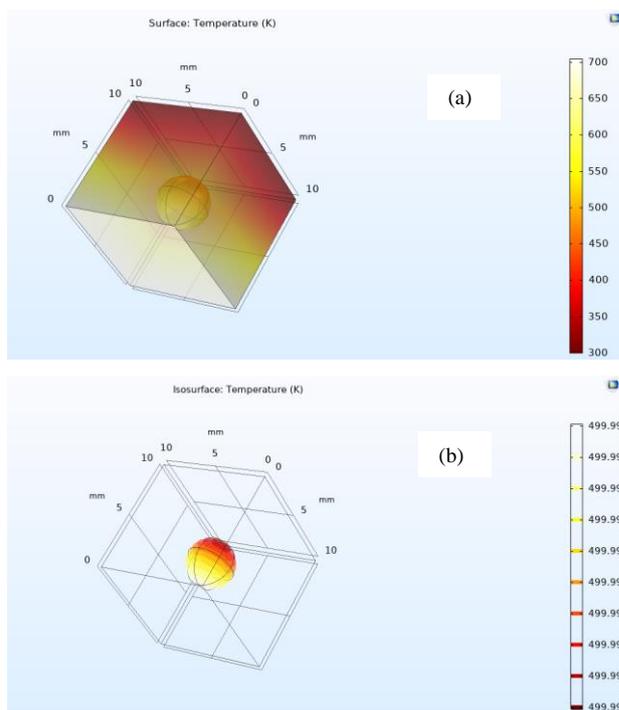


Figure 2. a. temperature distribution in the matrix phase, b. temperature distribution in the particle phase where the light-yellow color in both phases is the largest temperature. The solution presented in the Figure is independent of mesh size.

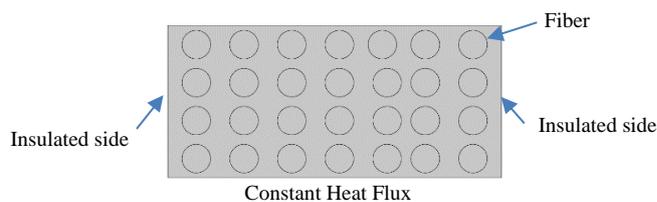


Figure 3. A diagram showing epoxy filled by fibers and the two sides are insulated and a constant heat flux applied on the lower side and transfer to the upper side

At the interface between the filler and the matrix Equation (3) is also imposed in this case. Similar to particulate fillers case, in each numerical experiments, the filler content (polypropylene fiber) has changed and the effect of this change on the temperature distribution has been observed. From this information, the composite effective thermal conductivity can be calculated using Fourier law. An example computation is shown in the Figure 4.

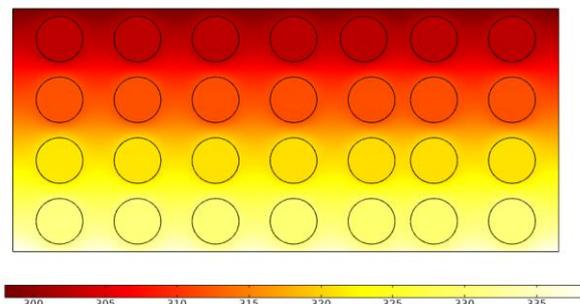


Figure 4. A diagram showing the temperature distribution changes with filler content where the light-yellow color in the right-hand Figures is the largest temperature

#### 4. RESULTS

The experimental and numerical results of the prepared composite samples over the range of fillers content are discussed in details below. CuO particles are added at four different concentrations (0.5, 1, 1.5 and 2% by weight). Polypropylene fibers loading is 20, 40, 60 and 80% by weight. It is important to mention here that composite thermal conductivities have been measured using Lee's disc technique (ASTM C518-04). For the mechanical properties, the composite samples were tested according to ASTM D-638, ASTM-D695, ASTM D7264/D7264M-21 and ASTM ISO 179 for tensile, compressive, flexural and impact strength, respectively.

##### 4.1. Thermal Behavior

The experimental and computational thermal conductivities for the three different composite systems are shown in Figure 5 where circles, squares and triangles are  $K_{eff}$  values of epoxy (DGEB)-CuO system, epoxy (DGEB)-fiber system and epoxy (DGEB)-CuO-fiber system, respectively and the dashed-red lines are the corresponding  $K_{eff}$  values computed numerically. The  $k$ -value of pure epoxy is shown as a red line on the y-axis. To include all the data in a single plot and hence a fair comparison is made, a logarithmic plot has been used.

It is better now to discuss first each system separately. In terms of DGEB-CuO system, it is obvious that  $K_{eff}$  increases steadily as the filler content increases. This could be attributed to the effect of CuO particles which have a much higher thermal conductivity than epoxy (about 250 times greater than the thermal conductivity of epoxy). The behavior observed in data is also possible to be explained in terms of "percolation theory". The particles/matrix percolate and hence make pathways, allowing the transmission of heat and that in turn may lead to increasing  $K_{eff}$ . [4]. This observation has been supported by the model results (red dashed-line). The model shows that the temperature difference increases CuO amount increases and therefore, an increase in  $K_{eff}$  values are expected. The model captures the experimental data very well up to 1% and then there is a clear divergence. This may be due an experimental error or even computational error, leading to such difference.

In terms of DGEB-fiber system, the behavior is different as expected.  $K_{eff}$  values decrease with fiber content. Similar to DGEB-CuO system, this might be the effect of fiber thermal conductivity which is less than that of epoxy by about 1.5 time. The model here follows the data perfectly, showing similar behavior. Finally, the hybrid system (DGEB-CuO-fiber) is more complicated. It is obvious that the increase in  $K_{eff}$  values occur, mainly, due to the effect of CuO particles which has the greatest thermal conductivity and follow percolation theory explanation.

##### 4.2. Mechanical Behavior

As mentioned, four mechanical tests have been made to assess the performance of the prepared composite samples. These are tensile, flexural, compressive and impact strength.

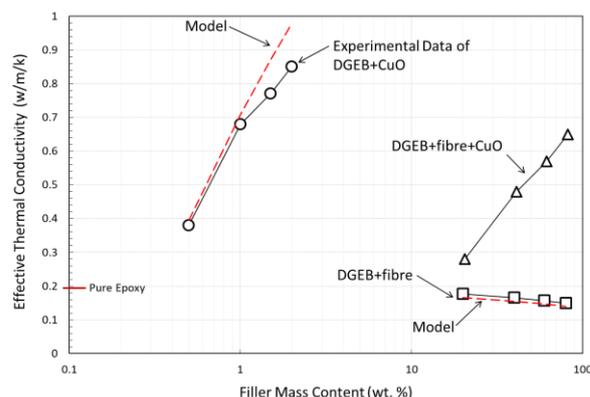


Figure 5. Experimental and computational thermal conductivities for the three studied composite systems where circles, squares and triangles are  $K_{eff}$  values of epoxy (DGEB)-CuO system, epoxy (DGEB)-fiber system and epoxy (DGEB)-CuO-fiber system, respectively and the dashed-red lines are the corresponding  $K_{eff}$  values computed numerically

Figure 6 shows the results of tensile strength for the three studied systems. The pure epoxy (red line on the y-axis) has a tensile strength of about 72 MPa. Adding CuO particles makes an improvement in tensile strength than pure epoxy, giving, surprisingly, maximum values of 96 Mpa at 1.5 (wt.%) of CuO loading. Increasing CuO content further leads to a decrease in composite tensile strength. Similarly, for the other two systems, the results are comparable to these of CuO particles but at loading level of 20 to 80% by weight of fiber.

The subsequent decrease in the tensile strength of the composite samples with increasing filler loading could be due to maldistribution of fillers (i.e., agglomeration and sedimentation begins to occur leading to form two non-mixed phases). This means the phase with less filler content cannot transmit stresses and hence a failure occurs and a decrease in tensile strength values above 1.5 and 60% of CuO particles and polypropylene fibers, respectively has been observed. Although, further characterization has not been done for the failed samples but the sedimentation of fillers and forming two non-mixed phases has been observed visually. For the hybrid system, the behavior is close to that of the other two systems as expected.

The results of flexural strength are given in Figure 7. The results are higher than those of tensile strength and show similar behavior (i.e., increases and then decreases at the same loading values of CuO and polypropylene). This behavior is probably not surprising since the flexural strength represents the value of failure stress when a specimen is exposed to a transvers bending load. So, it should be expected to be larger than the failure stress in tension. Again, for the same reasons, the decrease in flexural strength is, perhaps, due to overloading of filler content causing sedimentation and hence formation of two non-mixed phases.

Figure 8 shows the value of compressive strength. The neat epoxy (red line on the y-axis) has a compressive strength of about 107 MPa. It is clear that both CuO particles and polypropylene fibers increase the compressive strength epoxy by greater than 20%. While both fillers have approximately the same effect, it is

important to emphasize here that loading level of CuO particles is away smaller than that of fiber and easier in manufacturing.

Impact strength data are shown in Figure 9 and the general behavior is not that much different than the other properties. However, the maximum improvement in the impact strength reaches to about 80% for the three different systems. This indicate that the prepared systems can absorb more energy. The impact properties are, arguably, related to the morphological features of the polymeric materials. It is clear from the data given in Figure 9 that the addition of CuO and polypropylene fibers to epoxy leads to increasing the impact strength initially and then decreases. The results are in agreement with the results of the other properties discussed above and that supports that the best dispersion of fillers achieved at 1.5% and 60% for CuO particles and polypropylene fiber, respectively. Further loading, agglomeration starts to form and filler content increases the average size of nanoparticles domains reaches a micrometer level.

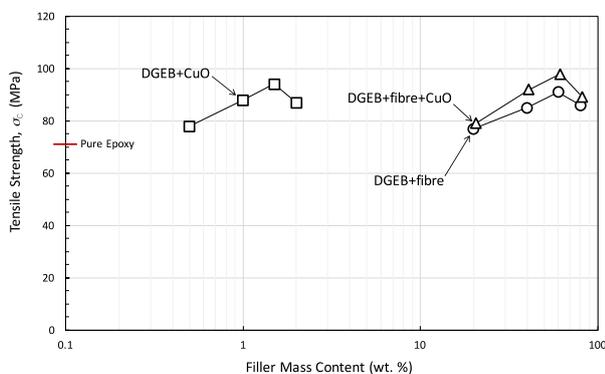


Figure 6. Tensile strength of the three prepared composite systems as defined in Figure 4

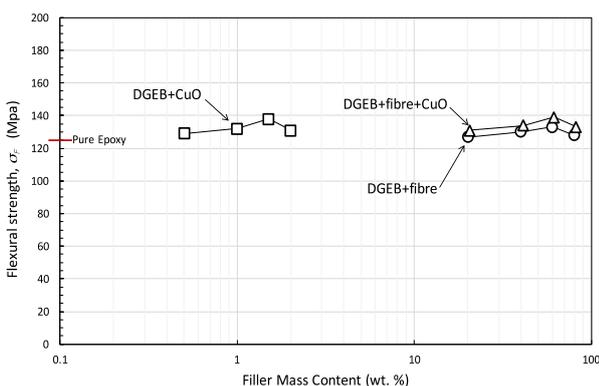


Figure 7. Flexural strength of the three prepared composite systems as defined in Figure 4

### 5. DISCUSSION

In general, the mechanical behavior of thermoset polymers relies on their microstructure and the density of the crosslinked chains [23]. For epoxy, these two factors are strongly affected by the presence of a filler. The more levels of fillers, the better interaction between the filler and the matrix and hence the better stress and heat transfer throughout the composite [24, 25].

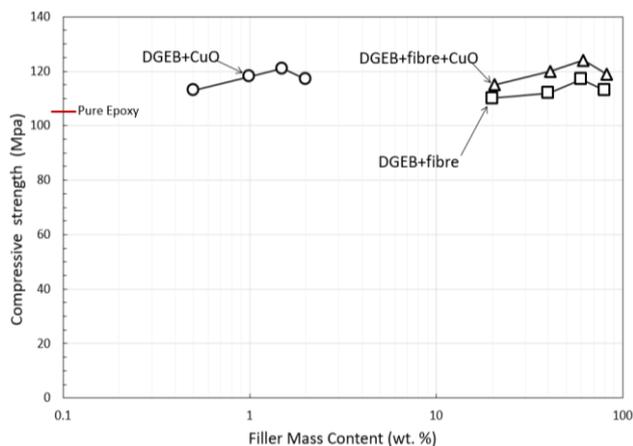


Figure 8. Compressive strength of the prepared composite systems

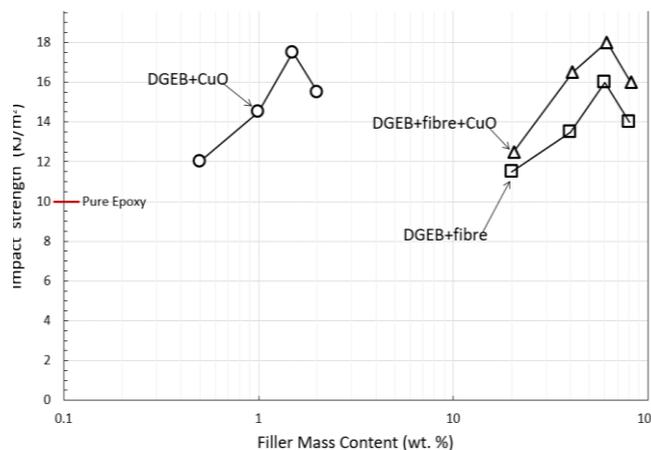


Figure 9. Impact strength of the prepared composite systems

Accordingly, the observed increase in composite thermal conductivity of nano-CuO composites is attributed directly to effect of filler content (Figure 5). This also explains the decrease in  $K_{eff}$  values of epoxy-fiber system observed in the same figure. Most importantly, the thermal model developed here supports these results, although there are some differences between its prediction and the experimental data.

For the mechanical properties the scenario is not different and it follows the similar principle of composite reinforcing. So, the general increase in composite mechanical properties (Figures 6-9) is attributed to the reinforcing effect of the filler which allow an effective transfer of stresses. However, agglomeration and sedimentation process tend to occur at a certain loading of fillers (as discussed earlier), leading to decreasing the properties in general. All these results are in good agreement with the results of the visual observations for the studied systems.

### 6. CONCLUSION

Different epoxy composites have been prepared in the current work using nano-CuO particles, polypropylene fiber and a hybrid system of these two fillers. The static mechanical properties and the thermal behavior of epoxy composites have been investigated as a function of filler content. Further, a model has been developed and used to

assess the thermal performance of the prepared composites. Except the decreases in  $K_{eff}$  values of epoxy-fiber with the filler content, the results showed that both nano-CuO particle and polypropylene fiber are efficient in reinforcing the thermoset epoxy. However, the comparison made here showed that reinforcing ability of nano-CuO particles can be considered more effective than fibers in terms of loading amount and ease of manufacturing. Also, the model developed here showed the ability to capture the experimental data very well. So, it is possible to be used as a useful tool to analyze and understand (at least qualitatively) the thermal behavior of composite material systems in general.

## NOMENCLATURES

### 1. Acronyms

DGEB Diglyceryl ether of bisphenol A

### 2. Symbols / Parameters

$C_p$ : Material specific

$k_i$ : Thermal conductivity

$K_{eff}$ : Composite thermal conductivity

$n_i$ : Vector normal to the matrix phase

$R$ : Interfacial thermal resistance

$t$ : Time

$T$ : Temperature

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