Journal of Minerals & Materials Characterization & Engineering, Vol. 11, No.2 pp.193-210, 2012
jmmce.org

Mechanical Properties of Potassium Titanate Whisker Reinforced Epoxy Resin Composites

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ABSTRACT

This paper deals with the study of mechanical properties of Potassium Titanate Whisker (PTW) reinforced epoxy based Polymer Matrix Composites (PMCs). Epoxy composites filled with PTW in various content of 0-20 wt% were prepared using the casting technique. Data on neat epoxy is also included for comparison. All tests were conducted at room temperature and as per ASTM standards. It was observed that inclusion of PTW affected most of the mechanical properties of neat epoxy. Density, hardness and heat deflection temperature of neat epoxy were found to increase with the PTW content. However tensile and flexural properties of the developed composites exhibited a varying trend with respect to PTW content. Epoxy filled with 10 wt% PTW showed good improvement in tensile strength and flexural strength of neat epoxy. It was observed that PTW is not beneficial in improving the impact strength of neat epoxy. Composites *with 20 wt% PTW exhibited least impact strength. This paper also highlights the possible reasons for variation in the mechanical properties of developed polymer composites.*

Key words: *Epoxy, PTW, PMCs, Mechanical Properties.*

1. INTRODUCTION

It is a common practice in the plastics industry to compound homo-polymers with fillers and fibers to dilute the manufacturing cost and/or attain desired properties [1]. By combining different fillers or fibers with various polymer matrices, polymer composites can be tailored to achieve property combinations which cannot easily be obtained from either the polymer matrices or the reinforcements alone. In past decades, many different substances have been used as reinforcements in composite preparation, although short fibers have attracted much more attention than other materials because of their low price and effectiveness in reinforcing polymers [2].

Whiskers are short fiber-shaped single crystals with high perfection and very large length-to-diameter ratios. According to theories of short fiber composites, the composites reinforced with thinner and stronger fibers can be anticipated to achieve much higher mechanical properties. Generally whiskers possess high strength and stiffness due to their nearly perfect crystal structure [3]. Therefore whiskers are reckoned as more effective reinforcements than traditional fibers such as carbon fiber and glass fiber. Recently various inorganic whiskers such as Calcium Carbonate ($CaCO_3$), Alumina (Al_2O_3), Silicon Carbide (SiC) and Potassium Titanate ($K_2Ti_6O_{13}$) were prepared and employed in the manufacturing of composites with different polymer matrices.

Several researchers have observed the significant changes in the mechanical properties of polymers reinforced with different kind of whiskers [4-19]. Youxi et al. [4] reported that optimal content of $CaCO_3$ whisker in PEEK composites is 15% to 20% combining both mechanical and tribological properties. The reinforcing effectiveness of $CaCO_3$ whisker contributing to increase in thermal stability, stiffness and load carrying capacity of PEEK was also reported by same investigators in another study [5]. Zhang et al. [6] investigated the mechanical and wear properties of silicon carbide and alumina whisker reinforced epoxy composites. It was observed in their study that both those whiskers significantly improved the flexural modulus and wear resistance of epoxy composites. However, Avella et al. [7] reported that addition of untreated SiC whisker into polypropylene lead to an enhancement of the modulus, but a decrease in the tensile strength.

Wang et al. [8] revealed that ZnO whiskers have better reinforcing effect with the nylon than the ZnO particles. ZnO whisker reinforced nylon exhibited higher tensile strength and hardness for most of the material combinations developed. Jang et al. [9] proposed modifications of matrix resin with thermoplastic particulates or ceramic whiskers as an alternative to rubber-toughening for improving the impact resistance of epoxy resins. They observed that dispersion of 10% of ceramic whiskers in an epoxy resin improved appreciably the impact energy, flexural strength and modulus of the epoxy.

Among the numerous inorganic fillers, potassium titanate whiskers (PTW, $K_2O.6TiO_2$) has been found to be a promising reinforcer for the wear resistant composites due to its unique properties, such as outstanding mechanical performance, low hardness (Mohs hardness 4) and excellent chemical stability. PTW is a kind of very fine micro-reinforcing material and it is suitable to reinforce the very narrow space in composites that conventional fillers are unable to do. In practice, it is an excellent fit for making products that have a complex shape, great precision and high polished surface. The price of the PTW ranges from one-tenth to one-twentieth of the cost of SiC whiskers [10]. In this regard, PTW have been used to reinforce most of the polymers. Many studies on PTW reinforced polymer composites have been carried out [11-19]. Generally Young’s moduli of the PTW reinforced composites increase with increasing whiskers content and impact strengths of the composites have opposite variation. But the tensile strengths of composites exhibit complicated variation.

Xing et al. [11] studied the effects of PTW on mechanical properties of PTW/PTFE composites and observed that PTW improved the properties of neat polymer. The range of PTW in PTFE for optimal integrative properties was found to be 5-10 wt%, which is similar to the behavior of nanoparticles. Chen et al. [12] fabricated PVC reinforced with controllably oriented PTW by hot pressing the precursor fibers that contained the whiskers. An increase of over 250% in tensile strength and 300% in flexural strength was observed for the composite containing 40% PTW. Shoubing et al. [13] systematically evaluated the tensile strength of a series of PTW filled castor oil-based polyurethane/epoxy resin interpenetrating polymer network and found an optimum increase in the tensile strength (25.5 MPa) of the composites at 3% of PTW. Demei et al. [14] reported a maximum PTW content limit of 20 wt% in polypropylene/polyamide blend matrix when the tensile strength of the composite increased with the increasing whisker content.

Tjong and Meng [15,16] observed increase in the tensile strength and modulus and decrease in strain at break and impact strength of PTW reinforced polyamide composites. Their study also revealed that surface treated PTW has better reinforcing effect with polymer matrices and hence enhance most of the mechanical properties. Increase in tensile strength and tensile modulus of PTW reinforced PEEK composites was also reported by Zhaung et al. [17]. It was revealed that compounding processes exhibit great influence on reinforcement efficiency of PTW. The composites pre-compounded with rheometer possessed higher mechanical performance than those pre-compounded with the extruder.

Zhu et al. [18] compared the effect of various whiskers on the performance of non-metallic (phenolic resin based) friction materials. The results showed that addition of the whiskers decreased the hardness of unmodified material and greatly improved its mechanical properties. Among them, magnesium borate whisker composite exhibited highest tensile strength and poorest Young’s modulus, calcium sulfate whisker composite exhibited best thermal stability and PTW imparted highest wear resistance to the composite. In a recent study on hybrid polymer composite friction materials, Mukesh et al. [19] observed that absolute friction effectiveness remained higher in the composites with ≥ 25wt% of PTW.

Most of the researches paid attention towards studying the influence of whiskers on thermoplastic materials and less work has been done by reinforcing the PTW with thermoset matrices. Epoxy resin is a well known thermoset polymer matrix as they possess better mechanical and thermal properties. They wet many substrate materials, absorb less moisture and can be processed with considerable degree of ease. The other advantages include excellent chemical resistance coupled with good electrical properties [20]. In this paper, study is focused on processing and mechanical characterization of PTW reinforced epoxy resin composites. The different mechanical properties such as density, hardness, tensile, flexural, impact properties and heat deflection temperature of the developed composites as a function of PTW content is measured to investigate the effect of PTW on epoxy resin system.

2. EXPERIMENTAL

2.1 Materials Used

Room temperature curing Epoxy resin system (LY556 + HY951 of M/s Hindustan Ciba Geigy Ltd, Mumbai) was used as the host matrix material. Potassium Titanate Whiskers (PTW, $K_2Ti_6O_{13}$) are ceramic micro- fillers and were used as the reinforcement. These ceramic whiskers are of splinter shape and properties are listed in Table 1.

Table 1. Properties of PTW

Diameter (μm)	Length (μm)	Density (g/cc)	Tensile strength (GPa)	Tensile Modulus (GPa)	Hardness (Mohs)
0.5 - 2.5	10 - 100	3.185	7	280	4

2.2 Fabrication of Composite Specimens

An open mold with cavity dimensions 300×300×6 mm was fabricated to cast polymer composites. The fillers were preheated to remove any moisture present and cooled to ambient temperature. The required quantities of filler were stirred gently into liquid epoxy resin, taking care to avoid the introduction of air bubbles. Hardener was then added to the resin in the ratio of 1:10 and then stirred to ensure complete mixing. The mixture was then poured into an open metallic mold coated with release agent to yield specimens of 300×300×3 mm upon curing and released from mold after 24 hrs.

Composition of the test specimens was varied up to 20% of filler loading at intervals of 5%. Extreme care was taken to avoid any undesirable filler settling effect by casting the slurry just prior to its gelling stage, all time keeping it in a stirred condition. This was done to ensure the uniform composition of cast specimens across its volume. Fig.1 outlines the casting procedure.

2.3 Mechanical Characterization

All tests were performed at room temperature and as per ASTM standards. The standards used are listed in the Table 2. All the reported values were calculated as averages over five specimens for each composition.

2.3.1 Density

Density is mass per unit volume and is usually expressed in g/cc. Densities of the composite were determined using the Archimedes principle. Distilled water at room temperature was used as the immersion fluid and the mass was measured using the high precision digital weighing balance (Shimadzu Japan, 0.1mg Accuracy). Mass of the specimen divided by the difference in the reading before and after the immersion of the composite specimen in the measuring jar gives density values for the specimens.

2.3.2 Hardness

Hardness of material is defined as the resistance to deformation, particularly permanent deformation, indentation or scratching. Hardness of developed composite samples was measured using Rockwell's Hardness Tester on M-Scale.

2.3.3 Tensile Properties

The tensile properties namely Tensile Strength, Tensile Modulus and Elongation at Break were investigated using Universal tensile testing machine (JJ Lloyd, London, United Kingdom, capacity 1–20 kN). The tensile test was performed at a crosshead speed of 10 mm/min considering a gauge length of 50 mm.

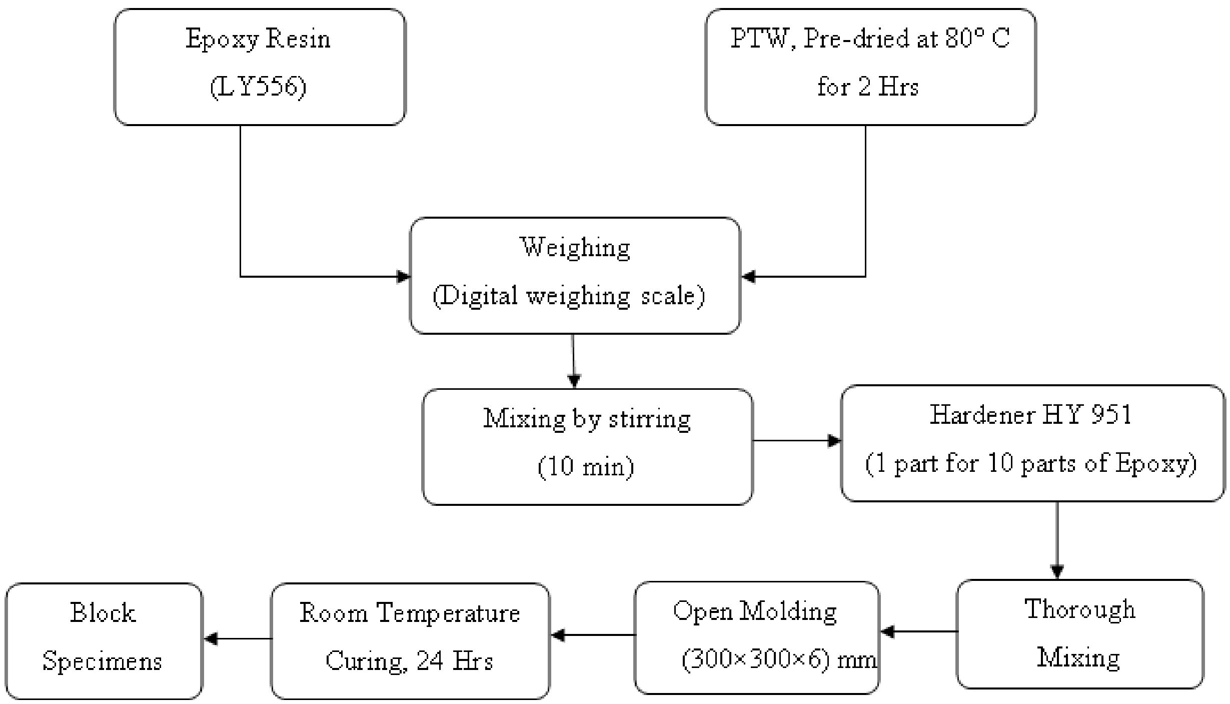

Figure 1. Schematic representation of the casting procedure

2.3.4 Flexural Properties

The flexural properties were investigated using Universal testing machine (JJ Lloyd, London, United Kingdom, capacity 1–20 kN). Three-point bending test was performed at a crosshead speed of 13 mm/min considering a beam span of 50 mm.

2.3.5 Impact Strength

Impact strength refers to ability of material to absorb the energy. Izod impact testing was done using CEAST pendulum impact testing machine (Max. capacity 25J). Unnotched specimens were fractured by impact input energy of 5.5 J.

2.3.6 Heat Deflection Temperature

Heat deflection temperature corresponds to the temperature that results in a deflection of 0.25mm at the mid span of the composite specimen during the three point bending conditions. This temperature was measured using a custom built set up.

Table 2. Standards used for Mechanical Testing

Mechanical Properties	Standards
Density	ASTM D792
Hardness (Rockwell M scale)	ASTM D785
Tensile properties	ASTM D3039
Flexural properties	ASTM D790
Impact strength (Izod test)	ASTM D256
Heat deflection temperature	ASTM D648

3. RESULTS AND DISCUSSIONS

PTW has modified the properties of neat epoxy in many ways. Significant improvements have been achieved by incorporating few wt% of PTW into epoxy matrix. Mechanical properties of filled polymer composites depend strongly on three important factors namely filler size, filler-matrix interface adhesion and filler loading [21]. Various trends of effect of PTW on composite properties have been observed due to interplay between these three factors which cannot be separated.

3.1 Density

Density of a composite depends on the relative proportion of matrix and reinforcing materials and is one of the most important factors determining the properties of the composites. Many mathematical models are developed for determining the densities of polymer composites. Rule of mixture [20] is one such simplest and widely used model for determining the theoretical densities of polymer composites. The variation in the density with respect to PTW content is reported in the Figure 2. It was observed that incorporation of PTW into epoxy matrix has increased the density of composites. This is mainly because of highly dense PTW fillers. Figure 2 also compares theoretical density values and experimental results. It was observed that experimental values are slightly lesser than that predicted by Rule of mixture. This was mainly because of micro voids present in the composite which was not considered in theoretical calculations.

The voids significantly affect some of the mechanical properties and even the performance of composites in the place of use. Higher void contents usually mean lower fatigue resistance, greater susceptibility to water penetration and weathering. The knowledge of void content was desirable for estimation of the quality of the composites. It was understandable that a good composite should have fewer voids. However, presence of void was unavoidable in composite making particularly through hand-lay-up route.

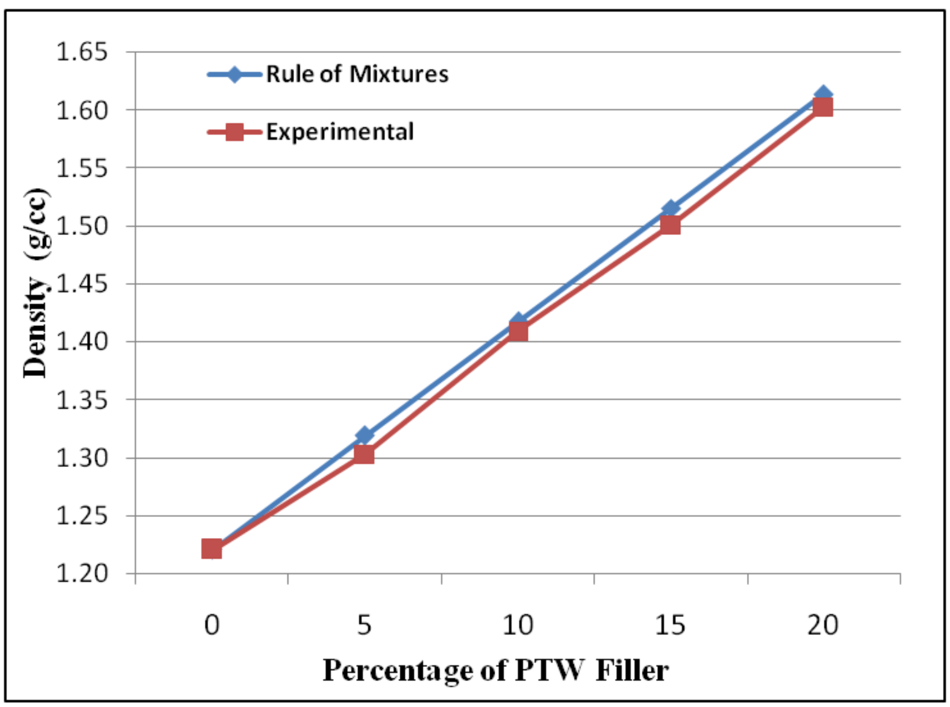

Figure 2. Effect of PTW content on the Density of neat Epoxy

3.2 Hardness

PTW was found to increase the hardness of neat epoxy (Table 3). Increase in hardness of neat epoxy with the increase in the content of PTW can be attributed to uniform dispersion of the harder PTW phase in epoxy matrix. A significant improvement in hardness was observed for 10 wt% PTW and hardness used to increase by small value at higher PTW contents. High strength PTW reinforcements may result in forming a network structure that improves the hardness of the composites. Whisker reinforcements such as SiC, Al_2O_3, $CaCO_3$ and PTW generally impart higher hardness to base polymer matrix [4,6,11].

3.3 Tensile Properties

Effect of PTW on tensile strength of the composites is depicted in Figure 3. Optimal increase in the tensile strength was observed at 10% PTW (50.94 MPa) compared to neat epoxy (44.65 MPa) which indicates a gain of nearly 14% in tensile strength. Reduction in the tensile strength at higher whisker loadings is explained by poor polymer-whisker interaction.

Ultimate strength of a composite depends upon weakest fracture path through the material. Hard particles such as PTW affect the strength in two ways. One is the weakening effect due to stress concentrations they cause and another is the reinforcing effect since they serve as barriers to crack growth [21]. In some cases, the weakening effect is predominant and thus composite strength is lower than matrix and in other cases reinforcing effect is more significant and then composites have higher strength than the base matrix.

Table 3. Hardness properties of Epoxy/PTW composites

Material	Hardness (Rockwell M Scale)
Neat Epoxy	90
Epoxy + 5% PTW	92
Epoxy + 10% PTW	95
Epoxy + 15% PTW	96
Epoxy + 20% PTW	97

Tensile modulus and percentage elongation variation with PTW loading is illustrated in Figure 4. Tensile modulus is found to decrease at 5 wt% of PTW and later increased considerably at higher filler loadings. It indicated that the composite has achieved superior stiffness at higher PTW content. Highest composite stiffness was observed at 20 wt% PTW which is a gain of 15% over neat Epoxy. Improvement in tensile modulus is an obvious effect of stiffer PTW particles. However elongation was reduced with the increase in the filler content. This indicated the loss in the ductility of the composite at higher PTW contents.

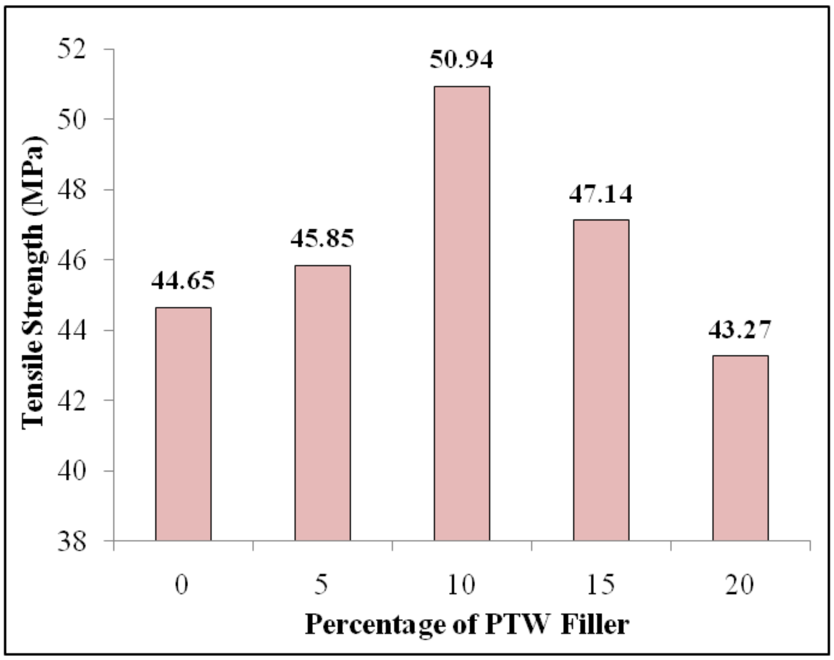

Figure 3. Effect of PTW content on the Tensile Strength of neat Epoxy

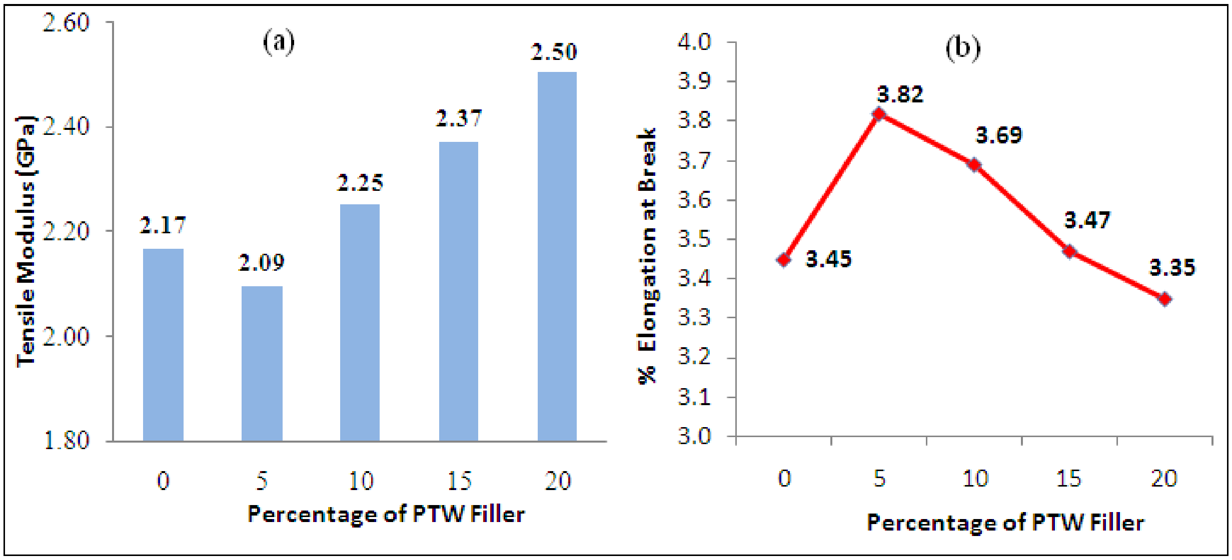

Figure 4. Effect of PTW on (a) Tensile Modulus (b) Percentage Elongation of neat Epoxy

Similar trend in the tensile behavior of PTW reinforced polymer composites with PEEK as matrix material [22, 23] and PP/PA blend [24] has been reported by previous researchers.

3.4 Flexural Properties

Flexural strength indicates ability of material to withstand bending forces applied perpendicularly to its longitudinal axis. In the present investigation a maximum strength of 72.48 MPa was observed for 10 wt% PTW which is an improvement of nearly 24% over the neat epoxy (Fig. 5a). However maximum flexural modulus of 4.30 GPa was observed at 5 wt% of PTW which is a gain of 13% over that of neat Epoxy (Fig. 5b).

Flexural strength was found to decrease at higher PTW content. Interfacial adhesion between PTW and epoxy molecules might be weaker than the intermolecular forces of epoxy and the micro-porosities of PTW/Epoxy composites tends to increase with the increasing PTW content, thus the flexural strength of composites was decreased at higher PTW content. In the present study flexural modulus obtained for composites are higher than that for neat epoxy for all material combinations. However at higher PTW contents, compatibility between the polymer matrix and PTW became poor and flexural modulus found to decrease. In the experimental range the best flexural properties were obtained with the composite with 10% PTW.

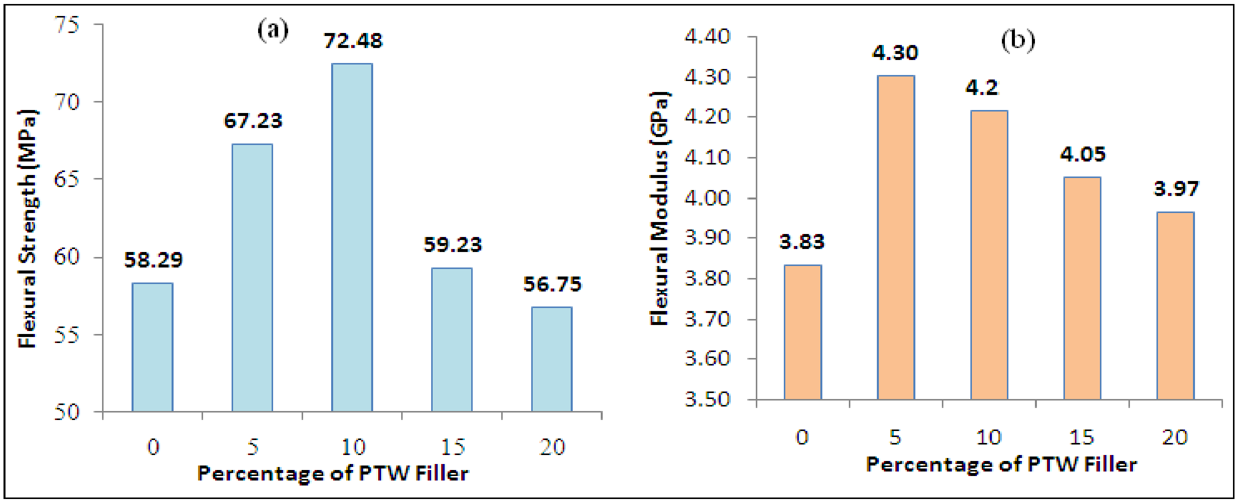

Figure 5. Effect of PTW content on (a) Flexural Strength (b) Flexural Modulus of Epoxy

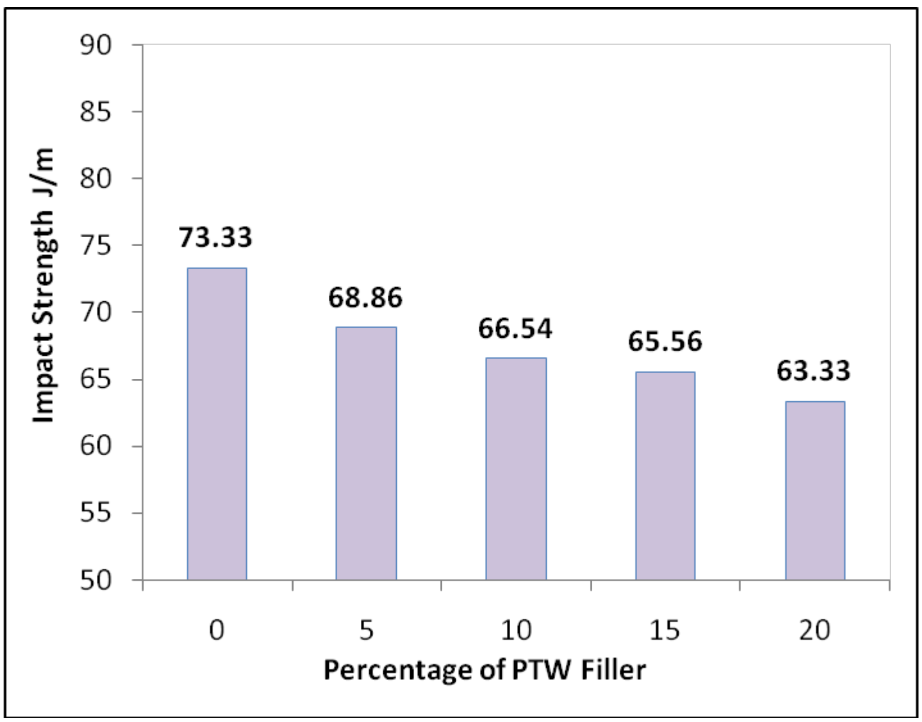

Figure 6. Effect of PTW content on the Impact Strength of neat Epoxy

3.5 Impact Strength

The impact property of polymeric materials is directly related to the overall toughness of the material. The objective of Izod impact test is to measure the relative susceptibility of a standard test specimen to the pendulum type impact load. The results are expressed in terms of kinetic energy consumed by the pendulum in order to break the specimen. The impact resistance results were very striking (Fig. 6). It was observed that impact strength of all materials combinations are less than that of neat epoxy. Composite with PTW content of 20 wt% exhibited the least impact strength (63.33 J/m) compared to neat epoxy (73.33 J/m) which is reduction of nearly 14%. It is obvious that PTW is not beneficial in improving the impact properties of neat epoxy.

Polymer based composite materials when subjected to impact type of loading conditions, energy is absorbed in the process of plastic deformation of matrix material, debonding at matrix/reinforcement interface and in the fracture of reinforcing material. The phenomena that absorbs least energy for its occurrence become prominent and leads to fracture [25]. In the present study, plastic deformation of epoxy matrix and debonding at interface could be the reason for decrease in impact properties of composites. However impact strength can be enhanced by proper surface treatment of whiskers resulting in improved adhesion between polymer and PTW and also by using suitable toughners for epoxy [15,16].

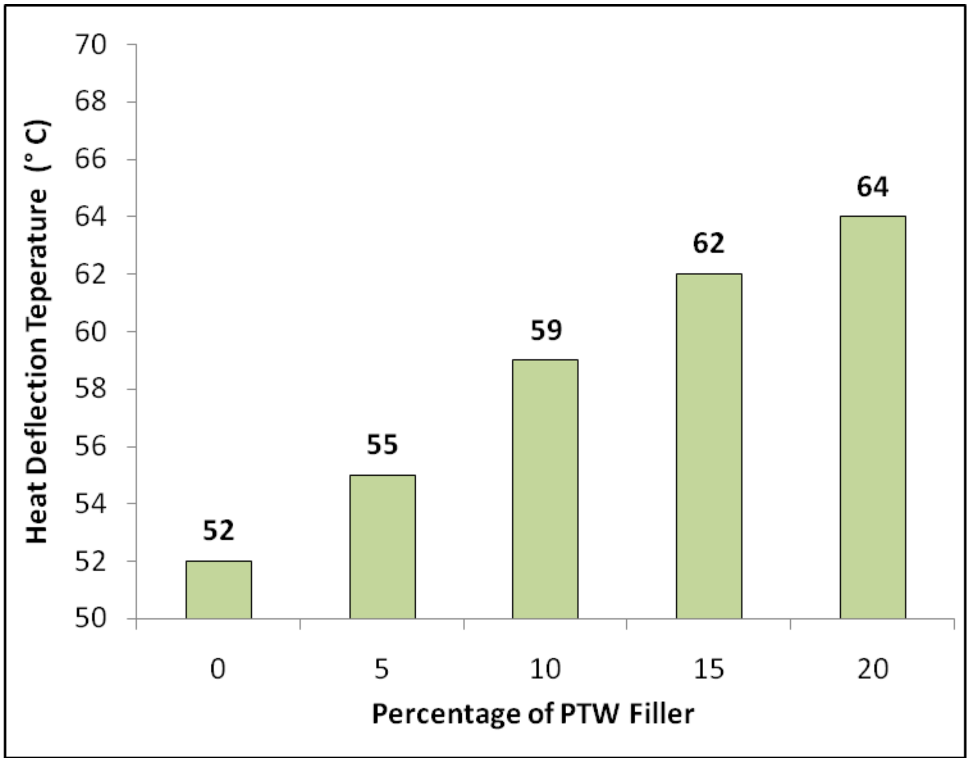

Figure 7. Effect of PTW content on the Heat Deflection Temperature of neat Epoxy

The decline in the impact strength with the increase in the PTW content was reported by Zhuang et al. [22] and Long et al. [26] also observed same trend of impact strength with the PTW loading in case of Polypropylene composites.

3.6 Heat Deflection Temperature

Heat deflection temperature is an indicator of general short term temperature resistance of materials. PTW was found to have positive influence on the distortion temperature of neat epoxy. An increase in the distortion temperature was observed with the PTW loading (Fig.7). At 20 wt% loading of PTW, a gain of 23% in deflection temperature was observed compared to the performance of neat epoxy. Increase in the deflection temperature is an obvious result of high thermal stability (1200°C in air) of PTW.

4. CONCLUSIONS

The effect of content of PTW on the properties of epoxy resin system was carefully studied. Study revealed a direct correspondence between the content of PTW and mechanical properties of composites and following conclusion were drawn.

1. PTW is an excellent performance whisker; it can improve density, hardness and heat deflection temperature of neat epoxy.

2. PTW filler addition shows significant improvement in tensile and flexural properties only at certain content (5-10 wt%). This performance is similar to that of nano-particles. Thus PTW can be an effective strengtheners and stiffners for thermosetting resins like epoxy.

3. Addition of PTW indicated a detrimental effect on impact strength of epoxy and it cannot be an effective toughners for epoxy.

PTW has immense scope on the fabrication of whisker reinforced polymer composites having vast number of industrial applications.

ACKNOWLEDGEMENTS

The authors would like to thank Director Fr. Valerian D'souza and Principal Dr. Joseph Gonsalvis, St. Joseph Engineering College Mangalore, for their cooperation and encouragement to carry out research work. Financial assistance provided by Sriram Charitable Trust Mudradi is highly acknowledged. Authors also express their sincere thanks to M/s. GTTC Mangalore for technical help and M/s. Brakes India Ltd. Mysore for providing testing facilities.

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