

Mechanical, Thermal and Wear Characteristics of Polymer Composite Material Reinforced with Calcium Carbonate Powder

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ABSTRACT

Quick mast-105 is a well-known epoxy-based adhesive used in fabric reinforcement, bonding of wooden parts and variety of metals. Its mechanical and thermal properties are still limiting factors for extended technological and industrial applications. Enhancement of mechanical, wear and thermal characteristics of this epoxy resin using a cheap filler of CaCO₃ powder is the focus of the present study. The mechanical properties are demonstrated in terms of the flexural modulus, hardness and impact strength of the reinforced cured compound while thermal properties are presented through thermo gravimetric analysis and thermal conductivity coefficient. Results show that addition of 15% by weight of CaCO₃ to the epoxy resin improves the mechanical properties and the thermal resistance of the end product paving the way for wider applications in industry and technology. Wear characteristics show that the reinforced product is suitable for high-speed applications.

Keywords: Polymer Composite, Epoxy, Reinforcements, CaCO₃ nanofiller, Mechanical Properties

الخواص الميكانيكية والحرارية وخاصة البلى لمادة بوليميري متراكبة مدعمة بمسحوق كربونات الكالسيوم

الخلاصة

ايبوكسي بعد (quick mast 105) من المواد الايبوكسية اللاصقة المعروفة والمستخدمة في مجال تدعيم النسائج ولصق الاجزاء الخشبية والمواد الاخرى . وتعتبر الخواص الميكانيكية والحرارية لهذا الايبوكسي من العوامل المحددة في مجال التطبيقات التكنولوجية والصناعية لهذه المادة . تهدف هذه الدراسة الى تحسين الصفات الميكانيكية والحرارية وخواص البلى لهذا الايبوكسي باستخدام مادة حشو رخيصة التكلفة من مسحوق كربونات الكالسيوم . لقد تم في هذا البحث استعراض الخواص الميكانيكية من خلال دراسة معاملات الانحناء والصلابة ومقاومة الصدمة للعينات الايبوكسية

المدعمة فيما تمت دراسة الخصائص الحرارية من خلال التصرف الحراري – الوزني ومعامل التوصيلية الحرارية للعينات المدعمة . وقد اوضحت النتائج ان اضافة كاربونات الكالسيوم بنسبة وزنية تعادل 15 % من وزن الايبوكسي تؤدي الى تحسين الخواص الميكانيكية والحرارية للعينات المدعمة مفسحا المجال لاستخدامها في مجالات صناعية وتكنولوجية اوسع . اما دراسة خاصية البلى فقد بينت ان مادة الايبوكسي المدعمة مناسبة للتطبيقات عالية السرعة.

INTRODUCTION

Polymer composites are materials used in diverse engineering applications such as gears and pumps impellers where the components undergo high erosive wear. The composite materials may present erosive wear that is typical of brittle materials[1,2]. Epoxy-based polymers are of much extensive use as coatings, adhesives, insulating parts and many others because epoxies are known for their excellent adhesion, high electrical and heat resistances and good mechanical properties[3]. Addition of nanopowders of different compounds such as Al_2O_3 [4-6], glass fibers[7,8], carbon nanotubes and carbon black[9-11], to epoxy-based polymers is believed to produce new composites of improved mechanical, thermal and wear properties in addition to being relatively cost-effective fillers compared to their original counterparts. This may consequently pave the way to new industrial applications and solutions[12-14]. These additives are believed to affect the tensile properties, packing characteristics and interfacial bonding of the polymer leading to enhanced properties for industrial and technological applications[15]. Improvement of mechanical properties has been attributed to formation of conjugated biphasic material due to addition of fillers. These fillers will then tolerate some of the applied tension to the polymer matrix leading to enhanced tensile properties of the end product.^[16] One of the most common fillers is the calcium carbonate ($CaCO_3$) which has been used as a cheap substitute to the Polyol for the production of flexible polyurethane foam leading (widely used in industry) to enhanced mechanical properties of the end product[17-19]. Incorporation of $CaCO_3$ in thermoplastic production is a common practice for reduced production cost in plastic industry[20].

Quick mast-105 is an epoxy resin for multipurpose uses like fabric reinforcement, bonding of wooden parts and variety of metals in addition of being used as adhesive for wide range of applications. It is known to have high flash point and no strong odor making it safer than polyesters. It also can be sanded and shaped after being cured and hardened. However, its mechanical and thermal properties are limiting factor for new applications and uses. It is therefore the aim of this study to enhance the mechanical, the wear and the thermal properties of the quick mast 105 epoxy resins by adding $CaCO_3$ nano-powder at an optimum ratio.

EXPERIMENTAL METHOD

Particle size distribution of $CaCO_3$. Calcium carbonate powder from natural sources was grinded for 10 hours to produce a fine powder of nanosize particles. The particle size was confirmed by measuring the particle size distribution of the $CaCO_3$ powder after grinding process. Particle size analyzer from MALVERN was used for the measurement. Equal amount of powders were dispersed in water and

sonication was performed for 10 minutes to achieve the required dispersion of the particles. Measurement was repeated three times to ensure consistency of results.

Samples Preparation. Five samples, with five different powder-to-epoxy weight ratios, have been prepared. The weight ratios of the CaCO₃ were 0.0% (Pure sample), 2%, 5%, 10% and 15% with respect to the epoxy. Initially epoxy resin (quick mast 105) and accurate ratio of CaCO₃ were mixed thoroughly for 15 to 20 minutes. The hardner was then added to the epoxy-CaCO₃ compound while continuously mixed to a homogeneous slurry. The mixture was then moulded in proper shapes and dimentions appropriate for the measurements setups requirements. The moulded samples (three specimen for each ratio) were left to cure at room temperature for three days before measurements were accomplished. Each measurement was repeated for at least three times and an average of the measured value was determined.

Mechanical and Thermal measurements. Flexural properties of the CaCO₃ reinforced quick mast samples were measured by 3-point bend method using ASTM-D790 bend tester from PHYWE instrumentation. For each load applied on the mid of the sample the deformation is measured accurately. From the slope of the load vs. deformaion graph one can determine the bending (flexural) modulus (E_b) from the following equation[2, 21]:

$$E_b = \frac{FL^3}{48 I \delta} \quad \dots(1)$$

Where F is the applied force, L is the distance on the sample between the outer holders, I is the moment of Inertia and δ is the measured deformation. The effect of the reinforcement is then investigated by plotting between concentration of CaCO₃ and the flexural modulus.

Hardness of shore durometer (D). Hardness of the prepared composite material was measured for all samples using shore hardness tester with shore hardness gauge, complied with DIN 53505 ISO 868 and ASTM D2240 from Thermimport quality control (TQC). Determination of hardness is based on applying the force on consistent manner without shock and measuring the depth of indentation at the surface of the samples.

Impact resistance. The impact fragility characteristics under shear stress of the prepared samples were measured using pendulum impact testing machine XJU-22. This measurement process is known as Charpy impact test which is based on determination of the amount of energy absorbed by the material during fracture (see for example [22]). Values of impact strength (G_c) and fracture toughness (K_c) are determined from this measurement. These two values are given by the following equations[21]:

$$G_c = \frac{U_c}{A} \quad \dots(2)$$

Where U_c is the energy required for sample breaking and A is the sample cross-sectional area.

$$K_c = \sqrt{(E_b \times G_c)} \quad \dots(3)$$

E_b is the flexural modulus in Pa unit.

Wear. The wear characteristics of the original and reinforced materials were measured by means of a pin-on-disk machine at different speeds and loads under dry sliding condition. Specimens were prepared in cylindrical shape of 10mm diameter and 15mm length. End-surfaces of each specimen were initially polished with 600 grade Silicon Carbide papers to ensure proper contact with the counter surface of the rotating disc. A carbon-steel disc was used as a counter surface rotating at 420 rpm. Prior to measurement, both specimen and disc surfaces were properly cleaned. Details of the wear machine and the experimental procedure are available elsewhere [23]. Wear characteristics were measured at constant sliding speed under different loads of 2, 5 and 10N and at constant load with varying sliding speed of 1.14, 2.73 and 3.08 cm/sec. The time of each measurement is fixed to 15 minutes. Wear rates were determined by accurate and careful measurements of weight losses (in mg) per sliding distance (in cm) under the above mentioned parameters of load and speed. During the test the pin was pressed against the rotating disc by applying the load. After running through a fixed sliding distance, the specimen were removed, cleaned, dried and weighed to determine the weight loss due to wear. Wear rate is calculated from the following equation :

$$\text{Wear rate (g/cm)} = \frac{\Delta M}{S} \quad \dots (4)$$

Where ΔM is the weight loss and S is the total sliding distance.

Thermogravimetric (TG) analysis. TG measurements were accomplished using high-performance modular thermogravimetric SETSYS Evolution analyzer from SETARAM instrumentation. The amount of the reinforced compound used for the measurement is about 15mg in each case. TG curves were obtained at constant temperature rate of 30deg/minute under flow of protective gas.

Thermal conductivity. The coefficient of thermal conductivity of the composite material under investigation was measured by Lee disc method which is appropriate for thermal conductivity measurement of poor thermal conductors. The coefficient of thermal conductivity k in W/m.K is determined from equation 5 below. The details of the measurements are described elsewhere [24].

$$k = \frac{msd(dT/dt)}{(A(T_1 - T_2))} \quad \dots (5)$$

where m , s , d are the mass, specific capacity and thickness of the lower disc in the Lees' apparatus. T_1 and T_2 are the temperatures of the upper and lower discs respectively (see reference [24]). The specimen is placed between the two discs of the apparatus.

RESULTS AND DISCUSSION

The particle size distribution of the CaCO_3 powder is shown in Figure(1) exhibiting sizes in the range 297nm – 2.38 μm . Figure (1) shows that the number of particles in the small size region of the distribution is higher than that in the large size region. The average size of the particles is found equal to 519.4 nm. Therefore, the grinding has produced a CaCO_3 powder. This powder is expected to mix

homogeniously with the epoxy resin providing reinforcement centers in the epoxy matrix at nanoscale dimension.

Figure (2) shows the dependence of the flexural modulus of the end-compound on the ratio of the CaCO_3 powder. The flexural modulus is found to decrease with increasing the additive concentration in the epoxy matrix. This behaviour could be attributed to a decrease in the elasticity property of the material due to increase of foreign particles of CaCO_3 . The highly concentrated samples with CaCO_3 were observed to require higher load for a similar deformation than the low concentrated samples. The increased concentration of particles increases the bonding strength of the epoxy as $(F/\delta)_{15\%} > (F/\delta)_{0\%}$ (see equation 1) which can also be measured experimentally. This is supposed to reflect on the hardness property of the reinforced compound such that the highly reinforced samples exhibit higher hardness. Figure (3), hardness of shore durometer, emphasizes this effect. Samples with higher concentration of CaCO_3 particles shows higher resistance to indentation force therefore exhibiting higher shore-D values. This could be attributed to the inherent hardness of the CaCO_3 particles compared with the epoxy which affects the overall hardness of the end-compound and becomes more prominent at higher concentrations. Figures (2 and 3) emphasize on the behavioural change of the mechanical properties of the end-compound which is believed to comply with the general strategy of the reinforcement technique. The additional CaCO_3 nanoparticles are thus working as tension-tolerating centers in the epoxy matrix leading to the exhibited mechanical changes. This is found to agree with previous observations of toughening mechanisms of other epoxies reinforced with silica nanoparticles [4, 6].

Figure (4) shows the impact strength (G_c) of the composite epoxy material before and after the reinforcement with the CaCO_3 particles. The impact strength, the energy required to break the sample, was found to increase with the increase of CaCO_3 concentration. From the energy conservation point of view the energy due to the applied shock should transfer to the sample material upon the impact. This energy transfer may take different forms; i.e. from kinetic energy to heat or deformation. But since the force is applied for a short time the energy transfer has to occur in this time scale. The response of the material to this effect is to behave like it is brittle leading to occurrence of fracture at the point of impact according to the principle of time-temperature superposition [25-27]. The energy absorbed in the present samples is found to increase with the increase of the CaCO_3 ratio in the epoxy matrix. The reinforcing particles are believed to absorb part of the delivered energy and prevent formation of fractures in the epoxy matrix. Yet these particles increase the stiffness of the specimen (i.e. sample hardness) as observed in figure 3. And this means that the material exhibits a loss in the elasticity property which is supported by Figure (2). The fracture toughness (K_c), presented in Figure (5), shows a decrease in value with the increase of CaCO_3 powder concentration. The fracture toughness is calculated from equation 3 which shows a direct dependence on the impact strength and flexural modulus. The behaviour of K_c in Figure (5) should therefore be attributed to the dominating effect of the flexural modulus (E_b) which appears as a second term in equation 3. This is equal to saying that K_c in Figure (5) resulted from a direct superposition of Figures (2 and 4). It is then justifying to reach at a conclusion that the addition of CaCO_3 nanopowders into the epoxy matrix had a more prominent effect on the flexural properties than on the

impact resistance. The addition of CaCO_3 made the quick mast 105 epoxy harder and less ductile product.

Wear characteristics of the original and reinforced specimens are presented in Figures (6 and 7). Figure (6) shows the wear rate dependence on the load variation for original and CaCO_3 reinforced specimens at sliding speed of 200 cm/sec for 15 minutes. It exhibits an increase of wear rate for all specimens with the increase of load. This is attributed to the effect of friction which increases due to load increase. Furthermore, the wear rate values increases with the increase of CaCO_3 ratio. Recalling that hardness increases with the CaCO_3 ratio, this indicates that the wear is non-adhesive in nature (increase of hardness and wear) [28]. It is known that with the increase of load the contact between the two surfaces increases leading to rapid deformation of epoxy protrusions because compressive and frictional forces dominate in dry sliding conditions [23]. Presence of reinforcing particles of CaCO_3 powder in these protrusions increases the wear rate. This could be attributed to weak adhesive forces between CaCO_3 particles and the epoxy resin leading to immediate removal of the former from the surface of the specimen during sliding. This again supports our conclusion of the non-adhesive characteristic of wear in the present composite material.

Figure (7) shows the wear rate dependence on sliding speed at constant load of 5N. It exhibits a decrease of wear rate with the increase of sliding speed for all specimen. At constant sliding speed the wear rate increases with the increase of the CaCO_3 concentration in the epoxy matrix (in agreement with the results from Figure (6)). These observations could be understood in terms of frictional forces acting between the sliding surfaces which decrease with speed. This reduction in frictional force will obviously lead to increase of wear resistance. As the wear rate depends on the weight loss and the sliding distance (see equation 4) which both increase with speed one expects that the dominating factor in equation 4 is the sliding distance. This means that the epoxy resin exhibits an inherent resistance to wear effect. However, this property is found to deteriorate with increase of CaCO_3 ratio. Nevertheless, Figure (6 and 7) both suggest that the reinforced epoxy could be more appropriate for high-speed applications than for high-load applications.

The TG curves of the reinforced composites are presented in Figure (8). All curves exhibit no major weight loss at temperatures as high as 350°C attributed to inherent characteristic of the original quick mast 105 epoxy resin. The thermograph shows an endothermic hump at about 150°C with no weight loss. This is attributed to a phase change in the reinforced composite material. An onset of weight loss followed by increasing loss rate is observed at onset temperature higher than 350°C . At 400°C 50% of weight loss is observed and attributed to decomposition of the epoxy resin. A careful investigation of this region (350°C - 400°C) and presence of exothermic peak indicates that the composite material undergoes a combustion reaction. At 470°C the TG curves can separately be identified from one another as material with higher CaCO_3 ratio exhibit less weight loss. Owing to the endothermic peaks at this temperature and the weight loss dependence on the epoxy resin content it is possible to attribute the TG curve to a final decomposition and evaporation of the epoxy resin. At 530°C the weight losses are only 80.15%, 76.2%, 73.9% and 66.54% for the 2%, 5%, 10% and 15% CaCO_3 reinforced samples respectively. The broad exothermic peaks between 530°C and 700°C is

believed to be due to another combustion of the remaining epoxy compounds. In the temperature range 700°C–800°C another weight loss is observed accompanied with an endothermic peak. These peaks are closely related to decomposition temperature of CaCO₃ bulk material (i.e. 840°C) according to the following equation:



Decomposition of CaCO₃ is known to occur at slightly higher temperature than the temperature observed here. In addition the endothermic peaks are observed to shift to higher temperature with the increase of the CaCO₃ ratio. The dependence of the decomposition temperature of CaCO₃ powders on particle size has been investigated by Mohamed M. *et al.* showing an increase of decomposition temperature and a decrease of decomposition rate with size [29]. Knowing that melting of CaCO₃ precedes its decomposition, this explains the shift of the decomposition peak, its distinction at higher concentrations of CaCO₃ powders and the rate of mass loss near the decomposition peak because melting produces higher amount of CaCO₃ that requires higher temperature of decomposition. Therefore, the present results agree with the observations made by Mohamed *et al.* [29]. At 780°C the TG curves exhibit no further weight losses and the remaining compounds can obviously be related to the residual CaO; the higher the starting ratio of CaCO₃, the higher is the residual CaO. The most important realization to make from the comparison of the TG curves is that the addition of CaCO₃ particles to the epoxy resin did not involve a chemical reaction between the epoxy and the powder. Therefore the mechanical properties of the new composite material could be explained on the basis of the tensile properties, the packing characteristics and the interfacial bonding characteristics in the epoxy matrix. The thermal conductivity in Watt/m.K of the CaCO₃ reinforced epoxy resin is shown in Figure (9) exhibiting a decrease in the conductivity value with increasing ratio of CaCO₃ powder. Figure 9 shows that addition of CaCO₃ enhances the thermal insulating property of the quick mast 105 epoxy resin. This is believed to pave the way for new applications where high thermal resistance is concerned.

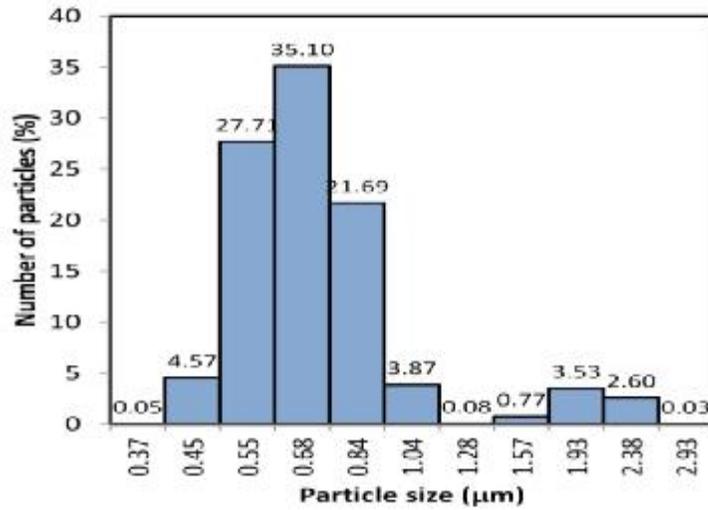


Figure (1) Particle size distribution of CaCO₃ powder measured after grinding of original commercial powder.

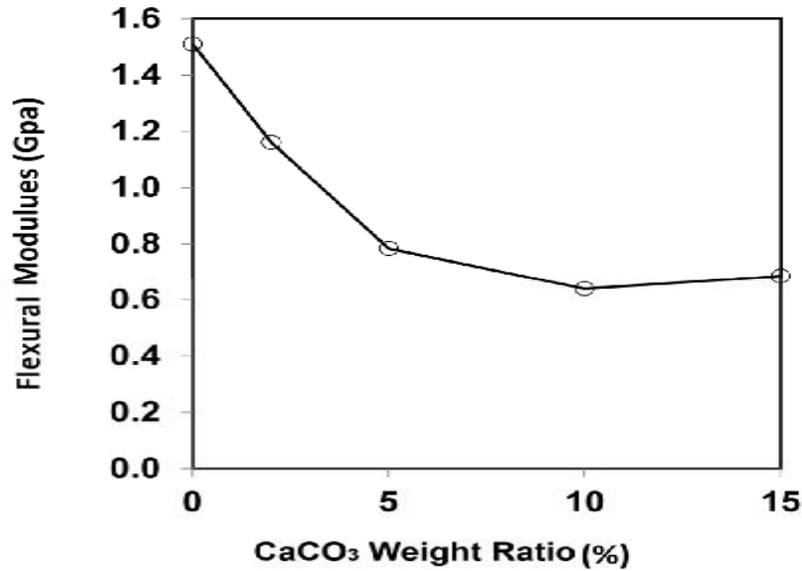


Figure (2) Dependence of flexural modulus of the reinforced epoxy polymer samples on weight percentage of CaCO₃ powder.

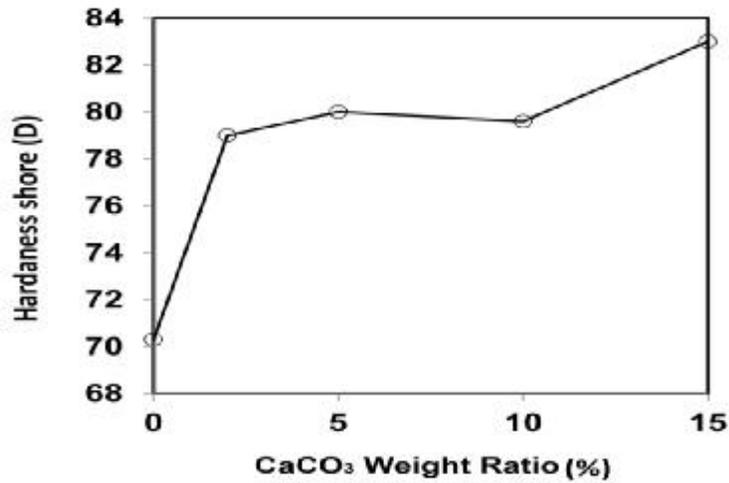


Figure (3) Behaviour of Hardness of shore durometer (D) on weight percentage of CaCO₃ powder.

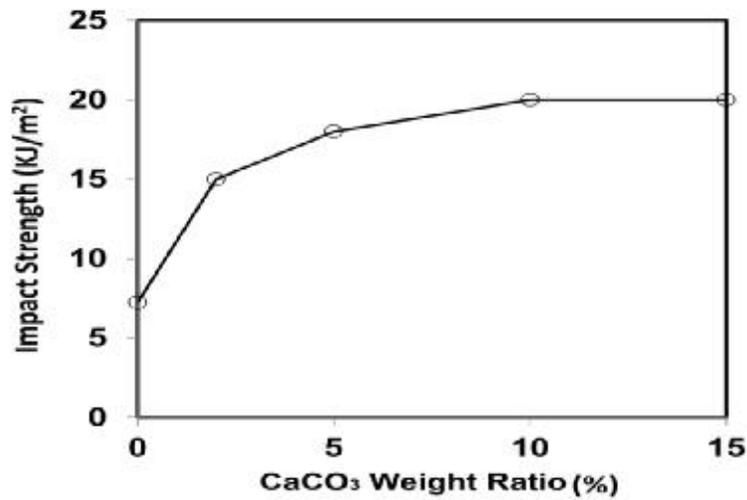


Figure (4) Impact strength dependence on the weight percentage of CaCO₃ particles of pure and reinforced epoxy polymer.

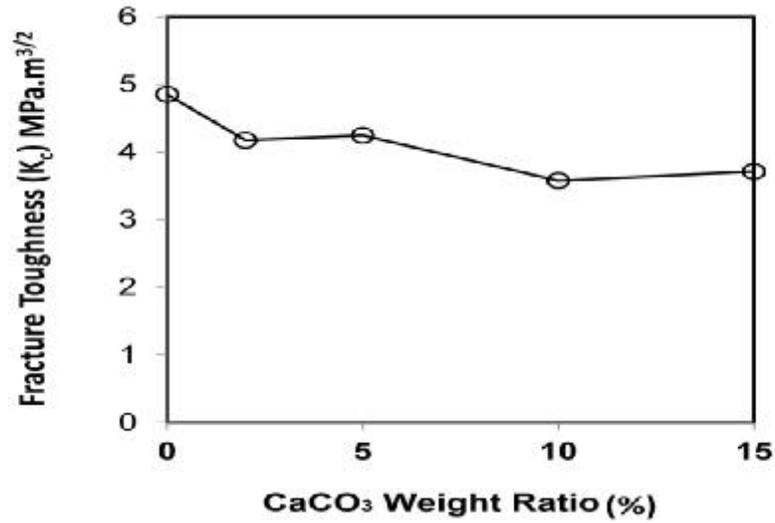


Figure (5) Behaviour of fracture toughness of the CaCO₃ reinforced epoxy polymer.

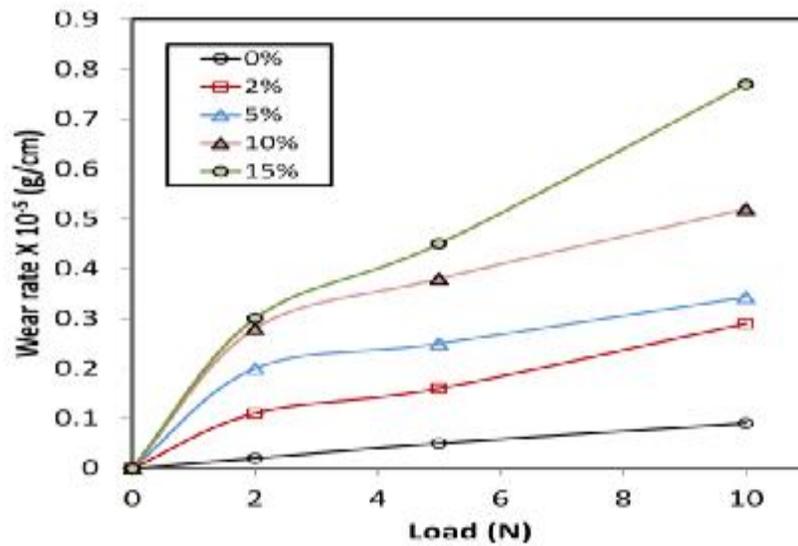


Figure (6) Wear rate dependance on load variation for pure and CaCO₃-reinforced epoxy polymer.

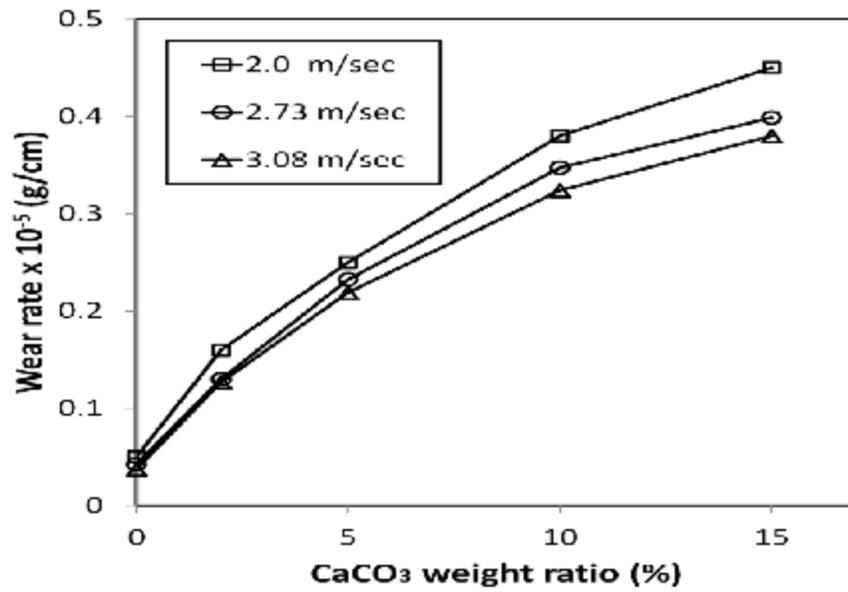


Figure (7) Wear rate dependence on CaCO₃ weight ratio at different speeds for pure and reinforced epoxy polymer.

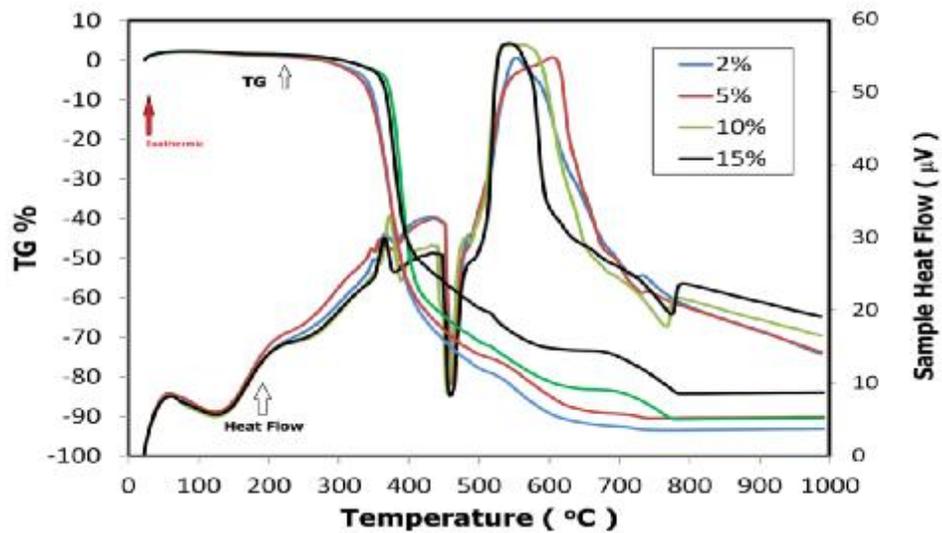


Figure (8) TG curves of CaCO₃ reinforced epoxy polymer.

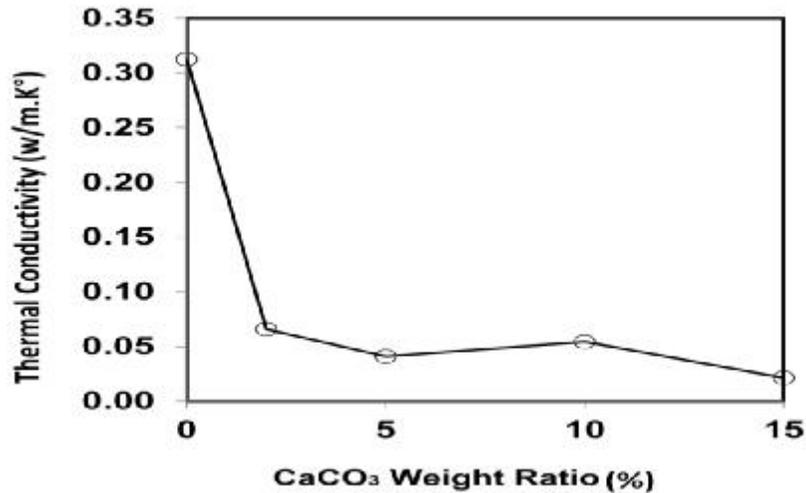


Figure (9) Coefficient of thermal conductivity of pure and CaCO₃-reinforced epoxy polymer.

CONCLUSIONS

Reinforcement of quick-mast 105 epoxy-based adhesivewith CaCO₃ powder has been achieved successfully. The enhanced mechanical, wear and thermal properties were demonstrated in terms of the flexural modulus, hardness, impact resistance, wear rate and thermal conductivity of the end-product. Results showed that addition of low quantities of CaCO₃ powder to the epoxy resin produces a harder, less ductile and better heat insulator product. Wear rate was found to increase with the increase of CaCO₃ ratio. Nevertheless, wear characteristics showed that addition of the CaCO₃ powder produced an epoxy-based composite suitable for high-speed applications. These findings are believed to pave the way for the utilization of the quick mast 105 in new industrial and technological applications.

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