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Assessment of mechanical and wear properties of epoxy-based hybrid composites

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ABSTRACT

Discarded florescent tubes and graphite rods obtained from dumped primary cells have been processed to obtain glass and graphite particles. 80 μm glass and graphite particles were used as reinforcements in epoxy resin, LY 556 cured with HY 931 hardener to produce epoxy resin hybrid composites. The morphology, mechanical properties, thermal stability and wear resistance characteristics of the epoxy resin glass/graphite hybrid composites were studied. The thermogravimetric analyser TGA 701 was used to examine the thermal stability of the epoxy resin glass particle/graphite composites. Addition of graphite and glass particles enhanced the strength, thermal stability and wear resistance of the epoxy resin. However, tensile strain and impact energy absorption of the epoxy resin hybrid composites started declining at 6 wt% of glass particle addition. The increase in wear rate of the composites with an increment in applied loads is attributable to increase in the normal reaction between the examined sample surfaces and the emery paper. Furthermore, the increase in wear resistance with an increment in wt% of glass particle additions is attributable to good interfacial adhesion between matrix and the fillers. The textural and appearance differences between the scanning electron micrographs of the control and epoxy resin hybrid composites is attributable to the presence of new phases due to exothermic and cross linking reaction between the matrix and the fillers. Hence, new vital engineering composites peculiar to automobile, aerospace and building industries have been produced.

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1. Introduction

Epoxy resins belong to a class of polymer under the aegis of thermoset [1]. They possess excellent mechanical strength, electrical and chemical resistance; good thermal characteristic and fine adhesion to many substrates after cure. They are used as matrix resins for reinforced composites, in aerospace industry, adhesives in car, as insulating materials for electrical and electronic industry. However, when compared to other light materials like aluminium, they have low mechanical strength and thermal resistance. Hence, filling of epoxy resin for improved mechanical, wear and thermal resistance properties is imperative. The mechanical performance of the fiber-reinforced composites usually depends on the properties of the matrix and fiber materials [2-4]. Reinforcement such as glass has good thermal resistance, wear resistance and high strength but they have low fracture toughness. Also, in the development of wide variety of composites for application in areas such as aerospace industry, automobile industries and sporting goods, carbon fiber has been used as the reinforcing material [5-6]. Carbon fibre reinforced polymer com-

posites are being used in a wide range of engineering applications because of increase in the impact energy absorption per unit weight, reduce noise and vibrations and excellent resistance to fatigue [7]. Hybridization of particles as a filler for a polymer gives rise to a new polymer based composite with enhanced mechanical properties [8]. Hence, the composites of these three engineering materials, i.e. polymer–matrix composites with small amount and size of glass and carbon particle reinforcements, could improve strength and impact energy of the composites. This is the thrust of this research i.e. to use the good properties offered by both glass and carbon particles to reinforce epoxy resin to develop composites with improved properties. Such composites made from high-performance particles (e.g., carbon and glass particles) embedded in compliant polymeric resins can be used in a wide range of fields such as aerospace engineering and sports utilities. The developed composites are expected to have high specific strength and toughness, superior manufacturability, as well as excellent corrosion resistance and fatigue tolerance.

Agunsoye et al. (2014) worked on the development and characterization of aluminium dross/epoxy resin composite materials [9]. Their results revealed that additions of particulate aluminium dross to epoxy resin enhanced significantly, the thermal and wear resistance of the epoxy resin aluminium dross composites. Hassan et al. (2012) worked on development of polyester/eggshell particulate composites [10]. Their results show that the addition of eggshell to the polyester slightly improved the mechanical properties of the produced composites. Allaouis et al. (2002) fabricated and studied mechanical and electrical properties of epoxy resin/carbon nanotube composites [11]. Results of the experimental examination revealed that the Young's modulus and the yield strength of the composites have been doubled and quadrupled for composites with respectively 1 wt% and 4 wt% nanotubes, compared to the pure resin matrix samples. Conductivity measurements on the composite samples showed that the insulator-to-conductor transition took place for nanotube concentration between 0.5 wt% and 1 wt%. Valášek et al. 2015 studied two-body abrasive wear of polymeric composites using waste abrasive Al_2O_3 particles as a filler [12]. Their results indicated 16 % improvement in hardness with composite made from 284 μm sized Al_2O_3 particles.

Many worked have been focused on enhancement of thermoset properties, the use of florescent tube glass particles and graphite particles obtained from discarded primary cell as reinforcements in epoxy is rare or not found. However, in this work, thermoset hybrid composites have been produced from epoxy resin (LY 556) reinforced with 80 μm glass and graphite particles. The mechanical properties, wear behaviour, phase morphology and distribution in the epoxy matrix of the produced composites were investigated. The glass particles used were obtained from processed discarded fluorescent tubes which have the potential of causing serious environmental harm due to mercury. The mercury from just one fluorescent tube is enough to pollute 30,000 l of underground water so that the water is no longer safe to drink [13]. Hence, this research was also aimed at eliminating this challenge by making use of this potential harmful material (discarded fluorescent tube) as reinforcement in the production of composites for engineering applications.

2. Materials and methodology

Materials used in this work are discarded florescent tube procured from Waste Dumping Centre of University of Lagos; graphite rods were obtained from discarded primary cells. Epoxy resin (LY 556), HY931 hardener and distilled water were purchased from Tony Nigeria Chemicals, Ojota Lagos. The major equipment used includes scanning electron microscope (SEM), Instron extensometer, model 3319; hot press, X-ray diffractometer and Avery Denison Universal Impact Testing Machine.

Broken glasses obtained from florescent tubes were submerged in a 100 l of boiling water contained in a plastic drum covered with 0.5 kg ground sulphur powder. This attempt was made to remove mercury from the florescent tube. The resulted suspension containing black particles was decanted off the drum after 24 h, leaving behind the broken glasses. The broken glasses were sundried at average daily temperature of 25 °C for 2 days. They were ground into powder using 87002 LIMOGES planetary ball mill, model 28A20 92 in accordance with [14]. Glass pow-

ders were classified using a set of sieves arranged in descending order of mesh sizes ranging from 50-300μm. Sieves were vibrated for 30 min using a sine shaker. Solid graphite electrodes were ground and sieved. Fig. 1 and Fig. 2 present glass and graphite particles.

Epoxy resin (LY 556) and hardener (HY 931) were mixed inside a beaker at 3:1 in accordance with manufacturer specification. The mixture was stirred by a hot plate and stirrer, model Jenway 1000 at 120 °C for 1 h and then poured into a metallic mould coated with petroleum jelly. Epoxy resin/hardener blends were left in the mould for 2 days at room temperature to obtain epoxy/hardener preforms.

Epoxy preforms were placed in a mould with different cavities patterned to the standard shapes for mechanical property analyses. The preforms were heated to 150 °C, held at the temperature for 1 h and then forged to obtain the standard samples for analyses (in accordance with ASTM D 3039 M959 and ASTM D 790-90), using a hot press.

For composite production, a mixture containing 80 μm sized graphite and glass particles were dispersed in 50 cm^3 of water in a beaker and then stirred manually with a glass rod for a period of 10 min. The mixture was added to 250 cm^3 epoxy resin (LY 556) in another beaker and then stirred for 15 min. 30 % HY931 hardener was added to the epoxy resin/filler mixture. The resulting mixture was stirred using magnetic mantle shaker for 1 h at a temperature of 120 °C to evaporate water molecules. The epoxy resin mixture was poured into a mild steel mould (coated with petroleum jelly as a dispatching/releasing agent) and then left in the mould for 2 days at room temperature. Epoxy graphite/glass particles hybrid composite preforms were placed in a forming mould. They were heated to 150 °C, held at the temperature for 1 h and then forged to obtain standard samples for mechanical property investigation. Six different composite samples produced contained 2 wt% graphite particles with increasing wt% of glass particles from 2-12 % at 2 % interval.

Phase identification of the produced epoxy samples were carried out using a Panalytical Empyrean X- ray diffractometer (XRD) in accordance with [15]. Morphology of the samples was examined with the aids of scanning electron microscope, ASPEX 3020 in line with [16].

Tensile samples of 80 mm gauge length and 7 mm width were subjected to tensile test using Instron extensometer, model 3369. Samples were gradually loaded at a strain rate of 10^{-3} 1/s [17]. This resulted in the simultaneous elongation and reduction in the cross section of the sample until the samples became fractured. The flexural test was carried out on the bended 100 mm square samples. The samples were loaded at the bended centre point until fracture occurred at the point. 150 mm diameter samples were subjected to compressive load using Instron extensometer.

The impact energies of the produced samples were determined with the aids of Avery Denison Universal Impact Testing Machine. The notched 60 × 10 × 10 mm^3 dimensioned samples were subjected to the Charpy impact test. First, the hammer pendulum was released to set the scale to zero point. Each sample was impacted with hammer pendulum of weight 300 J, released from the upper position of the machine. The impact energy absorbed by each sample was read and then recorded.

Fig. 1 Photograph of glass particles

Fig. 2 Photograph of graphite particles

The wear resistance of 60 × 10 × 10 mm^3 dimensioned samples of the control, epoxy resin hybrid composites containing 2, 4 and 12 % glass particles were investigated using Pin on Disc wear machine. Densities and the initial masses of the examined samples are expressed in two Cartesian system as follows (1.33 g/cm^3, 8 g); (1.28 g/cm^3, 7.7 g); (1.27 g/cm^3, 7.64 g) and (1.24 g/cm^3, 7.43 g) respectively.

The wear test was carried out on a 200 mm circular rotating disc with attached emery paper of 220 grit size according to [2]. The surface of the test sample was placed against the rotating disc for a period of 60 s under different loads at a speed of 2.36 m/s. The final mass after each test was measured and recorded. The mass loss during the investigation was calculated and recorded using the Eq. 1.

$$Mass\ loss\ =\ initial\ mass - final\ mass \tag{1}$$

The thermal stability of the control and the epoxy resin hybrid composites containing 2 and 12 % glass particles were examined using TGA 701. Each sample was heated from room temperature to a maximum of 900 °C for 200 min. The thermal stability was studied as a function of weight loss with heating temperature and time.

3. Results and discussion

3.1 X-ray diffractograms

X-ray diffractogram in Fig. 3 reveals the phases present in the epoxy resin (control) sample. It was observed from the result obtained that aluminium manganese titanium ($Al_3Ti_{0.78}Mn_{0.25}$), burnt lime (CaO), pyrolusite (P_2O_5), sodium aluminium sulphide (Na_4AlS_5) were identified at diffraction angles of 44.23°, 46.24°, 27.81° and 14.02° respectively.

Fig. 4 shows the X-Ray diffractogram of the epoxy resin hybrid composite containing 12 % glass particles. There are two peaks with many shoulders at their sides. This indicates higher degree of phase segregation and or straining within the matrix. They are attributable to chemical interaction among epoxy resin, glass and graphite particles. It was observed from the result that the phases were Iron titanium (FeTi), anatase (TiO_2), Titanium Zinc ($Zn_{0.6}Ti_{0.4}$). These phases were identified at diffraction angles of 50.01°, 28.23° and 85.23° respectively.

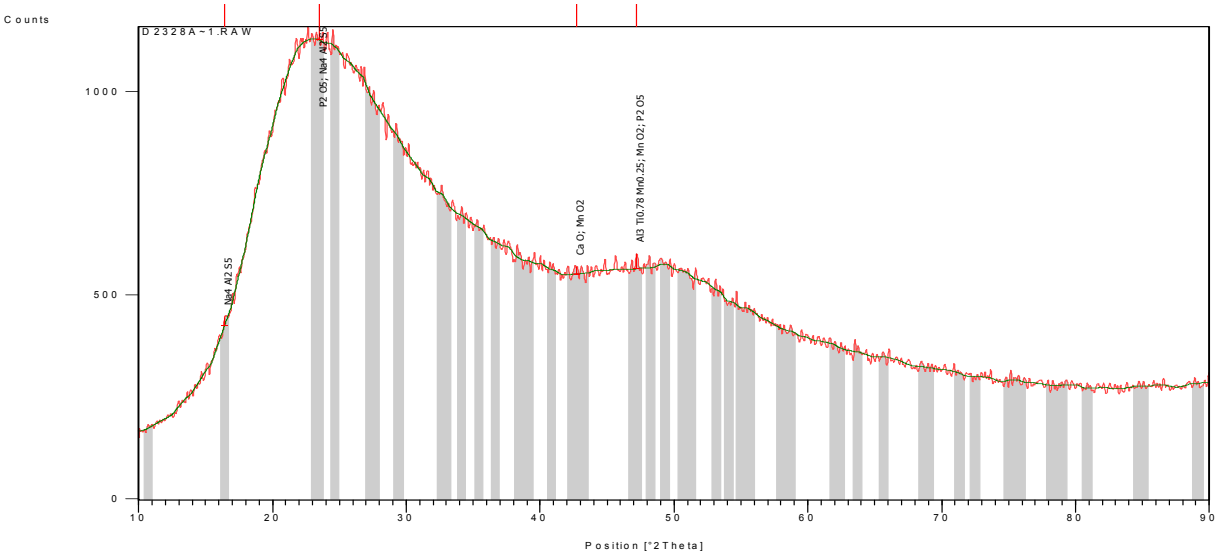

Fig. 3 X-ray diffractogram of the unfilled epoxy resin [2]

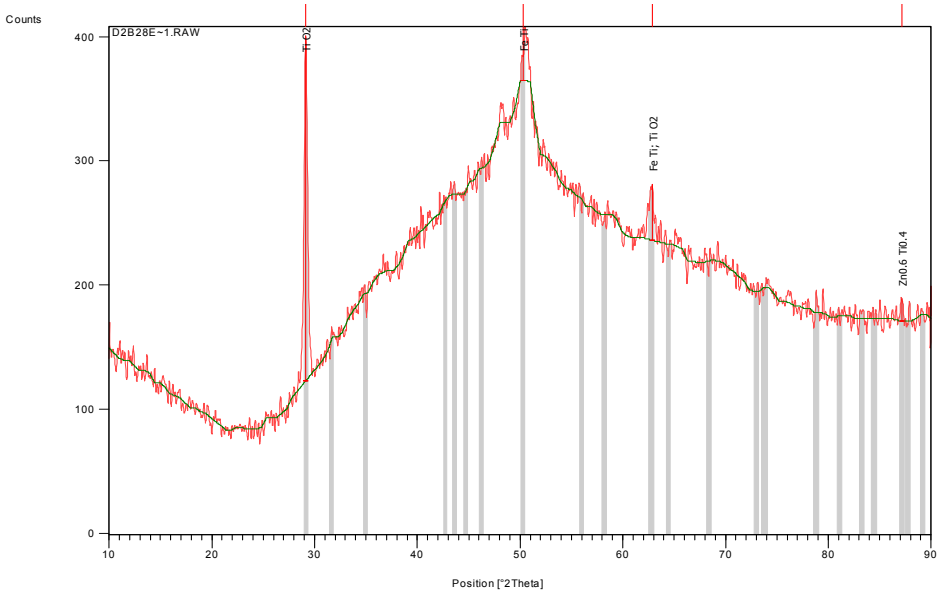

Fig. 4 X-ray diffractogram of the epoxy resin hybrid composite of 2 % graphite and 12 % glass particle additions

3.2 SEM micrographs

Microstructure of epoxy resin control sample is presented in Fig. 5. The structure reveals the white phases within the black matrix. The white phases are attributable to $Al_3Ti_{0.78}Mn_{0.25}$, CaO and Na_4AlS_5 as indicated by the diffractogram in Fig. 3. Fig. 6 presents the microstructure of the filled epoxy resin. The facial difference of the microstructure in Fig. 7 from that in Fig. 6 is attributable to glass and graphite particle additions. The microstructure appears rocky which indicates a good interfacial adhesion between the matrix and reinforcements.

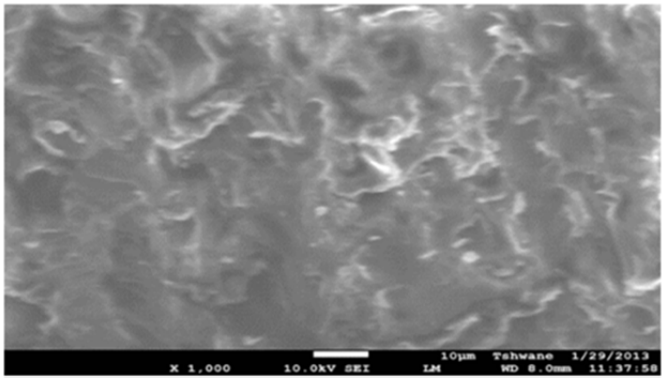

Fig. 5 Unfilled epoxy resin SEM micragh

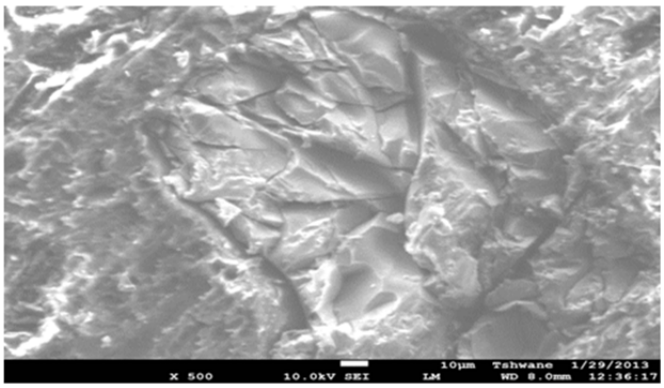

Fig. 6 Epoxy resin hybrid composite micrograph obtained from SEM

3.3 Mechanical properties

It was observed from Fig. 7, Fig. 8, and Fig. 9 that filling of epoxy resin with graphite and glass particles produced epoxy resin hybrid composites with enhanced tensile, flexural and compressive strengths. Strength of the epoxy hybrid composites increased with an increment in wt% of glass particle additions up to 10 wt% when the ultimate tensile and flexural strength start declining. This indicated epoxy resin saturation level. However, the increase in compressive strength beyond 10 wt% of glass particles may be attributable to brittleness of the phases within the matrix which enhance the strength in compression. Fig. 10 and Fig. 11 depicted that impact energy and tensile strain started declining at 6 wt% glass particle addition. This indicated a critical glass filling level such that glass particle addition above this level embrittled the epoxy hybrid composites. The decline in impact energy and tensile strain could be associated with brittleness of glass and graphite particles. During loading, the hybridized particles allowed the crack propagation in a more rapid manner than that in the case of epoxy hybrid composites at lower wt% of glass particle additions. This caused the 2 % graphite-6 % glass particle epoxy composites to fail at lower absorbed energy and percentage elongation.

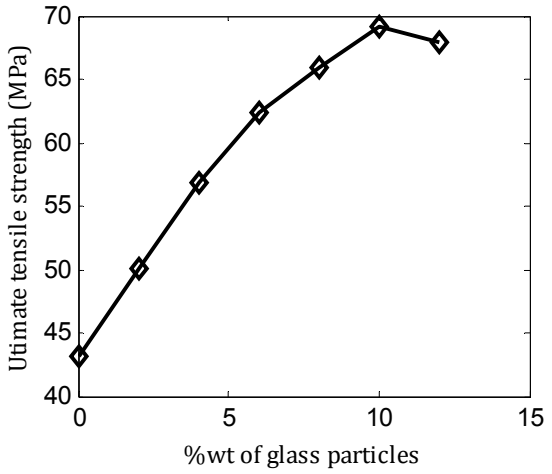

Fig. 7 Ultimate tensile with wt% of glass particles

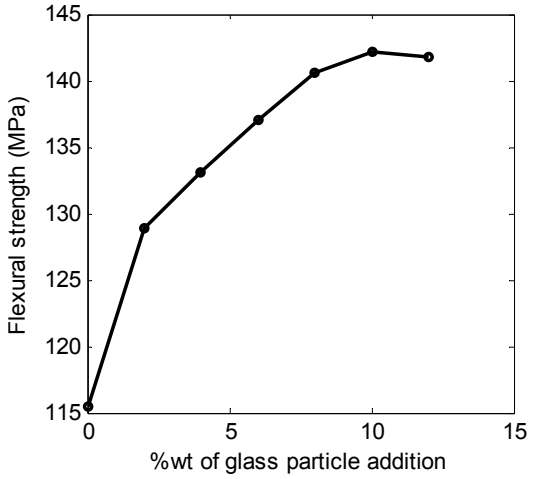

Fig. 8 Flexural strength with wt% of glass particles

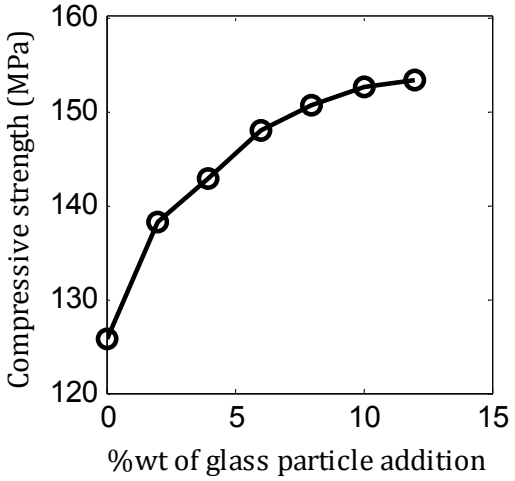

Fig. 9 Compressive strength with wt% of glass particle

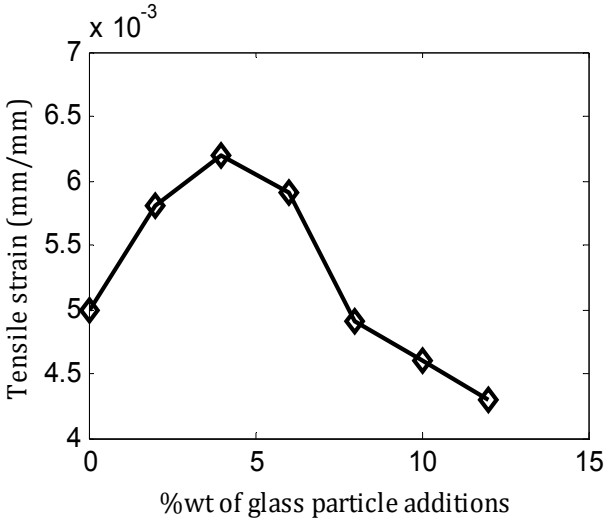

Fig. 10 Tensile strain with wt% of glass particles

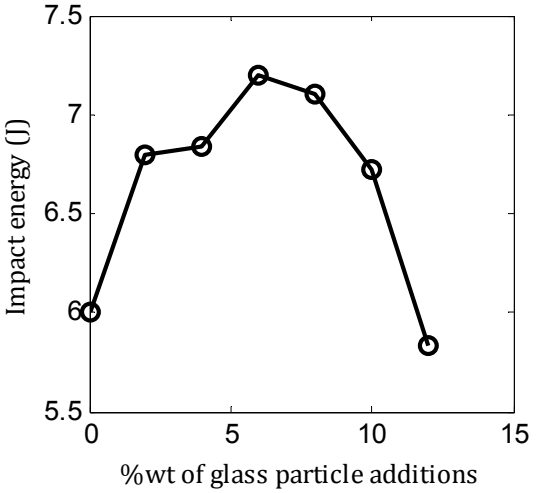

Fig. 11 Impact energy with wt% of glass particles

3.4 Wear resistance properties

It was observed from Fig. 12 that the specific wear rate increased with an increment in applied loads. This may be attributable to increased normal reaction which enhanced the friction between the epoxy samples and the emery paper. However, the decrease in specfic wear rate with an increment in glass particle addition is attributable to good interfacial bonding between matrix and the fillers; tough and rigid surfaces of the epoxy resin graphite-glass particles hybrid composites.

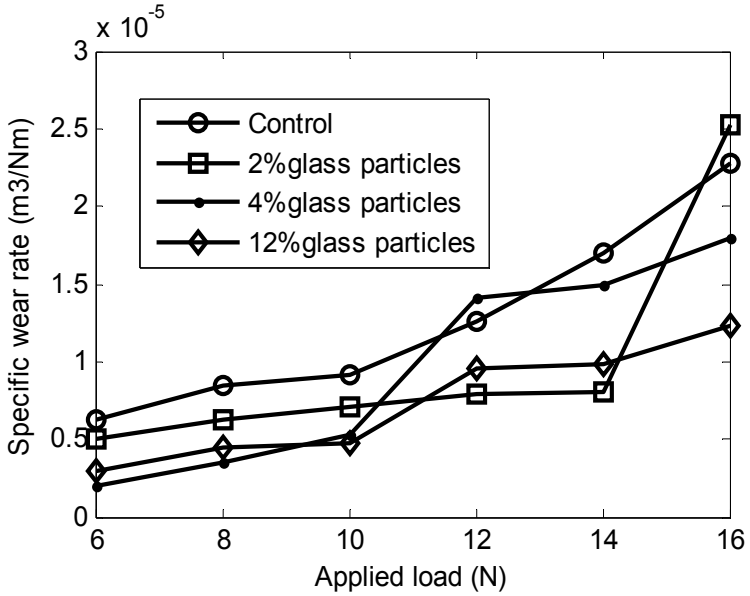

Fig. 12 Specific wear with applied load

3.5 Thermal resistance property

Fig. 13-15 show the variation of weight loss (%/min) as the heating time and temperature increased in a graph known as thermogravimetrogram. Fig. 13 shows the thermogravimetrogram of the epoxy resin (control) in which the weight loss at 900 °C was around 8 %/min, indicated by the highest peak of the curve. The various shoulders around the peak are functions of resistance of the epoxy resin to the thermal decomposition. The intersection of the base line and the curve is a measure of phase transition during heating regime. The maximum temperature at which intersection occurred is the glass transition temperature (Tg) of the examined sample. In Fig. 14, Tg of the control epoxy resin was around 430 °C. Fig. 14 and Fig. 15 show the thermogravimetograms of the epoxy resin hybrid composites of 2 % graphite containing 2 % and 12 % glass

particles respectively. In Fig. 14, the weight loss at 900 °C was around 3 %/min and Tg of the hybrid composite was around 460 °C while in Fig. 18, the weight loss was around 3.5 %/min and Tg is 600 °C. This is an indication of enhancement in thermal resistance of the epoxy resin hybrid composite. This is attributable to inherent refractoriness of the graphite and glass particles.

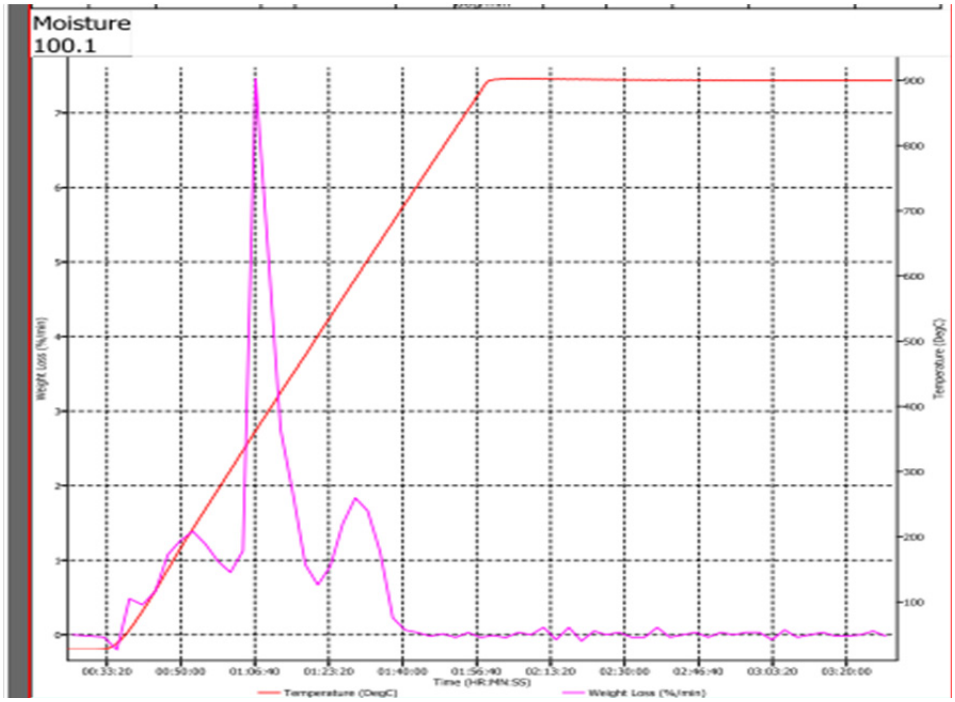

Fig. 13 Thermogravimetrogram of the epoxy resin

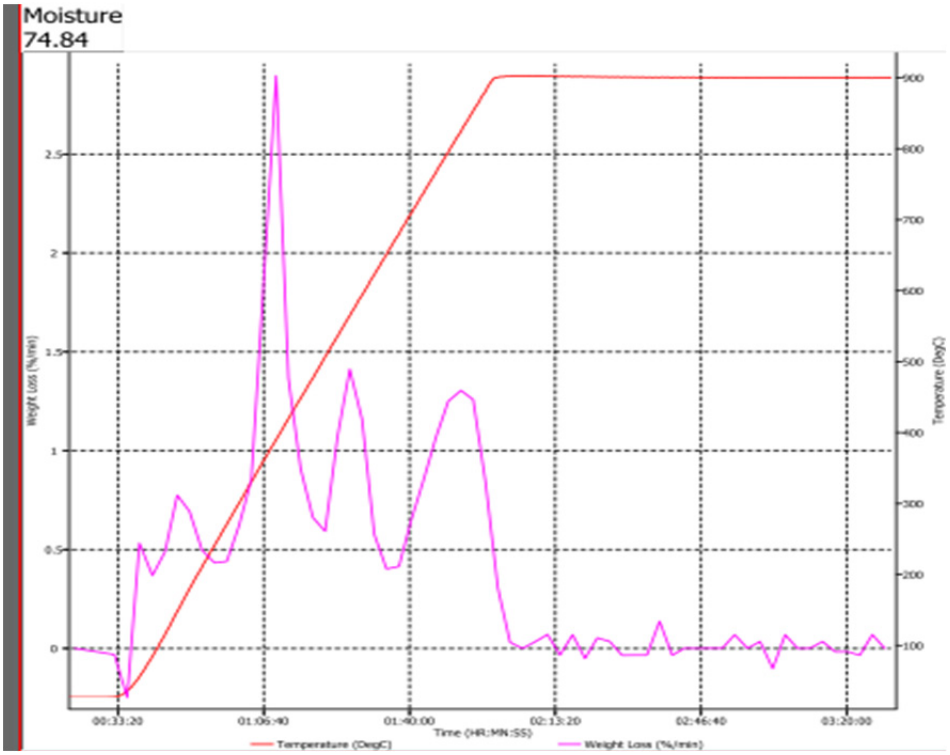

Fig. 14 Thermogravimetrogram of the epoxy resin hybrid composites of 2 % graphite and 2 % glass particles

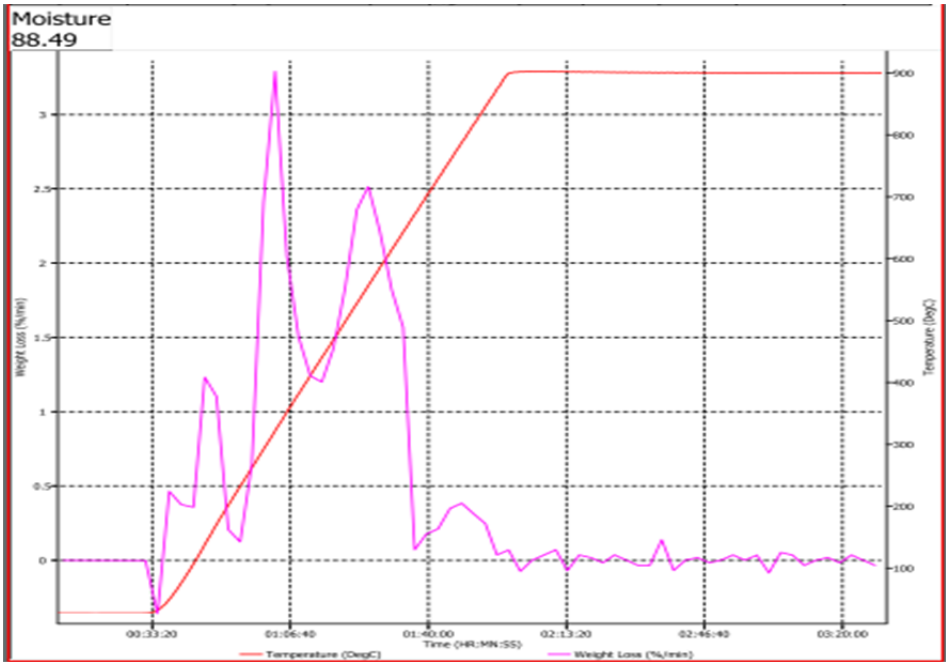

Fig. 15 Thermogravimetrogram of the epoxy resin hybrid composites of 2 % graphite and 12 % glass particles

4. Conclusion

Based on the results of experimental investigations in this work, the following conclusions can be made:

- Recycled materials obtained from graphite rods of discarded primary cells are essentially strong and tough reinforcements for thermoset polymeric materials.
- Recycled glass particles from florescent tube can be harnessed into materials development for various engineering applications
- Additions of graphite and glass particles to epoxy resin led to epoxy hybrid composites with enhanced mechanical, wear and thermal resistant properties of the epoxy resin.
- Compressive strength of the epoxy hybrid composites increased with an increment in wt% of glass particle additions.
- There are formation of new phases in consequent to exothermic reaction taking place during mixing and curing of the filler and the matrix.
- Enhancement in the properties of the epoxy resin is attributable to presence of glass and graphite particles within the matrix.
- The future work in this area will present the empirical model to characterise effects of hybridized particles on mechanical properties of the developed epoxy composites in order to validate the experimental results.

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References

[1] Bello, S.A., Agunsoye, J.O., Hassan, S.B., Kana, M.G.Z., Raheem, I.A. (2015). Epoxy resin based composites, mechanical and tribological properties: A review, *Tribology in Industry,* Vol. 37, No. 4, 500-524.

[2] Marieta, C., Schulz, E., Irusta, L, Gabilondo, N., Tercjak, A., Mondragon, I. (2005). Evaluation of fiber surface treatment and toughening of thermoset matrix on the interfacial behaviour of carbon-reinforced cyanate matrix composites, *Composite Science Technology*, Vol. 65, No. 14, 2189-2197, doi: 10.1016/j.compscitech.2005.05.008.

[3] Marieta, C., Schulz, E., Mondragon, I. (2002). Characterization of interfacial behaviour in carbon-fibre/cyanate composites, *Composites Science and Technology*, Vol. 62, No. 2, 299-309, doi: 10.1016/S0266-3538(01)00215-9.

[4] Wang, Y., Hahn, T.H. (2007). AFM Characterization of the interfacial properties of carbon fiber reinforced polymer composites subjected to hydrothermal treatments, *Composites Science and Technology*, Vol. 67, No. 1, 92-101, doi: 10.1016/j.compscitech.2006.03.030.

[5] Donnet, J.B., Bansal, R.C. (1990). *Carbon Fibers*, (2nd edition), Mercel Dekker, New York.

[6] Moutier, J., Fois, M., Picard, C. (2009). Characterization of carbon/epoxy materials for structural repair of carbon/BMI structures, *Composites: Part B*, Vol. 40, No. 1, 1-6, doi: 10.1016/j.compositesb.2008.09.003.

[7] Feraboli, P., Masini, A. (2004). Development of carbon/epoxy structural components for a high performance vehicle, *Composites: Part B*, Vol. 35, No. 4, 323-330, doi: 10.1016/j.compositesb.2003.11.010.

[8] Davoodi, M.M., Sapuan, S.M., Ahmad, D., Aidy Ali, Khalina, A., Jonoobi, M. (2010). Mechanical properties of hybrid kenaf/glass reinforced epoxy composite for passenger car bumper beam, *Materials & Design*, Vol. 31, No. 10, 4927-4932, doi: 10.1016/j.matdes.2010.05.021.

[9] Agunsoye, J.O., Talabi, S.I., Hassan, S.B., Awe, I.O., Bello, S.A., Aziakpono, E. (2014). The development and characterisation of aluminium dross-epoxy resin composite materials, *Journal of Materials Science Research*, Vol. 3, No. 2, 23-37, doi: 10.5539/jmsr.v3n2p23.

[10] Hassan, S.B., Aigbodion, V.S., Patrick, S.N. (2012). Development of polyester/eggshell particulate composites, *Tribology in Industry*, Vol. 34, No. 4, 217-225.

[11] Allaoui, A., Bai, S., Cheng, H.M., Bai, J.B. (2002). Mechanical and electrical properties of a MWNT/epoxy composite, *Composites Science and Technology*, Vol. 62, No. 15, 1993-1998, doi: 10.1016/S0266-3538(02)00129-X.

[12] Valášek, P., Müller, M., Hloch, S. (2015). Recycling of corundum particles – two-body abrasive wear of polymeric composites based on waste, *Tehnički vjesnik – Technical Gazette*, Vol 22, No. 3, 2015, 567-572, doi: 10.17559/TV-20131111140048.

[13] Parkinson, M., Visco, C. (2002). *Sustainable markets for waste glass from fluorescent tubes and lamps*, Final Report, The National Centre for Business and Sustainability (NCBS), Manchester, UK.

[14] Hassan, S.B., Agunsoye, J.O., Bello, S.A. (2015). Ball milling synthesis of Al (1050) particles: Morphological study and particle size determination, *Industrial Engineering Letters*, Vol. 5, No. 11, 22-27.

[15] Bello, S.A., Agunsoye, J.O., Hassan, S.B. (2015). Synthesis of coconut shell nanoparticles via a top down approach: Assessment of milling duration on the particle sizes and morphologies of coconut shell nanoparticles, *Materials Letters*, Vol. 159, 514-519, doi: 10.1016/j.matlet.2015.07.063.

[16] Bello, S.A., Hassan, S.B., Agunsoye, J.O., Kana, M.G.Z., Raheem, I.A. (2015). Synthesis of uncarbonised coconut shell nanoparticles: Characterisation and particle size determination, *Tribology in Industry*, Vol. 37, No. 2, 257-263.

[17] Bello, S.A., Raheem, I.A., Raji, N.K. (2015). Study of tensile properties, fractography and morphology of aluminium (1xxx)/coconut shell micro particle composites, (In Press), *Journal of King Saud University – Engineering Sciences*, doi: 10.1016/j.jksues.2015.10.001.