EFFECT OF ALUMINIUM PARTICLES ON MECHANICAL AND MORPHOLOGICAL PROPERTIES OF EPOXY NANOCOMPOSITES

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Bumper is a front or rear part of automobiles. It is designed and shaped to be impact absorbing and protecting automobiles from damage in low impact collisions. Initially, they were made from heavy steels, increasing the weight of automobiles and fuel consumption. Also, high impacts of steel bumpers on pedestrians during accidental collision cause fatalities and or disabilities. An effort to enhance fuel efficiency, safety, freedom of design and shape detailing, heavy alloys for automobile applications are now being replaced with polymeric composites. Aluminium micro particles and nanoparticles were prepared from aluminium cans through sand casting, lathe machine spinning, and ball milling techniques. Both types of aluminium particles were incorporated into a mixture of diglycidyl ether of bisphenol A (DGEBA, epoxy resin) cured with amine base hardener (ABH). Phases of the epoxy polymer and composites were identified using X-ray Diffraction (XRD). Spatial arrangement of the phases within the matrix and their elemental composition were examined using Scanning Electron Microscope with attached energy dispersive X-ray spectroscopy (SEM/EDX). Tensile, impact and micro hardness tests were conducted on the prepared epoxy/aluminium composites. Results of the XRD showed the presence of aluminium compounds/phases due to chemical reactions between aluminium particles and DGEBA/ABH system. SEM confirmed a homogeneous distribution of the phases within the epoxy matrix, and that there is a strong adhesion between the epoxy matrix and aluminium particles. Correlation between the mechanical properties of the prepared nanocomposite and the procured bumper materials exhibited a fair suitability of the prepared nanocomposites for automobile applications.

KEY WORDS: Aluminium; nanoparticles; correlation, mechanical properties, bumper material

INTRODUCTION

Nanotechnology is an area of research which has led to material restructuring, resulting in the enhancement of material properties. Nanoscience is the study of materials

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where some critical properties are attributable to an internal structure with at least one dimension less than 100 nm (1, 2). Nanoscience primarily deals with the synthesis, characterization, exploration, and exploitation of nanostructured materials (3). Nanostructured materials are low-dimensional materials comprising building units of a submicron or nanoscale size at least in one direction and exhibiting size effects. They are the products obtained owing to the research into nanotechnology. One of the nanostructured materials is the dispersion strengthening composite or nanocomposites. Nanocomposites are obtained through incorporation of nanoparticles in a matrix. Many studies have been focused on the development of particles reinforced composites to replace fibre reinforced counterparts because of the difficulties in their manufacturing and their anisotropic properties. However, some limitations in the properties enhancement have been found in the literature due to weak interfacial adhesion leading to poor mechanical and wear resistance properties of microparticle reinforced composites (4, 5). The development of dispersion strengthening composites/nanocomposites has been proposed to address the identified challenges noticed with microparticle reinforced composites (6). Since a nanoparticle is much finer than a micro particle, ultrahigh fineness known with nanoparticle could enhance its interaction with a matrix molecule leading to fortified adhesion, which is a basis for improvement in the mechanical properties of engineering composites.

An effort to enhance fuel efficiency, safety, freedom of design, and shape detailing, heavy alloy for automobile applications are now being replaced with polymeric composites (7-10). Due to high cost of glass, carbon or aramid fibre reinforced plastics, the evolution of automobile materials is limited to a high extent, especially in exotic cars (2). To expand the use of polymeric composites for automobile applications and their sustainability, adoption of cost effective recycling strategy is very important (11). Many reports have been found dealing with the development of ecofriendly reinforced plastics using particles obtained from agro and metallic wastes (12-19). However, reports on processing of aluminium cans to obtain aluminium nanoparticles (using ball milling technique) as reinforcement for development of epoxy/aluminium nanocomposites for automobile bumper application are very scarce if found at all. This work is aimed at studying the effect of aluminium particles on morphological and mechanical properties of epoxy/aluminium particulate nanocomposites. This attempt is very important since the potential benefits are related to not only environmental cleanliness, elimination of green gas emission and reduction in power consumption associated with the primary route for aluminium production, but also to the development of light weight materials for automobile applications.

MATERIALS AND METHODS

Epoxy resin, diglycidyl ether of bisphenol A (DGEBA) and amine based hardener (ABH) were obtained from Polymer Composite Institute (PCI), Ontario Canada through a local vendor in Lagos. Aluminium cans were obtained from the University of Lagos Management Centre, Lagos Nigeria. Aluminium cans were melted at 660±5 °C and cast

into bar using green sand mould. The bar was spun into particle/fibres using Colchester/triumph lathe machine, model 2000 at the Physics Department, University of Lagos. During spinning, jets of water were intermittently applied on the aluminium surface to cool and harden the surface. This aided the aluminium breakage. Aluminium particles/fibres were further broken using steel mortar and pestle. Prior to ball milling of aluminium, milling balls/carbonised coconut shell was charged into the 87002 LIMOGES planetary mill (model: 28A20-92), in the Federal Industrial Institute of Research Oshodi (FIIRO) Lagos, and the machine was operated for 30 minutes to coat the ball surfaces with carbonised coconut shells used as solid lubricants. Then aluminium particles were added and the ball milling was carried out for 40 hours at 8.5 charge ratios (CRs) and 192 rpm. An amount of carbonised coconut shell equivalent to 0.1 % of aluminium particles was used. Aluminium powders obtained at 40 hours were classified using a set of sieves in a descending order of grain fineness (2000-75 μm), vibrated mechanically for 30 minutes with the aid of a sine shaker in accordance with BS 1377, standard 1990 (14). The finest aluminium powders collected in a pan below 75 μm sieve were used as a precursor for the synthesis of Alnp. The precursor was further milled for 70 hours at 8.5 CRs according to the known procedure (20). The obtained particles were characterized for size determination using XRD and Transmission Electron Microscope (TEM).

Al micro particles (Almp) of the size of 56 μm and Al nano particles (Alnp) of the size 55.5 nm were incorporated into a DGEBA/ABH system in a 2:1 volume ratio, using mechanical shear stirring technique. The mixture was poured into a steel die open mould and allowed to set and harden at room temperature for 48 hours. Initially, 2 wt% of Almp was added to the DGEBA/ABH. The E/Almp composite samples were gently removed from the mould and then postcured at 130 °C for four hours using Shel lab vacuum oven, model SVA S2E, at the Department of Materials Science and Engineering, Kwara State University, Malete, Nigeria. The process was repeated with an increment in wt% of Almp additions, up to 10 % at an interval of 4 % in each case in line with (6). The same techniques and wt% of reinforcement additions were used to produce epoxy/aluminium nanoparticle (E/Alnp). The phases of the developed epoxy/aluminium composites were determined using XRD, spatial configuration and elemental composition were examined using SEM/EDX. Mechanical tests such as tensile, impact and micro hardness were carried out on the prepared composites. Figure 1 presents some of procedural steps taken in the development of E/Alnp nanocomposites.

Figure 1. Procedural steps for the development of epoxy aluminium nanocomposites

RESULTS AND DISCUSSION

Structure of the prepared aluminium nanoparticles

The X-ray diffractometry (XRD) profile of Alnp is shown in Figure 2. The three observed major peaks reveal Al at the diffraction angles (2θ) of 45°, 52.5° and 77.33°, while the minor peaks show Al_2O_3 at 29.48°, 41.03°, 50.08°, 61.85°and 67.95° and SiO_2 at 31.21°. The presence of SiO_2 can be linked to the carbonised coconut shell used as solid lubricant to prevent sticking of aluminium particles to the milling balls. Moreover, presence of Al_2O_3 suggests the oxidation of aluminium particle by cooling agent (water) during machine spinning and or milling environment during particle refinement process. The TEM image in Figure 3 displays relatively coarse Alnp surrounded by many fine Alnp. Average sizes of Alnp determined by XRD aided with Scherrer's equation and TEM supported with software are 55.5 and 70.4 nm, respectively.
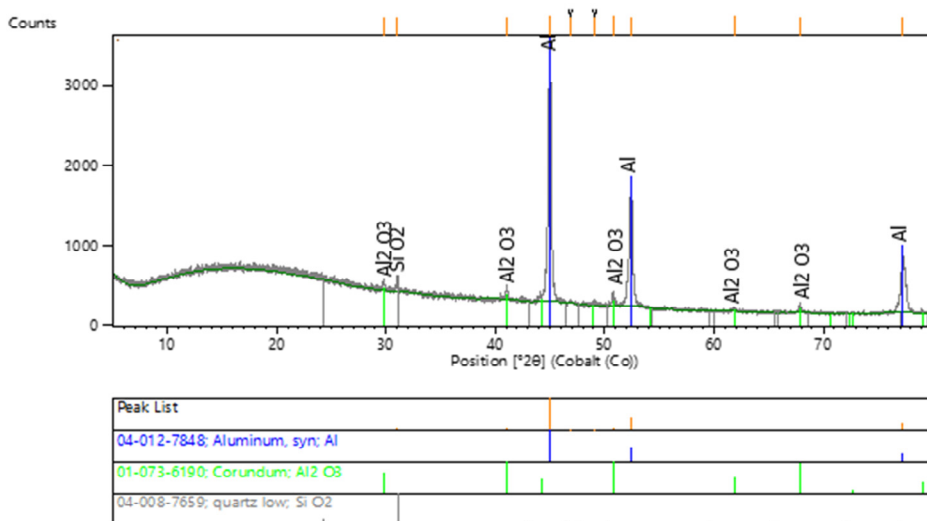

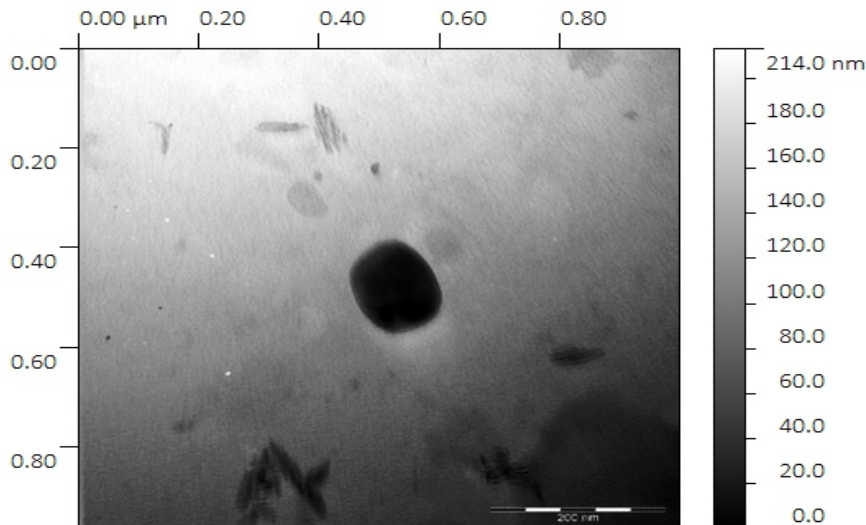**Figure 2.** XRD profile of aluminium nanoparticles

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Figure 3. TEM images showing size distribution of aluminium nanoparticles

Structure of the synthesized composites

Figure 4a-b displays the XRD profiles of epoxy polymer and E/Alnp nanocomposites. The major peaks observed in Figure 4a are due to $C_5H_9NO_2$, $C_{12}H_{12}N_2$, $C_5H_{14}ClN$ at 18.94, 18.94/42.34 and 42.34°, respectively. Their inter-planar spacing is 4.69, 4.49/2.13 and 2.13 Å. These phases are organic compounds, justifying chemical reaction between DGEBA and ABH that led to the formation of epoxy molecules. When Alnp was added to the DGEBA/ABH system, the stoichiometric balance between DGEBA and ABH was disturbed, leading to different cross linking reactions. This resulted in the formation of new phases such as $C_4H_9NO_4$, $C_4H_9NO_3$, $C_5H_4N_4$ and BrH_4N at 36.36, 36.36, 36.36, 36.36/44.54/64.92°, respectively, with the residual Al at 64.92° (see Figure 4b). Their inter-planar spacing varies between 1.2 and 4.7 Å, meaning that the orientation of the phases within E/Alnp nanocomposites has to do with mechanical properties of the nanocomposites (21). The observed phase orientations of E/Alnp nanocomposites are different from those of epoxy polymer having inter-planar spacing from 2.13 to 4.69 Å. The presence of second phase particles within the matrix of E/Alnp nanocomposites has reduced the spacing of the epoxy molecules. Since the enhancement in mechanical properties of dispersion strengthening composites depends on orientation of pha-ses/molecules, the particles wt% and sizes, interfacial adhesion, nature of stress transfer from a matrix to the filler, the differences in inter-planar spacing ranges of epoxy polymer and E/Alnp nanocomposite is an indication that the orientation of the phases present in the matrix of E/Alnp nanocomposites plays a significant role in enhancing

mechanical properties of the E/Alnp nanocomposites. However, the enhancement in the mechanical properties could also be ascribed to other factors mentioned above.
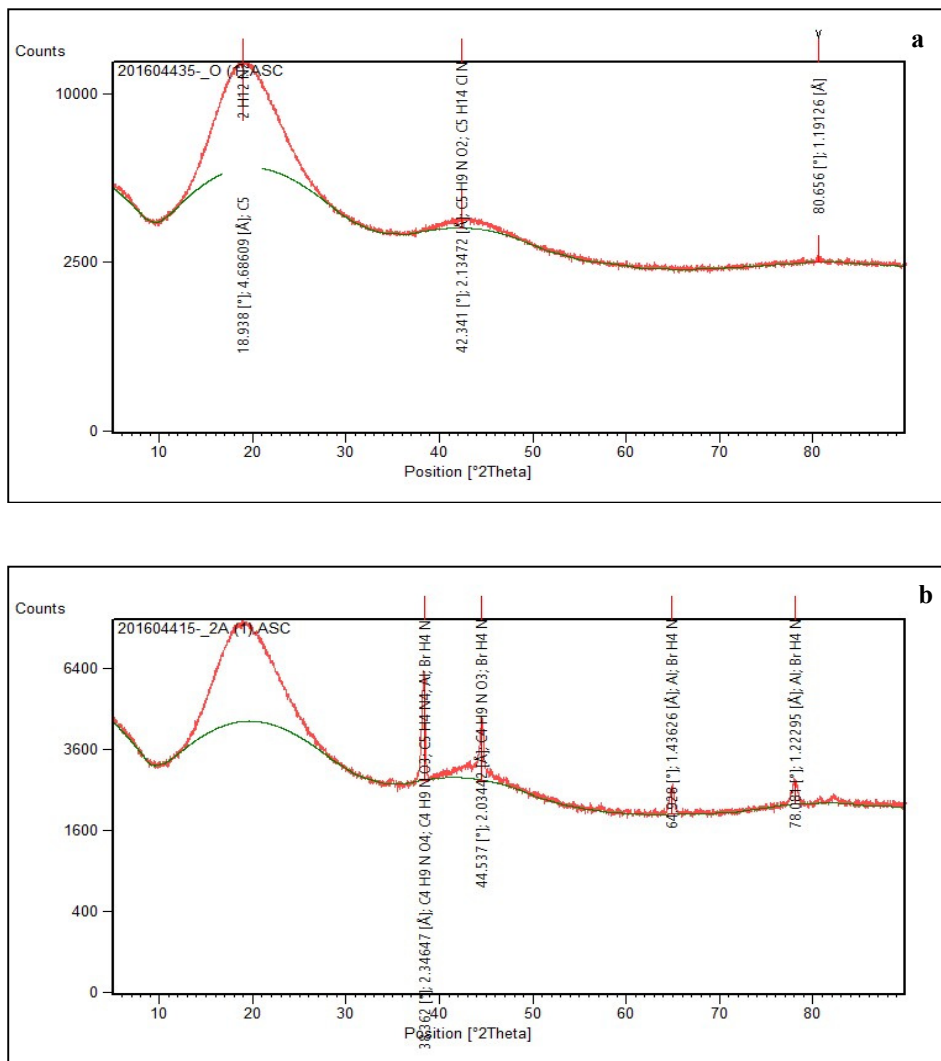

Figure4. XRD profiles of (a) epoxy polymer (b) E/Alnp nanocomposites

Spatial configuration of the epoxy composites

Figure 5a-b depicts the spatial configuration of the epoxy polymer and E/Alnp composites. Figure 5a reveals bonded segments representing infusible cross-linked structure

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of epoxy molecules. Both white and dull epoxy molecules phases are identified. These can be linked to epoxy compounds confirmed by XRD in Figure 2a. The EDX spectrograpms in Figure 5a indicate C (major peak), O and K, which are trace elements. The observed configuration and phases in Figure 5b are different from those in Figure 5a. This difference is linked to the addition of Alnp as a reinforcement to DGEBA/ABH system. The microstructure is homogenous with even distribution of second phase particles within the epoxy matrix. This indicates a good interfacial adhesion between the matrix and the reinforcement. With this structural integrity, a rapid load transfer from the matrix to the phase particles, which enhances the load bearing capacity of the E/Alnp composite, is expected.
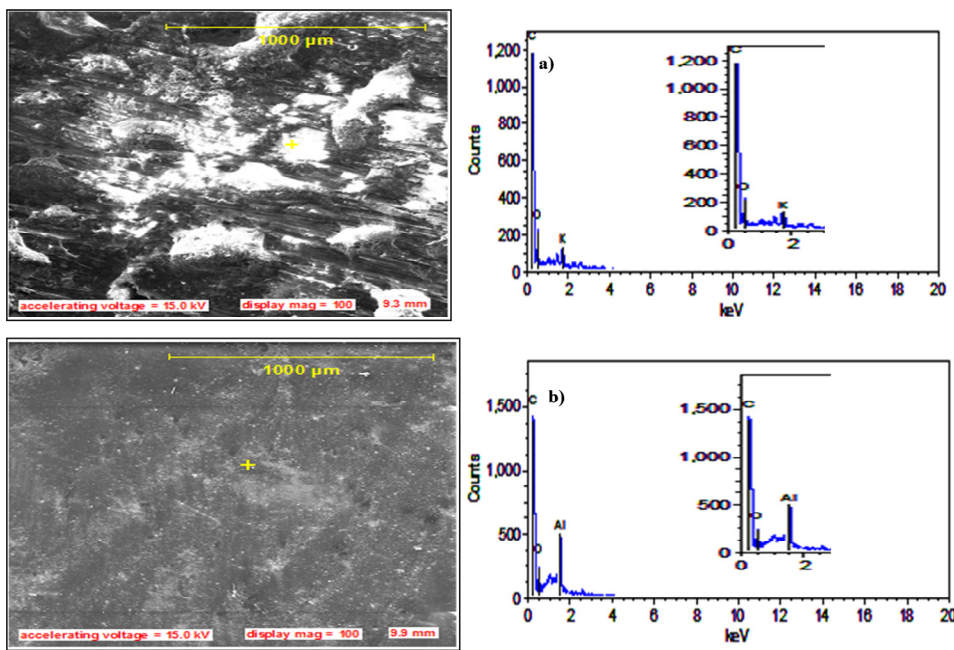

Figure 5. SEM/EDX of (a) epoxy polymer (b) E/Alnp nanocomposite

Mechanical properties of the epoxy nanocomposites

Figure 6 reveals an increase in the tensile strength of the epoxy/aluminium particulate composites with an increase in the wt% of aluminium particles. A progressive increase in tensile strength was noticed with Alnp additions while a decrease in tensile strength was observed above 6 wt% of Almp additions. Generally, the increase in the tensile strength can be attributed to the presence of second phase particles within the epoxy matrix. These phase particles are ceramic in nature and very rigid. Their strong adhesion to the matrix allowed effective and rapid stress/load transfer from the matrix to the particles which

acted as load bearers by their rigidity. Therefore, their presence within the epoxy matrix enhanced the load bearing capacity of the epoxy/aluminium composites. Moreover, the decrease in tensile strength found above 6 wt% of Almp could be ascribed to saturation of the matrix with Almp. This implied that above 6 wt% of Almp addition, the epoxy matrix was unable to join all Almp together. Therefore, there were domains of freely existing Almp within the epoxy matrix, which created region of discontinuity. Under uniaxial loading, the free existing particles interfered with mobility of the composite segment, creating voids through the movement from one point to another. These voids acted as stress raisers and caused the composite deformation at a stress level below the expectation. Similar explanation was found in literature (12, 15-17). A slight decrease in the percentage elongation with increased particle additions was noticed with E/Alnp while E/Almp displayed lower percentage elongation. This shows better mechanical behaviour of E/Alnp than E/Alnp which can be linked to smaller size of Alnp than Alnp which aided its better interaction with epoxy than Alnp.

Figure 7 illustrates a progressive increase in the impact energy with E/Alnp and E/Almp. This signifies an increase in the impact absorbing ability of the synthesized composites with a rise in wt% of particle additions. This increase can be linked to the softness of the epoxy matrix which relieved/damped the loading constraint and the rigidity of the second phase particles, which resisted the composite deformation. Higher impact toughness of E/Alnp than E/Almp can be associated with the fineness of the second phase particles found in the epoxy matrix of E/Alnp nanocomposites. Similar enhancement in the impact toughness of particle reinforced epoxy is found in the literature (22, 23). In addition, Figure 8 displays a small increase in the micro hardness of E/Almp micro composites, while enhancement in micro hardness of E/Alnp nanocomposites is highly noticeable up to 6 wt% of Alnp addition to epoxy while above this level, no significant improvement was noticed. This behaviour is expected because of the progressive increase in impact absorbing capacity of E/Alnp nanocomposites. It implies that up to 6 wt% of Alnp addition, there was an increase in the impact absorbing ability and resistance to indentation of E/Alnp, while above this level of reinforcement, the decrease in the resistance to surface indentation was compensated for an increase in the impact energy of the E/Alnp. The observed increase in micro hardness values is in line with the literature (23).

Correlation of mechanical properties of the prepared epoxy aluminium composites with those of existing automobile materials

The obtained results clearly demonstrated better mechanical behaviour of E/Alnp than E/Almp. Moreover, the tensile strength and impact energy of the developed E/Alnp composites were compared with Toyota (Sienna) and Nissan (Almera) bumper materials bought locally in Nigeria and tested using the same ASTM standards (ASTM D 3039 for tensile test, ASTM D 3763 for impact toughness) used for testing the prepared E/Alnp nanocomposites. The peak tensile strength was observed at 18.58 Nmm^{-2} for the 10 % Alnp addition to the epoxy. This value is greater than the tensile strengths of the Toyota and Nissan bumper materials (12.23 and 6.44 Nmm^{-2}, respectively). The impact energy of

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the Toyota bumper material was 21.18 J, that of Nissan bumper material 25.54 J, while the impact energy of the prepared E/Alnp nanocomposite was 14.01 J. This value is lower than that of either Toyota or Nissan bumper materials. In addition, this value is within the range of 10-18 J specified for Glass Material Thermoplastic for automobile bumper by Azdel (24). The micro hardness value of E/10% Alnp is 12.03 HV which is less than 442 HV peak hardness value specified in United Nation's Economic Commission for Europe (ECE)/324 Regulation No. 42 (25) for automobile bumper materials. This shows a fair suitability of the developed E/Alnp nanocomposites for automobile applications. The current research is focused on the development of epoxy/aluminium coconut shell particulate hybrid nanocomposite for automobile bumper application, which is just completed, and the results will be presented in another publication.
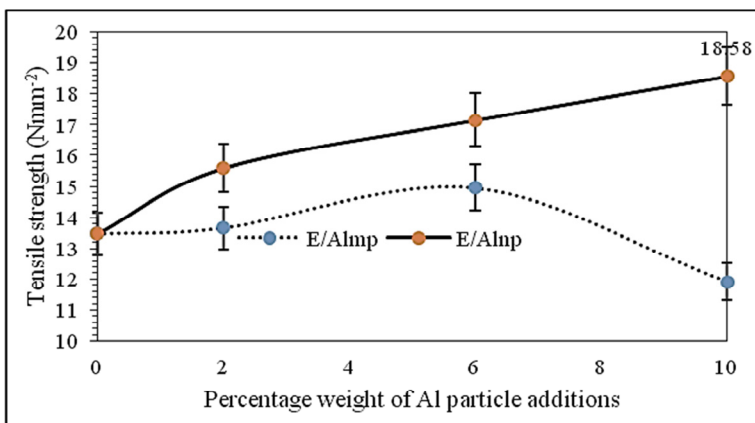

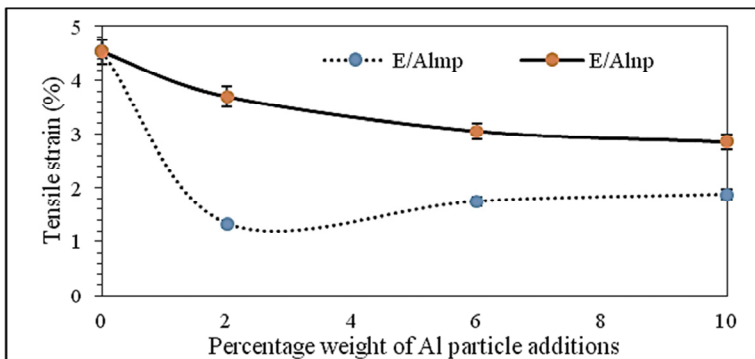**Figure 6.** Tensile strength of the epoxy/aluminium composites

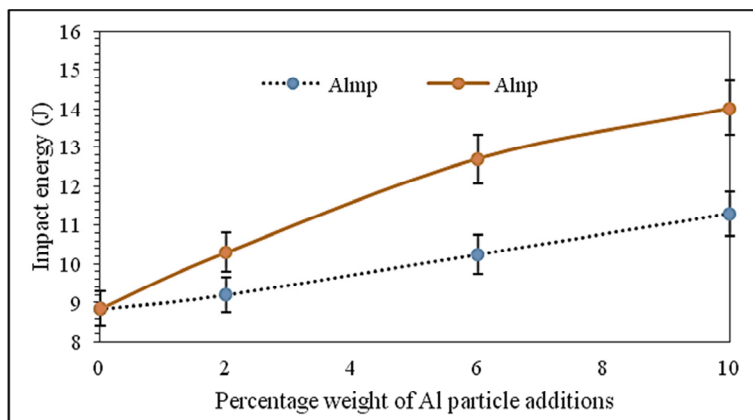**Figure 7.** Tensile strain of the epoxy/aluminium composites

Figure 8. Impact energy of the epoxy/aluminium particulate composites
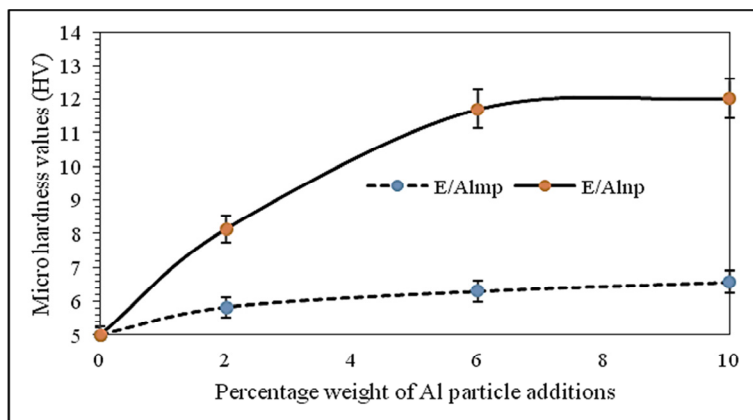

Figure 9. Micro hardness of the epoxy/aluminium composites

CONCLUSION

In conclusion, polymeric nanocomposites intended for automobile bumper application were developed from epoxy resin and disposable aluminium cans. Addition of aluminium particles to epoxy resulted in the improvement of mechanical properties of the synthesized epoxy composites. An increase of 37.84 % in the tensile strength of epoxy aluminium nanocomposites was obtained at 10 wt% of aluminium nanoparticle, while at the same wt% of aluminium microparticles addition to epoxy, a deterioration in tensile strength of epoxy aluminium micro composites was observed. Generally, better mecha-

nical behaviour of synthesized epoxy aluminium nanocomposites than their micro counterparts, is attributed to higher fineness of aluminium nanoparticle than aluminium microparticles which gives room for better interaction of aluminium nanoparticles with epoxy. Moreover, this study has given birth to an ecofriendly polymeric nanocomposite, whose large/industrial scale production is expected to lower cost constraint preventing widespread applications of polymeric composites in engineering structures.

Acknowledgement

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УТИЦАЈ ЧЕСТИЦА АЛУМИНИЈУМА НА МЕХАНИЧКЕ И МОРФОЛОШКЕ КАРАКТЕРИСТИКЕ НАНОКОМПОЗИТА

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Браник је предњи или задњи део аутомобила. Дизајниран је и обликован тако да апсорбује ударе и и штити аутомониле од оштећења у сударима ниског интензитета. У почетку су били израђивани од тешких челика, што је повећавало тежину аутомобила и потрошњу горива. Такође, снажни удари челичних браника у случајним сударима са пешацима резлтују у фаталним исходима и/или повредама. У настојању да се повећа ефикасност горива, безбедност, слобода у дизајнирању и детаљи у обликовању, тешке легуре за аутомобиле се сада замењују полимерним композитима. Алуминијумске микро- и нано-честице су добијене применом техника одливања у песку, обраде на стругу и млевења у млину са куглама. Честице алуминијума оба типа су биле инкорпориране у смесу диглицидил етар бисфенола (DGBA, епокси смола) и базног аминског очвршћивача (АВН). Фазе епокси полимера и композита су биле идентификоване помоћу дифракције X-зрака (XRD). Просторна структура фаза у матриксу и њихов елементарни састав су испитивани скенирајућом електронском микроскопијом у комбинацији са дисперзијом енергије X-зрака (SEM/EDX). Припремљени епокси/алуминијум композити су испитивани на истезање, отпорност на удар и микро чврстоћу. Резултати добивени помоћу XRD су показали присуство једињења/фаза алуминијума насталих реакцијом честица алуминијума са DGEBA/ABH системом. Примена SEM је потврдила хомогену дистрибуцију фаза у матриксу епокси смоле и да постоји јака адхезија између епокси матрикса и честица алуминијума. Корелација између механичких карактеристика припремљених нано-композита и коришћених материјала показала је прихватљиву погодност припремљених нано-композита за примену у аутомобилској индустрији.

Кључне речи: алуминијум, нано-честице, корелација, механичке карактеристике, матријал за бранике