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Suitability of Cow Horn as Filler in an Epoxy Composite

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ABSTRACT: This study focuses on assessment of cow horn as filler in an epoxy composite. A particle-reinforced composite was developed using horn particles (HP) and epoxy resin with filler of varying percentage weight (5%, 10%, 15%, 20%. 25%, 30%, 35%, 40 %) at particle sizes of 100 and 150 µm. The composites were developed by hand lay-up technique with varying process parameters. The properties of the developed composites were examined through tensile, flexural and impact tests. The results showed that the tensile properties of the polymers reduced with the incorporation of the cow horn as filler. But at higher curing temperature, a better strength was achieved. Meanwhile, the flexural and impact properties of the polymers increased with the incorporation of the fiber in no particular order. The composite materials with particle size of 100 µm with curing temperature of 80ºC exhibited higher tensile (37.58 MPa) and impact properties (74 J) than the lower particles. Generally, the cow horn was found to be a good potential filler in the composite if prepared using higher curing temperature as exhibited through its mechanical properties.

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Failure of materials in service and its consequences have been major concerns amongst Engineers leading to emergent of modern materials for different engineering applications. Modern engineering materials include metals, polymers, ceramics and composites. Ceramics, although are strong in compression, but generally weak in tension. Meanwhile, metals tend to have equal strengths both in tension and compression; composites have been developed to overcome the deficiencies of members of a particular class of materials (John, 1992). With extensive applications of polymers and its composites, due to their excellent mechanical properties, the demands for the materials are increasing (Fang *et al*., 2017). There are different composites that have been considered over time in lieu with optimizing materials to achieve good mechanical properties. Natural fibers are being considered as an alternative reinforcement in polymer composite due to their advantages over conventional glass and carbon (Saheb and Jog, 1999). These advantages include low cost, comparable specific tensile properties, renewability, recyclability, biodegradability, less health risk, non-irritation to skin, and non-abrasive to the equipment (Malkapuram *et al.,* 2009). Generally, polymers are classified as thermoplastics and thermosetting. Thermoplastic materials currently dominate as matrices for bio-fibres (Malkapuram *et al.,* 2009). The most commonly used thermoplastics for structural applications are polypropylene, polyethylene, and poly vinyl chloride (PVC); while phenolic, epoxy and polyester resins are the most commonly used thermosetting matrices (Malkapuram *et al.,* 2009). Most plastics possess low impact strength in their natural forms (American Chemistry Council, 2019); hence there is need for reinforcement which enhances the mechanical properties. Reinforced polymer composite has found its applications in variety of places such as the automobile industry like the car bumper, among others. Although, this bumper has been produced to possess good mechanical properties, but has tendency to break when subjected to little or no impact forces, which has become a problem to Engineers (Mazumbar, 2001). Meanwhile, studies revealed that the manufacturers were able to meet automotive requirements of cost, appearance and performance utilizing composites (Mazumbar, 2001). Currently, composite body panels have a successful track record in all categories from exotic sports cars to passenger cars to small, medium, and heavy truck applications. In 2000, the automotive industry used 318 million pounds of composites. Because the automotive market is very cost-sensitive, carbon fiber composites are not yet accepted due to their higher material costs.

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Automotive composites utilize glass fibers as main reinforcements (Mazumdar, 2001).

Epoxy resins are thermosetting polymers with good chemical resistance, high mechanical properties and thermal stability, high adhesive strength as well as high electrical insulation (Agarwal *et al*., 2017). For high performance applications in aerospace and marine structures, epoxy resins are used. This is as a result of its ease processing, hot and wet strength in conjunction with excellent mechanical properties in composites (Mukhopadhway, 2005). According to Mukhopadhway (2005), superior mechanical properties and better resistance to degradation made the performance of epoxy to be similar to that of polyester. Reinforcement could be either fiber reinforced, particle reinforced, flat flakes reinforced or filler reinforced. Fillers are added to a polymer formulation to reduce the costs and improve the properties. Fillers can either be solid, liquid or gas. They occupy space and replace the expensive resin with less expensive compounds without modifying other characteristics.

In this study, cow horn is being considered as the filler in an epoxy composite, being a material containing fibrous protein material called keratin (McKittrick *et al.,* 2012). It has been regarded as a viable reinforcing material. It is a tough, resilent, very ductile material that possesses highly resistant to impact with its reasonable amount of carbon present (Kumar & Boopathy, 2014; McKittrick *et al.,* 2012). This study therefore aims at testing cow horn as a suitable composite reinforcing material (filler) and imperative to produce composite with excellent mechanical properties which are also quite affordable as well as possess vast applications.

MATERIALS AND METHODS

The materials used in this study include cow horns; epoxy resin and catalyst which were respectively obtained at Sobi-Ilorin abattoir (Kwara State, Nigeria) and from a local vendor at Ojota, Lagos State, Nigeria. The cow horn was thoroughly washed and air dried to remove debris on it. Subsequently, the air-dried cow horn samples were oven dried using a conventional oven at 100ºC for 126 hours to completely remove moisture in the horn.

Figure 1 shows the cow horn samples in the oven for drying. The dried cow horn samples were crushed using a SNE FOURE Hammer Mill and then transferred to a "Broyeur-clero" ball mill (Figure 2). The milling operation was carried out for 22 hours. The milled cow horn was then sieved manually, using 100 microns and 150 microns sieves, to segregate two different sizes of horn particles.

Fig 1: (a) Samples of the cow horns (b) The cow horn samples in the oven ready for drying

Fig 2: Broyeur-clero" hammer mill used for crushing

Production of Epoxy Composite: The epoxy and each of 100 and 150 µm cow horn (were separately measured using an electronic measuring scale in different ratios as presented in Table 1 and kept separately in different containers (Figure 3a). In activating the resin, it was gradually mixed with the catalyst (hardener). A lot of care was taken at this stage, since rapid mixing might allow air bubbles to get trapped into the mixture. The weighted cow horn samples were then added to this mixture and mixed for about 5 minutes till homogeneity was attained. The Mixture of cow horn and resin is shown in Figure 3b.

The cow horn and epoxy were then poured into the wooden mould (Figure 4a) and allowed to cool. The moulds were left, after proper marking, for natural curing at room temperature for 72 hours (Figure 4b). To reduce the negative effects of polymerization shrinkage and increase hardness and wear resistance of the lightly cured resin composite samples (Figure 4c), post curing (heat treatment) of the specimens was done in a conventional oven at varying curing temperatures of 60 and 80ºC.

This process was also to further harden, set the cast epoxy resin composites and to increase its mechanical properties. This process was in line with the practice of earlier researchers (Irawan *et al*., 2011; Khondker *et al.,* 2005; Bello *et al.,* 2015).

Table 1: Mixing ratios of specimen

Specimens	P100	C40	C35	C30	C25	C20	C15	C10	C5
Cow horn (%)	0	40	35	30	25	20	15	10	5
Epoxy (%)	100	60	65	70	75	80	85	90	95

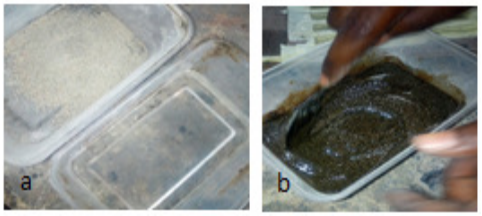

Fig 3: (a) Measured quantity of cow horn and epoxy, (b) Mixture of cow horn and resin

Determination of the Corn Horn Elemental Chemical Composition: The external cover of the horn (hoofs) were removed and soaked in water to make them free of blood and dirty materials. Subsequently, the cleaned horn was cut into smaller chips and rewashed in hot water and later sun-dried for 15 days. The elemental chemical composition of the corn horn sample was carried out using Shimadzu 720 XRF Analyzer (Maker: Shimadzu Cooperation, Japan).

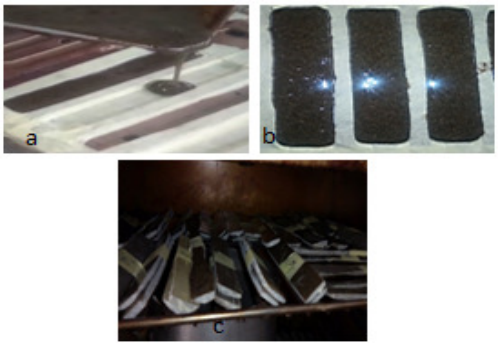

Fig 4: (a) Pouring of the cow horn mixture into the mould cavity, (b) Specimens at room temperature for curing, (c) Sets of specimens arranged in the oven for post curing

Characterization of the composite samples: The mechanical properties (such as tensile, flexural and impact properties) of the specimens were determined to characterize the composite produced, in line with the practices of Irawan et al., (2011); Khondker et al., (2005); Viviane et al. (2006); Kumar and Sankar (2019). The samples for tensile test were prepared and the test was conducted in accordance with the ASTM D638 / ASTM D3039 / D3039M – 17 standards.

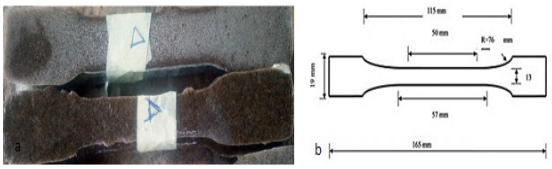

Fig 5: Tensile Samples

Figures 5 (a and b) shows the tensile test sample and its ASTM dimensions respectively. The tensile and flexural properties of the samples were determined at the National Centre for Agricultural Mechanization Ilorin, Nigeria Universal Testing Machine Laboratory using Win Test Analysis on Testometric Materials Testing Machine; Type DBBMTCL-5000 Kg, Serial No. 17819 (Figure 6) . The thickness of each of the samples was measured at three different positions along the length of the specimen and the average thickness was used for calibration. The test speed used was 5.0 mm/min with the gauge length fixed at 57.00 mm. Eight samples were tested for each test type.

Fig 6: Testometric machine with the specimen wedged between its grips

The flexural samples were prepared and the test was carried using ASTM D790-03 as a guide. The flexural test was evaluated using three-point bending flexural test, as recommended in ASTM D790-03 (Pham *et al.* 2014; Irawan *et al.*, 2011; Kumar and Sankar, 2019). Figure 7 (a and b) shows the pictorial flexural test sample dimensions as stipulated in ASTM standards and the impact machined used for the test respectively.

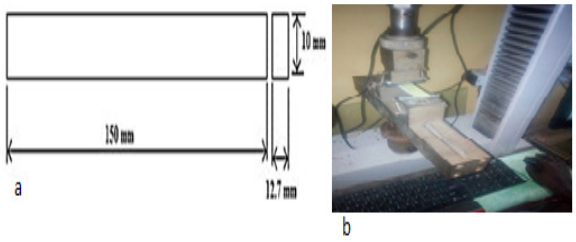

Fig 7: (a) Pictorial representation of specimen for flexural test with dimensions; (b) Testometric machine with a flexural specimen

The samples for the impact test were prepared as presented in Figure 8 and the test was carried out in accordance with the guidelines in ASTM D256-04 standard at Department of Mechanical Engineering, University of Ilorin, Nigeria.

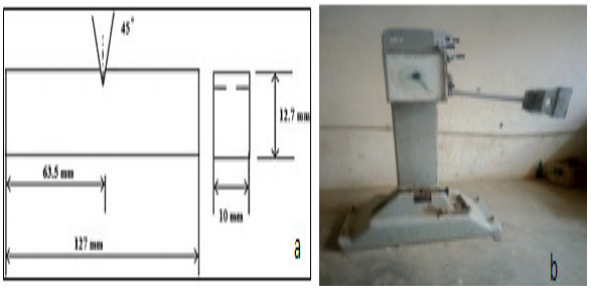

Fig 8: (a) Pictorial representation of specimen for impact test; (b) The Impact testing machine used

RESULTS AND DISCUSSION

The results of the elemental chemical composition of the corn horn sample are presented in Table 2. The major component was Sulphur (78.23 %), while calcium (8.10 %) and Molybdenum (5.80 %) constitute another significant element in the material. The values of the elemental composition of the corn horn were within the range values earlier discovered by Abdullahi and Salihi (2007).

Table 2: Elemental constituents of cow horn sample

S/N	Elements	Percentage
1	Sulphur	78.23
2	Calcium	8.10
3	Potassium	0.80
4	Copper	0.21
5	Zinc	2.00
6	Molybdenum	5.80
7	Aluminum	0.30
8	Silicon	0.13
9	Indium	0.5
10	Rhenium	2.3
11	Selenium	1.0

The various tensile strengths exhibited by the composite (cow horn/epoxy) samples were determined and studied considering various sizes of the composites (100 µm and 150 µm) at varying curing temperatures. The results are presented in Figures 9 and 10.

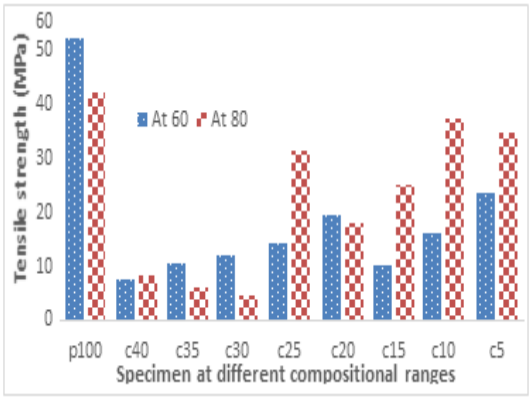

Fig 9: Tensile strength of specimens at 100 µm with different curing temperature variation

Figure 9 shows the tensile strength variation of the various specimens at 100µm. The results indicate that specimen p100 with curing temperature of 60°C exhibited the highest tensile strength of 52.29 Mpa, while that of 80°C curing temperature has strength value of 42.18 Mpa. This implies that curing temperature has significant effect on the tensile strength of the p100 sample. Generally, the values of the specimens' strengths were between 6.25 MPa and 52.29 MPa. The results revealed a drastic reduction in the strength values of the p100 with addition of cow horn. This might be as a result of poor compatibility between the matrix and cow horn particle. According to (Kumar *et al*. (2017), effective load transfer between the matrix and the particles serves as the base for the tensile strength of a particle-reinforced polymer matrix composite. Addition of cow horn as filler decreased the tensile strength of the composite in no particular order. Considering specimen c40, the tensile strength of the composite was found to be 8.29 MPa with curing temperature of 80^0C, but the specimen with curing temperature of 60°C recorded a low tensile strength value of 7.71 MPa. In the case of specimen c35 with curing temperature of 80°C, the tensile strength increased to 10.46 MPa, but the specimen with curing temperature of 60°C has a lower value of 6.25 MPa. The specimen C30 with curing temperature of 80^0C and one with curing temperature of 60°C respectively recorded tensile strength value of 4.71 MPa and 12.08 MPa. For specimen c25, the result shows a significant difference in the tensile strength with a value of 31.33 MPa (with curing temperature of 80°C) and 14.13Mpa (with curing temperature of 60°C). Specimen c20 has a tensile strength of 18.08 MPa at 80°C and 19.42 MPa at 60°C. Specimen c15 exhibited a tensile strength of 25.13 MPa and 10.04 MPa with curing temperature of 80°C and curing temperature of 60°C respectively. Specimen c10 shows an appreciable tensile strength value of 37.58 MPa (with curing temperature of 80°C). Specimen c5 has tensile strength value of 34.83 MPa and 23.46 MPa with curing temperature of 80°C and with curing temperature of 60°C respectively. The results analysis apparently revealed reduction in the strength of the composite. According to Duraisamy *et al*. (2017), horse particles in the composite create weakness in the adhesive force between the resin and the filler (horse particle) because horse particle acts as stress concentration points. Thus, the strength of the virgin epoxy composites decreases.

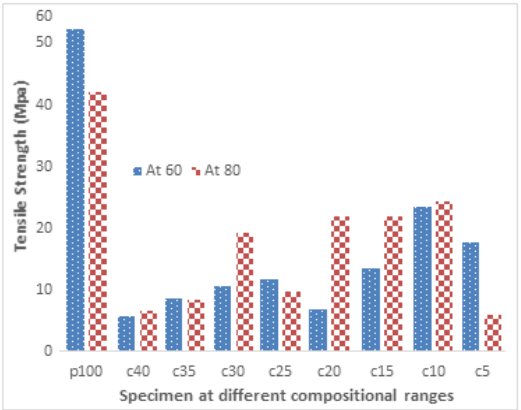

Fig 10: Tensile strength of specimens at 150µm with different curing temperature variation

Figure 10 shows the tensile strength variation of the various specimens at 150 µm. The results indicate that the specimen p100 with curing temperature of 60^0C has the highest tensile strength value of 52.29 MPa, while that of 80°C curing temperature has strength value of 42.18 MPa. This reflects significant effect of curing temperature on the tensile strength of the p100 sample. Generally, the values of the specimens' tensile strengths were between 7.71 MPa and 23.46 MPa for samples with curing temperature of 60°C and between 4.71and 37.58 MPa for samples with curing temperature of 80°C (with particle size of 100 µm). Meanwhile, samples with particle size 150 µm have tensile strengths between 5.77 MPa and 23.50 MPa (with curing temperature of 60°C), and 6.042 and 24.38 MPa (for samples with curing temperature of 80°C). The results revealed a drastic reduction in the strength values of the p100 (52.29 MPa and 42.2 MPa with curing temperature of 60°C and 80°C respectively) with addition of cow horn. Poor compatibility between the particles and cow horn particle-reinforced polymer matrix composite is likely to be a factor (Kumar *et al*., 2017). Addition of cow horn as filler decreased the tensile strength of the composite in no particular order. At specimen c40, the tensile strength of the composite with curing temperature of 80°C was found to be 6.71 MPa, while same sample with 60^0C curing temperature has a low tensile strength value of 5.79 MPa. For sample c35, the sample with 80^0C curing temperature and that with 60°C curing temperature exhibited strength value of 8.67 MPa and 8.33 MPa respectively. Sample c30 has tensile strength value of 19.21 MPa and 10.71 MPa for at 80^0C at 60^0C. At c25, the result shows a significant difference in the tensile strength with a value of 9.63 MPa (with curing temperature of 80^0C) and 11.63 MPa (with curing temperature of 60^0C). Specimen c20 have tensile strength of 21.88 MPa (with curing temperature of 80^0C) and 6.88 MPa (with curing temperature of 60^0C). For specimen c15, the tensile strength exhibited was 22.00 MPa for sample with curing temperature of 80^0C and a lower value of 13.58 MPa for sample with curing temperature 60^0C. Specimen c10 shows an appreciable tensile strength value of 24.38 MPa with curing temperature of 80°C. Specimen c5 for sample with curing temperature 80°C and sample with curing temperature of 60^0C has tensile strength value of 6.04 MPa and 17.79 MPa respectively. From these results (Figures 9 & 10), it is obvious that the specimens of 100µm particle size had better tensile strength values than specimens of 150µm particle size. According to Fu *et al*. (2008) particle size, good bonding strength between fibre particles and resins, and particle loading are parts of factors that affect the strength.

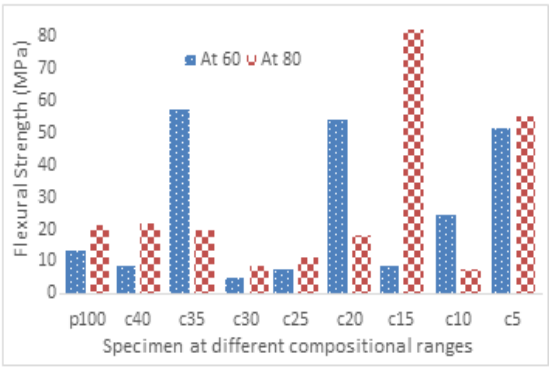

Fig 11: Flexural strength of specimens at 100 µm with different curing temperature variation

Figure 11 shows the flexural strength variation of the various specimens at 100µm. The results indicate that the flexural strength of specimen p100 with curing temperature of 60^0C was 13.23 MPa, while that of 80°C curing temperature's strength value was 21.58 MPa. This implies that curing temperature also has significant effect on the flexural strength of the p100 sample. Generally, the recorded values of the specimens' flexural strengths for samples of particle size of 100 µm were between 4.97 MPa and 57.11 MPa (samples with curing temperature of 60°C) and between 7.44 and 82.36 (samples with curing temperature of 80°C). The results also revealed an increase in the flexural strength values of the specimen p100 (13.23 / 60°C and 21.59 / 80°C) with addition of cow horn. Addition of cow horn as filler in the composite increased the flexural strength of the composite in no particular order. For specimen c40, the flexural strength of the composite sample with curing temperature of 80^0C was found to be 21.90 MPa, while sample with curing temperature of 60°C recorded a low tensile strength value of 8.62 MPa. At specimen c35, the flexural strength was 57.11 MPa for sample with curing temperature of 60^0C, but sample with 80°C curing temperature exhibited a lower value of 19.94 MPa. Specimen c30 recorded flexural strength value of 8.54 MPa and 4.92MPa for sample with curing temperature of 80°C and 60^0C respectively. For specimen c25, the result shows a significant difference in the flexural strength with a value of 11.31 MPa at 80^0C curing temperature and 7.58Mpa at 60^0C curing temperature. Specimen c20 has a flexural strength of 54.16Mpa with curing temperature 60^0C and 18.18MPa with curing temperature 80^0C. For specimen c15, the flexural strength was 82.36 MPa (with 80^0C curing temperature) but with a lower value of 8.54 MPa for sample with 60^0C curing temperature. Specimen c10 has a flexural strength value of 24.31 MPa with curing temperature of 60°C. Specimen c5 in its own case has flexural strength value of 55.27 MPa with curing

temperature 80°C and 51.73 MPa with 60°C curing temperature. The analysis of the results revealed that the post curing temperature has noticeable effect on the flexural strength of the composite. The analysis of flexural strength results shows that the specimens with smaller particle sizes of 100 µm exhibited higher flexural strength value than specimen of higher particle sizes of 150 µm, though at higher curing temperature. This result is in line with the findings of Duraisamy *et al*. (2017) that smallest horn powder size in composite gives better strength as a result of the fact that smaller particles have better dispersion and high surface area with the matrix. Figure 12 shows the flexural strength variation of the various specimens at 150 µm. Generally, the specimens' flexural strengths were between 5.31 MPa and 30.74 MPa (samples with curing temperature of 60°C) and between 3.97 and 41.34 MPa (samples with curing temperature of 80°C). The results indicate that the flexural strength of specimen p100 with curing temperature of 60°C has a low flexural value of 13.24 MPa as compared to when its curing temperature was 80°C (21.58 MPa). The general values of the specimens' flexural strengths were between 4.97 MPa and 55.27 MPa. The results revealed an increase in the flexural strength values of specimen p100 with addition of cow horn. Also, addition of cow horn of particle size 150µm as filler in the composite increased the flexural strength of the composite in no particular order.

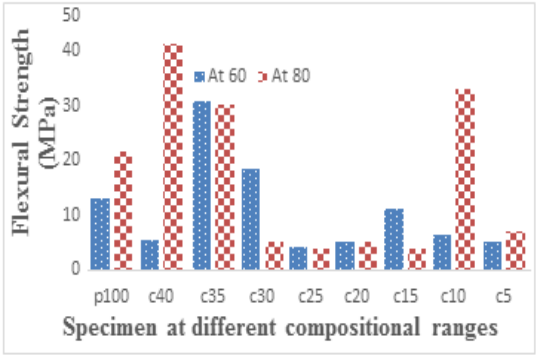

Fig 12: Flexural strength of specimens at 150 µm with different curing temperature variation

At specimen c40, the flexure strength of the composite was found to be 41.34 MPa for sample with curing temperature of 80°C, but recorded a low flexural strength value for sample with curing temperature of 60°C (5.398 MPa). Considering specimen c35, the flexural strength of sample with curing temperature of 80°C and that sample with curing temperature of 60°C were little or no difference in values of 30.21 MPa and 30.74Mpa respectively. This might be as a result of a good compatibility between the horn particles and the epoxy at that composition (Kumar *et al*. 2017). For specimen c30, there was a drastic drop in the flexural strength to 5.06 MPa for sample with curing temperature of 80°C and a value of 18.47 MPa for sample with curing temperature of 60°C. Low flexural strength values were recorded for specimens' c25 and c20 in an ascending order. Specimen c10 also exhibited a high flexural strength for sample with curing temperature of 80°C (33.05 MPa). At specimen c5, the flexural strength value was 7.09 MPa for sample with curing temperature of 80°C and 5.31 MPa for sample with curing temperature of 60°C. The results revealed that the post curing temperature has a noticeable effect on the flexural strength of the composite. From the obtained results, the highest flexural strength was obtainable with sample c5, 100 µm particle sizes with curing temperature of 80°C. Figure 12 shows the flexural strength variation of the various specimens at 150 µm. Generally, the specimens' flexural strengths were between 5.31 MPa and 30.74 MPa (samples with curing temperature of 60°C) and between 3.97 and 41.34 MPa (samples with curing temperature of 80°C). The results indicate that the flexural strength of specimen p100 with curing temperature of 60°C has a low flexural value of 13.24 MPa as compared to when its curing temperature was 80°C (21.58 MPa). The general values of the specimens' flexural strengths were between 4.97 MPa and 55.27 MPa. The results revealed an increase in the flexural strength values of specimen p100 with addition of cow horn. Also, addition of cow horn of particle size 150µm as filler in the composite increased the flexural strength of the composite in no particular order.

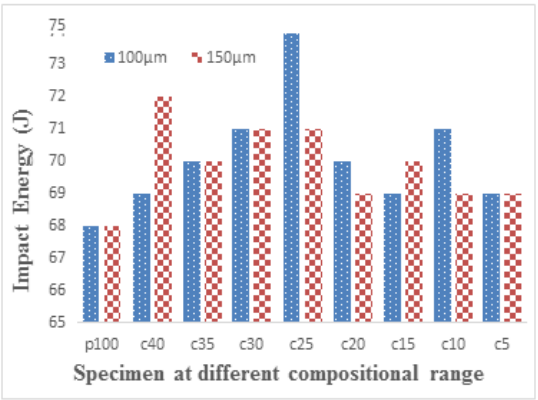

Fig 13: Impact strength of specimens with different size particles variation

In Figure 13, the impact energy variations for the various specimens at 60°C are presented. In general, the specimens' impact values ranged between 69 and 74 J for samples of 100 µm particle size, and between 69 and 72 J for samples of 100 µm particle size. The results indicate that the impact energy of p100 was 68

J. Specimen c40 has impact energy of 72 J at 100 µm particle size and impact energy of 68 J at particle size of 150 µm. Specimen c30 has the same impact value of 71 J at both size variations. Specimen c25 recorded the highest the impact energy of 74 J at 100 µm. Specimen c20 has an impact energy value of 70 J and 69 J at particle sizes of 100 µm and 150 µm respectively. Specimen c15 also recorded an impact value of 69 J and 70 J at 100 µm and 150 µm respectively. Specimen c10 has impact energy of 71 J at 100 µm particle size, while at 150µm particle size impact energy of 69 J was recorded. Specimen c5 also has the same impact energy at both size variations (100 µm and150 µm particle sizes) with a value of 69 J. From the results obtained, specimen c25 has the highest impact energy (74 J) with particle size of 100 µm. Though, c40 recorded very close impact energy value of 72 J with particle size of 150 µm. The increase in the impact strength of the new composite is an indication of good bonding strength of the specimens.

Conclusions: At higher curing temperature, better flexural, impact and tensile properties were achieved in the polymers with the incorporation of the cow horn as filler. Also, the composite materials with particle size of 100 µm with curing temperature of 80^{o}C exhibited higher tensile and impact properties. Therefore, the cow horn was found to be a good potential filler in the composite if prepared using higher curing temperature as exhibited through its mechanical properties. A composite prepared at 150µm mixture is highly recommended for an impact application of the composited especially for material engineering to be subjected to impact application. Further research works on the use of cow horn as filler in epoxy composite and also the effect of alkali treatment on the compatibility of the cow horn particles and epoxy are recommended

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