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# Effect of Fillers on Epoxy Resin

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***ABSTRACT:*** *The research work was focused on the study of effects of various filler materials on some Mechanical Properties of Unsaturated polyester and Epoxy Composites. For this purpose, 3 filler materials were selected and sieved into 3 different particle size 75, 105, and 150. The fillers used are – Aluminium, Clay and Coconut shell Powder. The composition selected was 70 %, 80 % and 90 %. Epoxy and Unsaturated polyester resin and 10 %, 20 % and 30% Fillers respectively. The fabrication method used was conventional hand lay-up technique. The tests carried out were Fourier Transform Infrared Radiation Analysis, Tensile Test, Flexural (3-Point Bending Test), Scanning Electron Microscopy and Water Absorption Test. The results indicated that all three filler types are suitable for improving the flexural and tensile strength of the unsaturated polyester and epoxy resin with the UP-Aluminium show a 9.83 % and 20.46 % increase in the tensile and flexural strength respectively. UP/Clay was 1.60 % and 0.11 % respectively, while for E-CL, it was 2.34 % and 1.24 % respectively. Coconut shell powder showed the best results, of all the three fillers used (18.48 % for Epoxy composite and 22.92 % for UPR), while the SEM analysis showed even dispersion of fillers in the epoxy composites. The water absorption test showed a retention capacity of 0.62 % and 0.43 % for epoxy resin and UPR respectively.*

***KEYWORDS:*** *Fillers, Epoxy resin, Mechanical Testing, Scanning electron microscopy, FTIR*
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## I. INTRODUCTION

The major problem in the application of polymers in engineering is their low stiffness and strength, compared to metals, the strength of these materials are approximately 5 times lower, which has led to the development of new materials called composites, where resins are reinforced with either particles or fibres (McCrum et al., 1988).

Composites are made by combining two or more natural or synthetic materials to maximize their useful properties and minimize their weaknesses. One of the oldest and most popular composites, glass-fibres reinforced plastic (GRP), combines glass fibres (which are strong but brittle) with plastic (which is flexible) to make a composite material that is tough but not brittle. Composites are typically used in place of metals because they are equally strong (on weight basis) but much lighter. Most composites consist of fibres of one material tightly bound into another material called a matrix. The matrix binds the fibres together somewhat like an adhesive and makes them more resistant to external damage, whereas the fibres make the matrix stronger and stiffer, thus helping it resist cracks and fractures. Fibres and matrix are usually produced from different types of materials. The fibres are typically glass, carbon, silicon carbide, or asbestos, while the matrix is usually plastic, metal, or a ceramic material (Chanap, 2012).

In contrast to conventional materials e.g., steel, the properties of the composites can be tailored considering the structural aspects. Composite characteristics, such as stiffness and thermal expansion, can be altered continuously over a wide range of values as desired by the designer. Careful selection of reinforcement type enables finished product characteristics to be tailored to almost any specific engineering requirement.

Fillers are a variety of solid particulate materials (inorganic, organic) that may be irregular, acicular, fibrous, or plate-like in shape and are used in reasonably large volume loadings in plastics. There is a significant difference in chemical structures, forms, shapes, sizes, and inherent properties of the various inorganic and organic compounds that are used as fillers. They are usually stronger and more rigid than the polymer, improving the strength and modulus of polymers, thus the mechanical property modification can be considered as their major function. Fillers were initially used as additives to reduce cost by replacing the most expensive polymer because of their unfavourable geometrical features (Xanthos, 2010). Other advantages of fillers include, improved processing, optical effects, thermal conductivity, flame retardancy and control of thermal expansion.

Epoxy resin is a generic name for compounds that have two or more oxirane rings (epoxy groups) in one molecule and are cured 3-dimensionally by a suitable curing agent. They are produced by reaction between bisphenol A and epichlorohydrin which is the most essential precursor in the production of epoxy resin (Yukisima, 1987). Epoxy resin acts as surfactants and are chemically well-suited to most substrate because of the oxirane ring they posses which provides the compound with its flexibility. Epoxy resin is also cured easily without evolution of by-products or volatiles, which makes it suitable for composite application. Epoxy resin, is very brittle and notch sensitive in structural application which led to increased research to improve toughness (May, 1987).

Several researches have been conducted over the years by researchers who have explored the possibility of modifying the behaviour of thermosetting epoxy resin by reinforcing it with different types of fillers like hard ceramics such as boron carbide($B_4C$), silicon carbide (SiC), and alumina ($Al_2O_3$) are found to enhance mechanical and wear properties of epoxy. (Abenoja et al., 2009). Graphite, molybdenumsulphide ($MoS_2$), and polytetrafluoroethylene (PTFE) are examples of lubricant fillers whic are sometimes used as filler for epoxy to make them suitable for low friction and low wear environments. To facilitate the interfacial interaction between the filler and the matrix, fillers are subjected to different pre-treatments. The results from this works showed improvement in the mechanical and tribological properties of composites i.e the friction lubrication and wear properties of the resin (Li et al., 2004., Jacobs et al., 2004).

The effect of filler types on carbon-epoxy resin using different types of filler types (Granite Powder, Aerosil (Fumed Silica) and Coremat). All filler types improved the tensile and flexural strength of epoxy resin. Aerosil filled composites however showed best results for Tensile strength whereas hardness and Bending results were good for Coremat filled ones (Anigol and Pol, 2015).

Bamboo fibre reinforced epoxy composites was fabricated using conventional filler (aluminium oxide ($Al_2O_3$) and silicon carbide (SiC) and industrial wastes (red mud and copper slag) particles as filler materials. Chosen particulate fillers (ceramic fillers) were incorporated into the bamboo-fibre reinforced epoxy, synergistic effects, as expected was achieved in the form of modified mechanical properties. Inclusion of fibre in neat epoxy improved the tensile strength and the flexural strength of the composites. But with the incorporation of particulate fillers, the tensile strengths of the composites are found to be decreasing in most of the cases. Among the particulate filled bamboo-epoxy composites, least value of void content was recorded for composites with silicon carbide filling and for the composites with glass fibre reinforcement minimum void fraction is noted for red mud filling.

Investigation showed that the tensile, flexural and impact properties are found to decrease with the increase in the filler particle size and filler volume fraction (Sandhyarani et al., 2010).

Mechanical properties of epoxy reinforced with different weight percent of silicon carbide nanoparticles were investigated. The experimental results indicate that the strength decrease with further increase in weight percentage of reinforcement. This could be due to the weak bonding between the matrix and the nanoparticles. The wear results show that Nano particles enhanced the wear resistance of unreinforced epoxy these is due to the ceramic Nano particles act as a rough surface relative to the counterface against which they slide (Nassar and Nassar, 2013).

Composites with 10, 20 and 30 wt % coconut shell powder epoxy composites were fabricated using Hand layup technique, and it was discovered after subjecting them to different mechanical tests, tensile strength increases up to 20 wt % of filler reinforcement beyond that it decreases. The flexural strength increased from 35 MPa in case of epoxy to 59, 78 and 67 in 10, 20 and 30 wt % composites respectively. Composites show better resistance to abrasion and wear as hardness increases from 13 HV of epoxy to 35 HV in E-30CSP composite (Srivastava and Maurya, 2015).

Studies of the tensile and flexural properties of composites made from coconut shell filler particles and epoxy resin was reported that the tensile and flexural strengths of the epoxy coconut filler composites were affected by the amount of filler in the composites. Maximum properties were observed at 15 % filler loading (Singh et al., 2013).

The mechanical and tribological characteristics of potassium titanate whisker (PTW) reinforced epoxy composites were studied and the effect of various test variables and material parameters on the friction and wear behaviour of epoxy/PTW composites systematically. Tribological tests were conducted on a pin-on-disc apparatus under dry sliding conditions. Addition of PTW was found to improve the wear resistance of the composites and 15wt % PTW filled epoxy exhibited lowest specific wear rate and highest friction coefficient compared to other test samples. PTW additions showed beneficiary effect on density, hardness, and stiffness properties of composites; however, strength properties and ductility were found to decrease with the increasing content of PTW. Scanning electron microscope (SEM) images of tensile fractured surfaces and worn-out surfaces of selected samples revealed different fracture mechanisms (Mudradi et al., 2014).

## II. MATERIALS AND METHODS

**Materials**

Epoxy resin plus hardener, unsaturated polyester, accelerator (cobalt octate), catalyst (methyl ethyl ketone peroxide, MEKP) and aluminium powder were purchased from a retailer, Nycil limited, Ibadan, while coconut shell was sourced at the kasuwa central market Kaduna. The coconut shell was then processed into powdered form using a mortar and pestle. Clay was gotten from Nigeria Defence Academy (NDA) old site. Aluminium powder was purchased in Aleshinloye market in Ibadan.

The Clay was air dried and made into clay powder by the use of the mortar and pestle in the chemistry laboratory of NDA, after which the powdered clay was then sieved into different particle sizes (75 µm, 105 µm, 150 µm) put in polythene bags and labelled (Cl75, Cl105, Cl150 respectively). The coconut shell was pulverised into powder form using a grinder after which they were sieved into different particle sizes and placed in different containers and labelled (C75, C105, C150). The same procedure was used in converting the aluminium into powder form at the metallurgical laboratory of Ahmadu Bello University, Zaria, and they were labelled for easy identification and to avoid mixing up different particle sizes. The aluminium powder was labelled Al75, AL105, and AL150.

**Composite Fabrication**

**Epoxy composites**

For the three different filler types and sizes, 5 g, 10 g and 15 g were weighed and transferred into separate beakers for 10 %, 20 % and 30 % filler load composites. For 10 % filler content, 5 g of aluminium powder (75 µm particle size) was weighed and put aside in a beaker. A 45 $cm^3$ volume of epoxy resin was then measured, after which the 5 g aluminium powder was poured into the pure resin and stirred gently till homogenous mixture was obtained. DETA (15 $cm^3$) was measured and then poured into the initial mixture of resin and filler; the mixture was then stirred gently and continuously for another 10 minutes after which it was transferred into the mould and allowed to cure over time.

This procedure was repeated for all filler types, at 20 % and 30 % filler content. The curing time for all the samples was 12 hours.

**Figure 1: Samples of composite fabricated**

**Equipments**

**Fourier Transform Infrared Analyser**

The fractured composite materials were placed in a pulveriser to granulate them, and then they were mixed with potassium bromate (KBr). After mixing with potassium bromate, they were placed under a hydraulic press, and formed into an oval plate like flat material. This material was then placed in the FTIR machine and infrared radiation was passed through it. The spectra was then transmitted and recorded. The fractured composites materials gotten from the tensile and flexural strength test were used to run this analysis. 30 % filler content was selected to run the FTIR and this test was carried out in the Multi-disciplinary Central Laboratory of the University of Ibadan.

**Tensile Test**

The tensile test was carried out using the OkhardUniversal Testing Machine, in accordance to the ASTM-D 3039 standards. Again five test per specimen were conducted, then average value obtained was recorded for data analysis.

The composite materials were first cut into dumb bell shapes as shown below. Each test specimen was placed between the two clamps of the Okhard machine. Then it was subjected to a tensile force which pulled the material apart till it fractured. This test was carried out in the material testing laboratory of the department of agriculture and environmental engineering, University of Ibadan.

**Flexural Strength Test**

Flexural test is a measure of the bending or transverse rupture strength; it is generally termed as the stress in a material just before it yields.

The flexural test was conducted using the Okhard Universal Testing Machine in accordance to ASTM-D790 standards. Five specimens were tested per sample and the average value taken for data analysis. Rectangular strips with a dimension of 3.22 mm x 12.7 mm x 125 mm were then cut out of the composite material and placed horizontally over two pivots of contact, a force was then applied at the middle of the test sample from the top by the loadcell at a rate of 0.02 KN/s until the composite material yielded. (Hodgkinson, 2000). This test was carried out in the Material testing Laboratory of the Department of Agriculture and Environmental Engineering University of Ibadan.

**Scanning Electron Microscopy (Sem)**

A scanning electron microscope (SEM) was used to observe the cross sectional images of the fractured samples. It was particularly used to study the fractured surface and cross-sectional area of the composite materials.

The principle of operation is based on using an electron microscopy machine which was used to focus a beam of electron on a pin pointed spot of the fractured surface on the composite materials. The image was then projected by an electronic signal reflected by the sample (Hashemi and Smith, 2010). Scanning electron microscopy of composite materials was carried out at Umaru Musa Yar'adua University, Katsina.

**Water Absorption Test**

Water absorption test was conducted to determine the quantity of water absorbed by the composite materials according to ASTM D570 standards.

Samples of both unsaturated polyester and epoxy filled composites at maximum filler loadings, (30 % filler content) were collected and weighed. They were then placed in water for 24 hours, the samples were then collected and patted dry with dry cloth, then were weighed again. The weight was then recorded as final weight and the percentage water absorption was calculated (intertek group). Water absorption test was carried out in the Chemistry Laboratory of Nigeria Defence Academy.

## III. REUSULTS AND DISCUSSION

**Effect of Filler Loading and Filler Type onTensile Strength of Epoxy Composite**

Figures 5 shows the variation in tensile strength of epoxy filled composites as a function of fibre type and fibre loading at different particle sizes, and are labelled respectively below.

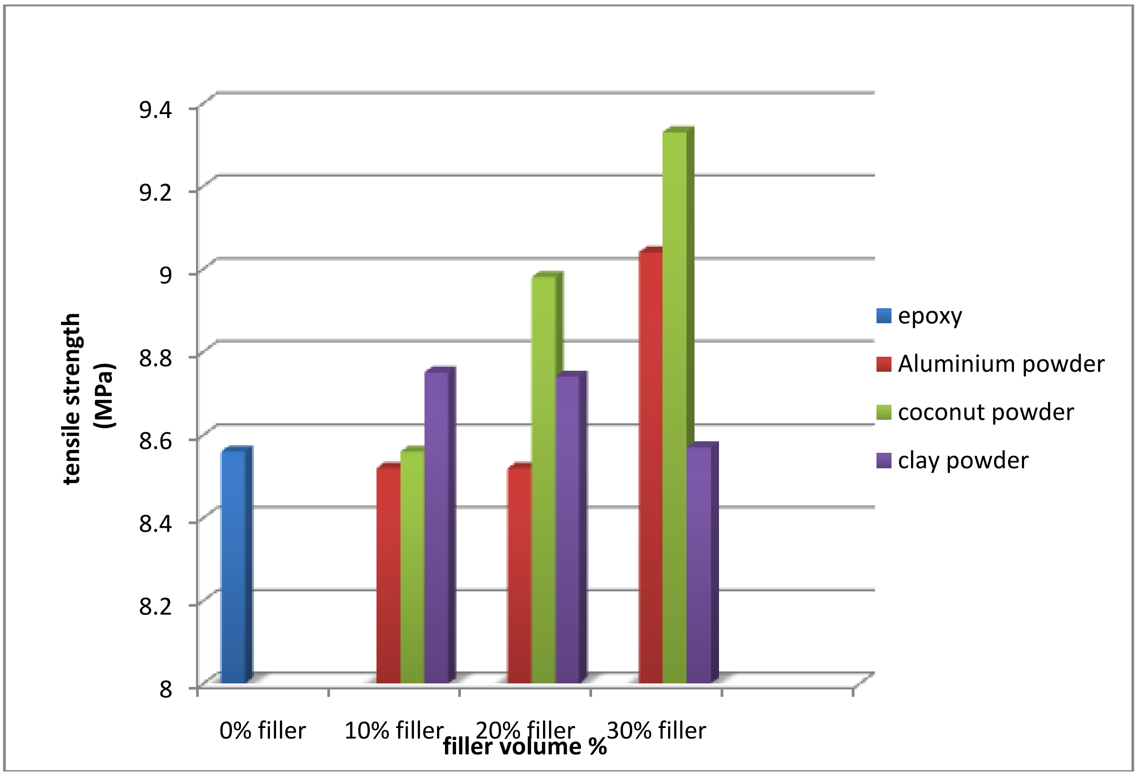


**Figure 2: Graph of Tensile strength versus filler loading of epoxy composites (105 µm)**

Generally as filler loading increases, the tensile strength increases also for all the filler types, with 30 % coconut shell powder filled composite showing the maximum strength. The reason for the high tensile strength exhibit by the coconut shell composite is immediately apparent as the FTIR spectrograph does not show any chemical interaction between the epoxy and the coconut shell powder. However the SEM micrograph showed very good dispersion of the coconut shell fillers in the epoxy and this may be the reason for the good tensile strength. The carbonyl and/or acetylene group introduced into the structure may lead to better interactions between the epoxy molecules. Hence the increase in tensile strength as the filler loading increase, increases the strength of the composite. For the clay fillers, the high agglomeration of the fillers as observed in the SEM test may be responsible for the slightly lower tensile strength observed with increase in filler loading. Furthermore, aluminium powder fillers showed they better dispersion as when compared to the clay fillers from the SEM test. The dispersion however was not even as compared to the coconut shell powder, this may be attributed to the increased strength observed for aluminium powder fillers which were close to the strengths recorded in coconut shell powder fillers.

**Effect of Filler Loading and Filler Type on Flexural Strength of Epoxy Composite**

Fig 6 shows that all filler types improved the flexural property of epoxy except for clay powder at 30 % filler content where a slight drop was recorded. However coconut shell powder showed the most improved result, where 9.36 MPa was recorded at 30 % filler content. It was also discovered that with every increase in filler content, the flexural property of coconut shell reinforced epoxy improved. This similar trend was recorded for aluminium powder reinforced epoxy as increase in filler content also helped improve the flexural property. The improvement in flexural strength for aluminium filled epoxy resin was however not as significant as that of coconut shell powder. Clay reinforced epoxy resin also improved in flexural strength as filler content increased, however, there was a drop in the strength at 30 % (8.80 Mpa) filler content even below the pure epoxy resin.

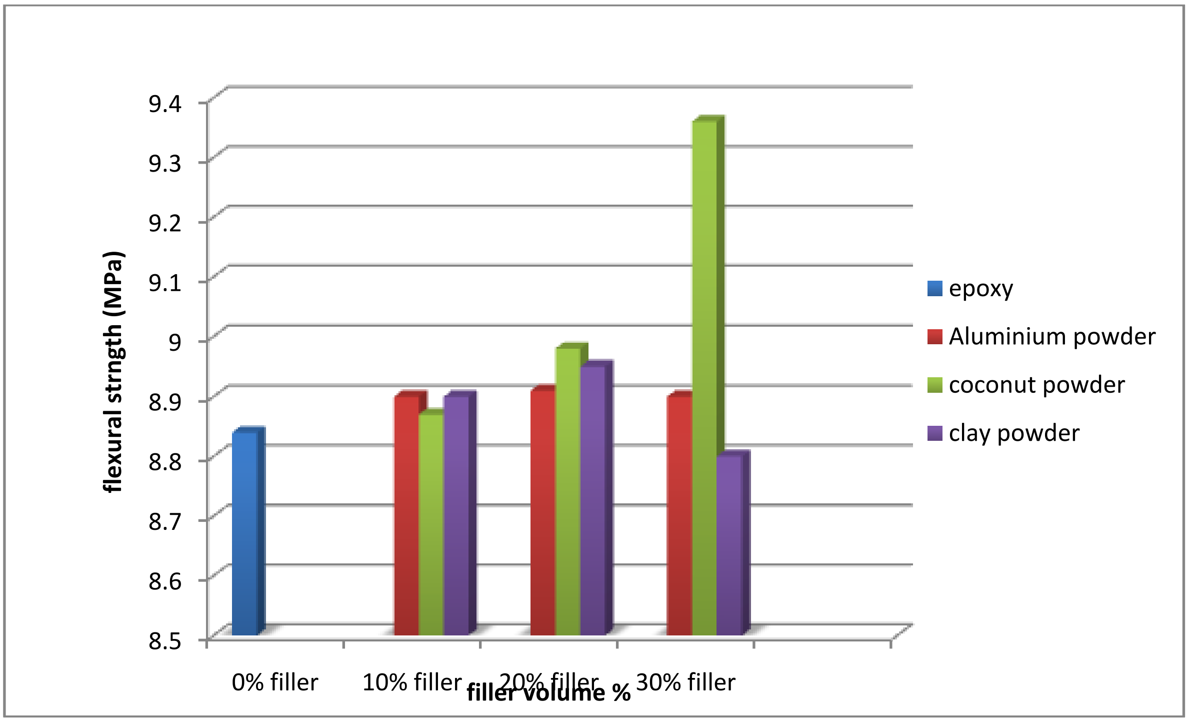


**Figure 3: Graph of flexural strength versus filler loading of epoxy composites (105 μm)**

**Scanning Electron Microscopy**

The SEM images below reveals the state of dispersion of the fillers and the detailed internal structure of the composites. It can be observed from figure 4 that the filler dispersed uniformly in the matrix and fairly strong interfacial bonding exists between the filler and resin. However some few pores and voids can be observed in the micrograph that may be as a result of particle pull out and entrapped air bubbles and these will ultimately affect the composite mechanical properties adversely.

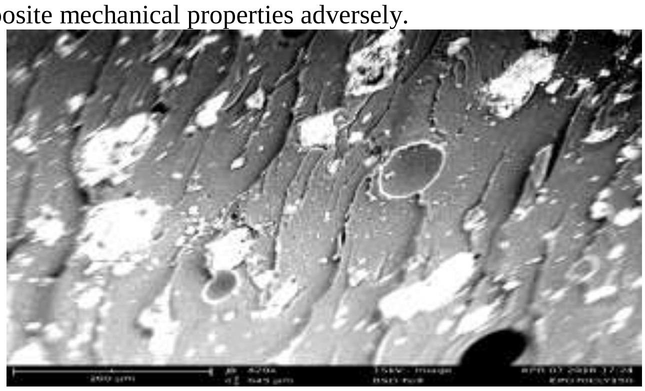

**Figure 4: SEM image Clay powder-filled Epoxy resin**

Figure 5 reveals the image obtained from SEM of coconut shell powder and epoxy matrix composite which shows good dispersion of the coconut powder filler and poor interaction between the filler and the matrix.Relatively high number of voids and pull-outs can be observed. The presence of large volume of voids can lead to crack propagation when bending load is applied to the structure.

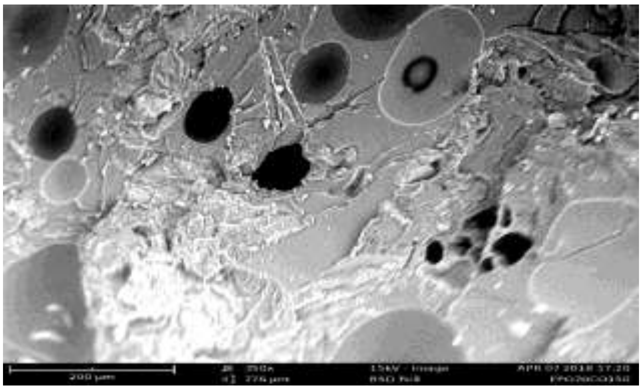

**Figure 5: SEM image of Coconut powder-filled Epoxy resin**

Figure 6 below, shows high amount of voids and poor interaction between the aluminium and the epoxy. However the dispersion of the aluminium particles is good, with less amount of agglomeration of the aluminium particles. Quite a number of line gaps or cracks may also be observed in the aluminium/epoxy micrograph.

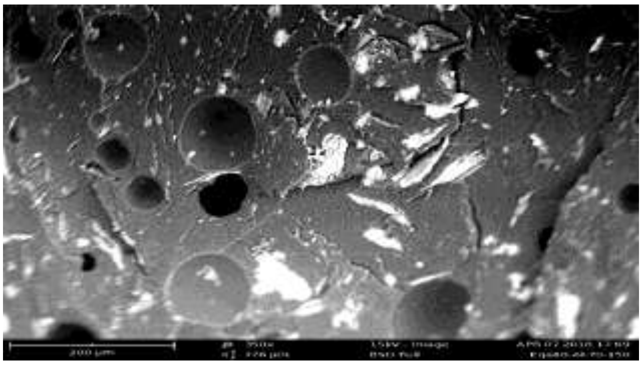

**Figure 6: SEM image of Aluminium-filled Epoxy resin**

**Water Absorption Discussion**

Water absorption test was conducted in accordance to ASTM D570. This test was conducted because in plastics, the moisture content is intimately related to properties suchas electrical insulation, mechanical strength, appearance etc. The effect on the properties depend the type of exposure (either humidity or immersion in water) and the geometry of the part.

**Percentage Water absorption =** $\frac{\textbf{wet weight} - \textbf{dry weight}}{\textbf{dry weight}} * \textbf{100\%}$

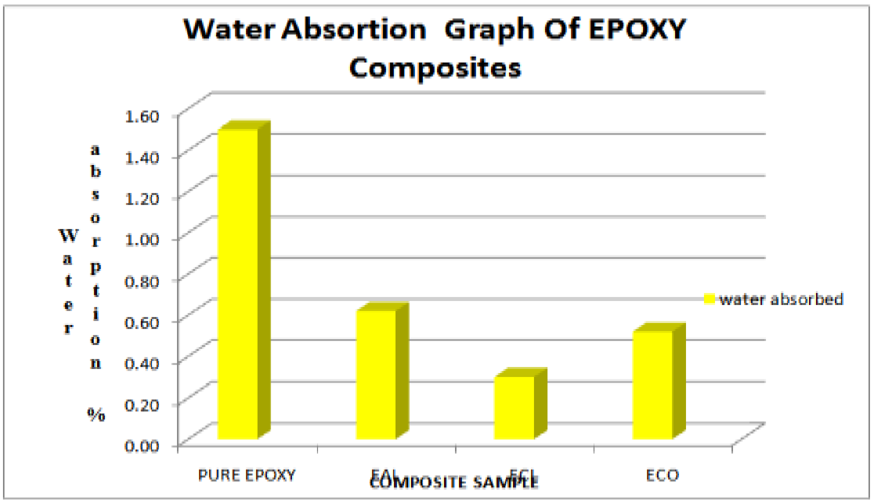


**Figure 7: Water Absorption Graph for epoxy composites**

## IV. CONCLUSION

All three fillers showed that they can be used to modify the properties of Unsaturated Polyester and Epoxy resin, Coconut shell powder which showed the best results in majorly all analysis is most suitable to modify this thermosets. Furthermore, aluminium and clay powder can be used interchangeably as the results from analysis show that they were in most cases close to each other. Epoxy Resin were subjected to FTIR analysis and the results obtained were studied, we were able to deduce that the introduction of certain functional groups and elimination of some functional groups when the fillers were incurred into the polymer matrix is responsible for the modification in strength, flexibility and water retention capacity of this materials. The tensile and flexural test results showed that coconut shell, clay and aluminium powder fillers showed good modification results, after comparing the results obtained. Coconut shell powder showed the best result of all three fillers for the tensile test, while Aluminium powder showed the best flexural result of all three fillers. The SEM plates showed good dispersion, agglomeration and voids in the composites, clay with tiny voids showed the best interfacial bonding as compared to the images obtained from the remaining two samples. Water absorption or retention was improved significantly in the composites, with coconut shell powder filled epoxy resin absorbing more water than that filled with clay and aluminium powder.

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**Table 1: Tensile Strength for Epoxy Composites**

| S/N | FILLER | FILLER % | **75 µm (MPa) *1000** | **105 µm (MPa) *1000** | **150 µm (MPa) *1000** |
|---|---|---|---|---|---|
| 1 | E-AL | **0 %** | 8.56 | 8.56 | 8.56 |
| 2 | E-AL | **10 %** | 8.76 | 8.52 | 8.48 |
| 3 | E-AL | **20%** | 9.11 | 8.52 | 9.48 |
| 4 | E-AL | **30 %** | 8.75 | 9.01 | 9.85 |
| 5 | E-CO | **0 %** | 8.56 | 8.56 | 8.56 |
| 6 | E-CO | **10 %** | 9.11 | 8.57 | 8.73 |
| 7 | E-CO | **20%** | 8.53 | 8.97 | 10.14 |
| 8 | E-CO | **30 %** | 8.50 | 9.33 | 8.76 |
| 9 | E-CL | **0 %** | 8.56 | 8.56 | 8.56 |
| 10 | E-CL | **10 %** | 9.10 | 8.76 | 9.52 |
| 11 | E-CL | **20%** | 8.59 | 8.73 | 9.08 |
| 12 | E-CL | **30 %** | 8.52 | 8.59 | 8.98 |

**Table 2: Flexural Strength For Epoxy Composites**

| S/N | FILLER | FILLER % | 75 µm (MPa) *1000 | 105 µm (MPa) *1000 | 150 µm (MPa) *1000 |
|---|---|---|---|---|---|
| **1** | **E-AL** | **0 %** | 8.84 | 8.84 | 8.84 |
| **2** | **E-AL** | **10 %** | 8.83 | 8.90 | 8.88 |
| **3** | **E-AL** | **20%** | 8.94 | 8.91 | 8.96 |
| **4** | **E-AL** | **30 %** | 8.90 | 8.90 | 8.94 |
| **5** | **E-CO** | **0 %** | 8.84 | 8.84 | 8.84 |
| **6** | **E-CO** | **10 %** | 8.94 | 8.87 | 8.89 |
| **7** | **E-CO** | **20%** | 8.83 | 8.98 | 8.90 |
| **8** | **E-CO** | **30 %** | 8.83 | 9.36 | 8.86 |
| **9** | **E-CL** | **0 %** | 8.84 | 8.84 | 8.84 |
| **10** | **E-CL** | **10 %** | 8.94 | 8.90 | 8.89 |
| **11** | **E-CL** | **20%** | 8.94 | 8.95 | 8.86 |
| **12** | **E-CL** | **30 %** | 9.01 | 8.80 | 8.93 |

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