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## Abstract

On the basis of some similarity in chemical composition with fly ash that has been previously established as an additive in epoxy resin-fly ash composite, the role of wood ash as an additive in the formulation of polymer matrix composite (PMC) was evaluated in varying percentages.

Chemical analysis was carried out using X-ray florescence spectrometry (WDXRE) to determine the chemical composition of wood ash. The compositions were compared with that of fly ash which has been determined by other researchers. The mechanical properties were evaluated and compared with the results previously obtained with the use of fly ash.

On comparative basis, the inclusion of wood ash in the formulation of polymer matrix composite (PMC) resulted in significant increase in impact and tensile strength. While the impact resistance was improved by two times as a result of incorporating wood ash, tensile strength of the PMC was enhanced by more than thirty-six percent (36%). The hardness of the formulated polymer matrix composites increases progressively with the increase in the wood ash content. The microscopic analysis revealed that the wood ash particles were uniformly distributed in the matrix without evidence of segregation.

**Keywords**: Fly ash, fibres, impact strength, polymer matrix composites, tensile strength, wood ash.

#### 1. Introduction

A composite material is a heterogeneous solid consisting of two or more different materials that are mechanically or metallurgically bonded together [8]. Each of the various components retains its identity in the composite and maintains its characteristic structure and properties. The composite material, however, generally possesses a unique combination of properties, such as stiffness, strength, weight, that is not possible with the components by themselves.

Several works have been carried out on the formulation of polymer matrix composites including the addition of various fillers and additives [1-6]. Fly-ash in particular, has been found to increase the comprehensive strength of

polymer matrix composites. In this work, the chemical composition of wood-ash is being determined and compared with that of fly-ash[1]. Their relatives influences on polymer matrix composite is being investigated with the aim of using wood-ash which is readily and cheaply available as property modifier for polymer matrix composites.

Industrially, the term 'plastic' is applied to a polymer to which one or more property-modifying agents have been added. Numerous types of additives are used by manufacturers and fabricators; in fact, virgin polymers are rarely used. Each additive has a specific function it performs, for instance, for service environment, anti-oxidants, anti-ozonants, anti-static agents, flame-radiants, ultraviolet radiation absorbers are used for easier processability, plasticizers are employed for toughness, fillers play major roles as additives in polymers [9].

A wide variety of fillers is used for polymers. In the case of thermosets, substances such as mica, glass fibre and fine sawdust are used to improve engineering properties and to reduce the cost of moulded products [9]. Polytetrafluoroethylene (PTFE) has been used as filler at a quantity level of 15% so as to improve the wear resistance of nylon components. Though polymers are usually non-conductive, they can be made conductive by loading them with appropriate filler such as electromagnetic shielding and specimen mounts in scanning electron micrograph analysis [9].

Additives are widely used for thermoplastics, thermosets, and elastomers. Typically, commercial plastics are mixtures of one or more polymers and a variety of additives such as plasticizers, thermal and light stabilizers, flame-retardant agents, fillers, colorants, processing aids, impact modifiers, and biocides [10]. The largest markets for additives are mostly for fillers and plasticizers [10]. These may be mixed with the polymer before processing by a variety of techniques such as dry blending, extrusion, compounding, compression/ transfer molding, thermoforming, calendaring and other methods [10].

Polymeric composites are physical mixture of a polymer (the matrix) and a reinforcing filler (the dispersed phase) that serves to improve some mechanical property such as modulus or abrasion resistance [11].

For most uses, additives are added to plastics to improve their mechanical properties, offer protection from service environment, reduce their cost, improve their moldability and /or impact colour for identification. These additive constituents are usually classified as fillers, plasticizers, lubricants, coloring agents, stabilizers, antioxidants and flame retardants. Some of the most common additives (fillers) include wood flour, cloth fibers, macerated cloth, glass fibers, asbestos fiber and mica [9] [18].

The high strength-to-weight ratios possessed by these new composites are the key factors that determine the engineering applications of these composites. Their early employment in aviation industry has led to its wide applications in other areas such as leisure, sports, automobile, defence industries and so on [8].

The influence of wood –ash as an additive on the mechanical properties of PMC is being investigated in this work.

# 2. Materials, equipment and experimentation

## 2.1. Materials and equipment

The major raw materials used include: Epoxy resin (Bisphenol-A-Co-Epichlorohydrine) and tetraethylenepentamine (curing agent) which were used as matrix materials in the fabrication of polymer matrix composite (PMC) samples. Wood ash particles were used as an additive while fibre glass from North Carolina State University, USA, was used as reinforcement.

The major equipment used in this research work are: Charpy impact testing machine, model 6703 (30Joules), Hounsfield tensometer, model 1.4F (20KN), Rockwell Hardness Testing Machine, model DFH – 100, hydraulic press with heating and cooling plates model ZBJ (50Tons-500Tons), Vacuum Electrical Oven model HTF 1800 (1400°C), two-high rolling mill, X-Ray Fluorescence Spectrometry, model XGT – 1700WR.

#### 2.2. Research methodology

Various methods are employed in the production of polymer matrix composites. These methods include hand methods (hand lay-up and the sprayup), moulding methods (matched-die moulding, pultrusion, forming methods employing gas pressure and low pressure-closed mould systems) and filament winding [7].

In the ongoing research effort, hand lay-up method was selected for its low production cost, little capital equipment and ease of formulation processes involved [7], [10], [14].

Polymer matrix composite material with glass fibre reinforcement using burnt wood ash as additive powder were homogenously mixed with low viscous epoxy resin. Both the manufacturing technology of PMCs and their resulting mechanical properties were evaluated in this work.

#### 2.3. General experimental procedures

Wood ash was prepared from an approximately 300g of woodchips (hard wood). The woodchips were pyrolyzed by heating to 400°C in a closed container. At the termination of devolatilization, the container lid was removed and the residual char was allowed to burn at 350°C for 5-8hrs. Prolong burning at this low temperature ensured complete burning of woodchips and prevented the burning off of some important elements in the wood ash.

Samples for X-ray fluorescence spectrometry were first finely ground and then poured in a glass tube to hold the ash on to sample compartment of the equipment for analysis. The powder was ground fine to ensure random orientation of the crystals so that there were sufficient amount of crystals to generate detectable signals at all angles.

A hand lay-up processing method in which a 10g of reinforcement (woven mat E-glass fibre) was put down to lie flat in a petri-dish. 3g of liquid epoxy resin was thoroughly dissolved in 15ml of acetone. The dissolved epoxy resin was then homogenously mixed with 0.5mltetraethylenepentamine that served as the curing agent. Thereafter, varying quantities of prepared wood ash (0.0-0.5g) was evenly mixed with the prepared resin-tetraethylenepentamine mixture. The mixture was then poured on 10g of fibre glass (reinforcement) placed in a petri-dish. This was allowed to cure naturally for 24hours and later transferred into vacuum electric oven for two hours at temperature of 60°C to attain uniform curing of the samples. Following this procedure, all the polymer matrix composite (PMC) samples were prepared and further processed for various experimental analyses.

The nomenclature shown in Table 1 below was used for identification of different compositions of wood ash in the fabricated composite (specimens). Based on the aforementioned procedure, specimen samples of the formulated polymer matrix with maximum thickness of 4mm were produced.

To meet the minimum standard thickness of 10mm required for the impact test, sandwiching of the layers of the produced polymer matrix was

adopted yielding impact specimen geometry of

10mm by 10mm by 100mm.

**Table 1: Formulated Polymer Matrix Composite Compositions** 

<b>Specimen Designation</b>	Qty of wood ash (%)	Qty of resin (%)	Qty of fibre glass
			(%)
A1	0.0	23.0	77.0
A2	0.8	23.0	76.2
A3	1.5	22.5	76.0
A4	2.3	22.7	75.0
A5	3.0	22.0	75.0
A6	3.7	21.3	74.0

The compounding process was carried out on the prepared samples with the aim of producing a unit specimen that would prevent delamination of sandwiched composite layers. This was achieved by blending natural butyldene rubber, high density

polyethylene (HDPE), sulphur, mecaptobenzothiazole (MBT), glass powder and trimethylquinolone (TMQ) in line with the quantities shown in table 2.

Table 2: Composition of Raw Materials for compounding process

Material	Total Weight (g)	% Composition		
High Density Polyethylene	210	56.4		
(HDPE)				
Sulphur	2	0.5		
Nitrile-butadiene Rubber (NBR)	90	24.1		
Mecaptobenzothiazole (MBT)	6	1.6		
Glass powder	60	16.1		
Trimethylquinolone (TMQ)	4.5	1.2		

Source: Akindapo, Oyinlola, Agboola [14]

The formulation was prepared by blending the raw materials at a temperature of  $170^{\circ}$ C using 2-high rolling machine. It was then pressed into a sheetform using compression moulding machine. The specimens were wrapped with the sheet forming an additional thickness of about 3-4mm around the rectangular surfaces of the fabricated composite. The prepared samples were placed in a metallic mould where they were pressed using hydraulic press at a temperature of  $150^{\circ}$ C and pressure of 7.5bar.

The compounded material were taken out of the mould and then carefully machined to standard geometry of 4mm by 10mm by 100mm for tensile test and 10mm by 10mm by 100mm for impact test.

#### 3. Mechanical tests

#### 3.1. Tensile test

The maximum tensile load that could be applied before the fabricated polymer matrix composite fractured was determined by tensile test in accordance with ASTMD 638 standard of measurement. The configuration of specimen was 4mm by 10mm by 100mm. The tensile testing machine was employed in subjecting the specimen to an axial elongation. The resultant loads on the specimens were measured at fracture.

#### 3.2. Impact test

The degree of resistance of the formulated composites to impact loading was determined by means of the charpy impact testing method in accordance with ASTMD 6110 standard method of measurement. A 25J of load was released at a velocity of 5.24m/s on the specimens while the various values of energy dissipated before fractures were recorded.

#### 3.3. Hardness test

The resistance to deformation and penetration was determined by Rockwell hardness testing machine. Polymer matrix composite samples were polished to give a very smooth surface. The hardness of the specimen was then measured using the F-scale of the equipment in accordance with ASTM D785 standard method of measurement.

#### 3.4. Microstructural examination

The microscopic analysis of the prepared samples was carried out to observe the distribution of wood ash particles in the matrix, resin-wood ash interface and glass fibre distribution in the matrix. Small fractured composite sample from impact test analysis was collected in a cleared and cleaned petri-dish and carefully studied under an electron microscope at magnifications of 100 and 200.

#### 4. Results and discussion

## 4.1. Chemical analysis of wood ash

The wood ash was analyzed for different concentration of chemical compounds using X-

Ray Fluorescence Spectrometry (WDXRE) and the result obtained which is comparable with the fly ash is indicated in table 3 below.

Table 3: Chemical Compositions of Wood Ash

Compound	CaO	K <sub>2</sub> O	$SiO_2$	$Al_2O_3$	$SO_3$	TiO <sub>2</sub>	MnO	Fe <sub>2</sub> O <sub>3</sub>	ZnO
Conc. (%)	78.50	8.79	7.20	0	1.10	0.31	0.09	1.27	0.06
C1	T/ O	D O	C.	O T	^	D . O	Mao	т	. 1
Compound	$Y_2O_3$	BaO	Ce	$O_2$	$u_2O_3$	$Re_2O_7$	MgO	10	otal

## 4.2. Tensile, impact and hardness tests

Variation of tensile, impact and hardness properties of the fabricated polymer matrix

composites under the investigated percentages of wood ash additive is presented in table 4.

Table 4: Variation of Tensile, Impact and Hardness Values with Various Percentages of Wood Ash as Additive

Specimen Designation	Qty of wood ash (%)	Qty of resin (%)	Qty of fibre glass (%)	Tensile Strength (N/mm²)	Impact Strength (J/mm²)	Hardness (HRF)
A1	0.0	23.0	77.0	76	50	23.4
A2	0.8	23.0	76.2	80	112	23.6
A3	1.5	22.5	76.0	96	105	24.0
A4	2.3	22.7	75.0	104	105	25.4
A5	3.0	22.0	75.0	102.7	78	26.9
A6	3.7	21.3	74.0	73.3	70	27.5

## 4.3. Miscroscopic Study of Fractured Surfaces

Micrographs showing the distributions of wood ash particles in the matrix and glass fibre-matrix

interfacial relationships are presented in Plates 1to 3

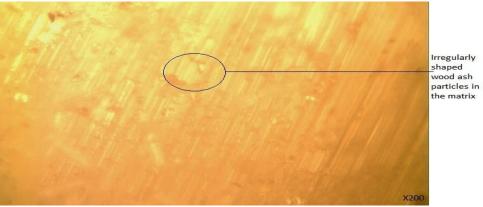
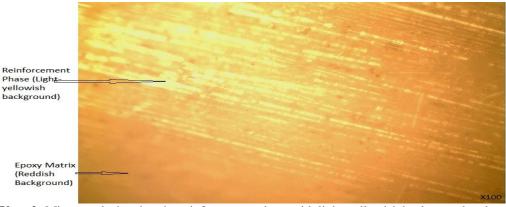


Plate 1: Micrograph showing the presence of irregularly shaped wood ash particles in the matrix (X200)



**Plate 2:** Micrograph showing the reinforcement phase with light-yellowish background and epoxy matrix with reddish background (X100)



**Plate 3:** Micrograph showing the disappearing reinforcement phase after rolling, reddish background showing the epoxy phase and dark background in the presence of NBR and HDPE mixture (X200)

#### 5. Discussion of results

#### 5.1. Chemical analysis of wood ash

On comparative basis the chemical compositions of the investigated wood ash has similar chemical compound but in varying compositions with the fly ash that has been established as an effective additive in polymer matrix composite.

The three major compositions in fly ash are  $SiO_2$  (54.9%),  $Al_2O_3$  (25.8%) and CaO (8.7%) [1], while wood ash has  $SiO_2$  (7.2%),  $K_2O$  (8.79%) and CaO (78.5%) as its major compositions.

Silica is generally used as abrasive material because of its hardness [15]. Therefore,  $SiO_2$  improves the hardness as well as chemical and thermal resistance of PMC.

Calcium oxide or quicklime (CaO) is a key ingredient in the wood ash. The work revealed that CaO improved the hardness and thermal resistance of the PMC as well as enhancing the bonding between the polymer matrix composite compositions.

Potassium oxide ( $K_2O$ ) is highly insoluble and thermally stable. It is non conductive and suitable for glass optics and ceramics applications. It is therefore used in improving the thermal and conductivity resistance of the matrix.

Aluminium oxide  $(Al_2O_3)$  which was present in fly-ash is cuspicuosly absent in wood ash is fairly chemically inert and white, thereby, is a favoured filler for plastics [15]. It is also known for its hardness and strength and therefore can be used to resist abrasive wear.

#### 5.2. Tensile strength

The various values of tensile strength of newly developed polymer matrix composites (PMCs) with different percentages of wood ash content as shown in figure 1 reveals a progressive increase in tensile strength of composite with increase of wood ash. The addition of 2.3% wood ash (sample **A4**) yielded an ultimate tensile strength (UTS) value of 104N/mm<sup>2</sup>. Addition of wood ash beyond 2.3% gave rise to a decrease in tensile strength due to reduction in the percentage of the matrix (epoxy

resin) that binds the composite. A brittle fracture, type of a failure, was noticed in the specimen. There was no observable plastic deformation or work hardening before the failure of the specimen except in specimen **A4** (2.3% wood ash). This is

traced to the fact that 2.3% of wood ash was able to modify the molecular arrangement of the polymeric composite which permitted little structural plastic flow.

## Tensile Strength (N/mm<sup>2</sup>)

150

100

50

0

0 0.8 1.5 2.3 3 3.7

## % Wood Ash

Fig. 1: Variation of Tensile Strength of the Formulated PMC with Percentage Wood Ash as Additive

Comparatively, the strength of the epoxy resin-fly ash composite increased with the fly ash content but at much higher percentage level (fig.2) compared with the newly formulated wood ash-composite. This could

be traced to the fact that **CaO** in wood ash gives a better binding property with toughness as it is the key ingredient in wood ash [11] therefore; smaller quantity was needed in the case of wood ash additive.

## Compressive Strength (N/mm<sup>2</sup>)

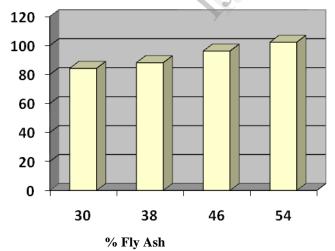


Fig.2: Variation of Compressive Strength of Resin-Fly Ash Composite with Percentage Fly Ash as Additive [5]

#### **5.3.** Impact strength

Figure 3 indicates that 0.8% addition of wood ash led to a significant increase in impact strength from 50 J/mm<sup>2</sup> (sample A1: Without wood ash) to 112 J/mm<sup>2</sup> (sample A2). Slight decrease in impact energy was observed from specimens A3 and A4. However, sharp decrease in impact strength was noticed from 3% addition of wood ash and above. This behaviour could be attributed to the effect of

wood ash on molecular flexibility of the polymer. Molecular flexibility plays an important role in determining the relative brittleness or toughness of the material [12]. This means the composite specimen was stiff, that is, the molecular segments were unable to disentangle and respond to the rapid application of mechanical stress and the impact produced brittle failure. Also, the decreased quantity of epoxy material with

increased particles of wood ash contributed to the observed brittle fracture with lesser energy.

## Impact Strength (J/mm<sup>2</sup>)

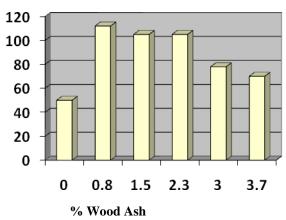


Fig. 3: Variation of Impact Strength of the Formulated PMC with Percentage Wood Ash as Additive

Comparatively, the impact strength of epoxy resin-fly ash composite also decreased with the fly ash content as shown in fig. 4 [1]. This followed the same trend

with the outcome of the epoxy resin-wood ash composite formulation in this research work.

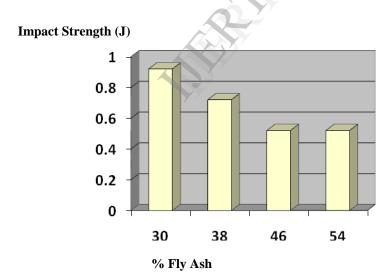


Fig. 4: Variation of Impact Strength of Resin-Fly Ash Composite with Percentage Fly Ash as Additive [5]

#### 5.4. Hardness test

Figure 5 shows that the hardness of the formulated polymer matrix composite increases progressively with the increase of wood ash content. This is because wood ash has CaO and SiO<sub>2</sub> which has

Hardness (HRF)

tendencies of improving the hardness and abrasive properties of the formulated polymeric composite. This affirms the role of additive in enhancing the hardness property of the polymeric materials in general.

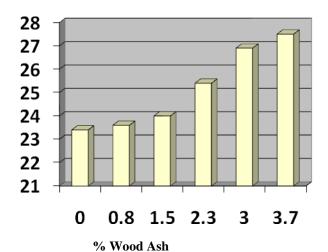


Fig. 5: Variation of Hardness Property of the Formulated PMC with Percentage Wood Ash as Additive

## 5.5. Microscopic study

In the microscopic analysis, it has been found that wood ash particles are uniformly distributed in the matrix. There was no evidence of wood ash particles segregation in the matrix. Also, the micro-examination of the prepared specimen shows clearly the two obvious phases of fibre reinforcement and the epoxy resin as matrix (plate 1& 2). The fibre reinforcement phase almost disappeared after the rolling and compounding of the composite with NBR and HDPE (plate 3).

## **6.** Conclusion and recommendations

#### 6.1. Conclusion

Polymer matrix composite with improved toughness and processability, which may be used for military and industrial purposes, by means of the incorporated wood ash additive has been successfully formulated in this research work. The results obtained in this work is in aggrement with the work of other researchers such as manoj single and Vickers chawla [1]. The following conclusions can be drawn from the research work:

- i. Lower quantity of wood ash was required as an additive in the newly formulated polymer matrix composite in contrast to fly ash that was needed in larger quantity for the previously established fly ash-composite.
- ii. Among the formulated samples, the addition of 2.3% wood ash revealed mechanical properties acceptable for the formulation of polymer matrix composite for industrial applications.
- iii. Additionally, wood ash was discovered to have good interfacial relationships with the matrix (epoxy) and fibre glass since there was no evidence of wood ash segregation in the formulated composite.

#### 6.2. Recommendations

It is hereby recommended that further mechanical tests such as ballistic test, dynamic mechanical analysis (DMA), flexural test and heat deflection temperature analysis be carried out on the prepared epoxy resin-wood ash composite in order to determine the ballistic properties and degradation modes of the composite.

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