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MECHANICAL BEHAVIOUR OF OIL PALM ASH-OIL PALM FIBRE HYBRID REINFORCED POLYESTER MATRIX COMPOSITES

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Abstract: The influence of oil palm ash-oil palm fibre weight ratio on the tensile stress-strain behaviour, tensile properties and flexural strength of polyester matrix composites has been investigated. Open moulding method was used to produce the polyester hybrid composites of 2, 4, and 6 wt% reinforcement consisting of oil palm ash (OPA) and oil palm fiber (OPF) in weight ratios of 1:0, 3:1, 1:1, 1:3, and 0:1 respectively. Density measurement, tensile and flexural test were used to characterize the composites produced. The results show that the open mould casting method adopted for the production of the composites was reliable judging from the low porosity levels (<0.5%) in the composites. The polyester matrix composites produced exhibited the same stress-strain deformation behavior as the unreinforced polyester irrespective of the reinforcement weight percent and weight ratio of OPA and OPF in the composites. However, the tensile strength, specific strength, strain to fracture, strain energy absorbed, and flexural strength of a good number of the composites produced were superior to that of the unreinforced polyester. The effect of the hybrid mix of OPA and OPF (using different weight ratios) on the mechanical behaviour of the composites produced. Keywords: polymer matrix composites produced.

1. INTRODUCTION

Before the last four decades, it was rather difficult to conceive of the design of high performance materials which were not metallic based. Today, due to the advances in materials science and technology, ceramics and polymer based materials for structural and other high performance applications are now available [1-3]. Composite materials with polymer matrices are competing favourably with metal based materials for a good number of industrial applications and in a number of cases have exhibited higher level of competence [4]. This is due to property advantages of polymer matrix composites (PMCs) such as versatile processing and fabrication routes, low processing cost, good wettability between the polymer matrices and the reinforcing materials, good range of mechanical properties among others [5-6]. There has been a general drive towards the reduction of the cost of producing PMCs while at the same time improving the mechanical and physical properties [7]. This has given impetus to research works targeted towards the use of agro based waste materials such as natural fibres and ashes processed from controlled burning of plant leaves and stems as reinforcements. The advantages of the agro wastes (natural fibres and fillers) are availability in large quantities, low cost of processing, low density, acceptable specific strength, good thermal insulation properties, reduced health threats such as dermal and respiratory irritation common with the use of synthetic reinforcing materials, renewability and ease of recycling, and creation of a more eco-friendly environment [8].

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There exists an enormous corpus of literatures on natural fibre and filler reinforced PMCs which have been applied in the design of components and parts such as door panels, seat backs, headliners, dashboards, interior parts, package trays, furniture, packaging, building and constructions and, even military vehicles and aircraft spare parts among others [9-11]. For these applications, polyester based composites are notably one of the most utilized PMCs. Polyester is a prominent thermosetting polymer which has been used widely for the development of polymer based composites. Its attraction is mainly due to its low cost in comparison with other notable thermosetting polymer matrix materials like epoxy, good cross linking tendency, excellent process-ability, its good wetting characteristics, and average mechanical properties when cured [12-13].

One of the most abundant agro resources in Nigeria is oil palm fruit tree. After the extraction of the oil from the fruit on maturation, the fibres are often discarded though in some cases used in the processing of paper products, building materials, absorbents and animal feeds [14]. It has also been successfully explored as reinforcement for PMC production [15-17]. However, there is currently limited literature on the use of oil palm filler-fibre as hybrid reinforcement in PMCs. The motivation for choosing to incorporate oil palm filler-fibre hybrid reinforcement into polyester is to ascertain the viability of developing polyester matrix composites with improved mechanical properties beyond the levels achievable with the use of only oil palm filler or oil palm fibres. The processing method does not result in additional production cost and efforts centred on the use of hybrid reinforcements of fillers and fibres have promise as it aims at harnessing the property advantages of both types of reinforcements [18-19]. In the present study, the mechanical behaviour of polyester matrix composites reinforced with varied weight ratios of oil palm ash and oil palm fibre is investigated.

2. MATERIALS AND METHODS

2.1. Materials. The materials utilized in this research work are: Unsaturated Polyester (UP) resin as-supplied with stability below 25°C (which served as matrix for the composites to be developed). Methyl Ethyl Ketone Peroxide (MEKP) and Cobalt Naphthanate which were used as the initiator and catalyst respectively were procured for the curing of the polyester matrices. Also oil palm fruit fibres which were utilized for the preparation of the reinforcements for the composite were obtained from an oil processing mill.

2.2. Reinforcement Preparation - Fibre Preparation. The oil palm fibres selected for direct use as fibre reinforcements were chemically treated to improve wetability between the fibres and the polyester matrix and reduction of moisture absorption capacity. The chemical treatment was performed using 0.1M NaOH in a water bath maintained at a temperature of 50°C for 2-4 hours. The Fibres were removed from the water bath and rinsed several times to remove any NaOH solution sticking to fiber surface and sun dried. The oil palm fibres before and after chemical treatment are presented in Figure 1.



Figure 1: Oil palm fibres (a) before chemical treatment, and (b) after chemical treatment **2.3. Filler Preparation.** The reinforcing filler was produced by burning some sun dried oil palm fibres using a metallic drum which served as burner. The dried oil palm fibres were ignited using charcoal and allowed to burn completely inside the drum. The ash obtained from the process was thereafter conditioned in a furnace at a temperature of 550°C for 4 hours to reduce the volatile and

b)

carbonaceous constituents of the ash. Sieve size analysis was carried out on the conditioned ash using a digital sieve shaker. Particle sizes below $50\mu m$ were utilized as the reinforcing filler. The nominal chemical composition of the oil palm ash is presented in Table 1. The oil palm ashes before and after the thermal conditioning is presented in

Figure 2.

Figure 1: Oil plam ash (a) before heat-treatment, and (b) after heat treatment

2.3. Composite Fabrication. Open moulding method was used to prepare the oil palm ash-fibre reinforced polyester hybrid composites. The process started with the determination of the quantities of oil palm ash (OPA) and Oil Palm Fibre (OPF) required to produce 2, 4, and 6 wt% reinforcement consisting of OPA and OPF in weight ratios of 0:1, 1:3, 1:1, 3:1, and 1:0 respectively (which amounts to 0, 25, 50, 75, and 100% OPA in the reinforcement phase). The determined

quantities of the polyester resin, OPA and OPF particles were measured on an electronic weighing balance. The polyester was poured into a beaker; 2% of Cobalt Naphthanate (catalyst) and 1% of Methyl Ethyl Ketone Peroxide (MEKP) (accelerator) were added and stirred with a rod manually to achieve homogenization. Thereafter, the reinforcing phases were added and stirred for few minutes until an even dispersion was achieved. The mixture was then

cast into already prepared aluminum moulds (Figure 3) which were pre-coated with polyvinyl alcohol (PVA) for easy removal of the cast samples from the moulds. The mixture was left for about one and half hours in the mould to cure before they were removed. This procedure was repeated for all samples prepared.

2.4. Density Measurement. The experimental density of each composition of composite produced was determined by dividing the measured weight of a test sample by its measured volume; while the theoretical density was evaluated by using the formula:

 $Q_{\text{polyester}/\text{OPA-OPF}} = (Wt_{\text{f} \text{ polyester}} \times Q_{\text{polyester}}) + (Wt_{\text{f}.\text{OPA}} \times Q_{\text{OPA}}) + (Wt_{\text{f}.\text{OPF}} \times Q_{\text{OPF}})$ (2.1)

where Qpolyester/OPA-OPF presents density of composite, Wtf. polyester and Qpolyester represents weight fraction and density of polyester respectively, wtf.OPA and QOPA represents weight fraction and density of oil palm ash respectively, while wtf.OPF and QOPF represents weight fraction and density of oil palm fibre respectively.

The experimental densities for each composition of the polyester matrix composites produced were compared with their respective theoretical densities; and it served as basis for evaluation of the percent porosity of the composites using the relations [21]:

% porosity = {
$$(QT - QEX) \div QT$$
} × 100% (2.2)

where, QT = Theoretical Density (g/cm³), QEX = Experimental Density (g/cm³)

2.5. Tensile Test. The stress – strain behaviour and tensile properties of the composites were evaluated with the aid of tensile tests performed following standard procedures in accordance with ASTM D3038M-08 [22]. The test was performed at room temperature using an Instron universal testing machine operated at a strain rate of 10⁻³/s. Three repeat tests were performed for



Figure 3. Composites in open mould

Table 1: Nominal Chemical Composition of Palm Oil Ash Khairunisa and Mobd [20]

Khairunisa and Mohd [20]				
Chemical constituent	OPA (%)			
SiO ₂	65.20			
Al ₂ O ₃	4.48			
Fe ₂ O ₃	5.44			
CaO	4.12			
MgO	2.25			
Na ₂ O	0.1			
K ₂ O	2.28			
SO ₃	2.25			
Loss on Ignition	13.86			

each composition of the composites produced to guarantee repeatability and reliability of the data generated. The tensile properties evaluated from the tension test are - the ultimate tensile strength (σ_u), the specific strength, the strain to fracture (ε_f) and strain energy absorbed (U).

2.6. Flexural Test. The flexural strength of the composites was evaluated by performing flexural three-point bending tests on the composites. The test was performed at room temperature using a tensiometric universal testing machine operated at a crosshead speed of 0.3mm/min. The testing procedure and flexural strength determination were performed in accordance with ASTM D7264M-07 standard [23].

3. RESULTS AND DISCUSSION

3.1. Density. The densities of the OPA-OPF reinforced Polyester hybrid composites produced are presented in Table 2. The densities of the OPA and OPF were found to be 1.1g/cm³ and 0.73g/cm³ respectively in comparison with the polyester resin which has density of 1.2 g/cm³. It is observed that there is a general decrease in the density of the composites with increase in the weight percent of the reinforcing phase that is from 2 to 6 wt% reinforcement in the composites. Also, it could be seen that the density of the composites decreases with increase in OPF content is obviously due to the lower density of the oil palm fibres (OPF) in comparison the oil palm ash (OPA). Hence, it can be stated that the combination of OPA-OPF hybrid reinforcing materials in the polyester matrix does not result in increased weight of the composites produced. It is also noted that the porosity levels in the composites are less than 0.5% which indicates that the open mould fabrication method adopted for the production of the composites is reliable.

Sample Designation	Composition OPA: OPF	Theoretical density (g/cm ³)	Experimental density (g/cm ³)	% Porosity
A0	0%wt	1.2	1.190	0.84
	2%wt			
X1	(1:0)	1.198	1.196	0.17
X2	(3:1)	1.196	1.195	0.084
X3	(1:1)	1.194	1.193	0.084
X4	(1:3)	1.193	1.191	0.17
X5	(0:1)	1.190	1.189	0.084
	4%wt			
Y1	(1:0)	1.196	1.194	0.17
Y2	(3:1)	1.192	1.191	0.084
Y3	(1:1)	1.187	1.185	0.17
Y4	(1:3)	1.185	1.182	0.25
Y5	(0:1)	1.181	1.1798	0.1
	6%wt			
Z1	(1:0)	1.194	1.190	0.34
Z2	(3:1)	1.189	1.187	0.17
Z3	(1:1)	1.183	1.179	0.34
Z4	(1:3)	1.177	1.174	0.26
Z5	(0:1)	1.172	1.170	0.17

 Table 2: Sample Designations with Densities of the Composites Produced

3.2. Stress strain behavior. The tensile stress-strain plots of the composites produced are presented in Figure 4. The general observation is that the tensile deformation behaviour of the composites is similar to that of the unreinforced polyester and is seen to progress in three well defined deformation stages as idealized in the schematic model presented in Figure 5. Thus it is noted that the deformation mechanism of the polyester matrix is very influential in explaining the deformation behaviour of the composites. Stage I is characterized by linear elastic deformation in which the stress acting on the composites is observed to increase linearly with strain; stage II which is characterized by visco-elastic deformation behaviour where it is observed that the plastic strain increases at a constant plastic (yield) stress; and stage III which is characterized by work hardening as the stress increases significantly with further increases in plastic strain. The

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deformation mechanism in stage I can be attributed to the little resistance posed by the composites to the stretching of the atomic bonds in polyester matrix which is a thermosetting polymer. At this stage, elastic deformation is influenced by the stretching of the polyester chains, a process which is reversible once the applied stress is removed. For stage II deformation the constant yield stress accompanying the increasing plastic strain of the composites may be attributed to the unwinding of the cross linked bonds in thermosetting polymers. This process can thus be sustained at low stress levels without accompanying increases in stress. The behavior observed in state III is due to the combined effect of the polyester chains motion past each other and the strengthening due to the presence of the reinforcing particles/fibres. The fibre/particle strength contributes immensely to the higher stress levels the polyester matrix sustains before fracture.



Figure 4: Stress - strain curves for the polyester hybrid composite with (a) 2 wt %, (b) 4wt%, and (c) 6 wt% oil palm ash – oil palm fibre reinforcement.



Figure 5: Idealized Stress - Strain tensile deformation schematic model for the Polyester matrix composites produced showing the three deformation stages

3.3. Tensile Properties. The results of the tensile properties of the composites produced are presented in Figures 5-8. It is observed from Figure 5 that majority of the composites had superior tensile strength values in comparison with the unreinforced polyester. In the case of the 2 wt% OPA-OPF reinforced composites (X series) it is observed that it is the composite composition containing only OPA (X1) that had superior tensile strength in comparison with the unreinforced polyester and the other grades of the polyester composites produced. It is also

observed that there appears to be a reduction of tensile strength for the OPA-OPF hybrid reinforcement with increase in the OPF weight ratio. However the composite grade containing only OPF is observed to also have superior tensile strength in comparison with the hybrid composite compositions suggesting that the hybrid compositions are not favorable for tensile strength improvement when 2 wt % of the reinforcements is utilized. It is noted that the increase in tensile strength for sample X1 (containing only OPA) is 52.13 % greater than that of the unreinforced polyester, 21.7% for composite containing only OPF (X5) and 28.9% for the hybrid composite composition (X2) with the best tensile strength value. For the 4 wt % reinforcement, it is observed that the tensile strength of the composites increases with increase in the weight ratio of OPF in the reinforcement reaching the maximum for X4 (having OPA-OPF weight ratio of 1:3)

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Thus it could be seen that the hybrid composition of OPA-OPF resulted in improvement in the tensile strength of the composites for this weight percent. The composite grade containing only OPF (Y5) is observed to have tensile strength values lower than that of the hybrid composite grades which is contrast with the behaviour observed for the 2 wt% composite grade containing only fibre reinforcement (X5). The hybrid composite grade Y4 having OPA: OPF weight ratio of 1:3 had the best tensile strength for all compositions and weight percents of composites produced in this research. When compared with A0 (which is the unreinforced polyester), Y1 (which is reinforced with only OPA) and Y5 (which is reinforced with only OPF), tensile strength increase of 64.9%, 28% and 45.9% respectively were obtained. For the 6 wt% a more consistent trend is observed with the tensile strength of the composites decreasing with increase in the amount of OPF in the reinforcement. The composite grade containing only OPF is observed to have the least tensile strength for this group of composites. Thus as far as tensile strength improvement is to be considered, the hybrid reinforcement of OPA and OPF appears to have good synergy when 4 wt% reinforcing phase is utilized. Also observe that for the single reinforced grades (X1, X5; Y1, Y5; Z1, Z5), the tensile strength of the composites decreases with increase in the weight percent of the reinforcements. For the hybrid composite grades, no consistent trend was observed with respect to weight percent but it is noticed that the 4 wt% reinforcement appeared to give the best tensile strength in comparison with the 2 and 6 wt % OPA-OPF reinforced composite grades.





Figure 5: Variation of Ultimate Tensile strength of hybrid reinforced Polyester/OPA-OPF composite

Figure 6: Variation of specific strength of hybrid reinforced Polyester/OPA-OPF composite

Figure 6 shows the plots of the specific strengths for the 2, 4, and 6 wt% OPA-OPF reinforced polyester composites produced. It is observed that the same trend observed for the tensile strength is followed in the case of the specific strength results. Sample Y4 which had the highest tensile strength of all grades of composites produced is observed to have specific strength increases of 66, 29.23, and 45.5% with respect to the unreinforced polyester (A0), Y1 (containing 4 wt% of OPA only), and Y5 (containing 4 wt% of OPF only) respectively. This shows clearly that the use of the hybrid OPA-OPF combination in ratio 1:3 results in improvement of the strength to weight ratio of the polyester matrix composites.





Figure 7. Variation of strain to fracture of hybrid reinforced Polyester/OPA-OPF composite Figure 8: Variation of strain to energy stored of hybrid reinforced Polyester/OPA-OPF composite

The results of the strains to fracture of the composite grades produced are presented in Figure 7. It is observed that there is no consistent trend of variation of the strains to fracture for all weight percents and weight ratios of the composites. It is however noticed that the best strain to fracture was obtained from sample Y4 (the 4 wt % reinforcement composition containing OPA: OPF in

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weight ratio 1:3) which incidentally had the best tensile and specific strength values. Also the strains to fracture of the 4 wt% reinforced composite grades were on the average superior to those of the 2 and 6 wt% reinforced composite grades. This is an indication that 4wt% reinforcement with OPA-OPF offers relatively the best tensile properties when the three weight percents worked on in this research are compared. The unpredictable strain to fracture behaviour may be due to the complexity of the interfaces formed between the filler, fibres and the polyester matrix [24].

The results of the strain energy absorbed (modulus of toughness) the composites produced is presented in Figure 8. It is observed that for the composite with 2%wt and 6%wt reinforcement, the strain energy stored (toughness) of the composites decreases with reducing ash content in the reinforcement (with the exception of X4). For the 4 wt% composite grade it is observed that sample Y4 (with weight ratio OPA: OPF of 1:3) had the highest strain energy absorbed. The strain energy absorbed is a measure of the amount of energy absorbed per unit volume at fracture. It provides a good measure of the ductility and well as the toughness of a material. Thus it could be said that sample Y4 has the best combination of strength, toughness, and ductility of all the composites grades produced.

3.6. Flexural Strength. The flexural strength values of the composites produced are presented in Figure 9. It is observed that the flexural strength which is a measure of a materials resistance to

deformation under load is higher for the composite grades containing only OPA. It is also observed that the flexural strength increases with increase in weight percent for the composite grades containing only OPA (X1, Y1, Z1). It is apparent that the hybrid mix of OPA and OPF in the composites reduced the flexural strength of the composites. This may be due to the complexity of the interface formed between



Figure 9: Variation of strain to flexural strength of hybrid reinforced Polyester/OPA-OPF composite

the ash, fibres, and the polyester matrix. For the 2 wt% and 6 wt%, this reduction is noticed to decrease with increase in the OPF weight ratio, but for the 4 wt% there is a departure from the afore-mentioned trend as slight increase in the flexural strength was observed with increase in the weight ratio of OPF in the OPA-OPF hybrid reinforced composite grades.

4. CONCLUSIONS

The influence of oil palm ash-oil palm fibre weight ratio on the tensile stress-strain behaviour, tensile properties and flexural strength of polyester matrix composites has been studied. From the results obtained, the following conclusions are drawn:

- The open mould casting method adopted for the production of the composites was reliable judging from the low porosity levels (< 2%) in the composites.
- The polyester matrix composites produced exhibited the same stress-strain deformation behaviour as the unreinforced polyester irrespective of the reinforcement weight percent and weight ratio of OPA and OPF in the composites.
- The tensile strength, specific strength, strain to fracture, strain energy absorbed, and flexural strength of a good number of the composites produced were superior to that of the unreinforced polyester.
- The effect of the hybrid mix of OPA and OPF (using different weight ratios) on the mechanical behaviour of the composites was not consistent for all the weight percents reinforcement worked on.
- The composite grade containing 4 wt % OPA and OPF in weight ratio 1:3, had the best combination of mechanical properties of all the composites produced.

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