

Effects of Cure Temperature on the Mechanical Properties of Coconut Shell and Snail Shell Particles Reinforced Polyester Matrix Composites

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Abstract:

The effect of cure temperature on the mechanical properties and glass transition temperature of snail shell and coconut shell reinforced polyester matrix composites was investigated in this study. Two batches of polymer composites with snail shell and coconut shell particles reinforcements of varied proportions from 5 – 25 wt. % were prepared by stir casting method. One batch of the samples was cured at room temperature while the other batch was heated to 1500 C and held for four hours. All the samples were subjected to tensile, impact, flexural, hardness and glass transition tests. Mechanical tests revealed that the samples cured at 1500 C exhibited better mechanical properties than the samples cured at room temperature. This could be as a result of better crosslinking within the post cured polyester composites. The micrographs revealed fair dispersion of the reinforcements within the matrix which contributed to the enhancement of the mechanical properties of the composites. The coconut shell and snail shell particles reinforced polyester composites at 20 wt. % and 10 wt. % reinforcements exhibited the highest resistance to elongation with values of 230.01 MPa and 193.64 MPa respectively. The snail shell particles reinforced composite and the coconut shell particles reinforced composite exhibited highest impact energy of 5.87 J and 5.40 J respectively at 15 wt. % reinforcement. The snail shell and coconut shell particles reinforced composites exhibited high hardness of 143.26 HV and 130.53 HV respectively. The improved properties exhibited by these composites is an indication of their suitability for engineering applications.

Keywords: Polyester Composite, Coconut Shell, Snail Shell, Cure Temperature, Mechanical Properties

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1. INTRODUCTION

Polymers may be thermoplastics, thermosets or elastomers. Thermoplastics can be reheated, softened and molded into new shapes while thermosets are hard and stiff cross-linked materials that cannot be softened or be molded when heated. Several types of polymers are being used as matrices in the development of composites. The most commonly used thermoset polymers are unsaturated polyester resins, epoxy resins, vinyl esters, phenolic, novolac and polyamide [1].

Unsaturated polyester resins (UPRs) are cross-linked by reaction with an unsaturated monomer or pre-polymer by virtue of the presence of carbon-to-carbon double

bonds in its polymer chain. UPRs are produced by chemical reaction of saturated and unsaturated dicarboxylic acids with alcohols. They form highly durable structures and coatings when they are cross-linked with a vinyl reactive monomer, most commonly styrene. UPRs are characterized by vinyl unsaturation in the polyester backbone. Generally, the unsaturated polyester is dissolved in the reactive monomer [2]. The properties of the cross-linked unsaturated polyester resins depend on the types of acids and glycols used and their relative proportions. UPRs are characterised by toughness, rigidity, and low moisture absorption. They have a wide variety of application in the transportation sector [3], manufacturing of water pipes, chemical containers,



offshore application, construction, and the paint industry [4].

Polymer matrix composites (PMCs) are developed using matrix materials such as polyester resin, epoxy resin, phenolic resin and vinyl ester resin with reinforcements such as fibres, particles and flakes. The matrix material transfers the applied load to the reinforcement which improves the physical and mechanical properties of the matrix. In recent times, the engineering applications of composites have increased tremendously because of the continuous advancement in the quality of composites produced. PMCs are the most common advanced composites. They provide great strength and stiffness along with resistance to corrosion.

Mechanical properties of a polymer can be controlled by the incorporation of well-defined modifier particles in the polymer matrix. Reinforcement of polymers with particulates plays an important role in the improvement of the mechanical properties of high performance materials. The negative environmental impact as a result of non-biodegradability of carbon/glass fibre reinforced polyester composites is creating pressure for the substitution of high energy consumption materials for natural and sustainable ones. Compared to synthetic fibers, natural fibers and particulates have shown advantages in aspects such as flexibility and toughness. Hence, there is a growing worldwide interest in the use of these fibers and particulates. The use of agro-based materials/wastes in reinforcing polymers have recently attracted the attention of researchers because of their advantages over other established materials. Agro-based materials are environmentally friendly, fully biodegradable, abundantly available, renewable, cheap and have low density.

Studies have been carried out on polyester composites with several agro wastes as reinforcements. Pothan et al [5] evaluated the tensile, impact, flexural properties and aging behaviour of short banana fiber reinforced polyester composites with special reference to the effect of fiber length and fiber content. The maximum tensile strength was recorded at 30 mm fiber length while the maximum impact strength was obtained at 40 mm fiber length. Incorporation of 40 % untreated fibers gave a 20 % increase in the tensile strength and a 341 % increase in impact strength. On treatment with silane coupling agent, composites showed a 28 % increase in tensile strength and a 13 % increase in flexural strength. Aging studies showed a decrease in tensile strength of the composites. Water absorption studies showed an increase in water uptake with increase in fiber content. Abbas et al [6] synthesized

polyester composites reinforced with 3 - 9 wt. % Acro shell (AS) particulate. There was considerable increase in tensile strength, young's modulus and hardness with increase in the filler content. The composites were found to have more impact strength when compared to unreinforced polyester. Nwigbo et al [7] produced a composite with a polyester matrix and chemically modified shells of castor seed (*Ricinuscommunis*) as reinforcement. The effect of the shell (filler) on the mechanical properties of the composite was experimentally quantified. The results of the mechanical tests revealed that the inclusion of the filler (shell) improved the strength of the base polyester matrix.

Bhat et al [8] investigated the Mechanical testing and microstructure characterization of glass fiber reinforced isophthalic polyester composites. The results indicated that the tensile strength and hardness of the polyester matrix increased with glass fiber reinforcement. Meenambika and Raghavendra [9] worked on the chemical resistance and flexural properties of bamboo/glass reinforced polyester hybrid composites. It was observed that the flexural strength of the hybrid composites increased with increased glass fiber content. These properties were found to be higher when alkali treated bamboo fibers were used in hybrid composites. The hybrid reinforced fiber composites showed better resistance to the acids. It was noted that the elimination of amorphous hemi-cellulose with alkali treatment leading to higher crystallinity of the bamboo fibers with alkali treatment might be responsible for these observations. Swain [10] worked on synthesis and characterisation of graphene based unsaturated polyester resin composites. Unsaturated polyester resin was reinforced with graphene nano sheets (GNS). The tensile strength and flexural strength of the reinforced polyester matrix composites increased reinforced.

Low cost particles are added to polymer matrix for economic reasons and improvement in molding characteristics. PMCs can exhibit unique properties with the combination of particles such as thermal conductivities, dielectric constants and ductility, scratch resistant and modulus, toughness and wear properties, impact performance, and compressive strength. Such polymers have also found their practical importance in several novel applications [11].

The importance of employing waste materials in developing useful and cost competitive engineering materials for domestic and industrial applications cannot be over emphasized. Hence, this research was carried out

in order to further reduce the environmental pollution caused by these waste(coconut and snail shells) and also develop an engineering material with improved mechanical properties suitable for automobile application.

2. MATERIALS AND METHODS

2.1. Materials

The coconut and snail shells used as reinforcements were obtained from a local market in Mushin, Lagos, Nigeria. The polyester resin (unsaturated), catalyst and accelerator were also obtained from a registered vendor in Lagos. The apparatus used in the preparation of the samples are wooden mould, masking tape, plastic containers, stirring rod, weighing balance and British standardised sieves (BSS). The photographs of some of these materials are presented in Figure 1.

2.2. Preparation of the Composite Samples

The coconut shell and snail shell were washed in water and sun dried for two days. Thereafter, they were ground using a pulverizer and sieved to 150 μm using the British standardised sieves (BSS). The polyester resin was weighed using an electronic weighing balance. The polyester resin was poured into a beaker and also weighed. The coconut shell and snail shell particulates were poured into a petri dish and weighed. This process was repeated for the five weight formulations used for this research. The formulation used for the polyester resin was $A/100 \times 100$ g, where A is the percentage of resin. The formulation used for the reinforcement was $B/100 \times 100$ g, where B is the percentage of reinforcement. The basis used for the preparation of samples was 100 g. The polyester and the particulates were mixed in different proportions while maintaining a total weight of 100 g. Five mixtures with particulate compositions of 5 wt. %, 10 wt. %, 15 wt. %, 20 wt. %, and 25 wt. % were obtained. The reinforcements were added in the right proportions to the polyester matrix and drops of catalyst and accelerator were also added. The mixtures were thoroughly stirred using a stainless steel rod in order to achieve uniform distribution of the reinforcements and avoid air bubbles. Each of the mixtures was poured into a wooden mould already coated with paper tape that acted as poly vinyl alcohol (PVA) to serve as a releasing agent. Thereafter, the samples were cured at 1500 C for 4 hours after which they were removed from

the mould. The 2nd batch of samples were left to cure at room temperature for 24 hours.



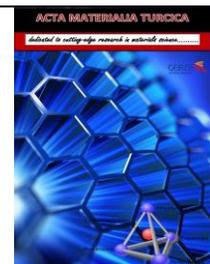
Figure 1. Photographs of the materials (a) as-received coconut shell (b) 150 μm coconut shell particles (c) as-received snail shell (d) 150 μm snail shell particles (e) wooden mould (f) some of the prepared composite samples in the mould.

Table 1: Quantity of materials

Reinforcement (wt. %)	Polyester resin matrix (wt. %)
5	95
10	90
15	85
20	80
25	75

2.3. Mechanical Tests

Tensile, flexural, hardness and impact tests were carried out on the samples. The tensile testing was performed using a testometric universal testing machine with serial number 25257 and capacity M500-25 KN. This test was operated at a cross head speed of 30 mm/min using a universal testing machine in accordance with ASTM D412 standard using dumbbell test piece. Each sample was subjected to tensile load and as the sample stretches, the computer generates graph as well as all the desired parameters until the specimen fractures. The tester automatically plotted the graph of load against extension



and various property of the specimen determined are tensile strength, tensile strain, modulus, tensile strain at break.

Three-point flexural test was carried out at room temperature on the samples of dimension 120 x 50 x 5 mm in accordance with ASTM D7264 standard. A cross-head speed of 30 mm/min was used while maintaining a span of 100 mm. Samples having a 2 mm deep V-notch at their centres were subjected to impact test using an Izod impact tester in accordance with ASTM D256 standard. Each sample was clamped vertically with the notch facing the striker and the striking pendulum was allowed to swing downward from a height of about 1.5 m with a velocity of 5 ms⁻¹ hitting the sample. The energy absorbed to break each sample was read from the dynamometer. The hardness of the samples was determined in accordance with ASTM D785 standard using a Rockwell hardness tester.

3. RESULTS and DISCUSSION

The results of this study showed that the samples post cured at 1500 C and held for four hours exhibited better mechanical properties than the samples cured at room temperature. This observation applied to both the coconut shell and snail shell particles reinforced samples. The reason for this could be that the post curing led to a higher level of crosslinking within the polyester leading to more efficiently cured samples and hence better mechanical properties. The results of the mechanical tests carried out on the samples post cured at room temperature and 1500 C for four hours are presented in Tables 2 – 5.

Table 2: Result of mechanical tests on the coconut shell reinforced composite samples cured at room temperature

Reinforcement (wt. %)	Bending modulus (MPa)	Impact strength (Joules)	Hardness (HV)	Young's Modulus (MPa)
Coconut Shell (5%)	170.6	4.94	82.53	70.97
Coconut Shell (10%)	189.3	4.90	85.4	116.9
Coconut Shell (15%)	202.76	5.12	92.0	185.17
Coconut Shell (20%)	210.37	5.14	110.7	199.17
Coconut Shell (25%)	180.56	5.2	105.8	162.0

Table 3: Result of mechanical tests on the snail shell reinforced composite cured at room temperature

Reinforcement (wt. %)	Bending modulus (MPa)	Impact strength (Joules)	Hardness (HV)	Young's Modulus (MPa)
Snail shell (5%)	158.77	5.04	108.3	95.63
Snail shell (10%)	172.53	5.10	99.03	159.73
Snail shell (15%)	142.1	5.33	110.67	125.63
Snail shell (20%)	155.93	5.36	96.27	160
Snail shell (25%)	170.36	5.04	83.53	200.53

Table 4: Result of mechanical tests on the coconut shell reinforced composites post cured at 150⁰ C

Reinforcement (wt. %)	Bending modulus (MPa)	Impact strength (Joules)	Hardness (HV)	Young's Modulus (MPa)
Coconut Shell (5%)	193.58	5.37	130.11	94.74
Coconut Shell (10%)	204.76	5.22	110.12	130.71
Coconut Shell (15%)	218.8	5.40	130.53	212.43
Coconut Shell (20%)	230.01	5.31	133.26	222.73
Coconut Shell (25%)	191.67	5.29	108.97	170.4

Table 5: Result of mechanical tests on the snail shell reinforced composites post cured at 150⁰ C

Reinforcement (wt. %)	Bending modulus (MPa)	Impact strength (Joules)	Hardness (HV)	Young's Modulus (MPa)
Snail shell (5%)	175.49	5.33	126.03	105.58
Snail shell (10%)	193.64	5.72	143.26	176.09
Snail shell (15%)	182.47	5.86	128.86	140.2
Snail shell (20%)	168.74	5.49	108.15	169.70
Snail shell (25%)	180.24	5.77	100.7	230.0

3.1. Microstructure

From Figure 2a, it was observed that there are some traces of impurities appearing as dark particles on the unreinforced composite sample. Figures 2b and 2c of both coconut shell and snail shell show some dark particles. These dark particles can be attributed to be coconut shell and snail shell reinforcements. The micrographs show that the reinforcements were evenly dispersed in the sample. It could be assumed that there was bonding between the reinforcement particle surface and the polyester resin. Figures 2d, 2e, 2f, 2g, 2h and 2i also show an increasing amount of coconut shell and snail shell reinforcements in the polyester composite. The reinforcement particles were also well dispersed in the polymer matrix. Micrographs of Figures 2j and 2k show that reinforcement at 25 wt. %, there was pronounced appearance of more dark patches with agglomeration of the particles. This may be as a result of the poor interfacial bonding between the particles and the polyester matrix. This was responsible for the reduction in the flexural strength, hardness and impact energy of the composites due to poor load distribution because of agglomeration. This is similar to the earlier report of Mechtali et al [12] and Rufai et al [13].

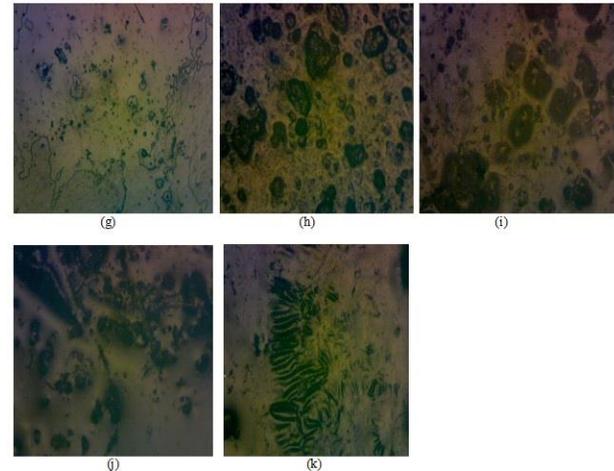
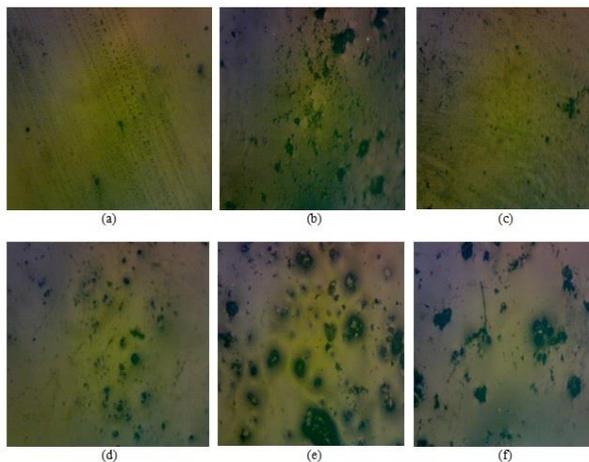


Figure 2. Optical micrographs of samples
 (a) unreinforced polyester resin
 (b) 5 wt. % coconut shell particles reinforced sample
 (c) 5 wt. % snail shell particles reinforced sample
 (d) 10 wt. % coconut shell particles reinforced sample
 (e) 10 wt. % snail shell particles reinforced sample
 (f) 15 wt. % coconut shell particles reinforced sample
 (g) 15 wt. % snail shell particles reinforced sample
 (h) 20 wt. % coconut shell particles reinforced sample
 (i) 20 wt. % snail shell particles reinforced sample
 (j) 25 wt. % coconut shell particles reinforced sample
 (k) 25 wt. % snail shell particles reinforced sample.

3.2. Flexural Strength

As illustrated in Figure 3, coconut shell particles reinforced polyester composite exhibited the highest flexural strength of 230.01 MPa at 20 wt. % reinforcement while the snail shell particles reinforced polyester composite exhibited the highest flexural strength of 193.64 MPa at 10 wt. % reinforcement. The flexural strength of the coconut shell particles reinforced composite increased with increasing reinforcement up to 20 wt. % after which a decrease was observed at 25 wt. %. There was an increase in the flexural strength of the snail shell particles reinforced polyester composite up to 10 wt. % after which a decrease was observed at 20 wt. %. However, there was a slight increase in flexural strength from 20 to 25 wt. %. The reduction in the flexural strength can be attributed to controlled mobility of matrix by filler

particles. As amount of reinforcement increases, there is reduction in total surface area available for matrix-filler interaction. This is similar to the earlier report of Raghad [14].

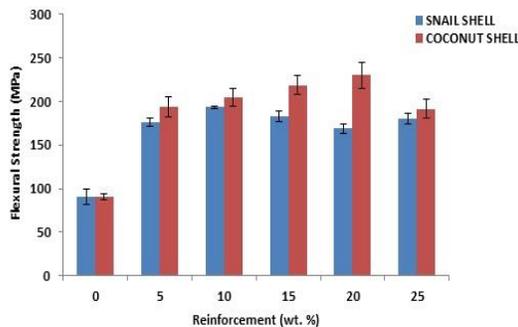


Figure 3. Bar chart of Flexural strength against wt. % reinforcement of the composites.

3.3. Impact Energy

The snail shell particles reinforced composite and the coconut shell particles reinforced composite exhibited highest impact energy of 5.87 J and 5.40 J respectively at 15 wt. % reinforcement before fracture as illustrated in Figure 4. Further increase in the concentration of the filler material reduced the ability of the polyester matrix to absorb energy which led to the reduction in the toughness of the composites. This may also be due to agglomeration of particles in the polyester matrix as revealed in Figures 2h – 2k. This is in agreement with the earlier report of Durowaye et al [15] and Rufai et al [13].

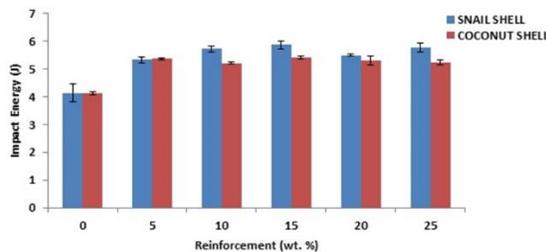


Figure 4. Bar chart of impact energy against wt. % reinforcement of the composites.

3.4. Hardness

There was an increase in the hardness of the coconut shell particles reinforced polyester composites when reinforcement increased to 10 wt. % while the hardness of snail shell particles reinforced polyester composites increased when reinforcement increased up to 15 wt. % as presented in Figure 5. The highest hardness of 143.26 HV was exhibited by the snail shell particles reinforced composite at 10 wt. % reinforcement while 130.53 HV was exhibited by the coconut shell particles reinforced polyester composites 15 wt. %. The hardness of the coconut shell reinforced polyester composite samples showed an undulating pattern could be due to the poor interfacial bonding or surface adhesion of the particles and polyester resin [8, 16].

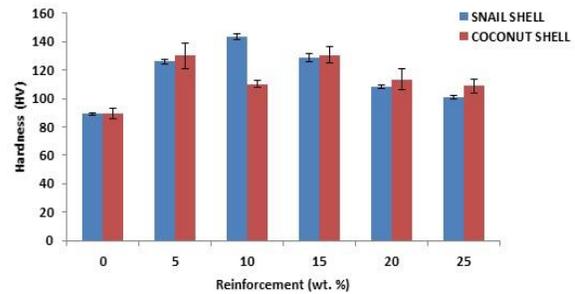


Figure 5. Bar chart of hardness against wt. % reinforcement of the composites.

3.5. Elastic Modulus

From Figure 6, both composites demonstrated a fair steady increase in elastic modulus as reinforcement increased. The snail shell particles reinforced polyester composite exhibited the highest elastic modulus of 230 MPa at 25 wt. % reinforcement while the coconut shell particles reinforced polyester composite sample exhibited the highest elastic modulus of 222.73 MPa at 20 wt. % reinforcement. The high elastic modulus of the composites could be due to interfacial bonding of the particulates and the polymer matrix [8, 17].

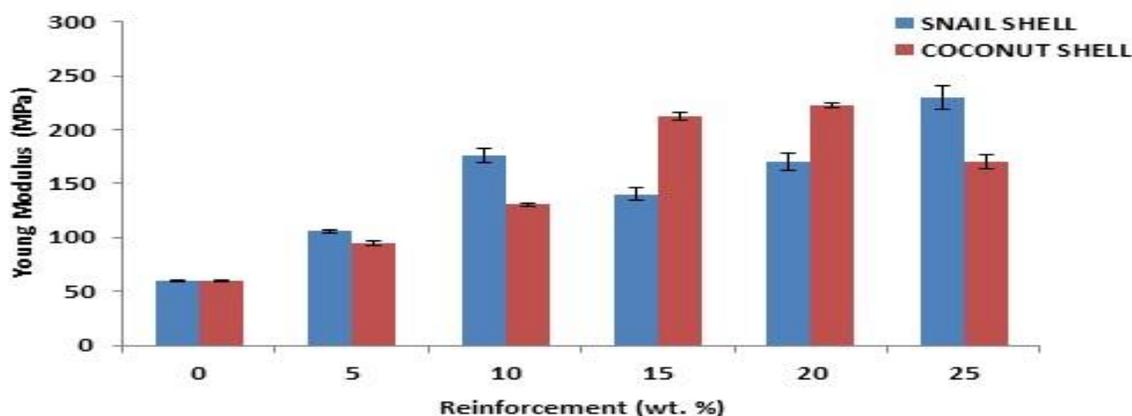


Figure 6. Bar chart of Young modulus against wt. % reinforcement of the composites.

4. CONCLUSION

It was seen that the mechanical properties of polyester composites can be improved by the agro waste and sea waste reinforcements of coconut shell and snail shell. The micrographs revealed a fairly even dispersion of the reinforcements within the matrix which contributed to the enhancement of the mechanical properties of the composites. The coconut shell and snail shell particles reinforced polyester composites at 20 wt. % and 10 wt. % reinforcements exhibited the highest resistance to elongation with values of 230.01 MPa and 193.64 MPa respectively before shattering relative to other samples. This is an indication that the coconut shell and snail shell particles reinforced polyester composites at 20 wt. % and 10 wt. % reinforcements can be used in place of the pure polyester for applications where flexibility is critical. The snail shell particles reinforced composite and the coconut shell particles reinforced composite exhibited highest impact energy of 5.87 J and 5.40 J respectively at 15 wt. % reinforcement before fracture which indicates both composites can be used in place of pure polyester in applications where impact strength is a necessary. The high hardness values of 143.26 HV and 130.53 HV exhibited by snail shell and coconut shell particles reinforced composites respectively is an indication that they can be used in place of the pure polyester for applications where hardness is a priority. The improved properties exhibited by these composites has proven their suitability for engineering applications.

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