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Effect Of Filler Loading And Particle Size On The Mechanical Properties Of Periwinkle Shell-Filled Recycled Polypropylene Composites.

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ABSTRACT: *Mechanical properties of composites of recycled polypropylene filled with periwinkle shell powder were investigated. Periwinkle shells used in this study were thoroughly cleaned, ground and sieved to three particle sizes of 150, 300 and 425 µm respectively. Filler loadings of 0 to 25 wt% were used in compounding the composites in an injection moulding machine and the resulting composites were extruded as sheets. Some mechanical properties were determined. Results showed that periwinkle shell powder improved the tensile strength, flexural strength, tensile modulus and hardness of polypropylene composites. These properties were found to increase with filler loading up to 15 wt% or 20 wt% loading (optimal filler loading) but decrease with filler particle size. However, elongation at break was found to decrease with increasing filler loading. Highest tensile and flexural strengths were obtained at 15 and 20 wt% respectively at 150 µm particle size. It is therefore concluded that periwinkle shell powder is suitable for the production of polypropylene composites.*
Keywords: *Polypropylene, periwinkle shell powder, filler, composite, particle size.*

I. INTRODUCTION

The wide use of polymeric materials such as plastics in various applications like engineering, medicine, automobile, sports, domestics, packaging, etc is increasing rapidly nowadays as a result of effective modification of the properties of plastics using additives. This enables polymeric materials to overcome its specific deficiencies during service and new environmental regulations which demand the search for materials that are eco-friendly [1].

Despite the successful utilization of many polymers in various fields, there is still a growing demand for materials that satisfy more stringent requirements such as high tensile strength, thermal conductivity, improved heat distortion temperature, lower thermal expansion and reduced material cost [2]. These requirements can be satisfied by utilizing a composite material whose constituents may act synergistically to meet the needs of particular application [3]. Plastic industry uses inorganic fillers such as talc, calcium carbonate, mica and glass or carbon fibres to modify the performance of thermoplastics. Inorganic fillers provide rigidity and temperature resistance; however, these fillers are costly and abrasive to the processing equipment [4], [5].

Recently, organic fillers produced from agricultural wastes have gained tremendous attention from plastic industry. The primary advantages of using organic fillers in thermoplastics can be listed as low densities, low cost, non-abrasiveness, high filling levels, low energy consumption, biodegradability, availability of a wide variety of fibres throughout the world and generation of a rural/agricultural-based economy [6]. Many research works have been reported on organic fillers reinforced thermoplastic composites, which have proved their applicability in various fields. Such fillers include snail shell powder [7], cocoa pod [4], oyster shell powder [1], groundnut shell, cocoa nut shell, palm kernel shell [8], bean pod ash [9], saw dust [10] and maize tassel [6].

Periwinkle shell (PS) is a by-product of agriculture. Periwinkle (*Littorina littorea*) is a species of small edible sea animal, a marine gastropod mollusk. The shell makes up over 70% of the weight of the animal. The shells have no identified use and are found littered around markets and homes [11]. Across the globe, much research efforts are geared towards possible ways of recycling wastes for reuse to keep environment clean and safe [9]. Hence, the use of periwinkle shell powder in producing recycled polypropylene composites is therefore hoped to convert wastes to wealth, tackle the problem of environmental pollution, reduce the material cost and produce biodegradable composites.

II. MATERIALS AND METHODS

Materials

Polypropylene (PP) used in this research was a product of SK Global Chemicals Limited, Korea obtained from CeePlast Limited Aba, Abia State. It has a melt flow index of 0.4g/10 min at 150 ^{0}C and density of 0.922g/cm^3. Recycled polypropylene was sourced from Ihiagwa village, Owerri West L.G.A., Imo State and was thoroughly washed, dried and sliced to tiny pieces. The compartibilizer; maleic anhydride-grafted-polyethylene (MAPE) used was bought from CEEPlast Industry, Aba, a product of Sigma Aldarich Company, U.S.A. It has the following properties: maleic anhydride = 0.5 wt%, viscosity = 500 cP (140 ^{0}C) and saponification value = 60 KOH/g. Periwinkle shell was obtained from Ihiagwa Market after the edible portion had been harvested. The shells were soaked in water for one week to thoroughly remove impurities. They were dried, ground and sieved to three particle sizes of 150, 300 and 425 µm. The chemical composition of the periwinkle shell is as shown in Table 1.

Preparation of Polypropylene Composites

The polypropylene composites of periwinkle shell powder were prepared by thoroughly mixing 100g of virgin polypropylene and 100g of recycled polypropylene with appropriate filler contents (5, 10, 15, 20 and 25 wt%) as shown in Table 2 and 7 g of maleic anhydride was added to enhance compatibility.

Each mixture was melted and homogenized with the filler in an injection moulding machine. The operation was carried out at an injection pressure of 100 kgf/cm^2 (9.81 MPa) and temperatures of 250 ^{0}C. The mixture was then extruded as sheets with dimensions of 150 x 150 x 4mm. Test samples were prepared from the sheets for the mechanical testing. Morphological analysis was also carried out using Scanning Electron Microscope (Phenom: product of Phenom World, Eindhoven, Netherlands; Model: ProX). The specimen was made conductive by introducing a minimum of a 5nm gold onto it and then cut into 2 x 2 mm using a sputter cutting machine. The sample was then placed on the column of the Scanning Electron Microscope (SEM) where the image was focused on navigation camera and then transferred to electron mode in accordance to the desired magnification e.g. 50, 80, 100 or 200µm. The samples were thus analyzed to determine the microstructure of the composites, from which the distribution, orientation and interaction of the fillers in the composite and the interfacial adhesion of the filler and polymer matrix were examined.

Testing

Tensile properties of each composite were determined according to standard (ASTM D 638) using a computerized universal testing machine (Instron Universal Tensometer, SSTM-Smart-1-Series-20KN; manufactured by Scientific Instrument Co. Ltd, U.S.A) at a crosshead speed of 5mm/min. Readings were recorded and calculated automatically by the instrument's software. Tensile properties obtained from this test are tensile strength, elongation at break, tensile modulus and yield strength.

Flexural strength was conducted according to standard (ASTM D 790) using Instron Universal Tensometer at a test speed of 2mm/min on a 3-point bending test. Loading was continued until fracture and values of flexural strengths were recorded automatically. Shore 'A' hardness was conducted in accordance with standard (ASTM D 2240) using a digital Durometer (JIS-K-6253). Readings were taken 10 seconds during the test period.

Morphological Analysis was done using Scanning Electron Microscope (Phenom: product of Phenom World, Eindhoven, Netherlands; Model: ProX). The specimen was made conductive by introducing a minimum of a 5nm gold onto it and then cut into 2 x 2 mm using a sputter cutting machine. The sample was then placed on the column of the Scanning Electron Microscope (SEM) where the image was focused on navigation camera and then transferred to electron mode in accordance to the desired magnification e.g. 50, 80, 100 or 200µm.
The samples were thus analyzed to determine the microstructure of the composites, from which the distribution, orientation and interaction of the fillers in the composite and the interfacial adhesion of the filler and polymer matrix were examined.

III. RESULTS AND DISCUSSION

Tensile Strength

Figure 1 shows the variation of tensile strength of PS-PP composite with filler loading. The filled samples showed greater tensile strengths than the unmodified sample except for sample containing above 20 wt% filler. At any chosen filler particle size, tensile strength increased with filler loading up to 15wt% before decreasing. The increase in tensile strength may be attributed to the strengthening effect of the filler incorporated into the polymer matrix. A filler is usually added to polymer material to improve its rigidity, making it stronger. The higher the content of filler incorporated, the stronger the polymer composite, provided the optimal filler loading is not exceeded. Highest set of values were obtained at 15wt% filler loading.

However, excessive incorporation of filler may lead to filler agglomeration (weak bonding) in the polymer matrix leading to formation of microfiller due to the difficulties in achieving a homogeneous dispersion of fillers. This results to stress concentration at the particle/matrix boundary region, leading to weakness of the particle/matrix

interaction and consequent reduction in tensile strength. This may be responsible for the reduced strength as indicated for filler loadings of 20 wt% and above. Also, it could be seen from Figure 1 that tensile strength decreased progressively with increase in particle size. This is because fine particles provide larger surface area and this encourages better dispersion and interaction between the polymer matrix and filler particles. This fair dispersion of filler in the matrix could be observed from the micrographs of the filled samples (e.g. Fig. 8, 9 and 10) when compared to the control (Fig. 7) revealed that there is enhanced spherulization owing to presence of nucleation sites provided by the incorporation of fillers. This consequently increases the ability of the composite to restrain gross deformation of the matrix. However, at larger filler particle sizes and at the same filler loading, there is poor dispersion of the fillers leading to cluster of the spherulites and subsequent increase in the size of the spherulites due to interaction with each other (See Figures 8, 9 and 10). This resulted to creation of pores in the system as could be observed in micrograph of PP-PS at 415 µm (Figure 10). Highest set of values were obtained at 150 µm particle size. Similar results were reported by [12,1,6,13,9].

Elongation at break

Figure 2 shows the variation of Elongation at Break of PS-PP composite with filler loading. Lower elongations at break were recorded for the modified samples than for the unmodified. It is obvious from the data that elongation at break decreased steadily with filler loading and particle size, with the highest set of values at 150 µm particle size. Elongation at break is a reflection of the ductility of a material, a direct opposite of brittleness. Filler incorporation into a polymer matrix increases the stiffness and hardness of the composite. This increase in hardness resulted in decrease in ductility of the material. Hence, as filler content increases, the ductility decreases. Such decreasing elongation at break with filler loading was also reported by [9,14,15].

Tensile modulus

Results of the variation of Tensile Modulus of PS-PP composite with filler loading are presented in Figure 3. Values obtained for the filled composites were higher than for the unmodified sample except at 25 wt% filler content. Generally, tensile modulus was found to increase with filler loading upto 15 wt% content and decreased with particle size. The highest set of values was obtained for 15 wt% content and at 150 µm particle size. Tensile modulus is an indication of the stiffness of a material. The incorporation of fillers into the polymer matrix improves the stiffness of the composites. This could be attributed to the fair distribution of the filler in the matrix, which efficiently hinders chain movement during deformation. On the other hand, smaller particles enhance better dispersion within the matrix than larger particles, hence the decreased tensile modulus with increase in particle size. Similar behaviour was also reported by [6,16,17,13]. However, [7] reported increase in tensile modulus with increase in particle size. It could be observed that all the values recorded for tensile modulus compared well with standard values (0.5 – 1.9 GPa) as recommended by [18].

Yield strength

Results of the Variation of Yield Strength of PS-PP composite with filler loading are presented in Figure 4. Higher values were obtained for the filled samples than for the control except at 25 wt% filler content. It could be observed that yield strength increased progressively with increase in filler loading up to 15 wt%, a behaviour similar to that of tensile strength. Above 15 wt% filler content, yield strength decreased. The increased yield strength may be as a result of the strengthening effect of the filler due to the fair dispersion in the polymer matrix. The yield strength increased with increase in particle size.

Flexural strength

Figure 5 shows the variation of Flexural Strength of PS-PP composite with filler loading. The filled samples were found to be higher than the unfilled sample. Flexural strength was found to increase with increase in filler content up to 20 wt% filler loading. The results also showed increase in flexural strength with increase in particle size except with 25 wt% at 425 µm which showed a sharp decrease in flexural strength. The increase in flexural strength could be attributed to the better rigidity and stiffness as a result of fair dispersion and distribution of the fillers in the polymer matrix, which efficiently hinders chain movement during deformation. Such an increase in flexural strength was reported by [19,6,20,16,1].

Hardness

Figure 6 shows the variation of Hardness of PS-PP composite with filler loading. Hardness of the modified samples were all higher than the unfilled sample. Hardness was found to increase progressively with both filler loading and particle size. This is attributed to the fact that this filler acts as a reinforcing filler. Incorporation of the filler into the polymer matrix enhanced the stiffness of the material. The higher the percentage of the filler incorporated, the harder the material, and the more rigid it becomes. [9,19] reported similar increase of hardness with filler loading while [7,22] reported decreased value with particle size.

IV. CONCLUSION

The mechanical properties of periwinkle shell powder filled recycled polypropylene composites were studied. The successful production of polypropylene composites filled with periwinkle shell powder was realizable. The fair distribution of the periwinkle shell powder particles in the polypropylene matrix is a major factor responsible for the improvement in the mechanical properties. Tensile and flexural strengths increased to a maximum with 15 and 20 wt% respectively at 150 µm particle size. Excessive incorporation of filler was believed to have led to filler agglomeration (weak bonding) in the polymer matrix leading to formation of microfiller due to the difficulties in achieving a homogeneous dispersion of fillers. Based on the results obtained, it is recommended that periwinkle shell powder be used as filler in producing recycled polypropylene composites.

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Table 1: Elemental **composition of periwinkle shell**

Elemental oxide	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	K_2O	Na_2O	Mn_2O_3	TiO_2	LOI
%	33.84	10.2	6.02	40.84	0.48	0.26	0.14	0.24	0.01	0.03	7.6

Table 2: Composition of Evaluated Formulations.

Code	**Virgin PP (g)**	**Recycled PP (g)**	**PS (wt%)**
V (Control)	200	0	0
P_1	100	100	5
P_2	100	100	10
P_3	100	100	15
P_4	100	100	20
P_5	100	100	25

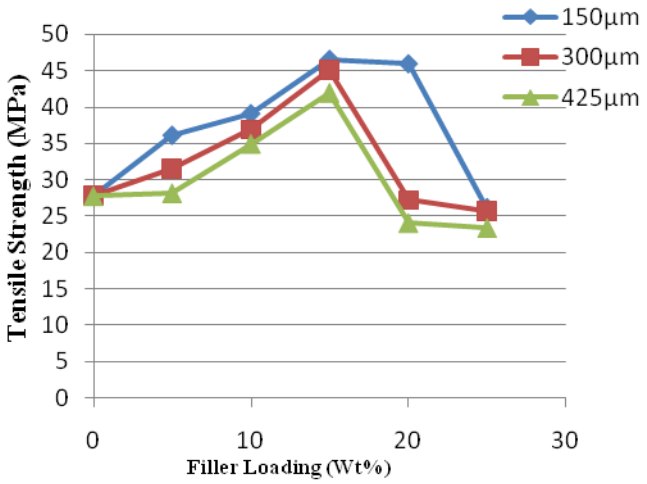

Fig. 1: Variation of Tensile Strength of PS-PP Composite with filler loading.

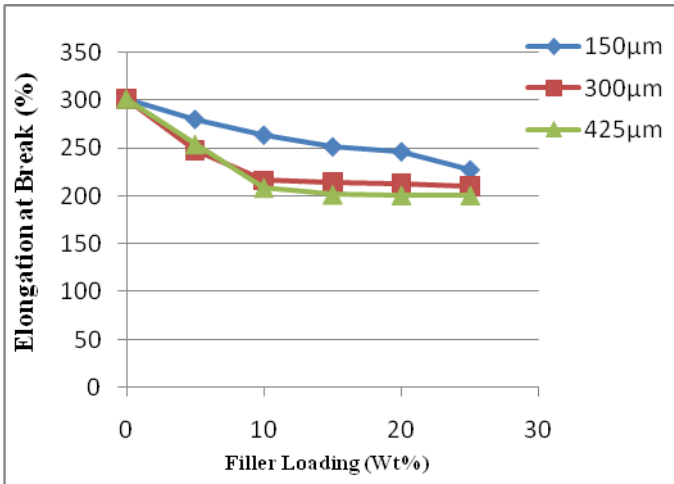

Fig. 2: Variation of Elongation at Break of PS-PP composite with filler loading.

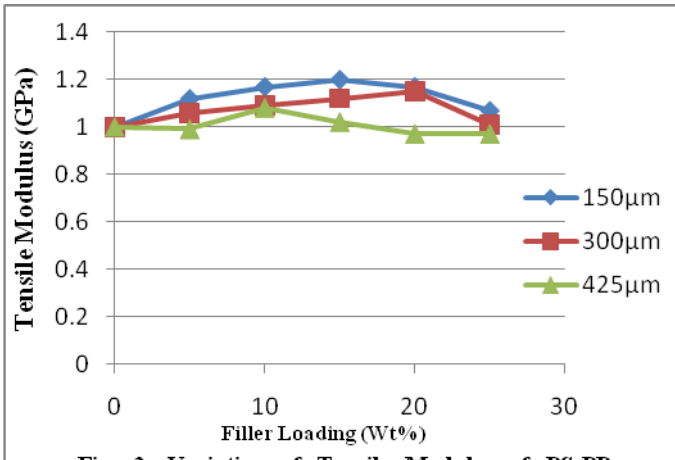

Fig. 3: Variation of Tensile Modulus of PS-PP composite with filler loading.

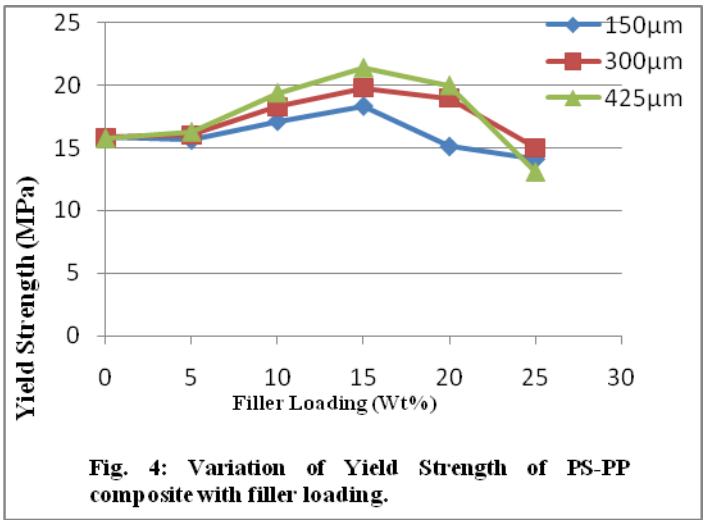

Fig. 4: Variation of Yield Strength of PS-PP composite with filler loading.

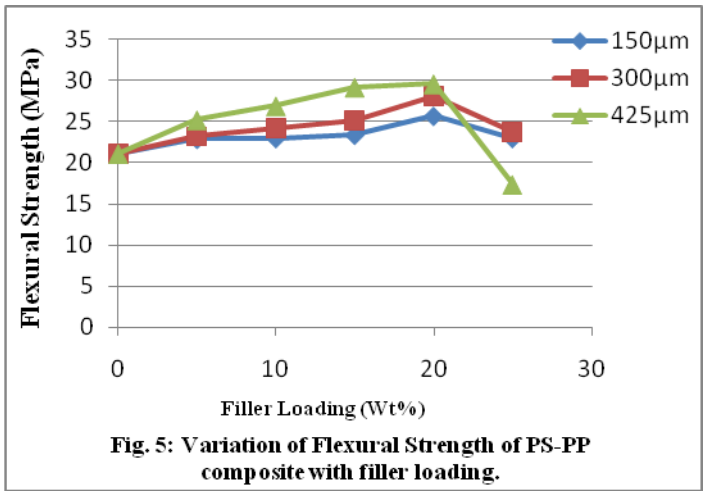

Fig. 5: Variation of Flexural Strength of PS-PP composite with filler loading.

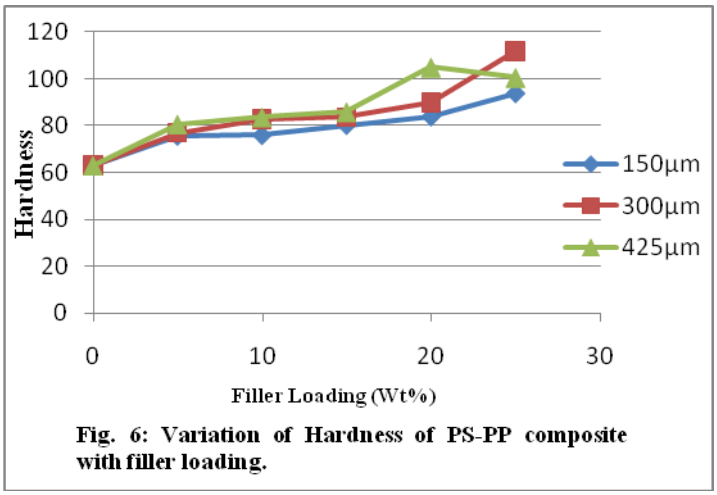

Fig. 6: Variation of Hardness of PS-PP composite with filler loading.

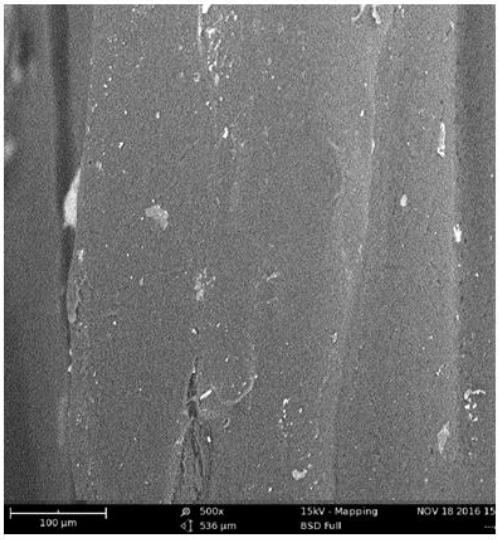

Fig. 7: SEM Micrograph of the control experiment.

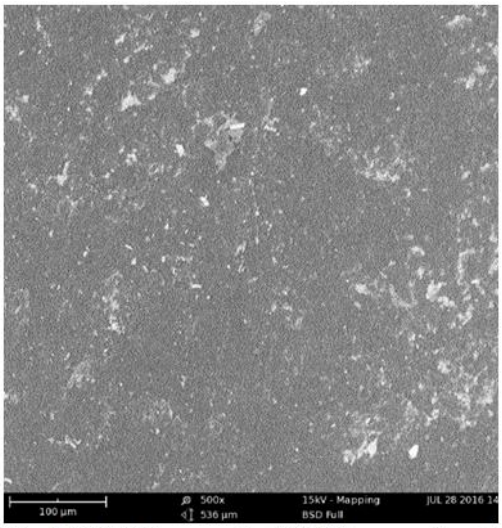

Fig. 8: SEM Micrograph of PP-PS composite at 15 wt% filler loading and 150 µm

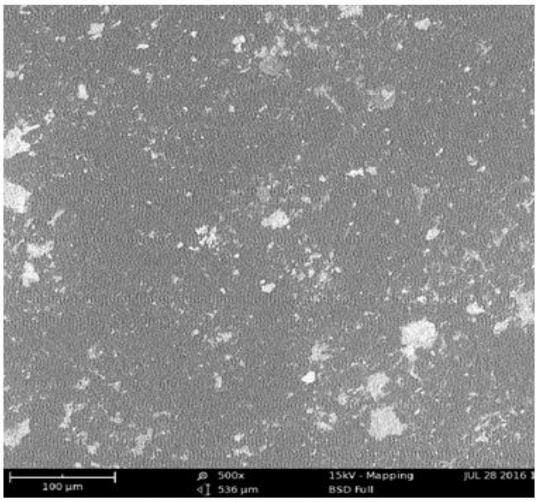

Fig. 9: SEM Micrograph of PP-PS composite at 15 wt% filler loading and 300 µm

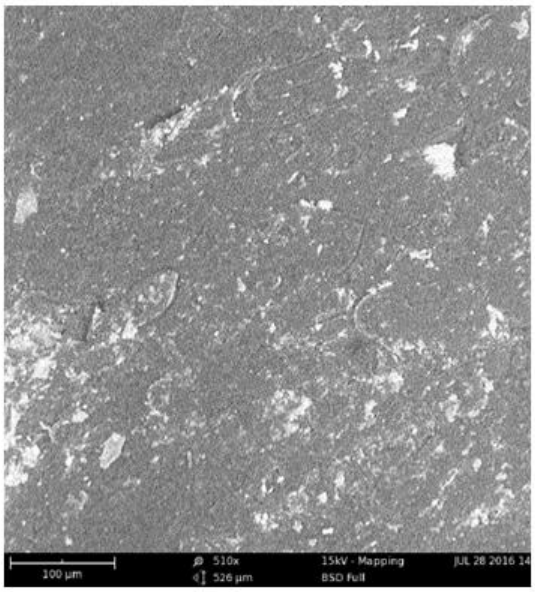

Fig. 10: SEM Micrograph of PP-PS composite at 15 wt% filler loading and 425 µm