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CHARACTERIZATION OF PERIWINKLE SHELL ASH REINFORCED POLYMER COMPOSITE FOR AUTOMOTIVE APPLICATION

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ABSTRACT

The Periwinkle shell (*Tympanotonus fuscatus*) is one of the most abundant wastes in the Calabar coastal region of Nigeria and needs to be put into proper use. The great need to shift attention towards waste materials with good mechanical properties to replace some materials used in the Automobile industries for Automobile products is paramount. This research focused on the Mechanical characterization of several composites developed from Periwinkle Shell Powder (PSP) as filler and four selected polymeric materials as the matrix. Recycled high-density polyethylene (rHDPE), Recycled linear low-density polyethylene (rLLDPE), Recycled polystyrene (rPP) and recycled polystyrene (rPS) from waste dumps were selected as the Matrix for the composites. The crushed periwinkle shell (CPSP) was subjected to a calcination (ashing) process. Ashed Periwinkle Shell Powder (APSP) was used to reinforce the rHDPE, rLLDPE, rPP and rPS at 0 to 40% filler loading. Mechanical tests carried out resulted in the 30%PSP and 70%PP composite having better tensile and flexural strengths, good flexural modulus, hardness, impact and moisture absorption results. Results obtained from the mechanical tests were comparable with values obtained from a tested existing vehicle bumper. The APSP-filled recycled polymer composites can serve as a suitable green alternative to existing vehicle bumpers.

Keywords: Automotive, Bio-composite, Periwinkle shell ash, Polymeric material, sustainability

INTRODUCTION

The daily increasing demand for new materials and great aspiration towards achieving a good combination of excellent qualities like strength, toughness, wear resistance, high-temperature performance and corrosion resistance in the automobile industries, is a welcomed development in our advanced world. Also, these new materials open doors in engineering (Buranyi, 2015) and research work is strongly focusing on the production of biodegradable and sustainable materials that can be green composites with the ideal application, replacing the old materials (Ubi et al., 2022; Ofem et al., 2020; Tobin et al., 2018).

These achievements in modern times, will strengthen the efforts pushed towards having strong growth, prosperity, security and quality of life for humans and the world at large. The ever-increasing cost of automobile parts will be reduced by putting a stop to almost 95% of the imported. Creating avenues for parts manufacturing locally, using available local composite materials. This dream of having a bio-based society with materials used or desired to be sustainable and harmless to the ecosystem is achievable and adds to the economy immensely (Ferguson et al.,2018).

In addition, sustainability has become the keyword in the world development plans. That is why, the United Nations (UN) in her world summit on the 5th of September, 2015, pleaded sustainability as the main topic to be digested. This meeting strongly advised and encouraged all nations to work towards achieving their objectives of sustainable development goal (SDG) by the year 2030. Their dream is to gradually push non-environmentally friendly and dangerous materials and replace them with sustainable engineering materials. The rapid increment in agricultural waste, especially waste from seafood (shells and bones) posing danger to the Ecosystem calls for attention (Yawas, et al.,2013) and the burning of these wastes, pollutes the environment and if stored, becomes excess for farm usage. And the high deposit of waste if controlled by usage in engineering will tackle environmental pollution (Amartey et al., 2023).

Consequently, in the world today lightweight materials are manufactured with a greater focus towards mechanical properties especially in the absorption of impacts, producing low stress and life safety. Periwinkle Shell, a nontoxic and low density, low or zero cost, renewable, available as waste and with lightweight quality is a material though mainly used in civil engineering can go a long way to replace some materials used in Automobile parts production. For good Mechanical properties, Periwinkles are usually collected fresh, especially in impact and hardness strength. The calcium oxide (CaO) and Silicon oxide (SiO_2) presence in the periwinkle shell accounts for the quality of hardness and strength, which give rise to compressive strength, high hardness quality and good density. And as such, for twenty years plus, works on shells especially in periwinkle shell and coconut in other to save the ecosystem have been paramount (Liman et al., 2020).

Periwinkle shells are usually found under the rocks, stones or pilling between high and low tide marks (Olusola et al., 2012), with typically mottled grey, white, and black. They are straight-sided or rounded cones with an obtuse point found within the region between low and high tides. They live near the ocean and spend part of their time underwater and prefer to be partially exposed to air (Soneye et al., 2016). There are eighty species of periwinkle in existence and ten of these species are found on the coast of West Africa (Dance, 1980) and out of these ten, comes the *Tympanotonus fuscatus* found in Calabar, the coast of Nigeria. The general processing method for years has been just traditional methods, involving washing, cooking, sucking/extraction and throwing away (Ekop et al., 2021). The tiny flesh is consumed as meat and the shells are thrown away as waste. Though they are commonly used for several purposes such as soil additives, brake pads, sandpaper, erosion control, ornament, aggregates in construction and many others (Solomon et al., 2017; Ekop et al., 2013; Pessu et al., 2014), but their main characteristics are yet to be exploited.

The *Tympanostomus spp* and *Pach mellania spp* are commonly found in the lagoon and mudflats of Nigeria, the Niger Delta between Calabar in the east and Badagry in the west (Dahunsi, 2003). With *Tympanotonus fuscatus* more in abundance. Researchers have reported that *Tympanotonus fuscatus* has a high level of $CaCO_3$ (88.22 ± 0.75%) and a relatively low but substantial level of $MgCO_3$ (10.25 ± 0.42%) (Orji et.al.,2017). And they are V-shaped spiral shells, very strong, hard in nature and brittle (Ogungbele and Omowole, 2012). These qualities bring out their importance, in construction and structural engineering (Dahunsi and Bamisaye, 2002). But despite these qualities, a greater portion is usually thrown as waste and this shell waste constitutes a problem for the area, hence the need to convert the waste for more applications, especially in the automotive sector will help the economic system of our dear nation. However, with this information on the mechanical and physical properties of agricultural waste, it will be very important to put the waste to sustainable use. And stored for future purposes, efficient design, maintenance of dimensional accuracy and transformation to different materials (Fakayode,2020; Mohite and Sharma, 2018; Ituen, 2015; Eke and Ehiem,2015). Figure 1 shows a picture of periwinkle shells.

Figure 1: Periwinkle shell Picture

Table 1: Elemental composition of a periwinkle shell (Ubong and Godwin, 2017)

Element oxide	SiO_2	Al_2O_3	Fe_2O	CaO	MgO	SO_3	K_2O	Mn_2O_3	P_2O_3	TiO_2	LO
%	33.84	10.20	6.20	40.84	0.48	0.26	0.14	0.24	0.10	0.03	7.60

Table 1, above, shows the percentage (%) of calcium oxide (CaO) which is 40.84% and Silicon oxide (SiO_2) which is 33.84% which sums up to 74.68%. These elements account for the quality of a periwinkle shell in terms of hardness and strength (Dahunsi et al.,2002). Yawas et al., 2013, also reported that periwinkle shells have good compressive strength, high hardness quality and good density with a decrease in size. Orji et al.,2017, also affirmed the fact that the Periwinkle shell has a very large deposit of $CaCO_3$, making it a probable source of $CaCO_3$. Research has shown that periwinkle shells that stay longer or get soaked in water lose strength (Powel et al., 1985; Amaziah et al., 2013) and as such must be collected fresh to obtain good characterization.

Polymeric materials are a great investment in the world if only researchers and manufacturers shift towards converting their waste into money. Otunyo *et al.* (2013), and Yawas et al. (2013), in their research, concluded that the mechanical properties and chemical properties of a polymer are crucial for almost all its engineering characteristics. There is a geometric growth in the area of polymeric composites due to some essential properties like low weight, affordability and minimization of environmental impact (Madueke *et al.*, 2014). Also, the recycling of polymers is beginning to gain attention and the danger to our ecosystem is reduced (Ubi *et al.*, 2022, Strapasson et al., 2005). To overcome the deficiencies of polymers and the call for new products makes this research on polymeric materials for human usage important, and following new environmental regulations guiding materials okay, with the strong conviction to save the ecosystem very important work (Nwanonenyi et al., 2013). Note that for rigidity and temperature resistance, the in-organic materials if used as fillers provide all, but are costly and abrasive to the processing equipment (Cletus, 2002; La Mantia et al., 2005). Organic waste fillers from farms have gained attention enormously as several industries want to use them and the plastic industry is one of them. These organic waste materials have low densities, low cost, non-abrasiveness, high filling levels, low energy consumption, biodegradability and availability as advantages they have become source materials (Ubi et al., 2023; Onuegbu & Madufor, 2012).

Figure 2: Polymeric material waste.

A car bumper is a structure integrated into the front and rear of a car to absorb impact in a minor collision, thereby minimizing the cost of repair. In summary, they have two safety functions, minimizing height mismatches between vehicles and protecting pedestrians from injuries (Jurewicz et al., 2016). Generally, the bumper has been an important feature in vehicles, protecting components from serious damage when in a low-speed collision and strongly improving the performance of the car by lowering the coefficient of drag. They perform this above by deflecting and protecting the vehicle when there is a collision with other moving vehicles or when a vehicle collides with a stationary Object. The bumper generally does provide security or protection to people, equipment or machines, especially during an accident. In equipment and machines, they reduce vibrations and in our household use, they are used at the edge of doors and windows to absorb impact reducing damage. However, the quality to absorb and nullify the effects of shock or refine the effects of strikes must be put into consideration in the selection of materials.

Today bumper produce is modernized to combine the strength, quality and properties of different materials to meet its function. They have materials with absorbing spring device usually gas-filled cartridge that allows the new bumper to absorb minor impact without damage or little damage. Kleisner and Zemeik, 2009, in their research, express their view that a bumper is designed to deform itself and absorb the force of impact during a collision. The design of the car bumper is to absorb shock, often spring-loaded to slow speed collisions, compress and extend back to its original position. A research work carried out by Sumaila and Ibhadode, (2016), showed that there is always a reduction of weight, which is about 25% in the reduction of weight of parts of a vehicle, saving about 750,000 barrels of oil consumption per day. This reduces the consumption of fuel by 13% and 101 million tons of CO_2 emission into the atmosphere.

However, this research is targeted towards having a light automobile car bumper, reducing the weight of vehicles, saving fuel, saving the cost of parts by converting waste to wealth and above all contributing to a safer environment. Lesser density completely means low fuel consumption and hence, fewer pollutants are discharged into the atmosphere (Sumaila and Ibhadode, 2016). Consequently, this information obtained made the use of periwinkle shell and polymer waste, as a material for reinforcement and binder respectively in composite production for application in the domestic automobile industry, having the weight and cost reduction in mind. This is with great enthusiasm to achieving the production of new material with an economic interest, carbon dioxide balance, health risk reduction, biodegradability, improvement of passengers and pedestrian safety in case of accidents becoming a targeted interest in the development of the car bumper in this research.

MATERIALS AND METHODS

The materials used in this research were sourced locally in Nigeria. Periwinkle which is sustainable, biodegradable and readily available in nature, was washed by the use of distilled water and sodium hydroxide (NaOH) to remove impurities and dried under sunlight to ensure dryness for a day. A jaw crusher manufactured by Retch, with model number 41074, type BB2 was used in crushing the periwinkle shell (Ps) to powdered form.

The Periwinkle Shell Powder was divided into two, a part was used calcinated (converted to ash), and the second part was left in its natural state and gave rise to Un-ash Periwinkle Shell (UPS). XRF characterisation was then conducted on the two samples to detect the element and oxides lost. Various waste polymeric materials were collected and used as the matrix for samples of composites. These polymeric materials have unique physical properties and are found around waste dumping environments, drainages, shallow rivers, streams and oceans. Their presence around dump sites and our eco-marine site poses a lot of danger to our environment and our aqua-life cycle.

The four (4) polymeric materials considered in this study are Polystyrene (PS), Polypropylene (PP), Linear Low-Density Polyethylene (LLDPE) and High-Density polyethylene (HDPE). The choice of these materials is due to their availability as waste and non-biodegradability nature. The density of the selected existing vehicle bumper material as obtained was 0.915 g/cm^3 and is in a similar range with the densities of the selected polymers. A Design of Experiment (DOE), full factorial with the use of eight (8) levels was introduced in the filler formulation, with periwinkle having weights of 5, 10, 15, 20, 25, 30, 35, 40 g (with steps of 5) while the matrix (polymer) was kept at 125 and 130 g for all samples as determined from the design of experiments. The least composition was 5/95 (filler to matrix ratio) and the most was 40/130 with a corresponding density of 0.988 and 1.11 g/cm^3 in the case of PS; 0.923 and 1.042 in the case of PP and so on. To obtain the appropriate densities, Microsoft Excel was employed in the mathematical computation and values from the rule of mixtures values of the density were noted as the various weight in grams was inputted. Only the mass contributors and density of the matrix were varied during the computation.

Collection and Preparation of Periwinkle Shell

Periwinkle shells were collected from Calabar South Central market, Calabar, Cross River State. It was sorted, washed, sun-dried, and crushed into smaller pieces using a Laboratory Milling Machine.

Dry Ashing of Periwinkle Shell

The Periwinkle shell particles were ash using Muffle Furnace. 1000g of the Periwinkle shell particles were spread in the ceramic sample tray, placed in the furnace chamber and firmly closed. The ashing was done at a temperature of 450ºC for 4 hours. The Periwinkle shell ash was removed and allowed to cool naturally at atmospheric conditions and reweighed to 732.66 g.

Table 2: Formulation Table

Material	Neat Sample	Sample A	Sample B	Sample C	Sample D	Sample E
Periwinkle shell ash	0	10	20	30	40	50
Polymer	100	90	80	70	60	50

Development of the composites.

Sample A, B, C and D were replicated for the five chosen polymers as shown in Table 2 and were labelled A_1, B_1, C_1, D_1, A_2, B_2, C_2, D_2, A_3, B_3, C_3, D_3, A_4, B_4, C_4, and D_4 respectively.

Mixing

Using the formulation shown in Table 2, the composite samples were produced by a mixing process involving the introduction of the polymer pellet while the rolls of the two-roll mill machine were in counterclockwise motion and softened for 5 minutes at a temperature of 170°C. Upon achieving a band and bank formation of the polymer on the front roll, the prepared periwinkle shell ash was introduced gradually to the bank, cross-mixed and allowed to mix for 3 minutes producing a homogenous sample. The composite was sheeted out and labelled accordingly.

Hot Pressing

The composite obtained from the mixing process was placed in a metal mould of dimensions 120 mm x 100 mm x 3.2 mm and put on a hydraulic hot press (Compression Moulding Machine) at a temperature of 150ºC and pressure of 2.5 MPa for 5 minutes. It was cooled, removed from the mould and labelled accordingly.

Tests

The composites developed from the selected polymeric materials were investigated for their suitability in replacing existing automobile car bumpers. Also, a new existing bumper from a vehicle was chosen as the control sample and was subjected to mechanical and physical tests. The rule of mixture was employed to aid the determination of formulation and related parameters of the materials to be used for the development of the car bumper composite.

Water absorption

The water absorption was carried out in accordance with (ASTM D570). Specimens were prepared with dimensions 50mm x 50mm x 3.2 mm. The water absorption test was done by total immersion of specimens in distilled water at room temperature for 7 days. The water absorbed was determined by weighing the samples at regular intervals of 24 hours, with the aid of a digital weighing balance. The percentage water absorption of the composite samples was calculated using Equation 1.

$$\%\text{water absorbed} = \frac{W_2 - W_1}{W_1} \times 100 \quad (1)$$

Where w_1 and w_2 are the final weight and initial weight of the sample respectively.

Tensile Strength

The tensile strength was carried out in accordance with ASTM D-638. Dumbbell-shaped samples were subjected to a tensile force and tensile strength, and tensile modulus percentage elongation at break for each sample was calculated and recorded automatically by the machine and the results were on the certificate.

Impact Strength

The impact test was carried out according to the standard specified ASTM D-156. The specimen was cut to dimensions 64 mm x 12.7 mm x 3.2 mm and 45º notched was inserted in the middle of the test specimens from all the produced composite samples. The impact energy test was carried out using Izod Impact Tester (Resil impactor testing machine). The specimen was clamped vertically on the jaw of the machine and a hammer of weight 1500 N was released from an inclined angle 150°. The impact energy for the corresponding tested specimen was taken and recorded.

Hardness

The hardness test was carried out in accordance with ASTM D2240 on a Mico Vicker Harness Tester. The test was carried out at different positions on each sample and average hardness was calculated and recorded.

Flexural Strength

The flexural strength test on the blends was carried out following ASTM D-790. The specimen measuring 100 mm x 25 mm x 3.2 mm was placed on a support span horizontally at 80 mm gauge length and a steady load was applied to the center by the loading nose producing three-point bending until the sample specimen failed. The maximum load (N) and the corresponding deflection (mm) were recorded accordingly as the sample specimen failed. The flexural strength and flexural modulus were calculated using Equations 2 and 3 respectively.

$$\text{Flexural Strength (MPa)} = \frac{3FL}{2bd^2} \quad (2)$$

$$\text{Flexural Modulus (MPa)} = \frac{FL^3}{4bd^3D} \quad (3)$$

Where, F is Maximum Load at break, L is the distance between the support spans at both edges of the specimen is 80mm, b is Sample width which is 25mm, d = Sample thickness is 3.2 mm

RESULTS AND DISCUSSIONS

The x-ray fluorescence result from the ash periwinkle shell powder (APSP) after calcination and an Un-ash periwinkle shell powder (UPSP) is presented in Table 3.

Table 3: X-ray fluorescence result for periwinkle samples

Element Oxide	SiO_2	Al_2O_3	Fe_2O	CaO	MgO	SO_3	K_2O	Mn_2O_3	P_2O_3	TiO_2
% UPSP	1.8	2.6	0.8	87.6	3.9	0.3	0.1	-	0.01	1.02
% APSP	2.05	3.63	0.24	92.1	0.04	0.2	0.15	-	-	0.01

Higher content of calcium oxide was observed for both samples as similarly reported by several researchers (Ubong and Godwin, 2017; Onueha et al., 2017; Badmus et al., 2007; Job et al., 2009; Koffi, 2008; Umoh and Olusola 2012), however, the percentage CaO content obtained for the UPSP and APSP in this research was significantly greater than that obtained in similar studies such as those reported by Ubong and Godwin (2017) and Onuoha et.al. (2017). The presence of P_2O_3 was negligible while other elements were present. Manganese oxide (Mn_2O_3) was absent in both samples compared. It was also observed that the APSP resulted in a significantly low percentage of magnesium oxide compared with the UPSP.

Observations from the tensile testing of the composites at different periwinkle shell ash filler loading showed that the polypropylene-filled APSP composites possess higher tensile strengths compared with the other polymer-filled APSP as shown in Figure 3.

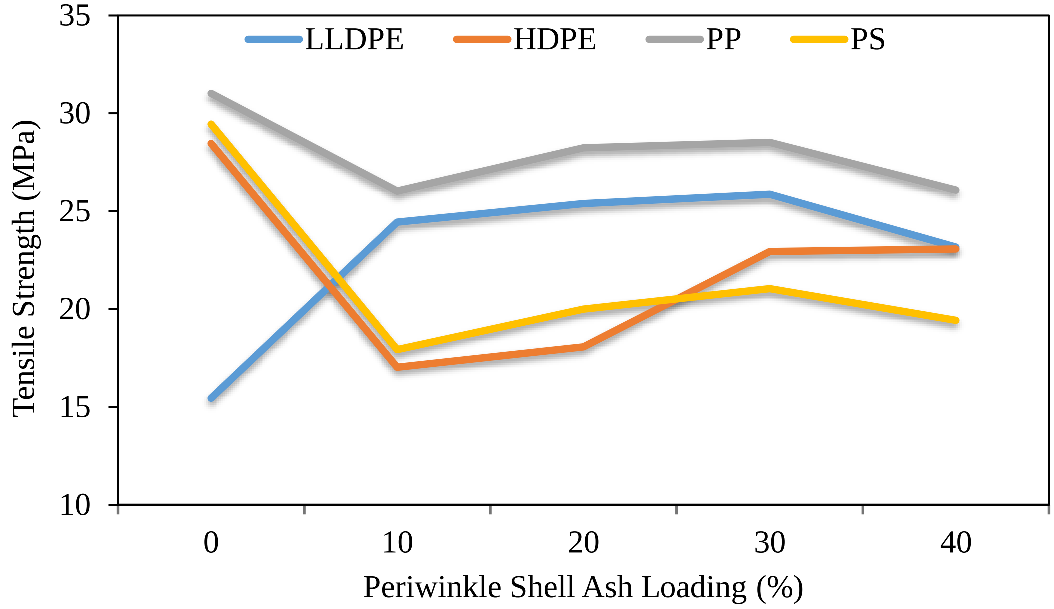

Figure 3: Effects of periwinkle shell ash loading on the tensile strength of polymer composites

The addition of periwinkle shell ash from the 10% filler loading level resulted in an increase in the tensile strength up to the 30% filler loading except for the recycled high-density polyethylene which resulted in a continuous increment in tensile strength up to the 40 % loading level. At filler loadings above 30%, it was observed that the APSP-filled composites exhibited brittle behaviour conforming to reports from Ogunghele and Omowole (2012) and Jamabo et al. (2009). At 30% increment, Composites of A3 resulted in a tensile strength of 26.03MPa, B3 yielded a tensile strength of 28.24MPa and C3 a tensile strength of 28.52MPa, thereby conforming to the reports of Herwandi and Napitupulu (2017) who asserted that good composite materials of such nature exhibit favourable tensile strengths. Shirkuma and Dibraka (2017) stated that with an increment of fillers, as in this case, the mechanical properties reduce and often show brittle properties as observed in this study.

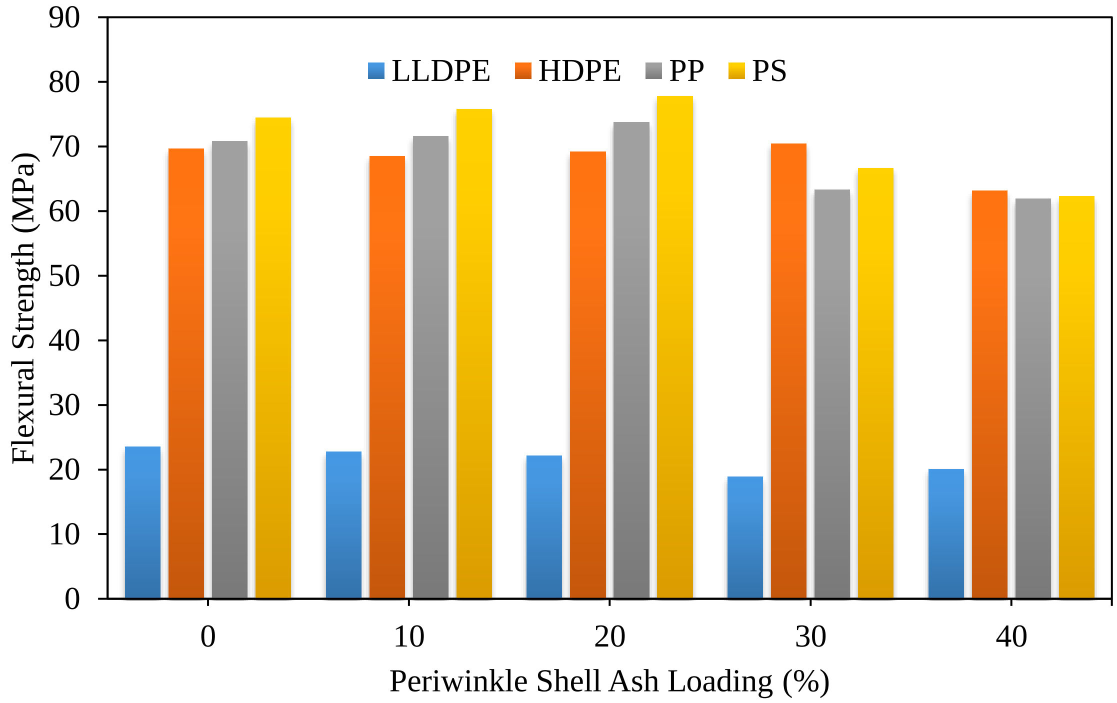

Figure 4: Effects of periwinkle shell ash loading on the flexural strength of polymer composites

The flexural strength of the composites was highest at the 20% filler loading level with the APSP-rPP composite resulting in the highest flexural strength. As shown in Figure 4. The flexural strength shows a significant increase, especially at the first 10% and 20% of APSP loading to the polymers but gradually dropped at 30% and 40% of APSP addition to the composite. This is due to the brittle nature of one of the filler materials (American Chemistry Council, 2014).

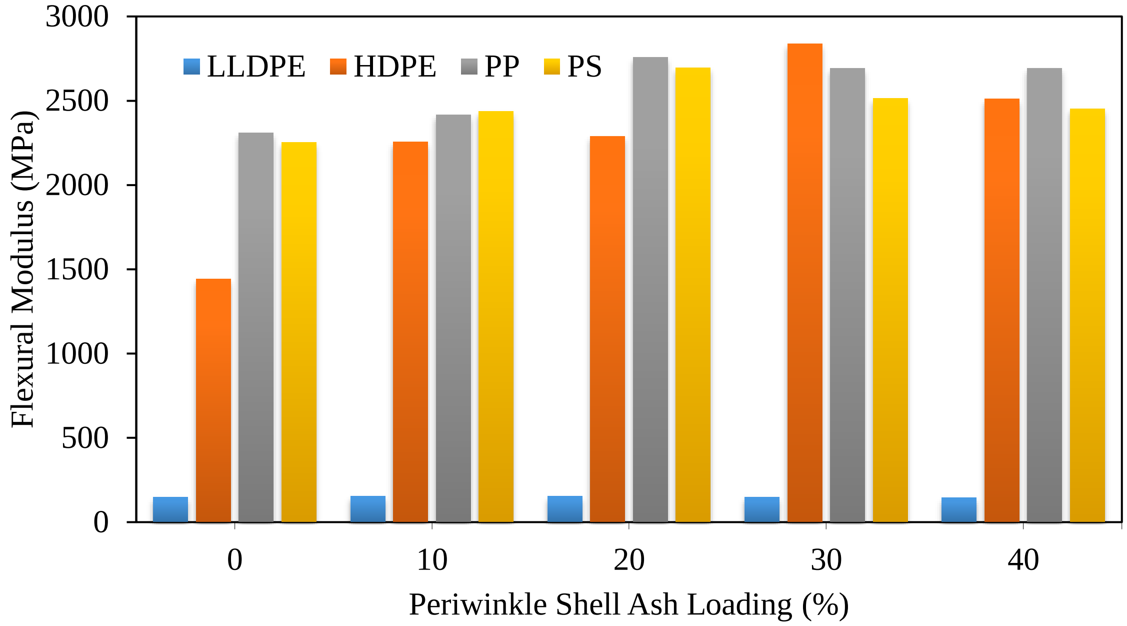

Figure 5: Effects of periwinkle shell ash loading on the flexural modulus of polymer composites

Flexural modulus, also known as bending modulus or modulus of elasticity in bending, measures a material's resistance to bending or flexing under a load. This indicates the stiffness or rigidity of the material. Across all filler loading levels, the APSP-LDPE composites showed a weak flexural modulus compared with other composites as seen in Figure 5. At the 20% and 40% filler loading, APSP-PP showed the highest flexural strength while at the 10% and 30% filler loading levels, the APSP-PS and APSP-HDPE respectively yielded the highest flexural strength. The best flexural strength among the samples was observed at the 30% filler loading. Herwandi and Napitupulu (2017) asserted that a good composite must express certain mechanical properties and its flexural modulus is expected to be excellent.

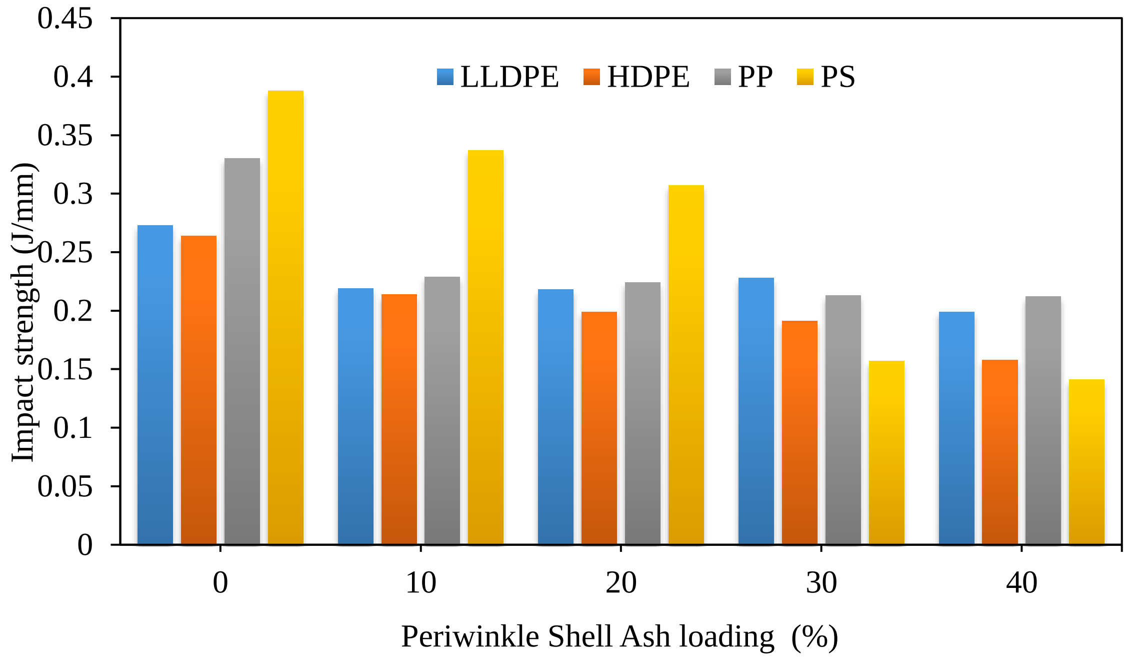

Figure 6: Effects of periwinkle shell ash loading on the impact strength of polymer composites.

The neat recycled polymers showed better impact strength properties compared with the composites filled with the ashed periwinkle shells powder. At 10% and 20% loading, the APSP-rPS composites resulted in the highest impact strength among other composites with the 10% APSP loading giving the highest tensile strength of 0.337 J/mm. As illustrated in

Figure 6, the APSP-rPP composites resulted in the second-highest impact strength at 10% and 20% loading. At 30% and 40% filler loading levels, the composites had lower impact strength compared to other loading levels. While the neat recycled polymers seem to have better impact strength property compared to the filled composites, the APSP-filled composite is still desirable owing to environmental and sustainability considerations since there exist filler loadings that exhibit similar acceptable impact characteristics. Aside from the APSP-rLLDPE composites with the highest impact strength of 0.228 J/mm experienced at the 30% filler loading level, other composites showed that the impact strength reduced with increasing filler loading.

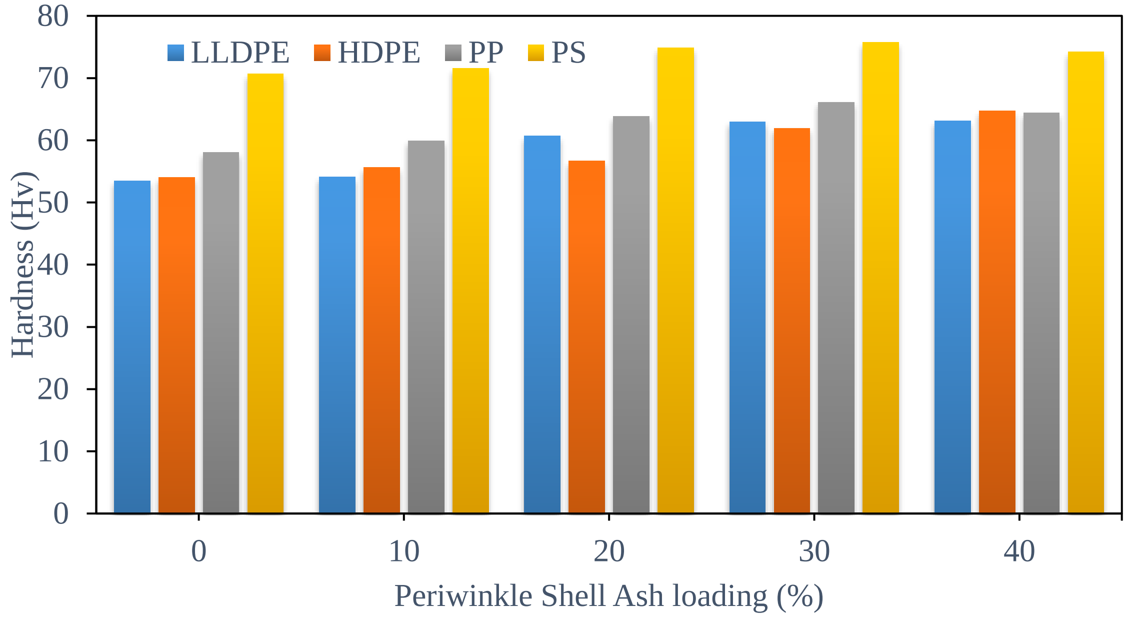

Figure 7: Effects of periwinkle shell ash loading on the hardness of polymer composites

As is common with most bio-filled composites, the hardness increases with filler loading and the composites tend to be brittle at higher filler loading (Dias et al., 2017). The results obtained for the composites in this study are illustrated in Figure 7.

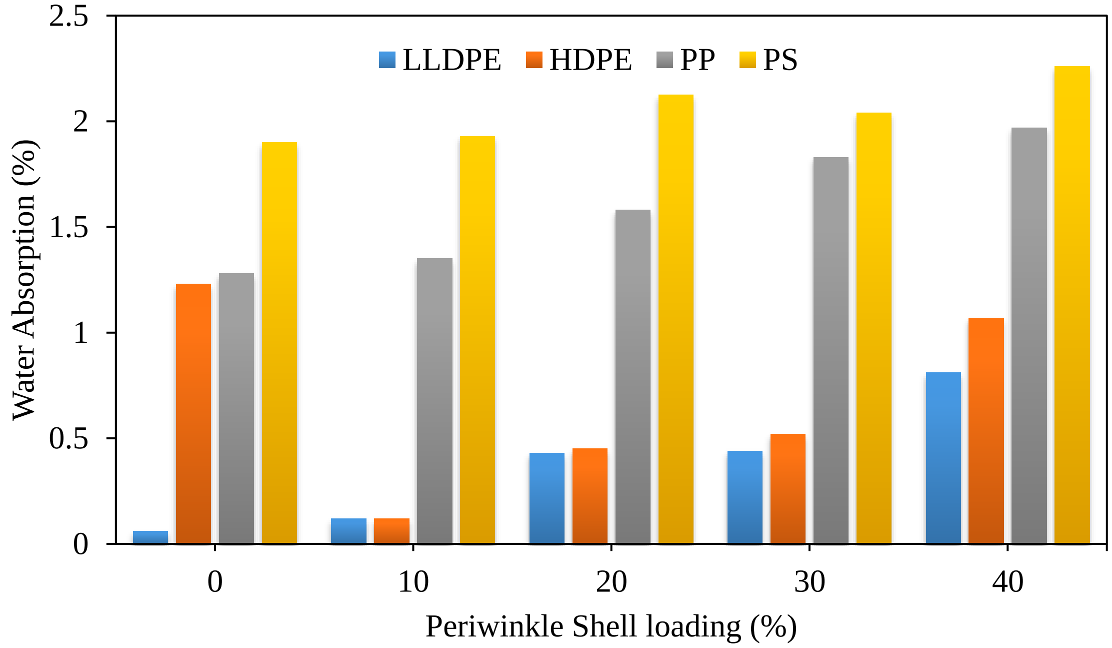

Figure 8: Effects of periwinkle shell ash loading on the water absorption of polymer composites

Powel *et al* 1985 and Amaziah *et al*. (2013) asserted that Periwinkle Shells in water for a long time, loses strength and Orji *et al.* (2017), also discovered that the Shell has a large quantity of $CaCO_3$, which is a good absorber of moisture, it's clear from Figure 8 that the developed composites absorbed moisture with the increment of APSP loading. The seven days of observation was enough time for the composites to absorb moisture. A lower loading levels, LLDPE and HDPE resisted the moisture absorption due to their inert bonding nature but gradually with an increment in the percentage of APSP, they started absorbing moisture due to the brittle nature of a Periwinkle Shell (Ogungbele and Omowole, 2012 and Jamabo *et al*., 2009).

CONCLUSION

The composite production of recycled LLDE, HDPE, PP and PS using periwinkle shell as reinforcement was investigated. It was observed that the recycled PP and PS gave better Mechanical Physical Properties in the combination. The composites having a filler loading level of 30% APSP to 70% recycled polymer yielded the desirable mechanical and physical properties desired for automotive bumper applications. The poor water absorption characteristics are due to the nature of the periwinkle shell and to mitigate this, further research needs to be carried out to see if chemical treatments of the filler before its introduction into the composites and the introduction of additives and use of emulsion will reduce the rate of moisture absorption. The developed composites can serve as cheap alternatives to existing epoxy glass fibre bumpers. The composites with reinforcement levels around 30% can yield composites with more than 100% increase in tensile strength and still offer lower weight and aesthetics.

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