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Empirical models for estimating the mechanical and morphological properties of recycled low density polyethylene/snail shell bio-composites



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Abstract The empirical models for estimating the mechanical properties and morphological of recycled low density polyethylene/snail shell bio-composites was investigated. The snail shell of particle sizes 75, 125, 250 and 500 μm with a weight percentage of 0, 5, 10 and 15 (wt%) with recycled polyethylene (RLDPE) were prepared by compounding and compressive moulding technique. Samples were cut from the panel and subjected to mechanical testing such as tensile, flexural and impact energy. Scanning electron microscope was used to analyse the fracture surface of the samples. Linear regression equation and analysis of variance (ANOVA) were employed to investigate the influence of process parameters on the mechanical properties of the samples. Results obtained showed that: as the wt% snail shell particles increased from 5 to 15, there was a raise in the tensile strength by (2.69) and the flexural strength (1.53). Also the increase in the snail shell particle size from 75 to 500 μm decreased the tensile strength by -5.46 , flexural strength -3.97 and impact energy by -1.97 . The predicted results obtained were in good agreement with the experimental results. Hence, the work can be used for indoor and outdoor structural applications.

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1. Introduction

Composites material have become important engineering materials all over the world because of the unique properties they offer when compared with polymer, metals or alloys. As a result most research and development are focusing on the

development of composite materials. Polymer composites have received the attention of researchers because of low strength, hardness and wear of plastics or polymer for most engineering applications. Polymer composites are now being used in both indoor and outdoor structural applications in housing, construction, auto-industry, aerospace etc.

Natural fillers in the form of fibres of particulate have gained the attention of researchers in recent time as reinforcing materials in polymers, metals and ceramics. They are eco-friendly, low cost, low density materials; they are renewable

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in a large amount when compared with the artificial fillers. Some of the previous studies on the use of natural fillers on the production of polymer composites are: [Patricio et al. \(2007\)](#) studied egg shell (ES) as a new bio-filler for polypropylene composites, water absorption and mechanical properties of high – density polyethylene/egg shell composite were studied by [Hussein et al. \(2011\)](#), the tribological behaviour of recycled low density polyethylene (RLDPE) polymer composites with bagasse ash particles as a reinforcement using a pin-on-disc wear rig under dry sliding conditions was reported by [Aigbodion et al. \(2012a\)](#), [Atuanya et al. \(2011\)](#) investigated the suitability of using recycled low density polyethylene (RLDPE) in wood composite board manufacture, [Aigbodion et al. \(2012b\)](#) studied high density polyethylene (HDPE) composite reinforced with 20 wt% orange peels ash particles and the effect of palm kernel shell on the microstructure and mechanical properties of recycled polyethylene (RLDPE) was reported by [Agunsoye et al. \(2012\)](#).

There are lots of snail shell and waste water sachet (recycled low density polyethylene) waste materials, these wastes constitute nuisance to the environment not only in Africa but the world at large. The ability to convert these wastes into useful engineering materials e.g. composites sharpens the focus of this present research work. From the available literature no investigation has been conducted on the application of the snail shell particles in polymer composite materials. A relationship between the mechanical properties of the polymer composites and the process parameters (particle sizes and weight percentage snail shell) will give a better understanding of the mechanical properties. Based on the above-mentioned situation, the study described in this work intends to study the empirical model for estimating mechanical properties of RLDPE composites reinforced with snail shell particles.

2. Materials and method

2.1. Materials

Pure water sachet (RLDPE) used were collected around the refused dump at the Faculty of Engineering, Nnamdi Azikiwe University, Awka, Anambra State, Nigeria. The RLDPE was washed, dried and pulverized to particles. The snail shell used in this work was brown snail shell obtained from Snail restaurants in Sabongidda-Ora, Edo-State Nigeria. The Snail shell was washed with water and ethanol, sun dried to remove the residual organic matter.

2.2. Equipment

The equipment used for this research were: metal mould, sieves, digital weighing balance, hack saw grinding machine, hydraulic press, compounding machine, Compressive machine and housefield tensometer, scanning electron microscope (SEM).

2.3. Method

40 kg of the dried Snail shell were oven dried at 105 °C for 5 h until all the moisture was completely removed ([Hassan et al., 2012](#); [Atuanya et al., 2014](#)). The dried Snail shell was charged

into a Denver cone crusher and was reduced to a size of 4–3 mm wider and then charged into a roll crusher to reduce the size of snail shells to between 2 and 1 mm. The products from the roll crusher were transferred into a ball milling machine and were left in the mill for 2 h; after which they were transferred into a set of sieves: 500 µm, 250 µm, 125 µm and 75 µm sizes and were sieved for 30 min using a sieve shaker machine. JOEL JSM 5900LV Scanning Electron Microscope equipped with an Oxford INCA™ Energy Dispersive Spectroscopy system was used to determine the Snail shell particle microstructure.

The Snail shell particles were dried in oven (UNB 100–500) for 24 h at 150 °C to a moisture content of 0.5–1% (based on dry weight) before composite production (75 µm, 125 µm, 250 µm, 500 µm) and (0, 5, 10, 15 wt%) were compounded with recycled polyethylene using a co-rotating twin-screw extruder. The temperature used was 160 °C with screw speed of 50 rpm ([Agunsoye et al., 2012](#)). Finally, the mixed samples come out in bulk form. The crushing machine was used to crush into particle form. The composite compounded was fabricated into size of 150 mm × 150 mm × 6 mm. The used temperature was 160 °C with a pressure of 25 Ton for 10 min. Each of the plate was cut into desired dimension for sample testing. Before the test, the samples were conditioned for 24 h at 23 °C and at 50% relative humidity ([Suresha and Chandramohan, 2004](#); [Singha and Thakur, 2008](#)).

The tensile test was determined in accordance with ASTM-D638-10 Standard Test Method for Tensile Properties of Plastics. This test method covers the determination of the tensile strength of unreinforced and reinforced RLDPE in the form of standard dumbbell-shaped test specimens. The samples were aligned properly to prevent bending moment occur during test. The cross-head speed was 5 mm/min and the gauge length was 40 mm. Three samples were tested for each composition and a mean of these samples was taken for Young modulus and tensile strength.

Flexural strength of the three-point loading system applied to a simply supported beam was used. The samples were aligned properly to prevent error measurement during test. The flexural strength was determined in accordance with ASTM D790-10 Standard Test Methods for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials. The test was conducted with a cross-head speed of 5 mm/min and a gauge length of 100 mm. Three samples were tested for each composition and a mean of these samples was taken.

The Izod test method was used to study the sample impact energy in accordance with ASTM D256-10 Standard Test Methods for determining the Izod Pendulum Impact Resistance of Plastics. The sample with dimension 55 × 10 × 4 mm was notched and placed vertically (Izod) in a vice with the notch positioned central to the top of the vice facing the swing patch of the pendulum. The pendulum having a known energy, strikes the sample and the swing height of the pendulum after breaking the sample is measured and subtracted from the calibrated swing height. The result is the absorbed energy which can break the sample.

A full factorial design of experiments of the type P^n ([Miller and Freund, 2001](#)) was used in the study of the mechanical properties where n corresponds to the number of factors and P represents the number of levels. Here i.e.: n corresponds to the number of factors (Particle size and wt% snail shell

particles) and p the number of levels ($P = 2$) (upper and lower levels of each variable, see Table 1). Thus, the number of trial experiments to be conducted for each material is 4 (i.e. $2^2 = 4$). If the response variable is represented by Y , the linear regression equation for these experiments is expressed as (Miller and Freund, 2001; Aigbodion et al., 2012a):

$$Y_{(1,2,3)} = a_0 + a_1A + a_2B + a_3AB \tag{1}$$

Where a_0 is the response variable at the base level, a_1, a_2 are the coefficients associated with each variable A (particle size) and B (amount of snail shell), a_3 the interaction coefficient between A and B within the selected levels of each variable and $Y_{(1, 2, 3)}$ represents tensile strength, flexural strength and impact energy respectively. The methodology for calculating the values for each regression coefficients, using the coded values A and B of each variable is described elsewhere (Buggy et al., 2005).

3. Results and discussion

Surface Morphology of Snail shell particles is shown in Fig. 1a. Snail shell particles were clearly seen to be solid in nature, but irregular in size. Some spherical shape particles and platy like structure can also be observed in Fig. 1a. The snail shell particles surface morphology plays a vital role in case of composite materials. The surface features of particles such as contours, defects and damage and surface layer were not observed in the SEM. The micro-analysis by EDS of the Snail shell particle morphology consists mainly of Ca, O, Si, Mg and Na elements (see Fig. 1b). The relative atomic percent of the atoms were obtained from the peak area and corrected with an appropriate sensitivity factor. The Snail shell particles showed a higher proportion of calcium atom. The higher proportion of calcium in the particles can be attributed to

the abundant presence of calcium in the Snail shell particles. The ratio of calcium to oxygen was 2.3:1.8 this ratio was attributed to also high level of oxygen because the Snail shell particles occurred in oxide form which was different from the ratio of $CaCO_3$. These results are consistent with the work of other co-workers that work on eggshell (Hussein et al., 2011; Patricio et al., 2007).

Fig. 2 shows the variation of tensile modulus and strength with wt% Snail shell particles. There was a significant increment in the tensile modulus and the strength as the snail shell

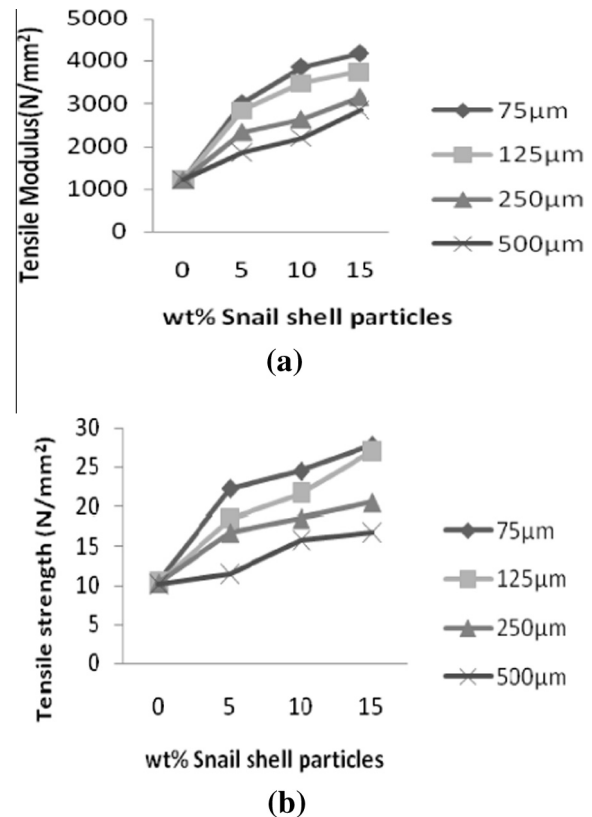
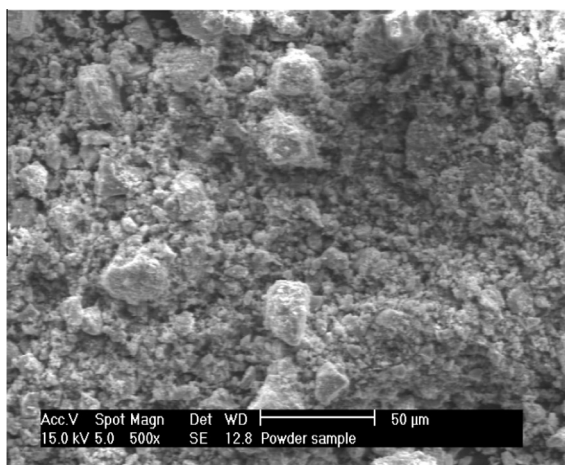


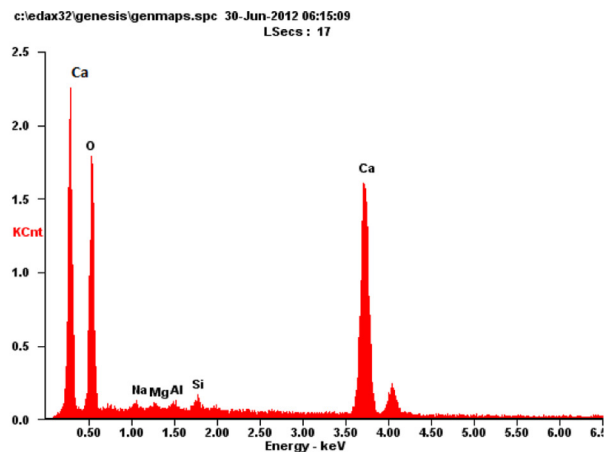
Figure 2 (a) Tensile modulus and (b) strength variation with wt% snail shell particles.

Table 1 Upper and lower levels of each factor along with their coded values.

S.No	Variables	Upper level	Lower level
A	Particle size (µm)	500 (+1)	75 (-1)
B	Amount of snail shell (wt%)	15 (+1)	5 (-1)



(a)



(b)

Figure 1 (a) SEM observation and (b) EDS analysis of the snail shell particles.

particle loading increased, smaller Snail shell particle sizes showed higher increments. The increment may be due to the platy structure of the Snail shell filler providing good reinforcement (Hassan et al., 2012; Hussein et al., 2011).

The increase in modulus of the Snail shell-filled composites indicates an increase in the rigidity of RLDPE related to the restriction of the mobility in RLDPE matrix due to the presence of snail shell particles. The modulus of these composites increased with increasing Snail shell particle loading. This suggests stress transfer across the polymer-particle interface. Snail shell in the matrix prevented movement in the area around each particle, contributing to an overall increase in the modulus. The high modulus values also support the use of the developed composites in indoor and outdoor applications which was in par with the work of Hussein et al. 2011. Fig. 3 depicts the variation in flexural modulus and strength with wt% Snail shell particles. The flexural modulus and strength rise with the increment in wt% Snail shell particles. The rate of increase of flexural modulus and strength was comparable to the increment in wt% Snail shell particles.

The size of the Snail shell particles influenced the tensile and the flexural strengths (see Figs. 2 and 3). Snail shell particles were divided into coarse particles (around 500 μm), large particles (around 125 and 250 μm) and fine particles (around 75 μm) according to their sizes. The mechanical interlocking between Snail shell particles and matrix (RLDPE) significantly affected the properties (Agunsoye et al., 2012; Bledzki and Gassan, 1999).

The high values of strength observed in this work may be due to the fair distribution of the Snail shell particles in the RLDPE matrix resulting in strong particles–RLDPE matrix interaction. The particle dispersion improved the particles–RLDPE matrix interaction and consequently increases the ability of the Snail shell particles to restrain gross deformation of the RLDPE matrix.

The impact energy of a composite is influenced by many factors: including the toughness properties of the reinforcement, the nature of interfacial region and frictional work involved in pulling out the particles from the matrix (Atuanya et al., 2014). Fig. 4 depicts the variation in impact energy with wt% Snail shell particles. The Impact energy decreased with increment in the wt% Snail shell particles. This may be attributed to the interference by the filler in the mobility or deformability of the matrix. This interference was created through the physical interaction and immobilization of the polymer matrix by imposing mechanical restraints (Hassan et al., 2012; Imoisili et al., 2013).

It can be seen that the impact energy of the composites slightly decreases with increasing filler loading. Increased filler loading in the RLDPE matrix resulted in the stiffening and hardening of the composite. This reduced its resilience and toughness, and led to lower impact energy which is in par with the work of Atuanya et al. (2014) and Raju et al. (2012). As the loading of Snail shell particles increases, the ability of the composites to absorb impact energy decreases since there is less ratio of the RLDPE matrix to particles. The results obtained in this work are within the standard level for bio-composites for indoor and outdoor applications (Hussein et al., 2011).

For the modelling of the mechanical properties, the upper level and the lower level of each variable along with their coded values used in this investigation are shown in Table 1. The design of the experiments and the values of respond

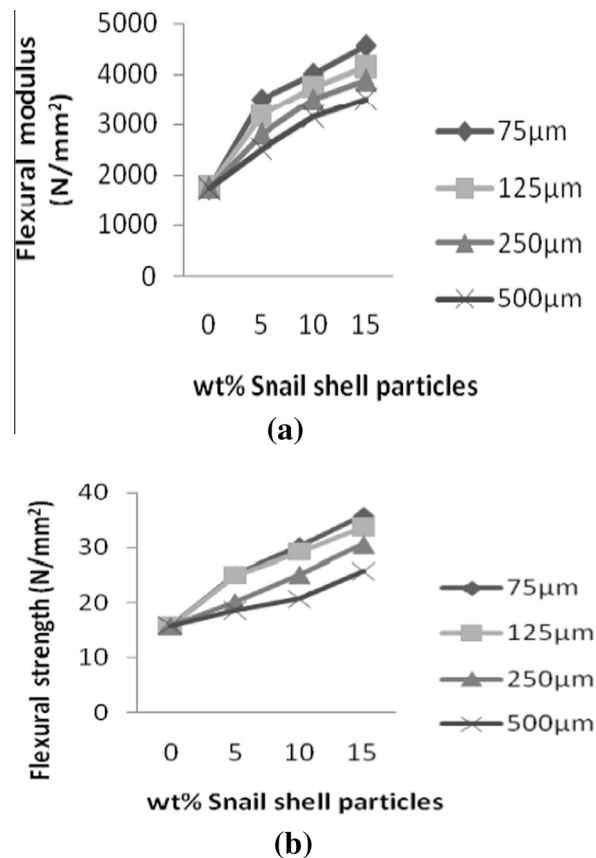


Figure 3 Variation of (a) the flexural modulus and (b) the flexural strength with wt% snail shell particles.

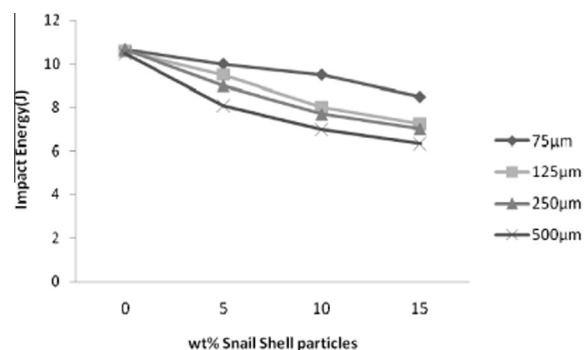


Figure 4 Variation of impact energy with wt% Snail shell particles.

variables corresponding to each set of trial are reported in Table 2. The respond variables in each trial represent the average of three measured data at identical experimental conditions.

From the factorial design and the stepwise variation of the two factors, the estimated response surfaces represents the best fit of the experimentally obtained values. Figs. 5–7 show the estimated response surface for the tensile strength, the flexural strength and the impact energy as a function of particle size and wt% Snail shell particles. It can be seen that both the tensile and the flexural strength were highly

Table 2 Matrix design for calculating the regression co-efficient and ANOVA.

S.No	A	B	AB	Tensile strength (N/mm ²)	Flexural strength (N/mm ²)	Impact energy (J)
S1	+1	+1	+1	16.71	25.70	3.71
S2	+1	-1	-1	11.5	18.50	7.30
S3	-1	-1	+1	22.26	25.10	10.50
S4	-1	+1	-1	27.8	35.76	7.50

Note: +1 = upper level, -1 = lower level.

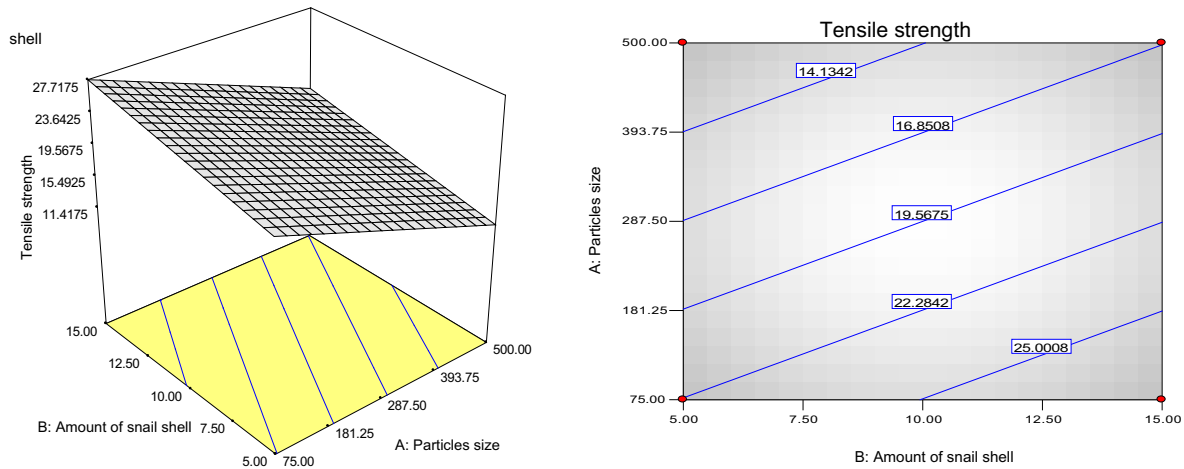


Figure 5 Response variation of tensile strength with interaction between snail shell particle size and wt%.

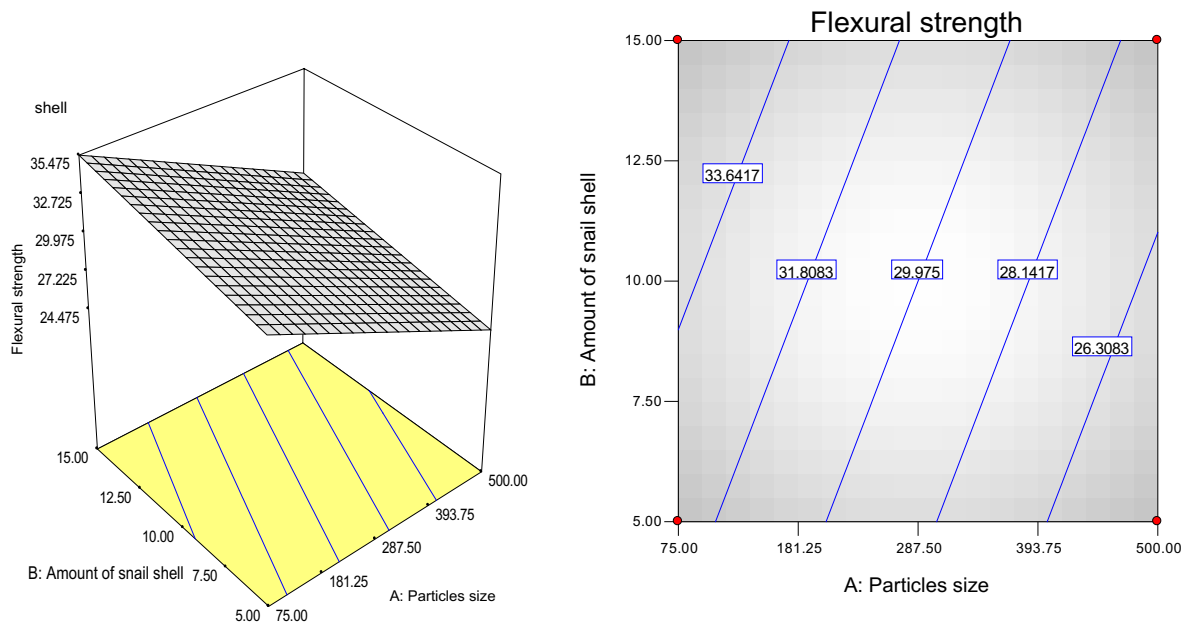


Figure 6 Response variation of flexural strength with interaction between snail shell particle size and wt%.

influenced by the Snail shell particle content. For the Snail shell particle size the tensile and the flexural strength were found to raise with decrement in the particle size from 500 to 75 μm . However, the estimated response surface indicates maximum composite strengths at 15 wt% Snail shell and 75 μm particle size (see Figs. 5 and 6). The impact energy

is highly influenced by the Snail shell particle content and particle size. The impact energy was found to raise with a decrease in the particle size (500–75 μm) and wt% snail shell particles (15–5). However, the estimated response surface indicates maximum composite impact energy at 5 wt% snail shell and 75 μm particle size (see Fig. 7).

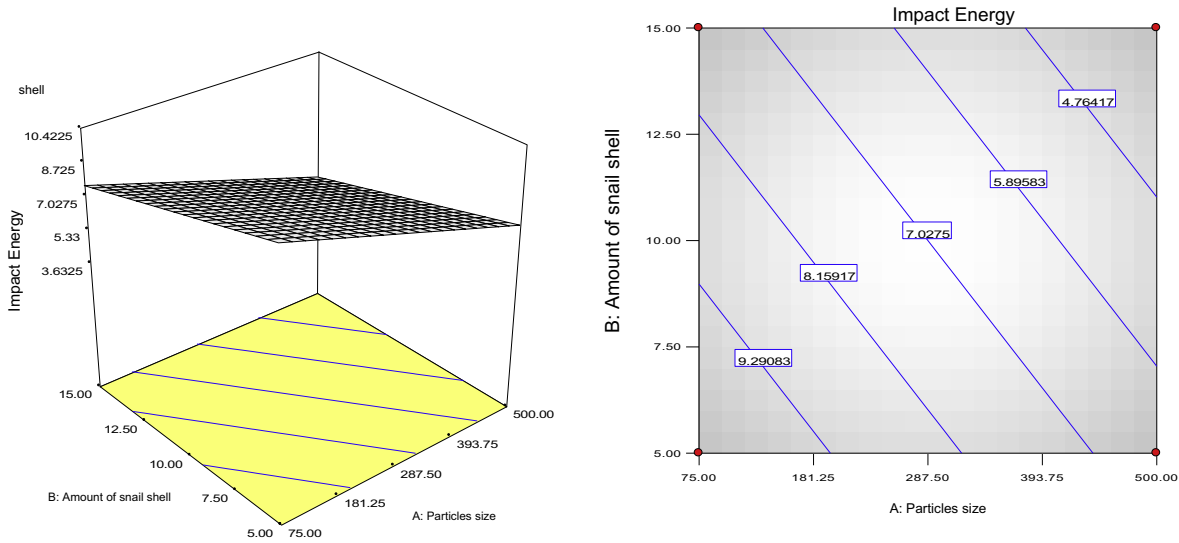


Figure 7 Response variation of impact energy with interaction between snail shell particle size and wt%.

ANOVA was used to determine the design parameters significantly influencing the tensile strength, the flexural strength and the impact energy. Table 3 shows the results of ANOVA. The analysis was evaluated at confidence level of 95%, that is for significance level of $\alpha = 0.05$ (Miller and Freund, 2001). The last column of Table 3 shows the contribution (P) of each parameter on the response, indicating the degree of influence on the results. For the ANOVA of Tensile strength Test, the fit was exact and the R^2 value is 0.9971. The Model F -value is 2722.61 implies, the model is significant, flexural strength the R^2 value is 0.9994. The Model F -value is 14501.00 implies, the model is significant and for impact energy the ANOVA was also fit, exact and the R^2 value is 0.9838 with Model F -value of 492.34 which, also implies that the model is significant. Values of “Prob > F ” less than 0.0500 indicate that the model terms are significant. In this case, A (particle size)

and B (wt% snail shell) were significant model terms. Values greater than 0.1000 indicate that the model terms are not significant (see Table 3).

The mechanical properties such as tensile, flexural, and impact strength were modelled using Design Expert statistical software. Eqs. (2)–(4) were the developed nonlinear regression models for tensile strength (Y_1), flexural strength (Y_2) and impact strength (Y_3) respectively.

$$\text{Tensile strength } (Y_1) = +19.57 - 5.46 \times A + 2.69 \times B \times \text{N/mm}^2 \quad (2)$$

$$\text{Flexural strength } (Y_2) = +29.97 - 3.97 \times A + 1.53 \times B \times \text{N/mm}^2 \quad (3)$$

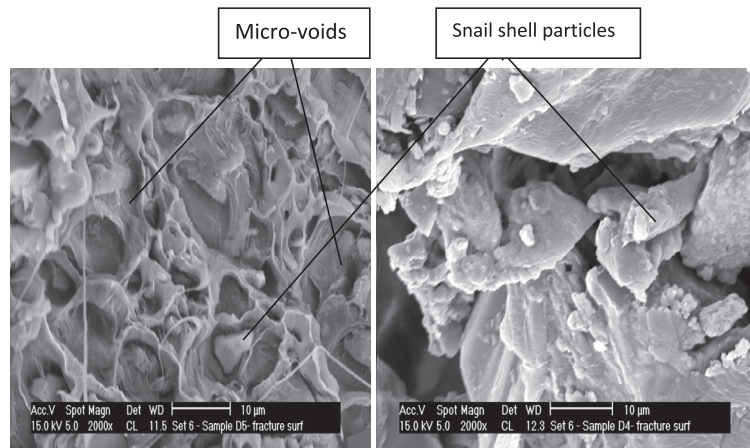
$$\text{Impact Energy } (Y_3) = +7.03 - 1.97 \times A - 1.42 \times B \times J \quad (4)$$

Table 3 ANOVA for Selected Factorial Model.

Source	Sum of squares	Tensile strength			P_{value}	Remarks
		DF	Mean square	F_{value}		
Model	148.25	2	74.12	2722.61	0.0136	Significant
A	119.36	1	119.36	4384.04	0.0096	
B	28.89	1	28.89	1061.18	0.0195	
Residual	0.027	1	0.027			
CorTotal	148.27	3				
<i>Flexural strength</i>						
Model	72.50	2	36.25	14501.00	0.0059	Significant
A	63.20	1	63.20	25281.00	0.0040	
B	9.30	1	9.30	3721.00	0.0104	
Residual	2.500E-003	1	2.500E-003			
CorTotal	72.51	3				
<i>Impact energy</i>						
Model	23.66		11.83	492.34	0.0319	Significant
A	15.56	1	15.56	647.78	0.0250	
B	8.09	1	8.09	336.90	0.0346	
Residual	0.024	1	0.024			
CorTotal	23.68	3				

Table 4 Validation of mathematical model.

Std.	Actual values			Predicted values			% of error		
	TS (N/mm ²)	FS (N/mm ²)	IM (J)	TS (N/mm ²)	FS (N/mm ²)	IM (J)	TS (N/mm ²)	FS (N/mm ²)	IM (J)
S1	22.26	32.40	10.50	22.34	32.47	10.42	0.36	0.22	0.76
S2	11.50	24.50	6.40	11.42	24.47	6.48	0.70	0.12	1.25
S3	27.80	35.50	7.50	27.72	35.47	7.58	0.35	0.08	1.07
S4	16.71	27.50	3.71	16.79	27.53	3.63	0.47	0.11	2.16
Average absolute error							0.47	0.13	1.85

a) Composite of 500 μm at 15 wt% snail shell, b) Composite of 75 μm at 15 wt% snail shell**Figure. 8** SEM tensile fracture surface of the composites.

Where (*A*) and (*B*) were the coded values of particle size and wt% snail shell particles respectively. The value of a_0 for tensile, flexural strengths and impact energy was 19.57, 29.97 and 7.03 respectively. It represents the respond variable value at the base level. By substituting the coded values of the variables for the experimental conditions in Eqs. (2)–(4), the tensile strength, the flexural strength and the impact energy for the composites can be calculated. It was noted that from Eqs. (2) and (3) that the coefficients of wt% Snail shell particles (*B*) were found to be positive. It indicates that increase in wt% Snail shell particles from 5 to 15 raises the tensile strength by (2.69) and the flexural strength by a factor of (1.53). Also, the coefficients of particle size (*A*) were found to be negative. It indicates that raise in particle size from 75 to 500 μm decreased the tensile strength by -5.46 , the flexural strength by -3.97 and the impact energy by -1.97 (see Figs. 4–6). Confirmation experiments were conducted for four sets of conditions. The actual values and the predicted values obtained from the Regression model were compared (see Table 4). The percentage of error was calculated using Eq. (5) for the validation of the Regression model (Miller and Freund, 2001).

$$\% \text{ of error} = \frac{\text{Actual value} - \text{Predicted value}}{\text{Actual value}} \times 100\% \quad (5)$$

From Table 4, the averages absolute error for the tensile strength, the flexural strength and the impact energy are found to be 0.47, 0.13 and 1.85% respectively, which means that a better accuracy was obtained using the developed Regression models.

The fracture surfaces of the composites were examined using JEOL JSM-6480LV scanning electron microscope. Fig. 8a shows the composite microstructure at 500 μm with 15 wt% Snail shell particles. The microstructure showed that the Snail shell particles are embedded in the polymer matrix. Snail shell particles are not broken and there were voids around the particle indicating poor interaction. The SEM of the fracture surface of the RLDPE and its composite at 15 wt% Snail shell particles at 75 μm is shown in Fig. 8b. Morphological properties' result shows that there is proper intimate mixing of Snail shell particles with the RLDPE in the bio-composites synthesized. These factors are responsible for the increases in the results of the tensile, flexural strengths and impact energy obtained in the composites with smaller particle size.

4. Conclusions

The effects of Snail shell particles reinforced RLDPE composites have been investigated as a function of filler loading and particle size. Based on the results and discussion above the following conclusions can be made: The incorporation of the Snail shell particles in the RLDPE polymer matrix as a reinforcement increases the tensile and the flexural strength of the material. Increases in wt% Snail shell particles from 5 to 15 raises the tensile strength and the flexural strength by a factor of 2.69 and 1.53 respectively. Increases in Snail shell particle size from 75 to 500 μm decreased the tensile strength by -5.46 , the flexural strength by -3.97 and the impact energy

by -1.97 . Factorial design of the experiment can be successfully employed to describe the mechanical properties of the samples and the developed linear equation models can be used in predicting the mechanical behaviour of the materials within the selected experimental conditions. Based on the results obtained in this study, it is recommended that the composites can be used in the production of indoor and outdoor applications.

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