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FFECT OF CALCINATED PALM KERNEL SHELL POWDER AS REINFORCEMENT ON THE PHYSICO-MECHANICAL PROPERTIES OF RECYCLED LOW-DENSITY POLYETHYLENE COMPOSITES FOR INTERLOCKS

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ABSTRACT

The change in physico-mechanical properties of recycled low-density polyethylene (rLDPE) composites with the incorporation of calcinated palm kernel shell powder was thoroughly investigated through the D-Optimality mixture design of the experiment. In particular, a focus was made on the long-term properties via mechanical and physical parameters of the thermoplastic composites. The mixing method adopted was a two-roll milling method at 150^{o}C operating temperature for 10 minutes. The matrix (rLDPE) was allowed to melt and formed a band around the roll before introducing the filler. The filler was added to the matrix. The addition was gradually through the roll nip with consistent cross-mixing with a cutting knife until a homogeneous mixture of the filler and the recycled polymer was achieved. Composites were produced using calcinated PKs powder as filler ranging from 10% to 50% at 10% loading intervals and then compressed with a a compression moulding machine at 150°C for 5 minutes under a pressure of 2.5 MPa. A correlation between the composite's compositions and properties was reported based on the obtained data. The results showed that thermoplastic composites' properties were significantly affected at p<0.05, and correlation coefficients for the proposed model equation were more than 9.0. Moreover, it was observed that increasing the filler content (CPKs) with a decrease in rLDPE showed better-combined characteristics for optimal selection. Therefore, Optimised variables for producing plastic interlocks have the proposed end use of the composite established at 60.8% rLDPE and 39.2% CPKs. Hence, the study revealed that using calcinated palm kernel shell powder as filler in recycled low-density polyethylene composites (rLDPE) enhances the physico-mechanical properties of the composites and is suitable for the fabrication of plastic interlocks.

KEYWORDS
Calcinated, recycled, reinforcements, composites, optimum, interlocks

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INTRODUCTION

Many polymer composites require materials with a combination of properties that the conventional metal alloys, ceramics and polymeric materials cannot meet. Most of these properties can be attained by combining at least two phases involving a matrix and reinforcing phases bonded together in a continuous phase to produce useful material with improved properties (Bledzki & Gassan, 1999). One of these phases is a continuous phase (matrix), while the other is a dispersed phase (reinforcement). In composites, materials are combined in such a way as to enable us to make better use of their virtues while minimising, to some extent, the effects of their deficiencies on the immediate environment via blockages of drainage (Yagyu, 2014). According to Hassan *et al.* (2012) and Apasi (2015), composite materials offer great potential in reducing weight because of natural fibre usage. Other advantages of natural fibres over inorganic fibres are renewability, biodegradability, abrasion resistance, low cost, good thermal properties, high toughness and non-corrosive (Tudu, 2009; Mohanty et *al.*, 2000; Malkapuram *et al.,* 2008).

It was evident that the utilisation of treated banana stalk fibre as reinforcement in LDPE and polyester improved the physico-mechanical properties of thermoplastic and thermosetting composites, respectively (Babatunde *et al.*, 2016; Emekwisia *et al.,* 2019). Muhammad and Ahmed (2020) confirmed that the use of natural fibre (banana-pseudo-stem-fibre) as reinforcement to epoxy sandwich structure and synthetic sandwich composites showed that optimal fibres improved the impact and CAI properties of the structure with low peak load and larger damage area in the optimal banana/epoxy structures. The physical, mechanical, and thermal properties of banana fiber-HDPE composites were studied by Neher *et al.* (2022). They investigated different properties' increment or decrease nature due to adding banana fibre in BF-HDPE composites.

Studies by Ahmed *et al.* (2019) and Essam *et al.* (2021**)** revealed the effect of pure polypropylene (PP) and PBS on the physico-mechanical properties of PP/HDPE and PBS/HDPE-g-MA composite, respectively. The results showed that PP offered better elastic modulus and strength due to the methyl attached to the carbon. Structural characterisation of the composites through FTIR spectra analysis confirmed that different ratios of PBS and HDPE-g-MA to the HDPE matrix resembled those of the neat HDPE. However, methods employed in the composites analyses comprised agro-fibre/filler as reinforcing phase over recycled low-density polyethene

needs to be addressed in the process variable domain, which could also cause drawbacks to the efficient use of natural fibre/filler. Hence, it is necessary to optimise these parameters toward physico-mechanical properties. This work aims to develop and characterise the thermoplastic composites using carbonised palm kernel shell powder as reinforcement in producing plastic interlocks and the influence of continuous phase (matrix) and discontinuous phase (filler).

MATERIALS AND METHODS

The recycled low-density polyethylene (rLDPE) and waste water sachets were sourced from various illegal dumping sites that pose environmental hazard threats to the inhabitants of the Nigerian Institute of Leather and Science Technology [NILEST], Samaru-Zaria metropolis and were free from foreign materials via washing with detergent and finally sun-dried. The rLDPE was further reduced into several small portions before being shredded into pieces by a plastic film shredding machine (DHS-20). The palm kernel shell was sourced from Sabon Gari Market, Sabon Gari Local Government, Kaduna State, Nigeria. Lubricant (processing oil) was utilised to lubricate the standard mold size 160 mm x 140 mm x 3 mm.

Calcination Process of Palm Kernel Shell

The Palm kernel shell, used as reinforcement in the composite, was crushed in a crusher machine (DPA200-3)**.** The crushed shell of 800g was packed in a crucible graphite and calcinated in an electric furnace (Muffin U.S.A, model 6019) at a temperature of 210 ^{0}C for 12 hours. The resulting black solid (calcinated palm kernel shell particles) was then grounded, sieved through a 75 μm to obtain fine powder and stored in controlled conditions at 50% relative humidity and 25 ^{0}C before use according to ASTM standards.

Design of Experiment by Mix-Design

The mixture Design of the experiment employed in this study, D-O, optimality mix-design together with point exchange, was considered the best design because it produces a design that best estimates the effect of factors and picks points that minimise the volume of the ellipsoid for the coefficient. These techniques are based on a multivariate model consisting of an experimental design to provide sufficient and reliable response values, provide a mathematical model that best

fits the data obtained from the experimental design, and determine the optimum value of the independent variables (Al-Salihi *et al.*, 2022).

Table 1: Selected process parameters at different levels

Component	Sieve Size	Name	Units	Type	Coded Low	Coded High
A		A: RLDPE	%	Mixture	+0 ↔ 50	+1 ↔ 90
B	75μm	B: Carbonized Shell powder	%	Mixture	+0 ↔ 10	+1 ↔ 50
				Total=	100.00	**L_Pseudo Coding**

Thermoplastic composites with the composition of calcinated PKs and rLDPE ratio were formulated by mix-design of the experiment as shown in Table 1, with each independent variable of low (0) and high (1) coded factors. Design Expert@ (13.0.1) software was used for the ANOVA, regression and graphical analysis of the test data statistically at probability ($p<0.05$). Mechanical and physical properties were taken as the response for the design of the experiment. The experimental data obtained by the above procedure was analysed by the mixture design of response surface methodology by design expert using empirical equation (1)

$$Y_{responses} = \beta_i X_i + \beta_J X_J + \beta_{iJ} X_i X_J + X_i X_J \sum_{i,j}^{n} \left[\beta_{ij}^{*}(A-B) + \beta_{ij}^{**}(A-B)^2\right] \quad (1)$$

Y is a response, i and j are linear coefficients, X_i and X_j are coded independent variables of A and B, respectively, β is the regression coefficient, and n is the number of factors studied.

Composites Formation

Table 2: Mixing ratio of thermoplastic composite for interlock production

Run	rLDPE	B:CPKs	Tensile Test	Hardness	Impact Test	Abrasion	Water Absorption
	(%)	(%)	(MPa)	(Shore)	(J/mm)	(%)	(%)
1	90	10	36.00	85.00	1.988	1.140	4.25
2	80	20	36.13	86.67	1.144	1.055	6.41
3	90	10	36.00	85.00	1.988	1.140	4.25
4	50	50	35.00	96.67	0.857	0.659	5.89
5	70	30	44.97	91.00	0.989	0.563	5.83
6	50	50	35.00	96.67	0.857	0.659	5.89
7	50	50	35.00	96.67	0.857	0.659	5.89
8	90	10	36.00	85.00	1.988	1.140	4.25
9	60	40	28.60	91.33	0.889	0.639	2.37
10	70	30	44.97	91.00	0.989	0.563	5.83

Each prepared sample of rLDPE and calcinated palm kernel shell powder at 75μm size was mixed on the two-roll mill (Model 5189) following the mixing ratio generated by design expert software (Table 2). The shredded waste water sachets (rLDPE) were introduced into the nip of the rolls rotating oppositely to each other at mixing conditions of 150°C for 10 minutes until the plastic paste was achieved, followed by the addition of calcinated powder of palm kernel shell, cross-mixed severally, sheet out and the samples obtained were labelled accordingly. Afterwards, composite test specimens were produced using a hot press compression moulding machine (Carver Inc., Wabash, U.S.A-Model 3851-0) at 130°C for 5 minutes at 2.5 MPa in a standard mould size of 160 mm x 140 mm x 3 mm. All samples were pre-heated before compression for five minutes under heat and cooled for three minutes after compression moulding.

Composite Characterisation

The various groups of prepared polymer composites were characterised by each sample's physico-mechanical properties and behaviours at different test conditions. Tensile, hardness, impact, abrasion and water absorption were considered in evaluating the produced composite. The hardness test was carried out according to the (ASTM D412, 1983) method by placing the indentor of the hardness testing machine (Muver Francisco, Munoz Irles- Model 5019) on the composite

sample at three different points, and the average was taken and recorded. The average value obtained via equation (2) was then utilised as a required response for the test.

The water absorption test, in accordance with ASTM D570 standard, was determined by taking the initial weight of the samples and the weight after dispersing them in distilled water for 24 hours at room temperature. The change in weight of the samples via digital weighing balance (Mehler Instruments Ltd AE 200) was determined by subtracting the initial weight of each sample from their respective final weight. The percentage water absorption of each sample was calculated using equation (3).

The mechanical strength of the composite was investigated through the tension and impact test. The tensile strength test was carried out following the ASTM standard D638 using the Housfield Monsanto tensometer (W6465) maintained at a cross speed of 10 mm/min. Dumbbell-shaped samples were subjected to tensile force, and tensile properties such as tensile strength, strain and modulus were determined. Three samples were tested in each case, and the average was recorded according to equation (4).

The impact test was carried out according to the ASTM D256 test standard using a real impact tester (CEAST real family 6957-500). This test is a standardised high-strain rate test, which determines the amount of energy absorbed by a material before fracture. This absorbed energy is a measure of composite materials' notch toughness. The specimen was cut into dimensions 80 mm x 30 mm x 3 mm from all the samples. The impact energy of the corresponding tested specimen was taken and recorded. These values were used to calculate the impact strength using equation (5).

The abrasion test was done manually using an abrader. After measuring each sample's initial value, the sample was placed on the machine's sample holder. Then, the machine switch was on, and the equipment rotated randomly. Then, the samples were measured to get the final value of each sample. The time taken for abrasion was one minute.

$$Average\ Hardness\ Test = \frac{Sum\ of\ test}{number\ of\ observation}\text{s} \quad (2)$$

$$\%\ Water\ Absorption\ = \frac{Final\ Weight - Initial\ Weight}{Initial\ Weight} \times 100 \quad (3)$$

$$Tensile\ strength = \frac{Force}{Cross\text{-}sectional\ Area} \quad (4)$$

$$Impact\ Test = \frac{Average\ impact\ strength}{sample\ thickness} \quad (5)$$

RESULTS AND DISCUSSIONS

Statistical Model Analysis

Table 3: Analysis of variance for thermoplastics composite

Source	**SS_R**									
	d_f	**Tensile Test**	**d_f**	**Hardness Test**	**d_f**	**Impact Test**	**d_f**	**Abrasion Test**	**d_f**	**Water Absorption**
Model	2	2.07*	1	214.50*	3	2.37*	2	0.5403*	3	12.30*
Linear Mix	1	1.64*	1	214.50*	1	1.91*	1	0.4194*	1	1.29
AB		0.4292 *				0.4154*		0.1210*****		0.1038
AB(A-B)						0.445*****				10.90*****

Key: **, **, significantly at p<0.01 and p<0.05*

The statistical model of the composite depends on significant terms to the proposed model equation. ANOVA analysis indicated that the process parameters (rLDPE/CPKs) have a significant ($p<0.05$) contribution to the model with p-values less than 0.05. This implies that the model terms are significant; otherwise, insignificant models with modification by transformation or reduction become necessary. Table 3 summarises the effect of mixture terms and product terms. Mixture components A and B were highly important for the preparation of the composite. At the same time, interaction products {AB and AB(A-B)} were found necessary to the proposed impact model alone because of the significant level ($p<0.05$) of interaction factor.

Table 4: Regression coefficients of coded variables

Coefficient	Tensile	Hardness	Impact	Abrasion	Water Absorption
β_i	6.00	84.76	1.98	1.2	4.18
β_j	5.00	96.25	0.85	0.66	5.82
β_{ij} *p-value*	1.90* (<0.0001)	-	1.87* (<0.0001)	1.01* (<0.0083)	0.94 (<0.595)
β_{ij}^{*}		-	-1.66* (<0.0013)	-	25.92* (<0.0012)
β_{ij}^{**}	-	-	-	-	-

Key: *, **, *significant at p<0.01 and p<0.05*

It was evident that the rLDPE and CPKs powder significantly (p<0.05) affected the physico-mechanical properties of the composite. However, interaction terms of AB and AB(A-B) were found to be insignificant to the hardness properties of the composite at (p<0.05). The sum of squares for each model was found significant at p< 0.05 with adequate precision greater than 4; this implies that the assumed models have consistent reproducibility and signal-to-noise ratio (S/N) above this value can be used to navigate the design space. The results from the regression coefficient (Table 4) implied that model equation (1) was reduced to equations 6 to 10, with only significant coefficients being considered in model reduction.

Results of Composite Characterisation

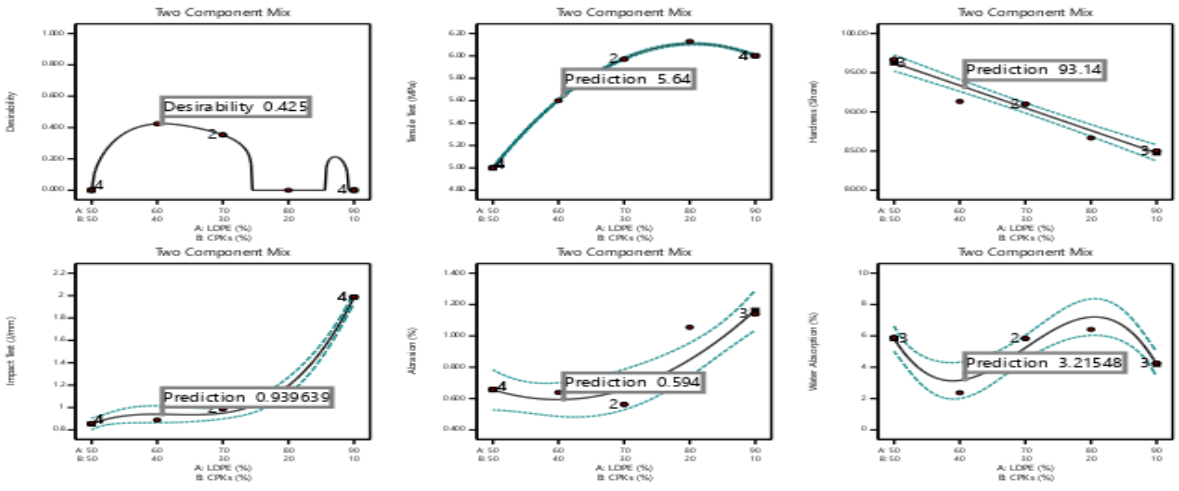

Figure 2: Analyses the effect of filler loading on composite properties

$$Y_{tensile} = 6X_i + 5X_J + 1.9X_iX_J \quad (6)$$

$$Y_{hardness} = 84X_i + 96.25X_J \quad (7)$$

$$Y_{impact} = 1.98X_i + 0.85X_J + 1.87X_iX_J - X_iX_J\sum_{i,j}^{n} \quad [1.66(A-B)] \quad (8)$$

$$Y_{abrasion} = 1.2X_i + 0.7X_J + X_iX_J \quad (9)$$

$$Y_{water\ abs} = 4.2X_i + 5.82X_J + X_iX_J\sum_{i,j}^{n} \quad [25.92(A-B)] \quad (10)$$

Figure 2 shows the analysed effect of filler loading on composite properties. The analysed results for tensile strength of produced thermoplastic composites increase with a decrease in filler loading (CPKs) until critical filler loading (20%) is exceeded; therefore, a steady decrease becomes noticeable. The decrease in tensile strength with increasing percentage filler loading could be due to the matrix intermolecular bonding disruptions by palm kernel shell particles resulting in intermolecular bond weaker of the matrix (Tudu, 2009) and other several factors, including filler size and processing methods (Obasi, 2015).

At the same time, a continuous increase is observed in the hardness properties with an increase in filler loading from 10% to 50%. The consistent increase in hardness properties of the composite with increased percentage filler loading could be related to the fact that waste paper in the polymer matrix may stiffen the polymer core, making it more resistant to indentation (Malcenji *et al.*, 2010). In addition, it was observed that as filler content increases, the impact effect decreases, indicating that composite resist resists action at higher filler loading.

The composite's weight loss (abrasion resistance) was observed in the sample filled with 40% CPKs with a low percentage. This could be due to proper filler-matrix distribution and interfacial solid adhesion that could have formed in the composite since filler–matrix composition affects the distribution and interfacial adhesion of the filler and the matrix, as evidenced by Okele

et al. (2018). The lower water absorption indicates better water absorption properties of the material.

The results show a progressive increase in percentage water absorption as the percentage filler loading increased from 10 to 20%, followed by a sudden increase with continued filler content increase. The progressive increase in water absorption could be related to the fact that the palm kernel shell is an agro-filler, which is expected to have the possibility of moisture absorption property (hydrophilic) (James, 2006). However, the unique trend shown by the sample filled with 40% filler loading could be due to better filler-matrix distribution and interfacial adhesion at this composition.

Model Development

Empirical models that describe interaction amongst constituents of the mixture and their significance level were investigated on the physico-mechanical properties of thermoplastic composites. However, obtained models, which can be used to predict composite properties for a given level of each factor, were developed in equation (6–10). In addition, these equations are utilised to compare the relative impact of the factors by comparing their coefficients. Coefficients represent the level of sensitivity, whereas the sign (positive and negative) stands for the proportionality of the variable to the generated response (Van Jaarsveld *et al.*, 2003; Varanda *et al.*, 2017).

Optimisation Parameters

The optimisation of individual responses was performed by numerical plots to achieve the right combinations of process parameters to produce plastic interlocks. Optimised properties were obtained by maximisation of tensile strength, hardness and impact strength with corresponding

minimisation of wear resistance and water absorption. Therefore, the plots show that a change in the ratio of RLDPE/CPKs causes a change in physico-mechanical properties of plastic inter-locks composite at a p-value less than 5%. The optimised parameters for individual process parameters, as predicted by the software, were 60.8% (RLDPE) and 39.2% (CPKs) at 35.64 MPa (tensile), 93.14 shore D (hardness), 0.94 J/mm (impact), 0.59% (abrasion) and 3.22% (water absorption).

Fabrication Procedures for Thermoplastic Interlocks

Figure 3: Produced plastic interlocks

Development of thermoplastic interlocks (Figure 3) involved carbonisation of palm kernel shell and sieving into powder, then mixing with recycled low-density polyethene (wastewater sachet) on the 2-roll mill (Figure 4). Aftermath, compression of the optimised mass ratio of CPKs/LDPE into metal mould (100mm×50mm×30mm) having desired interlock shape impression was hydraulically pressed at a temperature of 130^{o}C and pressure of 3 MPa for 3 minutes before it was cooled for 2 minutes under hydraulic pressure of 2.5 MPa.

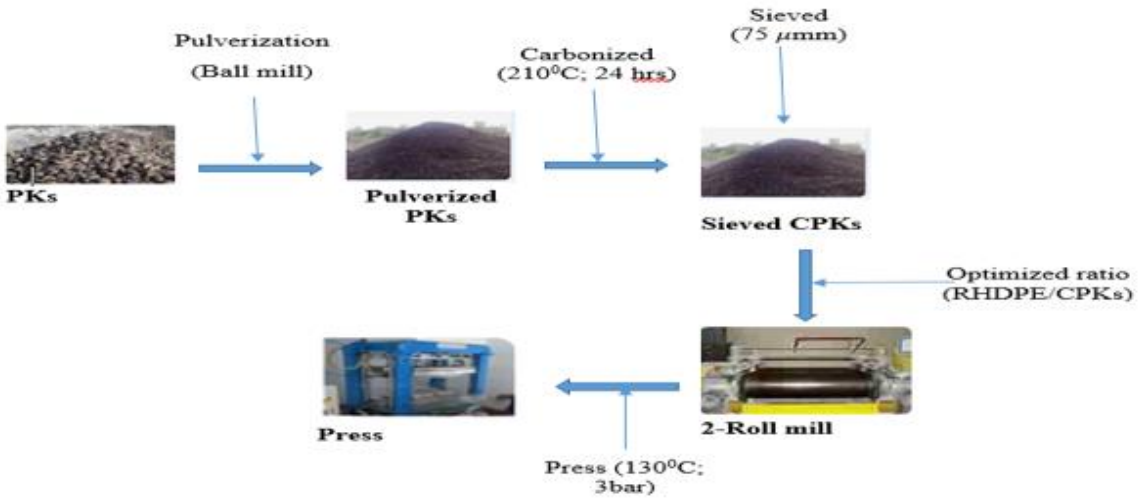

Figure 4: Thermoplastics Inter-lock Processes

CONCLUSION

The carbonised palm kernel shells (CPKs) activity in the polymer composite to produce plastic interlocks was successfully synthesised. Physico-mechanical properties of the composites were performed to evaluate the change of different properties due to the addition of a carbonised palm kernel shell in the composite. The physico-mechanical analyses revealed increased CPK content with optimum at 39.3% carbonised palm kernel shell powder and 60.7% recycled polymer (rLDPE).

REFERENCES

Ahmed, H. A., Ramadan, E. G., Ayman, A. W., & Mohamed, H. A. (2019). Mechanical and physical properties of polypropylene (PP) and high-density (HDPE). *Engineering Science*, *4*, 34-42.

Al-Salihi, S., Jasim, A. M., Fidalgo, M. M., & Xing, Y. (2022). Removal of Congo red dyes from aqueous solutions by porous γ-alumina nanoshells. *Chemosphere*, *286*, 131769.

American Society for Testing and Materials (ASTM) (1983). Standard specifications for composite properties determination. https://vteclabs.com/testing/physicaltestnig/astm-d256.

Apasi, A. (2015). Development of metal matrix/coconut shell composites for automobile application. PhD Thesis in Department of Mechanical Engineering, Ahmadu Bello University, Zaria, p31

Babatunde, O. O., & Temitope, G. O. (2016). Utilisation of banana stalk fiber as reinforcement in low-density polyethylene composites. *Revista Matéria, 21*(4), 953-963.

Bledzki, A. K., & Gassan, J. (1999). Composites reinforced with cellulose based fillers. *Progress in Polymer Science, 24*, 221-274.

Emekwisia, C. C., Aigbodion, V. S., & Ezema, I. C. (2019). Mercerization effect on the mechanical and physical properties of banana fiber polyester composite. *International Journal of Engineering Applied Sciences and Technology*, *4*, 124-132.

Essam, E., Walaa, M. W., Eslam, S., Salah, F. A., & Abbas, E. A. (2001). Study on the physical, mechanical, thermal properties and soil biodegradation of HDPE blended with PBS/HDPE-G-MA. *Springer Polymer Bulletin 79*(4). https://DOI:10.1007/s00289-021-03623-y.

Hassan, B. S., Aigbodion, V. S., & Patrick, S. N. (2012). Development of polyester/eggshell particulate composites. *Tribology in Industry, 34*(4), 217-225.

Malkapuram, R., Umar, V., & Yuvraj, S. N. (2008). Recent development in natural fiber reinforced polypropylene composites. *Journal of Reinforced Plastics and Composites, 28*, 1168-1189.

Mohanty, S., Verma, S. K., & Nayak, S. K. (2000). Dynamic mechanical properties and thermal properties of mape treated jute/HDPE composites. *Composites Science and Technology, 66*(34), 583-547.

Muhammad, R. M., & Ahmed, N. H. (2020). Effect of egg shell powder on some mechanical and physical properties of natural rubber (NR). *Iraq Journal of Mechanical and Material Engineering*, *12*(3), 160-167.

Neher, B., Hossain, R., Fatima, K., Gafur, M. A., Hossain, M. A., & Ahmed, F. (2022). Study of the physical, mechanical and thermal properties of banana fiber reinforced HDPE composites: *Materials Sciences and Applications, 11*, 245-262.

Obasi, H. C. (2015). Peanut husk-filled polyethylene composites: Effects of filler content and compatibilizer on properties. *Journal of Polymers.* http://dx.doi.org/10.1155/2015/18928.

Okele, A. I., Gimba, C. E., Mamza P. A., & Abba, H. (2018). Hybridisation of carbon black: cashew nut shell powder as fillers on the mechanical properties of natural rubber composites. *Composite Materials, 2*, 49-54. https://doi:10.11648/j.cm.20180202.12.

Tudu, P. (2009). Processing and characterisation of natural fiber reinforced polymer composites. B.Tech. Thesis Submitted to Mechanical Engineering Department, National Institute of Technology, Rourkela, Odisha-India.

Van Jaarsveld, J. G. S., Jannie, S. J., & Lukey, G. C. (2003). The characterization of source materials in fly ash-based geopolymers. *Materials Letter, 57*(7), 1272-1281.

Varanda, C., Portugal, I., Silver, A. M. S., & Silver, I. C. (2017). Optimisation of bitumen formation using mixture design of experiment (MDOE). *Construction and Building Materials, 156*, 611-620. https://doi.org/10.1016/j.conbuildmat.2017.08.146.

Yagyu, K. S. (2014). Study of effective thermal conductivity of fiber reinforced epoxy composites. A thesis submitted to Department of Mechanical Engineering, National Institute of Technology, Rourkela, Odisha (India), 60.