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Influence of Compression Molding Parameters and Particle Weight on Flexural Property of Bamboo Leaves Reinforced Low Density Polyethylene Composite

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Abstract

An entirely new eco-friendly thermo-plastic composite was produced from Recycled Low Density Polyethylene with chemically-treated Bamboo leaves particles as reinforcements, using compression molding. This work sought to study the effect of the compression molding parameters (such as press pressure, holding time and press temperature) and % particle weight on the flexural strength of the composite, and to suggest optimum conditions for obtaining optimum flexural strength for the composites using the central composite experimental design. Each of these parameters were taken in 3levels, and an experiment of 30 runs was conducted. Polynomial model with quadratic effect was selected, and model equation was generated for flexural strength using analysis of variance (ANOVA). The linear and quadratic effects of the chosen parameters were studied using contour diagrams. SEM analysis was also carried out on the optimized sample. Optimization showed a flexural strength of 5.51 MPa at press pressure of 6.00 MPa, press time of 5.50 mins and press temperature of 160.00^{o}C.

Keywords: Central Composite Design (CCD), Flexural strength, Compression molding, Polymer matrix composites (PMCs), Microstructure

1. Introduction

Polymer Matrix Composites (PMCs) have played a major role in revolutionizing the materials sector all over the larger society. This is largely due to the fact that much largely needed property combinations, which are improbable by other forms of material combinations, can be produced using PMCs. However, the production of PMCs using inorganic fibres like Glass fibres faces the problems of high cost of production, depletion of natural and non-renewable resources, and the high cost of accessing raw materials. This, therefore, presents a need for an affordable and effective alternative to inorganic fibres. Also, some inorganic fibres, like asbestos, are carcinogenic and injurious to health. Thus, there is a need to develop environmentally friendly and healthy fibres for use in PMCs. This has led the search for effective natural fibres and particles, which can serve as proper reinforcement for PMCs (Hardinnawirda and SitiRabiatull 2012; Atuanya et al 2013; Yawas et al 2013; Ojha et al 2012).

Apart from the natural fibers and particles added as reinforcements to these PMCs, the production route through which the PMCs passed has been shown to have some effect on the properties of these natural particles reinforced PMCs. Matoke et al, 2012, studied the effect of production methods and material ratios on physical properties of composites. For all compositions, therefore, the study showed that compression molding impacted better properties on any composite material than open casting, due to the pressure exerted on the composite during compression molding process. Thus compression molding has shown to be one effective way of producing PMCs, and has been utilized by many researchers (Kumar and Balachandar 2014; Aigbodion et al 2010; Atuanya et al 2013).

Natural fibers/particles (especially non-woody fibres), though good in environmental sustainability had always shown a draw-back from its susceptibility to water, and thus making PMCs manufactured with them prone to water absorption and thickness swelling, which can have negative effect on the properties of the composites thus produced. Srinivasababu et al 2014, studied the effect of chemical treatment of fiber on the mechanical and dielectric properties of Palmyra palm petiole (PPP) fiber reinforced Polyester composites. Two different set of samples containing chemical treated PPP (PPP-CT), and untreated PPP were produced by hand laying into polyester matrix, with varying volume fractions. Analysis carried out showed PPP-CT yielded better results in tensile, flexural and impact strengths than the untreated PPP. This was said to be because of the better adherence between the fibers and matrix after chemical treatment. Thus, chemical treatments carried out on natural fibers/particles before utilizing them as reinforcements helps to increase the mechanical properties. This is because chemical treatment removes the hydrophilicity of the fibres/particles thereby reducing their water absorption rate, and it also causes enhancement in mechanical strength as well as the dimensional stability of natural fibre/particle reinforced polymer composites.

Other major considerations which studies had shown to greatly affect mechanical properties of PMCs reinforced with natural fibers/particles are %fibre/particle weight, and process parameters (such as press pressure, time and temperature- for compression molding process route). Researches carried out in this direction are as follows:

Ojha et al 2012, studied the mechanical properties of orange peel reinforced polymer composites using epoxy resin as matrix, and hardener. The orange peels were crushed and sieved, and weight fractions were varied in the proportion; 5, 10, 20 and 30 wt% orange peel. Results showed that the density of the composite increased slightly with increase in particle content. Also, tensile strength, flexural strength and hardness also increased with increasing particle content, though these reached their maximum at 20wt%, and any further increase in particle content showed a slight decrease in these properties due to voids in the composite.

Hussain et al 2012, studied the effect of fiber composition on mechanical properties of short bamboo fiber reinforced polyester composites filled with alumina particulate. Two sets of samples with Polyester and bamboo fiber (15, 30 and 45 wt. %), and then polyester and bamboo fiber (45wt %) with Alumina particulate (5, 10 and 15 wt. %) were prepared. For the first sample, results showed that tensile strength increased with increase in wt% of bamboo fibers, while flexural strength was highest at 30wt% bamboo fibers. For the second set, tensile strength was highest at 15wt% Alumina content, while flexural strength was highest at 10wt% Alumina content. Comparatively, however, the mechanical properties of the bamboo-polyester composites filled with alumina particulate were found to be better than that of the unfilled bamboo-polyester composite.

Aireddy and Mishra 2011, studied the effect of particle composition on the mechanical properties of coir dust filled epoxy resin matrix composites. The particle composition was varied in the proportion: 10, 20, 40 and 60wt% coir dust. Results showed that erosion wear rate, specific wear rate, flexural strength, hardness and density decreased with increase in wt% of coir dust particles. This was attributed to the fact that the coirdust acted as only a filler, and not a reinforcement.

Kumar and Kumar, 2012, studied the effect of % weight fraction of coconut shell particle and coir fibers on the mechanical properties of both particles and particle/fiber reinforced epoxy composites. Two sets of samples were prepared with only coconut shell particle reinforced epoxy composites, and then with mixture of coconut shell particle/coir fiber reinforced epoxy composites. Analysis showed that density, tensile strength, modulus of elasticity, and fracture toughness decreased with increasing %weight fraction for the shell particle reinforced composite, while water absorption and hardness increased accordingly, with compressive strength and flexural strength increasing to a maximum and the decreasing with increase in %weight fraction for the shell particle reinforced composite.

Kumar and Balachandar 2014, studied the influence of hot press forming process parameters (press time, temperature and pressure) on flexural property of Glass/Polypropylene based thermoplastic composites using Box-Behnken experimental design. The research proved the fact that flexural strength is highly influenced by these process parameters; quadratic effects of some of the parameters and interaction effects of mold temperature and holding time were established. Analysis carried out showed that optimum flexural property was obtained at 11.1 bar mold pressure, 3.09 min holding time, and 220°C mold temperature. Their research also proved the importance of experimental design in studying the effect of process parameters on any mechanical property of PMCs, as other researchers also confirmed (Mehdinia et al 2013; Govindaraju et al 2014).

Agunsonye et al 2012, studied the effect of palm kernel shell (PKS) on the microstructure and mechanical properties of Recycled Low-Density Polyethylene (RLDPE) reinforced with PKS particulate composite as a new material for engineering applications. Analysis showed that composites produced with 150μm particle size had the best properties of the entire grade and that the grade can be used for interior applications and decorative purposes where high strength is not a critical requirement.

There was no generally agreed influence of increasing particle weights in physical and mechanical properties of natural particles reinforced composites. While some reported an increase in tensile, flexural, impact strengths and hardness, others reported a decrease or a variation at the most. The reason for these could be due to the fact that natural fibres have varying unpredictable behaviours which could affect their influence when used to reinforce a composite. Certain factors like the character and constituents of the fibres/particles, the particle sizes, production process variations, etc. make such behaviours 99% possible. Thus, each new fibre must be studied on its own merit.

Also, the analysis carried out on Recycled Low- Density Polyethylene (RLDPE) polymer composites showed that proper mixing and compounding during the production process resulted in a strong particle-matrix interface bond which is important for high mechanical properties. (Atuanya et al 2013). Processing parameters during molding significantly influence the properties and interfacial characteristics of the composites. Therefore, suitable processing parameters must be carefully selected in order to yield the optimum composite products. Most important hot press forming parameters that influence the mechanical properties are temperature, pressure, and heating time.

Despite many studies that have been done on biological wastes as fibres for PMCs, much attention has not yet been paid to Bamboo leaves. Owing to the much abundance of bamboo plants all over the world, especially Nigeria, bamboo stems have found a lot of uses in tropical regions ranging from building to paper and pulp production, and then to all forms of Composites. However, only little regards have been paid to its leaves as a reinforcement/filler for composite materials, except majorly as Bamboo leaf ash (BLA) in some metallic and ceramic composites in certain research works (Alaneme and Adewuyi 2013; Alaneme et al 2013; Asha et al 2014). This paper, therefore, presents for the first time the potential of using chemically-treated bamboo-leaf particles in reinforcing polymer composites, and takes into consideration the influence of production process parameters (press pressure, time and temperature) and particle weight on the flexural strength of the composite using the central composite design (CCD).

2.0 Material and methods

2.1 Raw materials

The raw materials used in this work were: Recycled Low Density Polyethylene (RLDPE) which was gotten from commercial sources at Onitsha, Anambra state, and bamboo leaves which were harvested from bushes within Enugu-Ngwo environs, Methanol and Sodium Hydroxide (NaOH),

2.2 Equipment/Apparatus

The following equipment and apparatuses were used in the research work:Electronic weighing balance, measuring cylinders, Rubber baths and stirrers, pulverizing machine, Electronic sieving machine, Metallic molds, Hydraulic-powered Compression molding machine, Hounsfield Monsanto Tensometer, Scanning electron microscope.

2.3 Experimental Details

Bamboo leaves were harvested from bushes within Enugu-Ngwo environs, and were washed and dried in the sun for three days. The RLDPE was also obtained from commercial sources at Onitsha, Nigeria. The dried leaves were chemically treated with NaOH and Methanol in order to reduce its hydrophilicity and improve fiber strength and adhesive properties.

The dried chemically treated leaves were crushed in a mill at Awka into particles, and sieved to obtain particle sizes of $\leq$ 300μm(as coarser sizes will reduce the effectiveness of the matrix-filler mix, and increase chances of porosity). Those which were above this size were returned to the mill for further crushing, or were thrown away as waste. Also, the RLDPE was sieved to obtain a uniform size of about < 2mm, for the purpose of obtaining a uniform mix of RLDPE and the pulverized bamboo particles.

2.4 Design of Experiments

The design of experiments is a powerful tool for modelling and analyzing the influence of control factors on performance output. Design Expert v. 11 software has been utilized to perform this task. The most important part of the design of experiments lies in the suitable selection of the control factors. (Kumar and Balachandar 2014). Central Composite Design (CCD) was used in this research. Factors chosen and their levels are presented in Table 1.

The significant variables like pressure, time, temperature and % particle weight were chosen as the critical variables and designated as A, B, C and D respectively. The low, middle, and high levels of each variable were designated as ─, 0, and + respectively.

Table 1:Table of variables for Polymer composite

PARAMETERS	LEVELS		
	-0	+	
(A)PRESSURE (MPa)	6	7	8
(B)TIME (mins)	5	7	9
(C)TEMPERATURE (^{o}C)	160	180	200
(D) PARTICLE WT. (%)	10	20	30

Placing these parameters into the Central Composite RSM yielded 30 experimental runs with six replications to minimize standard error, as shown in Table 2.

Table 2:Four-factor Central Composite Design Experiment with predicted response of dependent variable (flexural strength in MPa).

	Factor				Response
Std	**A:PRESSURE**	**B: TIME**	**C: TEMPERATURE**	**D:% PARTICLE WT.**	**FLEXURAL STRENGTH**
	MPa	**Mins**	**^{0}C**	**%**	**MPa**
1	6	5	160	10	5.14
2	8	5	160	10	6.47
3	6	9	160	10	5.78
4	8	9	160	10	4.98
5	6	5	200	10	2.74
6	8	5	200	10	5.03
7	6	9	200	10	3.69
8	8	9	200	10	4.09
9	6	5	160	30	4.03
10	8	5	160	30	2.99
11	6	9	160	30	5.30
12	8	9	160	30	2.26
13	6	5	200	30	2.40
14	8	5	200	30	2.46
15	6	9	200	30	4.43
16	8	9	200	30	2.41
17	5	7	180	20	6.26
18	9	7	180	20	5.72
19	7	3	180	20	1.79
20	7	11	180	20	2.17
21	7	7	140	20	4.82

22	7	7	220	20	2.31
23	7	7	180	0	6.26
24	7	7	180	40	3.37
25	7	7	180	20	3.91
26	7	7	180	20	3.80
27	7	7	180	20	3.70
28	7	7	180	20	4.01
29	7	7	180	20	4.00
30	7	7	180	20	3.91

2.5 Sample Preparation

A metallic mould of size 300 × 250 × 5 (mm) from which ASTM sizes for flexural tests were cut out.The calculations involved in the samples production were as follows:

Target density = 0.72g/cm^3
Volume of mould = 30 × 25 × 0.5 = 375cm^3
Therefore, total weight = density × vol = 0.72 × 375 = 270g (chemically treated bamboo leaves + Recycled low density polyethylene)

The mixture was formed by weighing out the needed amount of bamboo leaves particulate and the RLDPE, then mixing them manually in a bowl and pouring into the mould. Then the mould was placed in an electrically heated hydraulic press and the pressing was done in line with the outlined experimental design in Table 2. The composite boards thus formed, were removed and allowed to cool to room temperature. Prior to test, the composite samples were conditioned at constant room temperature and 68% relative humidity (RH) in accordance with ASTM D 618.

2.6 Flexural Test

The flexural test was also carried out at the University of Nigeria Nsukka Materials Testing Laboratory, using a Hounsfield Monsanto Tensometer, with the grips of a flexural setup/accessory (3-point flexural tester). The tests were carried out according to ASTM D 790.

The flexural strength of the composite material was then determined using the following equation:

$$\sigma = \frac{3FL^2}{2bt^2} \qquad (1)$$

where F is the maximum load (in newton), L is the distance between the supports (in mm), b is the width of the specimen (in mm), and t is the thickness (in mm).

2.7 Scanning Electron Microscopy (SEM)

The Scanning Electron Microscope (SEM) JEOL JSM-6480LV was used to examine the surface morphology of the base polymers and the optimized composite sample.

3.0 Results and Discussions

Table 2 showed the results for the experiments carried out on the samples using the Design of Experiments, and their response. This optimization process involves three major steps, performing the statistically designed experiments, estimating the coefficients in a mathematical model, and predicting the response and checking the adequacy of the model.

The ANOVA results are shown in Table 3. The Model F-value of 400.01 implies the model is significant. There is only a 0.01% chance that an F-value this large could occur due to noise. P-values less than 0.0500 indicate model terms are significant. In this case A, B, C, D, AB, AC, AD, BC, BD, CD, A^2, B^2, C^2, D^2 are significant model terms.

Values greater than 0.1000 indicate the model terms are not significant. The Lack of Fit F-value of 0.49 implies the Lack of Fit is not significant relative to the pure error. There is an 84.06% chance that a "Lack of Fit F-value" this large could occur due to noise. Non-significant lack of fit is good, because we want the model to fit.

Table 3: ANOVA for quadratic model of Flexural strength

Source	Sum of Squares	Df	Mean Square	F-value	p-value
Model	52.99	14	3.78	400.01	< 0.0001
A-pressure	0.6347	1	0.6347	67.08	< 0.0001
B- time	0.2459	1	0.2459	25.98	0.0001
C- temp.	9.00	1	9.00	950.90	< 0.0001
D-%particle wt.	12.65	1	12.65	1336.65	< 0.0001
AB	4.09	1	4.09	431.99	< 0.0001
AC	1.14	1	1.14	120.80	< 0.0001
AD	5.37	1	5.37	567.71	< 0.0001
BC	0.3299	1	0.3299	34.87	< 0.0001
BD	0.7036	1	0.7036	74.36	< 0.0001
CD	0.9722	1	0.9722	102.75	< 0.0001
A^2	7.22	1	7.22	763.24	< 0.0001
B^2	6.58	1	6.58	695.62	< 0.0001
C^2	0.2404	1	0.2404	25.41	0.0001
D^2	1.32	1	1.32	139.66	< 0.0001
Residual	0.1419	15	0.0095		
Lack of Fit	0.0704	10	0.0070	0.4926	0.8406
Pure Error	0.0715	5	0.0143		
Cor Total	53.13	29			

Table 4 shows the Fit statistics for Flexural strength of the composite. The Predicted R^2 of 0.9904 is in reasonable agreement with the Adjusted R^2 of 0.9948; i.e. the difference is less than 0.2. Adeq Precision measures the signal to noise ratio. A ratio greater than 4 is desirable. The ratio of 69.826 indicates an adequate signal. This model can be used to navigate the design space.

Table 4: Fit Statistics for Flexural strength

Std. Dev.	0.0973	**R^2**	0.9973
Mean	4.01	**Adjusted R^2**	0.9948
Coefficient of Variation%	2.43	**Predicted R^2**	0.9904
		Adeq Precision	69.8256

3.1 Model equation for Flexural strength

The Model equation (in terms of coded factors) for Flexural strength as suggested by Design Expert 11 is:

$$\text{FLEXURAL STRENGTH} = +3.888 - 0.163\,A + 0.101\,B - 0.612\,C - 0.726\,D - 0.505\,AB + 0.267\,AC - 0.579\,AD + 0.144\,BC + 0.210\,BD + 0.247\,CD + 0.513\,A^2 - 0.490\,B^2 - 0.094\,C^2 + 0.219\,D^2 \quad (2)$$

Here, A-D represent the independent factors, and the equation shows the quantitative effect of the factors on the response (Flexural Strength), with the response having both linear and quadratic effects on four variables studied. Coefficients with one factor represent the single effect of that particular factor while the coefficients with more than one factor represent the interaction between those factors. Positive sign in front of the terms indicates synergistic effect while negative sign indicates antagonistic effect of the factors.

Equation 2 can be used to make predictions about the response for given levels of each factor, and is also useful for identifying the relative impact of the factors by comparing the factor coefficients.

3.2 Contour Analysis and Optimization

Figures 1 to 6 represent the response surface and contour plots for the optimization of flexural strength. Figure 1 shows the response surface and contour plot for flexural strength as a function of press pressure and press time. It was observed that optimum flexural strength of 4.6 MPa was obtained at low press pressure (about 6.1 MPa) and high press time (8.7 mins). Figure 2 shows the response surface and contour plot for flexural strength as a function of press pressure and press temperature.It was observed that at decreasing press pressure and a corresponding decrease in press temperature, there was an increase in flexural strength, with optimum flexural strength of 5.2 MPa obtained at pressure of 6.1 MPa and temperature of 161°C.

Figure 3 shows the response surface and contour plot for flexural strength as a function of press pressure and % particle weight. Increase in flexural strength was favoured by a decrease in particle weight, and a corresponding increase in press pressure. Optimum flexural strength of 5.6 MPa was obtained at particle weight of 10.3% and pressure of 7.9MPa. Figure 4 shows the response surface and contour plot for flexural strength as a function of press time and press temperature. Optimum flexural property of 4.3 MPa was obtained at a time of 7 mins and temperature of 164°C. Figure 5 shows the response surface and contour plot for flexural strength as a function of press time and press % particle weight. A press time of 7mins and % particle weight of about 10.1% gave an optimum flexural strength of 4.75MPa.

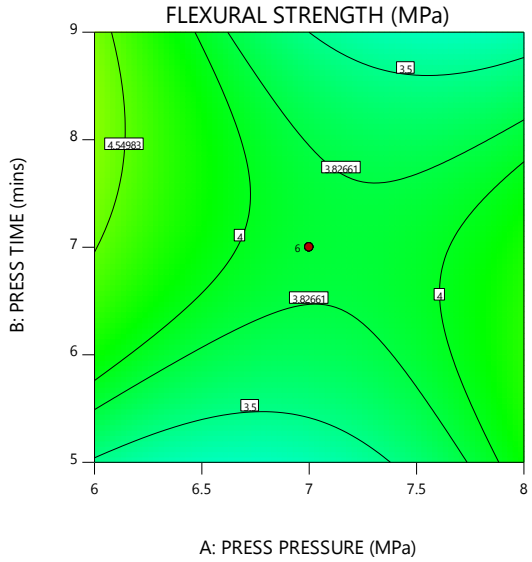

Figure 1:Contour between press pressure and press time keeping press temperature and % particle weight constant.

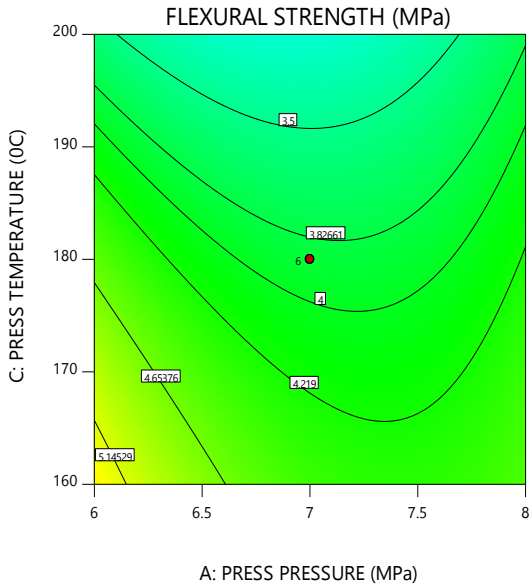

Fig. 2: Contour between press pressure and press temperature keeping press time and % particle weight constant.

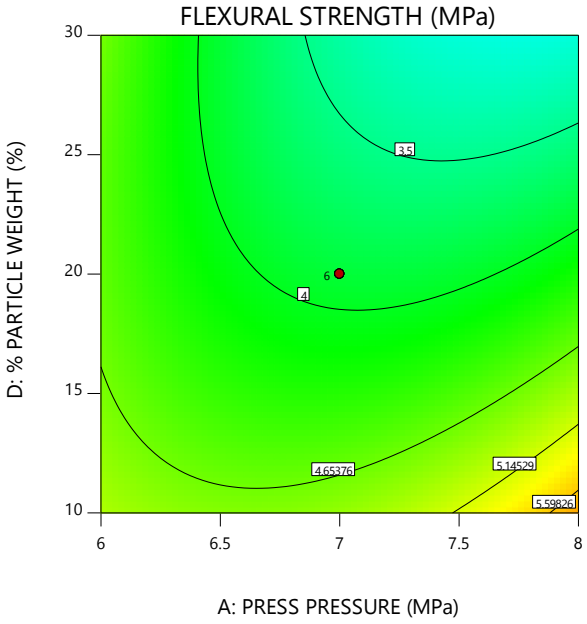

Fig. 3:Contour between press pressure and % particle weight keeping press time and press temperature constant.

Fig. 6 shows the response surface and contour plot for flexural strength as a function of press temperature and % particle weight. It was observed that high flexural strength was favoured by low press temperature and % particle weight, with optimum flexural strength of 5.5 MPa obtained at press temperature of 161°C and % particle weight of 10.5%. Generally, it was observed that low particle weight, low temperatures, medium time and medium pressures favoured an increase in flexural strength.

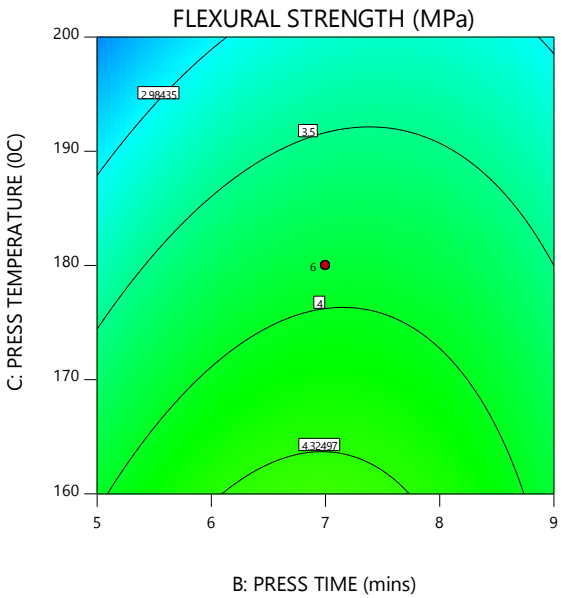

Fig. 4: Contour between press time and press temperature keeping press pressure and % particle weight constant.

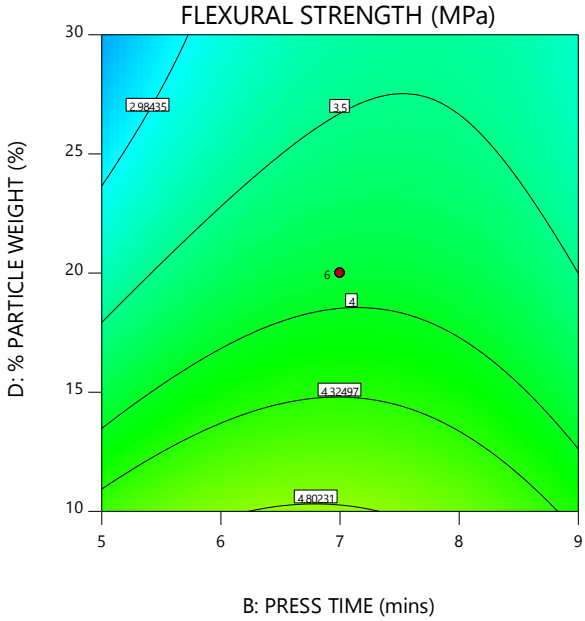

Fig. 5:Contour between press time and % particle weight keeping press pressure and press temperature constant.

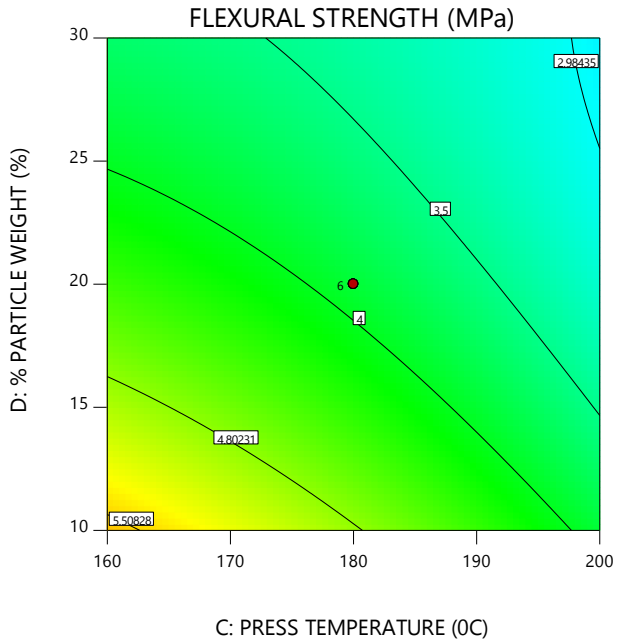

Fig. 6: Contour between press temperature and % particle weight keeping press pressure and press time constant.

The model (Eqn 2) was thus analyzed in order to select the optimum process conditions for maximizing flexural strength. The maximum flexural strength was predicted at 5.51 MPa, and the final optimized parameters were press pressure of 6.0 MPa, press time of 5.5 mins, press temperature of 160°C and % particle weight of 10.0%.

The optimized values were used to produce an optimum sample, and flexural test was then carried out on the sample at the University of Nigeria Nsukka Materials Testing Laboratory, using a Hounsfield Monsanto Tensometer, with the grips of a flexural setup/accessory (3-point flexural tester). The tests were carried out according to ASTM D 790. The result obtained, as shown in Table 5, yielded a good agreement. Thus the stated mathematical model is important for fabrication of Recycled Low-Density Polyethylene/chemically treated Bamboo leaf bio-composites.

Table 5:Table of Optimized/Actual Value

PERFORMANCE MEASURE	PREDICTED VALUES	ACTUAL VALUES	% ERROR
Flexural Strength	5.51 MPa	5.45	1.09%

3.3 Microstructural Analysis

Figure 7 showed the SEM/EDS of the Recycled Low Density Polyethylene. The EDS showed high amounts of oxygen, carbon and silicon, with trace elements of aluminium, iron, potassium, calcium, sulphur and phosphorus. Figure 8 showed the SEM/EDS of the Optimized Composites. The EDS showed high amounts of carbon and oxygen. The presence of Na (sodium) and chlorine (Cl- in trace amounts) in the optimized sample showed the effect of the mix with chemically-treated bamboo particles.

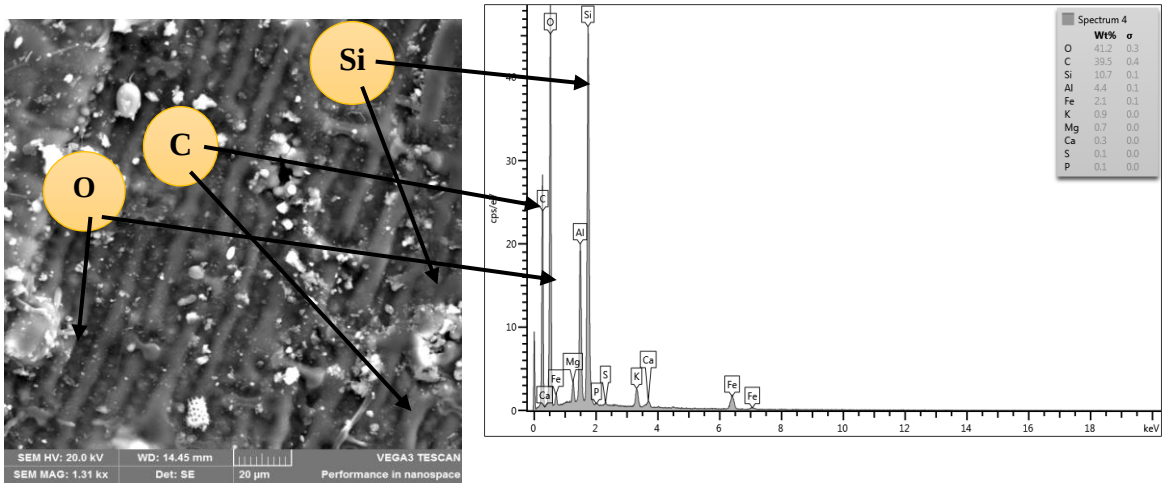

Figure 7:SEM/EDS of RLDPE

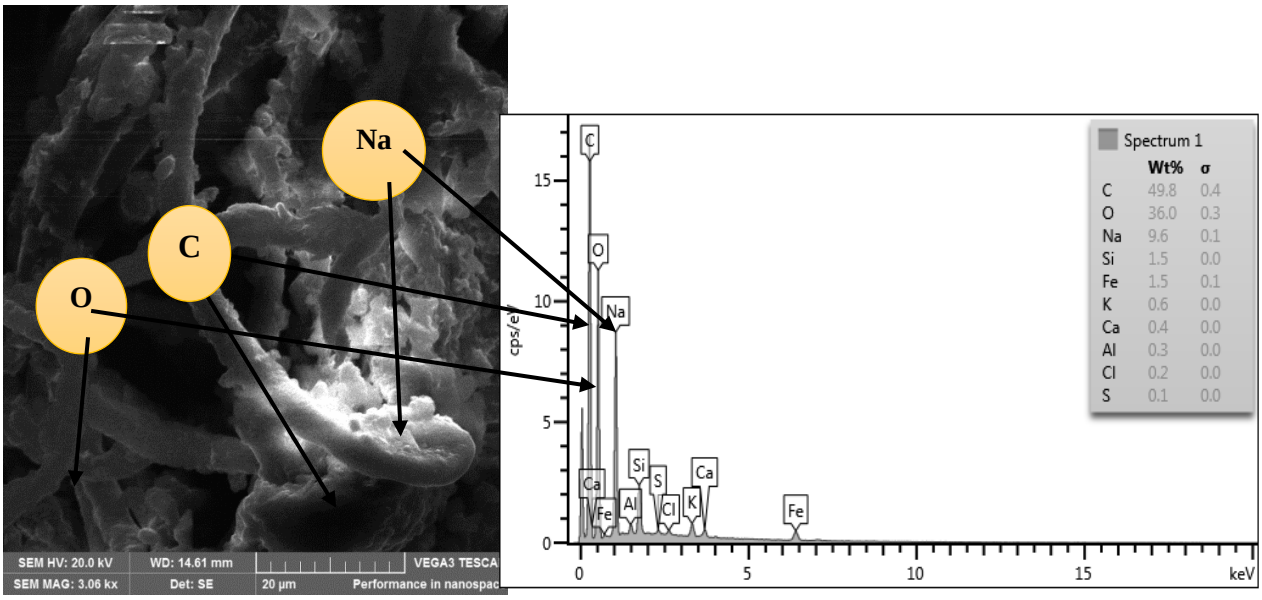

Figure 8:SEM/EDS of the optimized sample

4.0. Conclusion

From the results obtained from this research, the following conclusions are made: The research work has developed as it were an entirely new possibility for one of the most common, but rarely considered waste material- *bamboo leaf*, thus reducing the effect of waste material burning prevalent in Africa, and yielding an environmentally friendly product. The research work, also, has provided information on predictive models for Flexural strength of Bamboo-Leaf reinforced Polymer composite, and also suggested optimum process parameters for the best yield. The result from the optimization met the standards for interior roofing of automobiles (headliner).

5.0 Recommendation

1. Other polymer materials could also be used as a base polymer to discover new possibilities for further use of the BambooLeaf.
2. This grade can be used for interior applications such as car seat, dashboard, and car interior for decorative purposes or other interior parts of an automobile where high strength is not considered a critical requirement
3. The attention of Plastic and Composite industries and other research institutions are drawn to this new possibility for bamboo leaf.

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