Effect of filler carbonization on agro-waste based ceiling board

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Abstract
This work presents the use as a filler of carbonized breadfruit seed coat and recycled low density polyethylene as the binder in ceiling board manufacturing. The depulped bread fruit seed was carbonized for 2 h at a temperature of 500°C. The experimental design was set up using the Design Expert software. A total of 30 experimental tests were developed for four parameters and three responses. The parameters are carbonized bread fruit seed coat/recycled Low Density Polyethylene mass ratio (filler-binder mass ratio), compaction time, compaction temperature and compaction pressure while the responses are thermal conductivity, thickness swell and water absorption. The models developed have been validated using the Study of Variance (ANOVA). Using the 3D surface map, the influence of the parameters on the responses was studied. The optimization method of the Design Expert program was used to evaluate the optimal level of the parameters that will produce the best possible result from their combination. The result gave optimal values of 16.206% filler/LDPE, 9.406 minutes compaction time, 200°C compaction temperature and 11 MPa compaction pressure, which gave 0.246% Water Absorption, 1.998% Thickness Swell and 2.898 W/M.K Thermal Conductivity.

Keywords
Thermal conductivity, thickness swell, water absorption, carbonization, pressure, temperature

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Introduction
Nigeria has a lot of agricultural produce, producing more than ten million tons of agricultural waste annually. These waste items include: seed coat of breadfruit, rice husk, straw, bagasse, coconut fiber, maize husk, etc. Most of these wastes are often poorly discarded, causing pollution through two main ways, namely indiscriminate dumping (land, water and air pollution) and burning (greenhouse effect). However, most of these agro-waste products can be recycled into new, more environmentally sustainable products that can add value to Nigeria’s economic growth in the same way.

Filler materials reinforce the matrix by enhancing the elastic modulus and the strength of the resulting composite. Its incompatibility is the main drawback of integrating agricultural fillers into a synthetic polymer. Natural fibers are naturally hydrophilic, whereas synthetic polymers are hydrophobic. The hydrophilic properties of natural fibers lead to a high absorption of moisture, causing the fiber to change its dimensions, resulting in the swelling of the composite created and eventually rotting through fungal attack. The key causes of this biodegradation are the presence of hemicelluloses in natural fibers, while lignin is thermally stable but vulnerable to ultraviolet degradation. The goal of carbonization is to curb the shortcomings of natural fibers for use in rubber vulcanization.

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Low density polyethylene is a durable and versatile polymer marked by long branches that do not pack well into crystallites. The molecules are able to pack more tightly as the chains become more linear, such as high density polyethylene.1

In order to add unique properties to the finished product, natural cellulose fibers have also been used as the reinforcement component in polymers. A variety of polymer composite materials based on cellulose fibers have been produced using different synthetic techniques.2 These include rice husk-saw dust,3–5 jatropha curcas seedcake,6 watermelon peels,7 bamboo,8 breadfruit seed coat,9,10 palm kernel fibre,11–13 cork-based composite,14,15 cane wood,15,16,17 etc. Aguele and Madufor,13 studied the effect of carbonized coir on the physical properties of natural rubber composite. Compared with carbon black reinforced composite, the result obtained was favorable. With increasing filler loadings, they sustained a growing pattern. This work is therefore aimed at studying the effect of the carbonized breadfruit seed coat on the ceiling board’s physical properties.

Materials and method

Recycled low-density polyethylene (rLDPE) and Breadfruit seed coat (carbonized) are the materials used, while the machinery is; carbonization furnace, hot pressing unit, weighing balance, double grinding mill Asiko II, screw gauge micrometer, sieve, mold.

Preparation of materials

Upon processing, the breadfruit coat was dried and carbonized at the Scientific Equipment Development Institute (SEDI) in Enugu State, Nigeria, at a temperature of 500°C for 3 h using a carbonization furnace. The carbonized coats were ground and sieved to a particle size of 150 µm, adopted from the Ezenwa et al.9 report. See Figure 1 for Breadfruit Seed coat.

Development of Samples

Using the experiment developed by the design software expert, thirty test samples were made. The production of the sample involved mixing the filler and rLDPE according to the ratio stipulated by the experimental design for each of the samples. Based on the compaction time, compaction temperature and compaction pressure for each sample, the mixture was then hot-pressed into a rectangular mold using a hot pressing system, see Figure 2 for recycled Low Density Polyethylene.

Experimental Design

Using the Design Expert 10 program, the Response Surface Methodology (RSM) under the Central Composite Design was used for the experimental design. Four parameters of carbonized breadfruit seed coat/recycled low density polyethylene mass ratio (filler-binder mass ratio), compaction temperature, compaction time and compaction pressure were considered against three responses: thermal conductivity, water absorption and thickness swell. A total of 30 experimental runs consisting of 14 core points, 10 star points and 6 center points have been produced. For each factor in the design, the star points reflect new extreme values (low and high). The center points allow curvature to be calculated, while the key experimental points are the core points. The Study of Variance (ANOVA) was used for the validation of the developed model equation.

Determination of the Responses

The answers for the analysis are swelling thickness, absorption of water and thermal conductivity. After partially submerging the sample in water for 24 h, the thickness swell is expressed as the ratio of the increase in thickness to the initial thickness, while the water
The absorption rate is the ratio of the weight increase to the initial weight. The thickness swell and water absorption are computed using equations 1 and 2 respectively with the initial thickness $T_0$, initial weight $W_0$, final thickness $T_1$ and final weight $W_1$. Thermal conductivity is a function of the heat source's temperature, thickness and thermal flux. Using the thermal conductivity tester, the thermal conductivity of the samples was calculated by putting the samples between the tester’s cold and hot surfaces, $T_1$ and $T_0$, respectively. The thermal conductivity $K$ is evaluated using equation (3) with $\rho$ heater thermal flux, $t$ sample thickness, and $A$ sample surface area.

$$T_s = \frac{T_1 - T_0}{T_0} \quad (1)$$

$$WA = \frac{W_1 - W_0}{W_0} \quad (2)$$

$$k = \frac{\rho t}{A(T_o - T_1)} \quad (3)$$

### Optimization of the Production Parameters

The Design Expert software optimization tool was used to optimize the output parameters. All the parameters were set to a minimum in the optimization setup in order to achieve the desired result, which is to minimize the absorption of water, thermal conductivity and thickness swelling. The optimization produced the combination of production parameters that would give the best result to be obtained.

### Results and Discussion

#### Results

For the experimental design, the response surface technique was used, the Analysis of Variance (ANOVA) was used to validate the developed model, while the optimization tool was used to optimize output parameters.

### Experimental parameters and responses

As shown in Table 1, four experimental parameters were studied (filler-binder ratio, compaction time, compaction temperature and compaction pressure). In accordance with the research conducted by Ezenwa et al., the maximum and minimum values of the parameters as shown in Table 1 were set specifically to determine the yield effect of carbonizing the breadfruit seed coat and to draw a contrast between the results obtained by the use of the uncarbonized breadfruit seed coat.

The design expert software as shown in Table 2 produced thirty experimental runs. It consists of 14 core points, 10 points of the star and 6 points of the center. The parameters under analysis (filler-binder ratio, compaction time, compaction temperature and compaction pressure) and the corresponding responses (thermal conductivity, swelling of thickness and absorption of water) for each test run are also shown in Table 2.

### Experimental parameters and thermal conductivity

**ANOVA and Lack of Fit Test for thermal conductivity.** The ANOVA for thermal conductivity shown in Table 3, gave a Model F-value of 17.34 implying that the model is significant and has a 0.01% chance to occur due to noise. Significant model terms are identified with "Prob > F" values less than 0.0500, thus from the table it can be seen that C, D, AB, AC, AD, BD, CD, A$^2$, B$^2$, C$^2$ are significant model terms, hence the final equation is reduced to:

$$Thermal Conductivity = + 30.06276 - 0.32871 C + 0.52260 D + 0.00384 AB - 0.00115 AC + 0.004125 AD + 0.016500 BD - 0.003875 CD + 0.002575 A^2 + 0.0059 B^2 + 0.0099375 C^2 \quad (4)$$

The lack of fit test for thermal conductivity is shown in Table 4, with a “Lack of Fit F-value” of 3.94 implying that it has a 7.17% chance to occur due to noise, thus the model developed is good.

**Normal probability plot for thermal conductivity.** The normal plot of residuals for thermal conductivity shown in Figure 3 gave a closely packed points around the diagonal indicating that the chosen model is adequate to predict the response variable.

**Combined effect of parameters on thermal conductivity.** The 3D surface plots for thermal conductivity.
Table 2. Experimental parameters and responses.

| Runs | Parameters | Compaction time min | Compaction temperature °C | Compaction pressure MPa | Responses |
|------|------------|----------------------|-----------------------------|--------------------------|-----------|
|      | Filler-binder ratio wt% |                       |                             |                          | Thermal conductivity W/MK |
|      | Compaction temperature °C |                       |                             |                          | Thickness swell % |
|      | Compaction pressure MPa |                       |                             |                          | Water absorption % |
| 1    | 10          | 5                    | 180                         | 7                        | 0.68       |
| 2    | 20          | 5                    | 180                         | 7                        | 0.69       |
| 3    | 10          | 10                   | 180                         | 7                        | 0.46       |
| 4    | 20          | 10                   | 180                         | 7                        | 0.66       |
| 5    | 10          | 5                    | 200                         | 7                        | 0.9        |
| 6    | 20          | 5                    | 200                         | 7                        | 0.64       |
| 7    | 10          | 10                   | 200                         | 7                        | 0.64       |
| 8    | 20          | 10                   | 200                         | 7                        | 0.54       |
| 9    | 10          | 5                    | 180                         | 11                       | 0.77       |
| 10   | 20          | 5                    | 180                         | 11                       | 0.78       |
| 11   | 10          | 10                   | 180                         | 11                       | 0.74       |
| 12   | 20          | 10                   | 180                         | 11                       | 1.16       |
| 13   | 10          | 5                    | 200                         | 11                       | 0.5        |
| 14   | 20          | 5                    | 200                         | 11                       | 0.54       |
| 15   | 10          | 10                   | 200                         | 11                       | 0.68       |
| 16   | 20          | 10                   | 200                         | 11                       | 0.72       |
| 17   | 5           | 7.5                  | 190                         | 9                        | 0.68       |
| 18   | 25          | 7.5                  | 190                         | 9                        | 0.74       |
| 19   | 15          | 2.5                  | 190                         | 9                        | 0.62       |
| 20   | 15          | 12.5                 | 190                         | 9                        | 0.58       |
| 21   | 15          | 7.5                  | 170                         | 9                        | 0.96       |
| 22   | 15          | 7.5                  | 210                         | 9                        | 0.74       |
| 23   | 15          | 7.5                  | 190                         | 5                        | 0.49       |
| 24   | 15          | 7.5                  | 190                         | 13                       | 0.49       |
| 25   | 15          | 7.5                  | 190                         | 9                        | 0.44       |
| 26   | 15          | 7.5                  | 190                         | 9                        | 0.46       |
| 27   | 15          | 7.5                  | 190                         | 9                        | 0.48       |
| 28   | 15          | 7.5                  | 190                         | 9                        | 0.52       |
| 29   | 15          | 7.5                  | 190                         | 9                        | 0.44       |
| 30   | 15          | 7.5                  | 190                         | 9                        | 0.5        |

Table 3. Analysis of variance for quadratic model of thermal conductivity.

| Source                | Sum of Squares | df | Mean square | F Value | p-value | Prob > F |
|-----------------------|----------------|----|-------------|---------|---------|----------|
| Model                 | 0.77           | 14 | 0.055       | 17.34   | <0.0001 | Significant |
| A-filler-binder ratio | 9.600 E-003    | 1  | 9.600 E-003 | 3.04    | 0.1017  |           |
| B-compaction time     | 1.667 E-005    | 1  | 1.667 E-005 | 5.278 E-003 | 0.9430  |           |
| C-compaction pressure | 0.062          | 1  | 0.062       | 19.64   | 0.0005  |           |
| D-compaction temperature | 0.019       | 1  | 0.019       | 0.20    | 0.6628  |           |
| AB                    | 0.036          | 1  | 0.036       | 11.43   | 0.0041  |           |
| AC                    | 0.053          | 1  | 0.053       | 16.75   | 0.0010  |           |
| AD                    | 0.027          | 1  | 0.027       | 8.62    | 0.0102  |           |
| BC                    | 6.250 E-004    | 1  | 6.250 E-004 | 0.20    | 0.6628  |           |
| BD                    | 0.11           | 1  | 0.11        | 34.49   | <0.0001 |           |
| CD                    | 0.096          | 1  | 0.096       | 30.43   | <0.0001 |           |
| A²                    | 0.11           | 1  | 0.11        | 36.00   | <0.0001 |           |
| B²                    | 0.037          | 1  | 0.037       | 11.81   | 0.0037  |           |
| C²                    | 0.27           | 1  | 0.27        | 85.78   | <0.0001 |           |
| D²                    | 2.411 E-003    | 1  | 2.411 E-003 | 0.76    | 0.3960  |           |
| Residual              | 0.047          | 15 | 3.158 E-003 |        |         | Not significant |
| Lack of fit           | 0.042          | 10 | 4.203 E-003 | 3.94    | 0.0717  |           |
| Pure error            | 5.333 E-003    | 5  | 1.067 E-003 |        |         |           |
| Cor total             | 0.81           | 29 |             |         |         |           |
conductivity are shown in Figure 4(a) to (f). From Figure 4(a) it can be seen that increasing the filler-binder ratio increases the thermal conductivity while that of compaction time gives a negative trend. Figure 4(b) and (c) also show a similar trend for filler-binder ratio while increasing compaction temperature and compaction pressure gave a decrease in thermal conductivity as seen in Figure 4(f). From Figure 4(d) and (e) the reverse effects of compaction time, compaction pressure and compaction temperature are seen to repeat.

Experimental parameters and thickness swell

**ANOVA and lack of fit test for thickness swell.** The ANOVA for thickness swell shown in Table 5, gave a Model F-value of 6.35 implying that the model is significant and has a 0.05% chance to occur due to noise. Significant model terms are identified with “Prob > F” values less than 0.0500, thus from the table it can be seen that D, AD, BC, BD, A², B² are significant model terms, hence the final equation is reduced to:

\[
\text{Thickness swell} = + 2.62448 + 0.07000D
- 0.008375AD - 0.00335BC - 0.03025BD \\
+ 0.0068A^2 - 0.0112B^2
\]  

The lack of fit test for thickness swell is shown in Table 6, with a “Lack of Fit F-value” of 1.00 implying that it has a 53.66% chance to occur due to noise, thus the model developed is good.

**Normal Probability plot for thickness swell.** The normal plot of residuals for thickness swell shown in Figure 5 gave a closely packed points around the diagonal indicating that the chosen model is adequate to predict the percentage thickness swell.

**Combined effect of parameters on thickness swell.** The 3D surface plots for thickness swell is shown in Figure 6. The combined effect of filler-binder ratio ratio and compaction time on thickness swell in Figure 6(a), shows that increasing filler-binder ratio ratio increased thickness swell while increasing compaction time decreased thickness swell. The effects of filler-binder ratio ratio on thickness swell were seen to repeat in Figure 6(b) and (c), while that of compaction time is also seen in Figure 6(d) and (e). From Figure 6(f) it can be seen that increasing both the compaction temperature and compaction pressure decreases the thickness swell.

Experimental parameters and water absorption

**ANOVA and lack of fit test for water absorption.** The ANOVA for water absorption shown in Table 7, gave a Model F-value of 34.29 implying that the model is significant and has a 0.01% chance to occur due to noise. Significant model terms are identified with “Prob > F” values greater than 0.01, thus from the table it can be seen that A, C, D, AB, AC, AD, BD, CD, A², B², D² are significant model terms, hence the final equation is reduced to:

\[
\text{Water absorption} = -0.11477 - 0.64060A + 0.020885C \\
+ 1.08401D + 0.00505AB + 0.0036125AC \\
- 0.019187AD - 0.007625BD - 0.00540625CD \\
+ 0.00237083A^2 + 0.021483B^2 + 0.014193D^2
\]  

**Figure 3.** Normal probability plot for thermal conductivity.
The lack of fit test for water absorption is shown in Table 8, with a “Lack of Fit F-value” of 0.72 implying that it has a 68.99% chance to occur due to noise, thus the model developed is good.

Normal probability plot for water absorption. The normal plot of residuals for water absorption shown in Figure 9 gave a closely packed points around the diagonal indicating that the chosen model is adequate to predict the percentage water absorption, see Figure 7 for Normal Probability plot for water absorption.

Combined effect of parameters on water absorption. Figure 8 shows the 3D surface plots for

Figure 4. 3D surface plots of the effect of parameters on thermal conductivity: (a) Compaction time versus filler-binder ratio, (b) compaction temperature versus filler-binder ratio, (c) compaction pressure versus filler-binder ratio, (d) compaction temperature versus compaction time, (e) compaction pressure versus compaction time, and (f) compaction pressure versus compaction temperature.
water absorption which gives the combined effect of two parameters at each instance. The combined effect of compaction time and filler-binder ratio in Figure 8(a) shows that water absorption increases with increasing filler-binder ratio while the increase in compaction time decreases water absorption. Similar effect of filler-binder ratio content on water absorption is seen in Figure 8(b) and (c) while the reverse effect of compaction time on water absorption is repeated in Figure 8(d) and (e). The effects of compaction temperature and compaction pressure is seen in Figure 8(f), as increase in both parameters decreases water absorption.

**Optimization of the central composite design.** Table 9 shows the various solutions obtained from the optimization process. The selected solution shows parameter levels of 16.205% filler-binder ratio, 9.569 min compaction time 200°C compaction temperature and 10.956 MPa compaction pressure. These optimized parameter levels will yield a response of 0.584 W/MK thermal conductivity, 0.772% thickness swell and 0.26% water absorption.

**Validation of result.** Figure 9 shows the contrast between the optimization result obtained and that of the ceiling.
board based on the uncarbonized breadfruit seed coat. It demonstrates that the carbonization of the breadfruit seed coat increases the ceiling board’s thermal conductivity while reducing the rate of absorption of water and thickness swell.

**Discussion of Result**

From the optimization result the combination of the production parameters; 16.205% filler-binder ratio, 9.569 min compaction time 200°C compaction temperature and 10.956 MPa compaction pressure, will yield a

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**Figure 6.** 3D Surface of the effect of the parameters on thickness swell: (a) Compaction time versus filler-binder ratio, (b) compaction temperature versus filler-binder ratio, (c) compaction pressure versus filler-binder ratio, (d) compaction temperature versus compaction time, (e) compaction pressure versus compaction time, and (f) compaction pressure versus compaction temperature.
ceiling board of 0.584 W/MK thermal conductivity, 0.772% thickness swell and 0.26% water absorption. 0.584 W/MK thermal conductivity which is quite close to that of the uncarbonized breadfruit seed coat based of 0.367 W/mK reported by Ezenwa et al.\(^9\) and better than that of 0.07 kW/MK and 0.082 kW/MK for waste paper and rice husk based.\(^18\) The water absorption of 0.26% obtained is better than 12.3–23.35% water absorption for saw dust based\(^19\) and that of 7.5–14.5% for waste paper and rice husk based.\(^20\) The thickness swell of 0.772% obtained from is much better than that of 9.83% for jatropha curcas seedcake by Olorunmaiye and Ohijeagbon.\(^6\) Thus carbonising the breadfruit fruit seed coat has shown good physical properties for ceiling board production.

### Conclusion

The study investigated the effect of carbonized breadfruit seed coat as a ceiling board production filler material. Four parameters that cut through the composition of material (filler-binder ratio) and production parameters (compaction time, compaction temperature and compaction pressure), were considered. Three responses (thermal conductivity, water absorption and thickness...
swell), which are the physical properties of the formed ceiling board, have been studied against these parameters. For optimization of these parameters, the central composite design of the Design Expert program was used. To view the effects of these parameters on the responses, 3D surface plots were also produced. The result obtained gave parameter levels of 16.205% filler-binder ratio, 200°C compaction temperature of 9.569 min compaction time and 10.956 MPa compaction pressure to generate a response of 0.584 W/MK.

Figure 8. 3D surface plot of the effect of the parameters on water absorption: (a) Compaction time versus filler-binder ratio, (b) compaction temperature versus filler-binder ratio, (c) compaction pressure versus filler-binder ratio, (d) compaction temperature versus compaction time, (e) compaction pressure versus compaction time, and (f) compaction pressure versus compaction temperature.
thermal conductivity, 0.772% thickness swell and 0.26% water absorption. Compared to current designs, these findings indicate that the carbonized breadfruit seed coat is a good filler material for the development of ceiling boards.

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