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# **Research article**

# Influence of chemical treatment on the properties of cement-paper hybrid composites for ceiling board application



<sup>a</sup> Department of Mechanical Engineering, Landmark University, Omu-Aran, PMB 1001, Kwara State, Nigeria

<sup>b</sup> Department of Metallurgical and Materials Engineering, Federal University of Technology, Akure, PMB 704, Ondo State, Nigeria

<sup>c</sup> Faculty of Materials Science and Chemical Engineering, Ningbo University, China

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# ABSTRACT

This study evaluates the effect of treated and untreated jute fiber/eggshell particulate reinforced cement-paper matrix composites for ceiling board application. Treated jute fiber (TJF) was obtained by immersing untreated jute fiber (UJF) into 1.25 M sodium hydroxide (NaOH) solution in a shaker water bath maintained at 40 °C for 4 h. Eggshells (ESP) were pulverized and sieved to -75µm. Samples were prepared by varying the fiber volume fraction from 0.5 to 2.5 wt.% in the composites. While other constituents such as the binder (cement) and eggshell were kept constant. An hydraulic press cold compaction molder was utilized in the production of the hybrid composites in a predetermined mix ratio designed based on previous research. The samples produced were cured for 7 and 14 days, then sundried for 36 h. The physical, thermal, mechanical and wear behaviour of the produced composites were evaluated while the surface morphology of the fractured splitting tensile samples were analyzed. The result reveals that TJF/ESP hybrid composites had better performance than UJF/ESP hybrid composites in most of the tests carried out. Increase in the number of curing days was found to also enhance the properties of the composite developed. While 2.5 wt. % TJF/ESP showed an optimum properties among the composites tested. When compared with standard, it is concluded that the hybrid composites developed can be suitable for ceiling boards and also find possible application in wall partitioning.

#### 1. Introduction

Building can be regarded as one of the most important needs in the history of human evolution, the problem of housing deficit increases even with the government intervention to ensure the availability of shelter for their citizens [1]. All over the world, pressure has been mounted on building materials such as cement, roof frames, bricks, and windows, roof tiles, ceiling frames, and ceiling tiles. Owing to this increasing demand, there is a corresponding increase in the price of these materials [2, 3, 4]. Global warming has become more prominent in recent years, characterized with change in weather, ocean currents, a major rise in average temperature and climate change in different parts of the world [5, 6]. For this reason, there is a need to develop building materials with improved insulation properties coupled with good mechanical characteristics. Various policies have been enacted to reduce the use of fossil fuels and adoption of minimal carbon energy technologies [7, 8]. Ceiling is

regarded as a very important part of a building; it reduces the amount of heat energy dissipated into the room when absorbed by the roof tiles. This suggests that ceiling materials must possess good thermal insulation property and high specific heat capacity while the strength of the material is not compromised below the required standard. The use of waste material such as paper waste has a significant role to perform in the reduction of housing material deficit. Paper is a product of wood and its primary constituent are cellulose, lignin, hemicellulose and ash [9, 10]. After use, these materials are regarded as waste thereby littering the environment and causing environmental pollution [11]. Reprocessing of paper waste can potentially reduce the consumption of wood (a product of trees) which has been reported to control the effect of greenhouse gases in the environment, reverse desertification trend and also serve as a good substitute for materials that a large amount of fossil fuel are needed in their production [12, 13]. An adequate way of utilizing paper waste is the recycling of paper and the addition of other additives such as cement,

\* Corresponding author.

E-mail addresses: adediran.adeolu@lmu.edu.ng, dladesoji@gmail.com (A.A. Adediran), tobalogs39@gmail.com (O.A. Balogun).

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<sup>\*\*</sup> Corresponding author.

starch, and natural fibers to develop new material which can be employed in ceiling, packaging, bricks, and other light weight applications [14, 15]. Natural fibers are agro wastes that are biodegradable in nature, they are renewable, cheap and characterized by their light weight. These fibers and their extracts in recent years have been embedded into various matrix material such as paper, polymers, metals, ceramics (clay, bricks and concrete); so as to improve their mechanical, thermal properties and durability [16, 17, 18]. The drawback of natural fiber is mainly linked to their hydrophilic nature which leads to loss of mechanical properties by the formation of poor interfacial adhesion with the intending matrix and they can decompose when used in sand and cementitious based matrix materials [19]. A way of overcoming this challenge is by subjecting natural fibers to alkali treatment which reduce their hydrophilicity, enhance their adhesion to matrix, and reduces effectively the rate at which these fibers decompose in sand and cementitious based matrix materials [20, 21]. Various researchers have studied the effect of natural fiber addition as reinforcement in paper and cementitious matrix while employing cement or starch as binder [22]. Akubueze et al [22], reinforced plaster of Paris (POP) with kenaf fiber modified with 10 % Sodium Lauryl Sulphate. The result of the research shows an increase in the tensile strength of the developed composite, which seems to suggest that natural fibers can be used as reinforcement in brittle materials in order to enhance their resistance to deformation when mechanical stress is applied. Oladele et al [23], added sponge fiber to paper matrix while utilizing cement as the binder and it was concluded that the incorporation of sponge fiber enhance the mechanical performance of the matrix up to 4 wt. %, followed by a decrease. The performance of Scirpus grossus fiber when compared with fiberglass in the development of ceiling materials further suggests that natural fibers can be adopted and investigated for the production of cheap, light weight and eco-efficient materials in building applications [24]. This research investigates the influence of chemical treatment on the properties of cement-paper matrix while utilizing untreated jute fiber (UJF), treated jute fiber (TJF) and eggshell particulate (ESP) as reinforcing materials. In addition, the thermal, mechanical and wear behaviour of the hybrid composite were studied.

# 2. Materials and method

# 2.1. Materials

Sodium hydroxide (NaOH) and distilled water were acquired from a chemical store in Omu-Aran, Kwara state, Nigeria. Waste paper, cement, and eggshell were procured from a local store in Akure, Ondo state, South Western part of Nigeria while the jute plant used was obtained from a local farm in Awule, Ondo State, Nigeria.

# 2.1.1. Processing of the jute fiber

Jute plants were harvested from a local farm in Awule, Ondo State. The Harvested jute plants were tied together in bundles (20 jute plant per bundle). Jute plant bundles were immersed in water for a period of 14 days to allow the swelling of the jute plant inner cell. The water used in soaking the plants was changed daily to ensure the final jute fibers produced were clean. Stripping of the jute fibers from the stalk bundles was carried out while the plants were still immersed in water. Afterwards, jute fibers were sundried for seven (7) days and divided into two portions, the first portion was left untreated while the other portion was treated with 1.25 M sodium hydroxide (NaOH) solution in a shaker water bath at a temperature of 40 °C for 4 h. Thereafter, the rinsing of the treated fiber in distilled water was carried out while the neutrality of the fibers was tested using litmus paper, which thus confirmed the neutrality. Untreated and treated jute fibers shown in Figure 1(a) and (b) were trimmed to 50 mm length and sun-dried for 3 days to ensure the removal of water before been used as reinforcement in cement paper pulp matrix.

#### 2.1.2. Processing of eggshell

The eggshell procured were washed with detergent and rinsed in water to ensure the removal of the residual organic materials. This was succeeded by sun drying for 6 h. After which eggshell was pulverized and sieved to particle size of -75  $\mu$ m. The eggshell and pulverized eggshell were as shown in Figure 1(c) and (d).



Figure 1. Showing (a) Untreated Jute fiber (b) Treated jute fiber (c) Eggshell and (d) Pulverized eggshell.

# 2.1.3. Production of paper pulp

Paper waste were shredded using paper shredder with further reduction of the shredded papers into smaller seizes with the aid of scissors. The paper (cut into smaller sizes) were collected and soaked in hot water initially boiled to 100 °C. The mixture was then allowed to cool and left for 4 days in the bucket. On the fourth day, the paper pulp was ground using a paper pulp grinding machine. The slurry as shown in Figure 2a was stirred minutes using a wooden rod for 15 min. Afterwards, de - watering was effected by pouring the slurry into a sack bag. A representative sample for splitting test is as shown in Figure 2(b).

# 2.2. Mould dimensions

Various mould dimensions were used for production of the test samples. Rectangular test molds ( $200 \times 50 \times 50 \text{ mm}^3$  and  $64 \times 12.7 \times 6.4 \text{ mm}^3$ ) and cylindrical molds ( $25 \times 20 \text{ mm}$  and  $50 \times 100 \text{ mm}$ ) were utilized in the preparation of samples used for physical, thermal, mechanical and wear tests.

# 2.3. Composites development

Cement-paper composite were developed using hydraulic press cold compaction molder. Dried pulp, ordinary portland cement, particulate eggshell, water and (treated and untreated) jute fiber were measured using electronic weighing balance, composite production was carried out in a predetermined proportion as represented in Table 1. Dry paper pulp was initially mixed manually with cement in a container; water was added in order to make the paper soft and improves its binding capacity with the additive. Mechanical mixing was employed to ensure homogenous and proper mixing; the ratio of water to cement adopted was 0.68–0.75. Each weight fraction was mixed and used in filling the rectangular and cylindrical molds employed. Compaction of the admixture was done for a period of 4 min at a temperature of 27 °C and an applied pressure of 0.25 kPa using hydraulic press cold compaction molder by placing each test filled mould between the upper and lower plates. After compaction the samples were cured in a polyethylene bag (for 7 and 14 days) and then sundried for 36 h. The splitting tensile samples were as shown in Figure 2b.

# 2.4. Preliminary characterization of samples

The initial test such as tearing resistance (420 mN), bursting strength (0.159 MPa), grammage (74 g/m<sup>2</sup>), specific heat capacity (1.410 J/gK), drainage time (5 min), thermal conductivity (0.047 W/mK), and consistence (medium) were assessed to determine the inherent properties of waste office paper used in accordance with existing standards [25, 26, 27, 28, 29, 30, 31]. Compositional analysis, surface morphology of jute fiber and the chemical composition of the eggshell particulate were studied.

# 2.5. Test method for physical properties

#### 2.5.1. Porosity and water absorption

Prior to evaluating the percentage porosity and water absorption, samples used in were sundried for 8 h. Evaluation of the amount of porosity was necessary to determine the degree of void present in the composite produced while % water absorption is necessary determines the behaviour of these materials when exposed to water. Porosity and water absorption and were measured in compliance with [32], adopting Eqs. (1) and (2) respectively

(%) 
$$PC = \frac{W_2 - W_1}{W_2 - W_3} \times 100$$
 (1)

(%) WC = 
$$\frac{W_2 - W_1}{W_1} \times 100$$
 (2)

where; PC-porosity of the composite; WC - water absorption of the composite;  $W_1$  - dry weight of samples when suspended in air;  $W_2$  - saturated mass of the samples suspended in air;  $W_3$ -saturated mass of the sample when suspended in water.

#### 2.5.2. Bulk density

The samples used to determine the bulk density of the composite were prepared in a similar way to that of porosity and water absorption. Beforehand, samples were also sundried for 8 h. Bulk densities of the samples were assessed in commensuration with [33] while employing Eq. (3).

$$BC = \frac{W_1}{W_2 - W_3} \times \text{density of water}$$
(3)

where; BC-bulk density of the composite;  $W_1$ - dry weight in of samples when suspended in air;  $W_2$ - saturated mass of the samples suspended in air;  $W_3$ -saturated mass of the sample when suspended in water.

# 2.5.3. Linear shrinkage and volumetric

Linear and volumetric shrinkage was analyzed with the aid of vernier caliper and carried out in agreement with [34]. This property was evaluated to determine the dimensional stability of the produced samples while utilizing Eqs. (4) and (5) respectively.

(%) 
$$LSC = \frac{L_2 - L_1}{L_1} \times 100$$
 (4)

(%) VSC = 
$$\frac{V_2 - V_1}{V_1} \times 100$$
 (5)

where; LSC-linear shrinkage of the composite; VSC-volumetric shrinkage of the composite;  $L_1$ -initial length of samples after compaction;  $L_2$ -length



Figure 2. Showing (a) Paper slurry (b) Splitting tensile samples.

# Table 1. Mix ratio of the composite.

Designation	Paper (%)	Cement (%)	Fiber (%)	Eggshell (%)
Control	85	15	-	-
0.5	74.5	15	0.5	10
1	74	15	1	10
1.5	73.5	15	1.5	10
2	73	15	2	10
2.5	72.5	15	2.5	10

of the sample after curing and oven drying;  $V_1$ -initial volume of samples after compaction,  $V_2$ -volume of the sample after curing and oven drying.

# 2.6. Test method for thermal properties

#### 2.6.1. Thermal conductivity

Thermal conductivity was evaluated using guarded hot plate method, this property determine the rate of heat transport by a mechanism called conduction through the cement-paper fiber board. Thermal conductivity of the composite samples was assessed in line with [30], using Eq. (6).

$$TC = \frac{wcp(\emptyset_1 - \emptyset_2)4x}{\pi d^2 (T_1 - T_2)t}$$
(6)

where; TC-thermal conductivity; w-weight of the plate; cp-specific heat capacity of the plate;  $\emptyset_1$ - initial temperature of plate B;  $\emptyset_2$ -final temperature of plate B; d-diameter of the composite; x-thickness of the composite; T<sub>1</sub>-initial temperature of plate A; T<sub>2</sub>-final temperature of plate A; t-time taken to attain a stable temperature.

#### 2.6.2. Specific heat capacity

Specific heat capacity was performed in line with [28], Differential scanning calorimetry was used in the determination of the specific heat capacity for each sample. Composite sample of height 20 mm and diameter 25 mm were utilized.

#### 2.7. Test method for mechanical properties

The samples used for evaluating the mechanical properties were initially oven dried at a temperature of 50  $^\circ$ C for a period of 12 h.

# 2.7.1. Compressive strength

Evaluation of the compressive strength of the composite samples was carried out the aid of universal testing machine as per [35] using Eq. (7). Samples with height of 100 mm and diameter 50 mm were used for this evaluation. Three samples were tested for each composition and the average obtained was used as the compressive strength.

$$C = -\frac{P}{A}$$
(7)

where; C- compressive strength; P- load applied; A-cross sectional area of the sample.

# 2.7.2. Flexural strength

Flexural strength of the composite produced was performed in agreement with [36] using Instron Universal testing machine. Eq. (8) was employed in computing the flexural strength while the gauge length of the sample is  $200 \times 50 \times 50 \text{ mm}^3$ . Three samples were tested for each composition and the average obtained was used as the flexural strength.

$$F = \frac{3PL}{2WT^2}$$
(8)

where; F-flexural strength; P-maximum load; L-length of the sample; W-sample's width; T-thickness of sample.

#### 2.7.3. Tensile strength

Instron Universal testing machine was used for the evaluating the tensile strength of the developed samples. Tensile strength was carried out in line with [37] and the estimation of the resistance of each sample to a stress in tension was done using Eq. (9). Samples with dimension of 100 mm height and 50 mm diameter were used. Three samples were evaluated for each composition and the average was used as representative value.

$$T = \frac{P}{A}$$
(9)

where; T-tensile strength; P-maximum tensile load; A-cross sectional area of the sample.

#### 2.7.4. Split tensile strength

Split tensile strength of composite samples was evaluated using Instron Universal testing machine. The test was carried out in accordance with [38] and computed using Eq. (10). Samples with dimension of 100 mm height and 50 mm diameter were used in the determination of split tensile strength. Three samples were evaluated for each weigh fraction and the average was used as the value for split tensile strength.

$$Ts = \frac{2P}{\pi dl}$$
(10)

where; Ts-split tensile strength; p-applied load; d-diameter of composite sample; l-length of composite sample.

#### 2.7.5. Impact resistance

A measure of resistance to Impact was determined in order quantity the samples resistance to sudden load while utilizing Izod impact testing machine. The test was observed in accordance with [39]. Samples used were of 60 × 12.7 × 3.2 mm<sup>3</sup>. Three samples were evaluated and the average was adopted as representative value for the resistance to impact for each weight fraction.

# 2.7.6. Bursting strength

Bursting strength of the composite samples was evaluated as per [26], using a ZwickRoell burst tester which employs pressure in bursting of the samples, the composite samples were made into a board with gauge length  $20 \times 15 \times 4$  mm<sup>3</sup>. The test measure the resistance of the paper samples to increasing pressure, a fixed volume flow was used in the determination of bursting strength for all weight fractions. Three samples were evaluated for each weight fraction and the average was computed as the bursting strength.

#### 2.7.7. Hardness

Evaluation of the resistance of the developed sample was done in line with [40], Vickers micro hardness tester was employed. The diamond pyramid indenter and a load of 0.5 kg were used for the static indentation. Computation of the hardness for each sample was done using Eq. (11). Samples were indented on three different occasions.

$$HV = 1.854 \frac{P}{M^2}$$
(11)

where; HV-vickers hardness; P-load used for indentation; M<sup>2</sup>-mean of the diagonal.

#### 2.8. Wear behaviour

Evaluation of the abrasive wear of the composite samples was evaluated as per [41] using Taber abraser. The initial weight of the sample was determined using electronic weighing balance, followed by mounting of the sample on a rotating platform at a fixed speed of 100 rpm and the abrasion wear was carried out to determine the effect of jute fiber and eggshell particulate addition on the wear loss at composite samples surfaces. The final weight of the samples was also measured and the difference in weight was determined as wear loss index using Eq. (12). Three samples were evaluated and their average was used as a representative value for each composition.

$$W = \frac{W_i - W_o}{W_i} \tag{12}$$

where; W-weight loss index;  $W_i$ -initial weight of sample and  $W_o$ -final weight of sample.

## 2.9. Material characterization

Scanning electron microscope (JOEL JSM-7600F) was used for the characterization of the surface morphology of jute fiber and fractured split tensile samples.

#### 3. Result and discussion

#### 3.1. Preliminary analysis

#### 3.1.1. Elemental composition of particulate eggshell

The EDX carried out on the eggshell was as shown in Table 2, revealed the presence of Ca and Fe in considerable amount. These element can combine in the presence of oxygen to form compounds such as calcite (CaCO<sub>3</sub>) and ferric oxide (Fe<sub>2</sub>O<sub>3</sub>) are good re-enforcers that can improve the properties of the developed hybrid composite [42].

# 3.1.2. Compositional analysis of paper, jute fiber, and morphology of jute fiber

The waste office paper used contains cellulose, hemicellulose, lignin ash and other constituent. Cellulose dominates the constituent of paper, from Table 3 cellulose constitute 86.73 % while other constituent are present in 1.5 %. Paper is a product of wood and the lignin content present in the paper used has been reduced to 1.3 % during production. As revealed in Table 3, untreated jute fiber is made up of 57.45 % cellulose, 25.32 % hemicellulose, 15.41 % lignin and other constituent in proportion of 1.872 %. Alkali treatment afforded the fiber led to a

Table 2. Elemental composition of particulate eggshell.

Compounds	Wt. %
Si	0.12
Ca	79.4
К	5.1
Mg	0.3
0	11.8
Al	0.6
C	0.08
Fe	2.1
Na	0.5

decrease in lignin, hemicellulose, and other constituent [43] as shown in Table 3 and Figure 3(a) and (b) respectively. It is noteworthy to mention the increase in the cellulose present in jute fiber, this leads to increase in cellulose present on fiber surface ready for contact with the matrix and hence, interfacial adhesion is improved [44, 45]. Also, the hydrophilicity of the jute fiber was reduced due to removal of the lignin (which is non-reactive) and the modification of the cellulose [46]. Therefore, treated fiber was observed to possess rough surface which assist in the formation strong interfacial adhesion and interaction between components of matrix and reinforcement.

#### 3.2. Physical properties

#### 3.2.1. Porosity

Porosity test was used in estimating the amount of pores present in the JF/ESP hybrid composite. From Figure 4, it is revealed that the amount of porosity observed in the developed JF/ESP was reduced when compared to the control sample. Treated samples were found to have lower porosity than the untreated ones, this feat can occur on the account of the chemical treatment on the interfacial adhesion between the matrix and the reinforcement. Chemical treatment increases the surface area of cellulose in contact with the matrix. Hence, adhesion was improved [47]. The presence of the cement which serves as a binder in the present of water is also responsible for the bond strength in the composite samples. Therefore, increase in the number of curing days results in improved hydration and better compactment of the composite. Curing ensures the presence of free water content during hydration; this aids complete hydration of the cement [48]. The result presented highlights that 0.5 wt. % TJF/ESP addition had the lowest porosity of all the samples produced with values of 33.48 % pores after the sample was cured for 14 days. This indicates that 0.5 wt. % TJF/ESP had 31.6 % reduction in porosity when compared with the control sample cured for the same number of days. Among untreated samples, 0.5 wt. % UJF/ESP composite also had the lowest porosity with the best performance obtained at 14 days curing. The result of obtained agrees with the work of Ahmad et al [49].

#### 3.2.2. Water absorption

The performance of JF/ESP hybrid composite to water absorption was as represented in Figure 5. The behavior of the sample to water absorption was similar to Figure 4, where porosity reduced with fiber addition when compared to the control sample. 0.5 wt. % TJF/ESP addition at 14 days curing had the lowest water absorption capacity. 0.5 wt. % TJF/ESP gave a value of 35.86 and 34.11 % compared to the control sample with values of 42.30 and 38.12 % water absorption at 7 and 14 curing days, indicating a reduction of 17.98 and 11.76 % at 7 and 14 curing days. The chemical composition of the paper used as shown in Table 3, revealed that paper is mainly composed of cellulose, the cellulose content in paper has high affinity for water molecules [50]. Since paper content reduces with the addition of ESP and TJF which occupies more volume and less affinity for water, the reduction in water absorption is justified. However, TJF/ESP composite had lower water absorption capacity their untreated counterparts. This behavior may be attributed to the alkaline treatment given to the fiber, which is responsible for the removal of lignin and hemicellulose content present in the fiber. The treated fiber has been reported to possess increased surface area which aids the evaporation of water rather than retention of water in the case of untreated fibers [51]. Therefore, the hydrophilic nature of developed composite is reduced. Chemical treatment is a potent way of reducing the water absorption property of natural fibers and their incorporation into the matrix ensue reduction in water absorption when compared to the untreated ones. The behavior observed corroborates with the findings of Owodunni et al [52].

# 3.2.3. Bulk density

The density of the produced composite samples was determined and the result was as revealed in Figure 6. It was observed that 5 wt. % UJF/

# Table 3. Compositional analysis of paper and jute fiber.

Constituents	Paper pulp (%)	Untreated jute fiber (%)	Treated jute fiber (%)
Cellulose	86.73	57.45	69.45
hemicellulose	8.37	25.32	19.81
lignin	1.3	15.41	9.47
ash	2.1	-	-
others	1.5	1.82	1.27

ESP addition had the best performance in terms of density (which is a property where lower value is desired) with a value of 0.590 g/cm<sup>3</sup> at 7 curing days, which gives a reduction of 18.05 and 24.35 % compared to control samples cured for 7 and 14 days respectively. The trend observed was increase in bulk density at 0.5 JF/ESP addition, followed by a progressive decrease from 1-2.5 wt. % JF/ESP. Since JF and ESP possesses density that are higher than that of the paper [53, 54, 55]. Therefore, initial increment in bulk density may ensue and further reduction in bulk density may be attributed to increasing volume of JF. However, it was



Figure 3. SEM images of (a) Untreated jute fiber (b) treated jute fiber.



Figure 4. Variation of porosity with untreated and treated JF/ESP hybrid cement-paper composites.



Figure 5. Variation of water absorption with untreated and treated JF/ESP hybrid cement-paper composites.

observed that UJF/ESP hybrid composite samples possess lower density that the treated ones, due to increase in the density of treated fibers [56]. It is noteworthy to mention that samples cured for 14 days possess higher bulk density compared to samples cured for 7 days. Similar result was reported by Iswanto et al [57] for Sorghum based fiber board. In recent years, materials with reduced density are well appreciated in engineering applications due to easier construction, economical transportation, and labour convenience [58, 59]. This This research highlights the potential of JF/ESP eco-friendly reinforcements that can be incorporated into matrix material in order to reduce their bulk density while other properties are also modified.

# 3.2.4. Linear shrinkage

The result obtained shows the response of JF/ESP hybrid composite samples to linear shrinkage. From Figure 7, it was deducted that the control sample had the highest percentage linear shrinkage with values of 0.51 % and 0.67 % at 7 and 14 days of curing respectively. Among the JF/ESP hybrid composite, 2.5 wt. % UJF/ESP had the best linear stability of 0.41 and 0.45 % at 7 and 14 days respectively. This reduction observed can occur because of the presence of JF/ESP in the hybrid composite, which reduces the volume of paper matrix available for shrinkage. However, the chemical treatment given to the fiber had no effect on the shrinkage of the hybrid composite samples. The trend observed was increase in percentage linear shrinkage with higher number of curing days, this occurrence may be attributed to the incomplete hydration process at 7 days. The free water present in the composite were further employed in hydration process, therefore, hydration is continued. At the completion of this process, the product may completely fill up the pores present in the samples. Incapability of the hydration product to completely fill up these pores, ultimately cumulate in the reduction in size and shape of the composite [60]. All hybrid composite samples satisfied the minimum requirement for linear shrinkage of ceiling boards according to ASTM C 209 [61].

#### 3.2.5. Volumetric shrinkage

Figure 8 shows the result of the composite sample produced in terms of volumetric shrinkage. The performance of the samples was similar to that of linear shrinkage where the control sample had the highest percentage linear shrinkage as revealed in Figure 7. A value of 1.19 and 1.22 % was recorded as the percentage volumetric shrinkage at 7 and 14 days of curing respectively. It was observed that volumetric shrinkage was promoted with increase in the number of curing days and the addition of JF/ESP had a reduction effect on the shrinkage of the hybrid composite samples. 1.5 wt. % UJF/ESP composite was observed to possess the best stability in terms of volumetric shrinkage among the reinforced hybrid composited. Chemical treatment had no effect on the percentage change is volumetric shrinkage. This result uphold the claim of [62], indicating

that the incorporation of JF/ESP into paper matrix improves the dimensional stability of the composite developed.

# 3.3. Thermal properties

# 3.3.1. Thermal conductivity

Figure 9 shows the response of the various weight fractions of the developed samples to thermal conductivity. From Figure 9, it was noted that 2.5 TJF/ESP sample cured for 14 days had the highest thermal conductivity with a value of 0.156 W/mK, showing an enhancement of 25.80 % when compared with the control sample cured for the same number of days. Followed closely by this, is 2 wt. % TJF/ESP composite sample which has the second highest thermal conductivity with 19.35 % enhancement when juxtaposed with control sample. Thermal conductivity was observed to reduce with increasing UJF/ESP addition; indicating that 2.5 wt. % UJF/ESP sample had the best thermal insulation characteristics. This supports the claim made by [63] who reported the same behaviour with addition of date palm fiber to natural mortal. Surface modification of the fiber give rise to a linear increase in the thermal conductivity of TJF/ESP hybrid composites, this is attributable to increase in the surface charge of treated fibers. Hence, thermal conductivity is enhanced, as emphasized in previous study by [64] who reported the role of treated fiber in improving thermal conductivity. The value reported for asbestos as stated by Obam [65] is 0.096 W/mK which is in the same range with was observed in this study.

A common trend observed was the increase in thermal conductivity of UJF/ESP and TJF/ESP hybrid composites as curing progressed from 7-14 days, upholding the findings of [66]. In ceiling applications, a material with lower thermal conductivity is desirable, in order to reduce the amount of heat energy conducted into the building on a sunny day. All UJF/ESP hybrid composites have thermal conductivity lower than the control material, this suggest that these samples are more efficient than the control in applications where thermal insulation property is essential.

#### 3.3.2. Specific heat capacity

Specific heat capacity of a material is a function of density and heat [67]. From Figure 10, where it was deducted that 2.5 wt. % TJF/ESP hybrid composite cured for 7 days shows the most significant improvement in specific heat capacity with a value of 1.24 J/gK. This is consistent with the research of [68] who reported an increase in specific heat capacity with the addition of treated sisal fiber. Untreated samples were found to experience reduced specific heat capacity with increasing fiber addition while for the treated samples it was the other way round. 0.5 wt. % UJF/ESP hybrid composite had a specific heat capacity of 1.195 J/gK, which is the highest among UJF/ESP hybrid composite experienced a 1.23 % reduction in specific heat capacity when matched with the control sample cured for the same number of days.



Figure 6. Variation of bulk density with untreated and treated JF/ESP hybrid cement-paper composites.



Figure 7. Variation of linear shrinkage with untreated and treated JF/ESP hybrid cement-paper composites.



Figure 8. Variation of volumetric shrinkage with untreated and treated JF/ESP hybrid cement-paper composites.



Figure 9. Variation of thermal conductivity with untreated and treated JF/ESP hybrid cement-paper composites.

Increasing the number of curing days of samples led to a decrease in the specific heat capacity value. This is ascribable to the reduction of free water present in the composite and the formation of more hydration products which fills up the pores present in the composite at increase number of curing day. Therefore, specific heat capacity is reduced [69]. However in a building application, materials with higher value of specific heat capacity is desired; this insinuate that samples reinforced with treated fibers and cured for 7 days had better performance than other breeds of samples produced.

#### 3.4. Mechanical properties

#### 3.4.1. Compressive strength

The behaviour of the composite samples to a compressive loading was as shown in Figure 11, where it was observed that 2.5 wt. % TJF/ESP hybrid composite gave the best performance, in terms of resistance to compressive loading. Similarly, 2.5 wt. % TJF/ESP had a compressive strength of 0.74 and 0.81 MPa which is 45.1 and 50 % higher than the value obtained for the control sample at 7 and 14 days of curing



Figure 10. Variation of specific heat capacity with untreated and treated JF/ESP hybrid cement-paper composites.

respectively. The entire composite produced had better compressive strength than the control sample. This may be attributed the addition of JF/ESP since compressive strength improves with reduced in porosity, the addition of ESP serve as a filler and reduced the volume of pores present in the composite. Paper as a material is characterized with the presence of poor calcium silicate hydrate (C-S-H) cohesion, the addition of ordinary portland cement which possess strong C-S-H bond (known to serve as gel) improves the bond strength of the hybrid composite; hence, compressive strength is improved [70, 71]. The common trend noticed was the improvement in compressive strength with increasing fiber addition as reported by Oladele et al. [72] in their research, where an increased compressive strength was reported for cement-paper matrix reinforced with cow hair. Samples cured for 14 days had better performance than those cured for 7 days, this justifies the claim made by [73] who reported improvement in compressive strength as curing days progressed. Increase in curing days brings about continuation of hydration process of the JF/ESP composite; hence, compressive strength is enhanced. Among the untreated fiber reinforced composites 2.5 wt. % UJF/ESP also had the best compressive strength of 0.7 and 0.77 MPa at 7 and 14 curing days respectively; this highlight the effective stress transfer achieved along with good interfacial adhesion observed in Figures 20 and 21. The result obtained shows that JF/ESP which are regarded as agro waste has potential of improving mechanical properties of matrix material, when employed as reinforcements for various applications.

# 3.4.2. Flexural strength

Figure 12 reveals the response of composite samples to bending load, this property indicate the produced hybrid composites samples resistance

to deflection. The result obtained was similar to that of compressive strength as revealed in Figure 11. The 2.5 wt. % TJF/ESP hybrid composite had the best resistance to bending load with flexural strengths of 0.48 and 0.57 MPa when compared to the control sample where 0.25 and 0.29 MPa were obtained at 7 and 14 days of curing respectively. This agrees with the work of Oladele et al [23, 72] who reported increased flexural strength of cement-paper composite with sponge fiber addition up to 2 wt.% followed by a decrease. However, all samples had better flexural properties than the control. Similar to the behaviour observed in Figure 10, all hybrid composite produced had better flexural strength than the control material. It was noted that samples produced with treated fibers performed better than the untreated ones; this behaviour can be linked to the chemical treatment given to the fibers. Alkaline treatment has been reported to remove impurities, waxes, lignin, and hemicellulose from fiber surface [73]. Therefore, the percentage of cellulose present at the fiber surface increased and enhanced interfacial adhesion between the matrix and the fiber was achieved as highlighted in Figures 20 and 21. The 2.5 wt. % UJF/ESP also had the optimum value of flexural strength among the UJF/ESP hybrid composites with 64 and 79.31% enhancements at 7 and 14 days of curing respectively as in the case of compressive strength. This justified the use of UJF/ESP as capable reinforcing materials. Incorporation of second phase into matrix serve as impediment to dislocation movement, the barrier created ultimately cumulates in improved strength provided the second phase possess high strength and the interfacial adhesion between the reinforcement and matrix material is strong. The eventual passage of any dislocation results in the creation of stress field, making dislocation movement more difficult, hence, accumulation of these dislocations yields improves stiffness.



Figure 11. Variation of compressive strength with untreated and treated JF/ESP hybrid cement-paper composites.



Figure 12. Variation of flexural strength with untreated and treated JF/ESP hybrid cement-paper composites.

Nonetheless, flexural strength was discovered to improve with increasing number of curing days. This correlates with the finding of [66] who claims an increase in flexural strength with increase in number of curing days.

#### 3.4.3. Direct tensile strength

The response of the composite produced was as represented in Figure 13. Tensile strength of a material indicates the resistance of the material to applied tensile load that tends to pull the material apart. Figure 13 shows that the control sample had a tensile strength of 0.024 MPa at 7 days of curing and 0.011 MPa after the control samples was further cured for another 7 days, showing a 0.013 MPa reduction in tensile strength. The addition of 0.5 wt. % JF/ESP gave a value of 0.007 and 0.009 MPa for untreated and treated sample after 7 days of curing. Further increase in curing to 14 days resulted in a decreased tensile strength in both the treated and untreated sample. The initial decrease in tensile strength observed can be linked to the low volume of fiber and the presence of particulate eggshell. The best performance in tensile strength was recorded for 0.25 wt. % TJF/ESP at 7 days of curing, this sample had 150 % enhancement when compared to the control sample cured for the same number of days. The trend observed was a decrease in tensile strength at 0.5 wt. % JF/ESP addition, followed by progressive increase due to increase in the volume fraction of jute fiber which results into enhanced formation of interconnecting network between the fiber and reinforcement, therefore higher stress is needed to fracture the material [74]. Modification of the fiber surface with alkali treatment was discovered to improve the tensile properties of the developed composites. Therefore, treated samples possess better tensile properties at both 7 and 14 curing days due to improvement in the surface cellulose roughness which aids better adhesion between the matrix and reinforcement. Also, treatment of fibers has been reported to improve aspect ratio and tensile properties of the fiber, causing improvement in the load bearing capacity of the produced composite and this was highlighted by the good adhesion observed in the SEM images (Figures 19, 20, and 21) [75, 76]. Among the UJF/ESP hybrid composites, 2.5 wt. % fiber addition had optimum performance with tensile strength of 0.042 MPa at 7 days of curing, showing an enhancement of 108.3 % than the control sample cured for the same number of days. Increase in curing days in all samples was observed to result into a decrease in tensile strength.

#### 3.4.4. Splitting tensile

The splitting tensile behaviour of the developed composite samples was as revealed in Figure 14, where it was observed that the control sample had a value of 0.081 and 0.087 MPa at 7 and 14 days of curing respectively. Addition of 0.5 wt. % JF/ESP improved the splitting strength of the matrix to give a value of 0.089 and 0.105 MPa for UJF/ESP while 0.090 and 0.109 MPa were obtained for TJF/ESP at 7 And 14

days respectively. 2.5 wt. % TJF/ESP composite had the highest performance of all the hybrid composite samples produced with a value of 0.144 at 14 days of curing, showing an improvement of 0.102 and 0.057 MPa when compared with the control sample cured for 7 and 14 days respectively. The improvement observed can occur on the account of increase in the volume of fibers (as shown in Figures 19, 20, and 21) which serve as a barrier to dislocation movement, further movement of dislocation needed to fracture the material would require higher amount of stress. Hence, improved strength is achieved [74], the result of this research buttress the findings of [77] who in their research work reported enhancement in splitting tensile strength with fiber addition. However, an increment in splitting tensile strength of the samples was noted as the number of curing days progressed from 7-14 days. Control sample had an improvement of 0.06 MPa while an increase of 0.042 MPa was recorded for 2.5 wt. % TJF/ESP composite with increase in the number days of curing. This asserts the work of [78] who emphasized improvement in splitting tensile strength as the number of curing days increased.

Untreated samples show lower fracture resistance against splitting tensile load than their treated counterparts. This can occur owing to the hydrophilic nature of natural fibers which makes them absorb moisture readily from the atmosphere, leading to swelling of the fibers and the formation of microvoids and microcrack at the fiber-matrix interface. Hence, lower amount of stress is needed to fracture the composite [79]. Among the untreated composite, 2.5 wt. % UJF/ESP had the best performance at 14 days of curing with a value of 0.110 MPa.

#### 3.4.5. Impact strength

Impact strength was evaluated to reveal the resistance of the composite samples produced to fracture when a sudden load is applied. Resistance of the samples produced to impact load was as represented in Figure 15, where it was observed that the control sample had impact strength of 2.1 and 2.4  $J/m^2$  at 7 and 14 days of curing respectively. Addition of 0.5 wt. % JF/ESP led to increase in impact strength, where 2.19 and 2.25 J/m<sup>2</sup> were obtained for 0.5 wt. % UJF/ESP while 2.31 and 2.65 J/m<sup>2</sup> were recorded for 0.5 wt. % TJF/ESP at 7 and 14 curing days respectively. The optimum number resistance to impact load was obtained for 1.5 wt. % TJF/ESP composite sample cured for 14 days. The increase in impact strength observed may occur owing to the presence of fiber, which is responsible for reducing the rate at which inherent crack present in the material propagate. This upholds the findings of Ekunobi et al [80], who reported an increased impact strength of ceiling board produced from waste paper with fiber addition. Impact strength was noted to increase as curing progressed from 7-14 days, this is in agreement with the findings of [81], revealing the effect of curing on impact strength and concluded that impact strength of cementitious materials improve with increase in curing days.



Figure 13. Variation of tensile strength with untreated and treated JF/ESP hybrid cement-paper composites.



Figure 14. Variation of splitting tensile strength with untreated and treated JF/ESP hybrid cement-paper composites.

It is noteworthy to mention that samples produced with treated fibers performed better than the untreated ones, when an impact load was applied on the samples. This is attributable to increase in surface roughness and wettability of jute fiber which improved the interfacial adhesion as a result chemical treatment given to the fibers [82]. This interfacial adhesion was confirmed in the SEM micrograph (Figures 19, 20, and 21). Among the untreated samples, 1.5 wt. % UJF/ESP had the highest impact strength of 2.43 and 2.57 J/m<sup>2</sup> at 7 and 14 days of curing respectively.

#### 3.4.6. Bursting strength

Bursting strength was evaluated to determine the degree of resistance of the produced composite samples to rupture. From Figure 16, it was observed that the control sample possess a bursting strength of 0.621 and 0.684 MPa after 7 and 14 days of curing respectively. Addition of 0.5 wt. % JF/ESP led to a decrease in bursting strength to give a value of 0.574 and 0.630 MPa for UJF/ESP while for TJF/ESP 0.6 and 0.672 MPa were obtained after 7 and 14 days of curing respectively. This indicates that a 7.56 and 7.35 % reduction was recorded for 0.5 wt. % UJF/ESP while



Figure 15. Variation of impact strength with untreated and treated JF/ESP hybrid cement-paper composites.





3.38 % and 1.75 % reduction was observed for TJF/ESP after 7 and 14 curing days respectively. The reduction observed can be related to the presence of ESP and the low volume of fiber present in the hybrid composite at that weight fraction, bursting strength has been reported to improve with fiber addition while particulate reinforcement reduce the resistance of the samples to rupture when high pressure is applied on the material [83].

2.5 wt. % TJF/ESP had the highest bursting strength, showing a value of 0.780 MPa after 14 days of curing which is 14.03 % enhancement when compared with the control cured for the same of days. This feat was attained based on increased volume of fiber at that weight fraction as revealed by the microstructural morphology in Figure 21(a) and (b). The trend observed was an initial decrease in the resistance of the hybrid composited when a bursting pressure was applied. At 0.5 wt. % JF/ESP, followed by an increase from 1 - 2.5 wt. % JF/ESP addition in both treated and untreated samples.

As the number of curing days increase, bursting strength was found to also increase in all samples, because of the formation of more hydration product at higher number of curing days. TJF/ESP samples are characterized with better bursting strength before ultimate rupture due to adequate force transfer and enhanced energy absorption capacity than the UJF/ESP samples [84]. The incorporation of these agro-wastes into matrix material as a substitute for synthetic reinforcements can be utilized for the development of eco-friendly material to improve their properties and service life.

#### 3.4.7. Hardness

The ability of a material to resist indentation is usually referred to as the hardness of the material. This property is used in the determination of

deformation occurring in a material when an external load is applied [85]. Figure 17 reveals the hardness of various weight fractions of samples developed, where it was observed that all hybrid composite samples produced have improved hardness than the control sample cured for the same number of days. This suggests that the rigidity of the samples have been improved, therefore, the ability of the samples to resist indentation is enhanced. The trend observed was increase in hardness with increasing fiber addition and the peak value for hardness was obtained at 2 wt. % JF/ESP, followed by a decrease at 0.25 wt. % JF/ESP. 0.5 wt. % JF/ESP had the lowest value of hardness for both TJF/ESP and UJF/ESF hybrid composite. This corroborates with the study of Ekpunobi et al [80] whose research highlight the increase in fiber improve the hardness of ceiling boards produced from waste paper. However, the values obtained at that weight fraction of reinforcements were found to be better when matched with the control sample. Furthermore, curing was noticed to improve hardness in all samples produced; the reason for this may be linked to the continuation of hydration process up to 14 days of curing.

# 3.5. Wear loss index

Variation in the wear behavior of the composite samples developed was as shown in Figure 18. The control sample had wear index that is higher than the ones obtained for the reinforced samples cured for the same number of days. This shows that the incorporation of JF/ESP as partial replacement for the matrix material enhance the resistance of the hybrid composites developed to wear loss, this can be attributed to the addition of the JF/ESP as the second phase. From Table 2, eggshell shows the presence of calcium and iron, these elements can combine with oxygen to form CaCO<sub>3</sub> and Fe<sub>2</sub>O<sub>3</sub> which are regarded as good



Figure 17. Variation of hardness with untreated and treated JF/ESP hybrid cement-paper composites.



Figure 18. Variation of wear index with untreated and treated JF/ESP hybrid cement-paper composites.



Figure 19. SEM images of (a) 0 wt. % TJF/ESP, (b) 0.5 wt. % TJF/ESP hybrid cement-paper composite.

reinforcing materials [86]. At 2 wt. % TJF/ESP addition, the wear resistance was at its highest and the wear loss recorded was 29.29 % lower than the control after 14 days of curing. 2.5 wt. % TJF/ESP hybrid composite cured for 14 days possess a wear index of 8.5 which shows a 14.14 % reduction when compared to the control sample cured for the same number of days.

Samples cured for 14 days were found to have wear resistance greater than those cured for 7 days. Among the sample cured for 7 days, 2 wt. % TJF/ESP hybrid composite exhibited a wear loss index of 7.5 which is lower than the other samples cured for the same number of days. Generally, wear loss was observed to decrease with increasing JF/ESP addition up to 2 wt. % followed by a decrease. Treated samples show better wear resistance than the untreated samples; this can be linked to improved specific interactions owing to surface modification of the fiber [87]. 0.5 wt. % UJF/ESP had the lowest wear resistance among UJF/ESP hybrid composite. However, when matched with the control sample, its wear property is superior and can be selected above the control sample in applications where better wear resistance is needed.

#### 3.6. Morphological evaluation of composites developed

Figures 19, 20, and 21 show the SEM images of the splitting tensile sample fractured surface of the control and TJF/ESP hybrid composites. Figure 19(a) highlights the fibrous nature of the paper employed and the presence of porosity in the cement paper matrix. Figure 19(b) shows the addition of 0.5 wt. % TJF/ESP, the SEM image captured the presence of jute fiber in minimum proportion however some of the fibers agglomerated. This occurrence may be responsible for the low mechanical properties observed at that weight fraction. From Figure 20(a), it was observed that JF and ESP were evenly dispersed when compared with



Figure 20. SEM images of (a) 1 wt. % TJF/ESP, (b) 1.5 wt. % TJF/ESP hybrid cement-paper composite.



Figure 21. SEM images of (a) 2 wt. % TJF/ESP, (b) 2.5 wt. % TJF/ESP hybrid cement-paper composite.

Figure 19(b) while the surface morphology of 1.5 wt % TJF/ESP hybrid composite as shown in Figure 20(b) was characterized by fiber pull out. This composite sample shows even distributions of JF which highlight the reason for the improved resistance against crack propagation observed. Figure 21(a) and (b) shows the SEM micrograph of 2 wt. % TJF/ESP and 2.5 wt. % TJF/ESP respectively. These samples were characterized with homogenous dispersion of fiber, which begat effective stress transfer. However, good interfacial adhesion demonstrated between matrix and reinforcements at these weight fractions underline the improvement mechanical properties observed.

#### 4. Conclusions

The physical, thermal, mechanical and wear behaviour of JF/ESP reinforced hybrid composites were studied, from the results obtained, the following conclusions were drawn.

- chemical treatment was observed to enhance interfacial adhesion between the matrix and the reinforcement. Therefore, the physical, thermal, mechanical and wear properties of the developed hybrid composites were improved when compared to the untreated ones.
- ii. as the number of curing days increases, the properties of the developed composite improved except in the case of tensile strength where a reduction was observed.
- iii. 0.5 wt.% UJF/ESP had the least performance of all the composites developed. However, 2.5 wt. % TJF/ESP sample had the optimum performance out of all the hybrid composites developed.
- v. JF and ESP are suitable reinforcing materials in cement/paper matrix and can therefore be use in ceiling board application.
- vi further research should be carried out on cement/paper matrix reinforced with other particulates and fibers at higher fiber fractions, for possible improvement in flexural strength of the composite with potential applications in ceiling board, and wall partitioning.

# Declarations

### Author contribution statement

Adediran, A.A & Adesina, O.S: Analyzed and interpreted the data; Wrote the paper.

Balogun O. A & Akinwande A. A: Conceived and designed the experiments; Performed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data; Wrote the paper.

Olasoju O. S: Contributed reagents, materials, analysis tools or data; Wrote the paper.

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#### *Competing interest statement*

The authors declare no conflict of interest.

#### Additional information

No additional information is available for this paper.

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