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Effects of Alkali Treatment on the Physiochemical and Mechanical Performances of *Grewia mollis* Fibre Reinforced Waste FARO Water Bottle Composites

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Abstract: Reinforcing polymer matrices with natural fibres has currently gained momentum among many researchers around the world. This study investigated the effects of alkali treatment on the physicochemical and mechanical performances of *Grewia mollis* fibre-reinforced waste FARO water bottle composites. The hand layup technique was employed in formulating the composites and their physiochemical and mechanical performances were studied according to ASTM standards. The physiochemical analysis showed that moisture absorption behavior decrease while density properties increase as the concentration of NaOH increases. According to the analysis, the untreated composites absorb more moisture owing to their hydrophilicity nature. The analysis proves that alkali treatment decrease moisture content and increase density properties of the green composites. The mechanical analysis confirmed that the treated composites recorded high tensile and compressive values of (5% = 35.25, 10% = 40.85, 15%= 44.05, 20% = 38.25, 25% = 34.06 and 5% = 47.20, 10% = 63.20, 15%= 67.50, 20% = 53.40, 25% = 51.00) MPa compared to the untreated ones which recorded low tensile and compressive values of (32.85 and 43.02) MPa. The mechanical performances were significantly improved as the concentrations of NaOH raises up to a threshold point of 15% and latter declined from threshold points of 20-25%. According to the analysis, composites treated with 10–15% exhibited better improvements than 20–25% and the optimum improvements were found to be 15%. The analyses of alkali treatment were correlated with the physiochemical and the mechanical performances of the treated and untreated composites. The findings confirmed that the alkali treated composites exhibited better physiochemical and mechanical performances as likened to the untreated composites based on the studied characteristics and were found to be recommendable for structural applications.

Keywords: *Alkali Treatment, Characterization, Composite, Fibre, Grewia mollis, Reinforcement, Waste FARO Water Bottles.*

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I. INTRODUCTION

The issues of global warming and environmental threats have recently been attracting a significant attention globally [1]. These issues have prompted researchers around the globe to search for suitable remedies and seeking for alternative materials and green technology in many industrial segments. The second are relying on the cost of production of the composites and their functional applications such as transportation, building and construction industries and furniture packaging [2]. The green composites are prepared using natural fibres obtained from plants, which comprise of different chemical components such as cellulose, hemicellulose, wax, and ash [3, 4].

Natural fibre composites (NFCs) are increasingly becoming center of attention from both academia and industries for their diverse applications in various industrial sectors. These materials possess unique behaviors such as biodegradability, eco-friendliness, eco-efficiency nature, high modulus, improved specific strength, low rate of carbon emission, and ease of fabrication [5, 6]. Natural fillers that used to reinforce polymer matrices provide a new class of materials that exhibit notable mechanical properties with diverse applications [3, 7]. The plant natural fibres are sourced from various parts of plants, such as the bark, stems, leaves, and roots. The major commercially employed cellulosic fibres comprise of seed fibres, bast fibres, and leaf fibres. Root fibres (*grewia mollis*), and stem fibres such as banana, flax, *grewia ferruginea*, *grewia mollis*, hemp, jute, kenaf, ramie, roselle, saccharum, and sun hemp are also applied [8-11].

Natural fibres (NFs) obtained from *grewia* species are versatile cellulosic fibres and are broadly employed in the domain of diverse industrial applications. Birlie and Mamay [11] extracted and characterized cellulosic fibre from plant stem of *grewia ferruginea*. The FTIR analysis confirmed the presence of biopolymers in the fibre according to their findings. The thermo-gravimetric analysis revealed that the fibre demonstrated thermal stability and has the potential to be utilized as sustainable reinforcement material in polymeric composites according to their report. *Grewia mollis* fibres can be derived from the bark, leaves, stems and roots of *grewia mollis* plant. They consist of cellulosic fibres with hemicelluloses, lignin and pectin as a bonding material. The mechanical performance of the green composites depends on numerous parameters including the volume fraction of fibres, length, shape, arrangement, fibre polarity and the interfacial bonding with the polymer matrices [12]. Despite a number of advantages of natural fibres as reinforcements, there are some limitations on using natural fibres in composite formulation including lack of adequate adhesion between fibres and the polymer matrix, poor thermal stability, and hydrophilic nature as they contain strongly polarized hydroxyl group [1, 13].

Polymer matrices, such as polypropylene (PP) and polyvinyl chloride (PVC) are axiomatically incompatible with natural fibres, as these matrices have hydrophobic nature. This limitation leads to weak interfacial bonding between the fibres and the polymer matrices which, as a result, lead to deboning and failure in the end use production [14]. The interface between polymer matrix and the reinforcing fibres plays a critical role in determining the physicochemical and the mechanical performances of the green composite. Basically, the characteristics of natural fibre composites are unambiguously connected with the nature of the natural fibres and their compatibility with polymer matrices. Therefore, it is pertinent to reduce the moisture content and hydrophilic nature of natural fibres by applying a proper surface treatment to enhance fibre compatibility with various polymer matrices [15].

Several studies revealed that surface treatment to natural fibres enhance the mechanical properties of the green composites. There are a number of chemical treatments that can be used to enhance the compatibility between fibre and matrix and increase the functionality of natural fibres. Alkali treatment stand the most useful chemical treatment for natural fibres to eliminate wax and oil covering some parts of these fibres and to increase the roughness of fibre surfaces that lead to better interlocking of fibres with the polymer matrix [14].

GU [16] used NaOH solution, with concentrations varied from 2.0 to 10% separately on freshly brown laminated coir fibres to study their tensile properties. He reported an enhancement in coir fibre adhesion with polypropylene after the chemical treatment. He also reported a decline in tensile strength values as the concentration of NaOH increased. One of his main conclusions was that, as the concentration of NaOH is higher than 8%, the adhesion of the fibre with the polymer matrix is enhanced. Similarly, Karthikeyan and Balamurugan [17] used the same solution with various concentrations (2.0, 4.0, 6.0, 8.0 and 10) % at ambient conditions for 10 days to treat freshly retted long combed coir fibres. The treated fibres were reinforced epoxy resin to study the mechanical properties. The treatment with 6% NaOH exhibited better result in impact strength, while higher alkali concentrations lowered fibre strength and subsequently the impact strength.

The issue encountered by manufacturers is the choice of natural fibre which is the weightiest procedure for polymer composites with optimum physicochemical, mechanical, and other functional characteristics [11]. Recent discoveries on the composites produced from waste plastic matrix and natural fibres such as groundnut husk, wood dust particles, tea leaves waste and g*rewia mollis* fibres were mentioned in the literature. Previous studies confirmed that *Grewia mollis* fibre (GMF) has been utilized in composites, significantly due to its availability, cost efficiency, good mechanical performance, and other characteristics such as good light weight, modulus, specific strengths, and finally economic viability [11]. But no report was found on the effects of alkali treatment on the physiochemical and mechanical performances of this fibre in composite fabrication. This study aimed at investigating the effects of alkalil treatment on the physiochemical and mechanical performances of *Grewia mollis* fibre (GMF) reinforced waste FARO water bottle composites (WFWBCs).

The notable factors that affect the physiochemical and the mechanical performances of the composites are the surface treatment for the reinforcing materials. The physiochemical performance represented by moisture absorption and density properties and the mechanical performance represented by tensile and compressive strength for the untreated and the treated fibres and the composites were investigated and reported with a peculiar focus on the failure mechanism. The major concern of this study is to develop composite panels by choosing abundant waste Faro water bottles (WFWBs) with the hope to reduce waste disposal in our environment and choosing a cost effective approach of manufacturing and investigating the effects of alkali treatment on the physiochemical and the mechanical performances of the produced composites as well as to justify their optimum alkali treatments, which if found suitable would be recommended for structural applications. The aforesaid investigation will potentially aid in failure prediction approach for systems designed by utilizing these abundant WFWBs.

II. MATERIALS AND METHODS

- *Sample Collections*

The waste FARO water bottles (plate-1) were assembled from Jumeta town, Yola South Local Government Area of Adamawa State, Nigeria. *Grewia mollis* is a widespread species of flowering plant (plate-2) in the family Malvaceae, native to tropical Africa, Yemen, Oman, Senegal and Zimbabwe [18]. It is also widely distributed in northern Nigeria especially north-eastern part of the country [23]. The GMF was gathered from Bagale Mountain (plate-3) sited at Girie Local Government Area of Adamawa State, Nigeria.

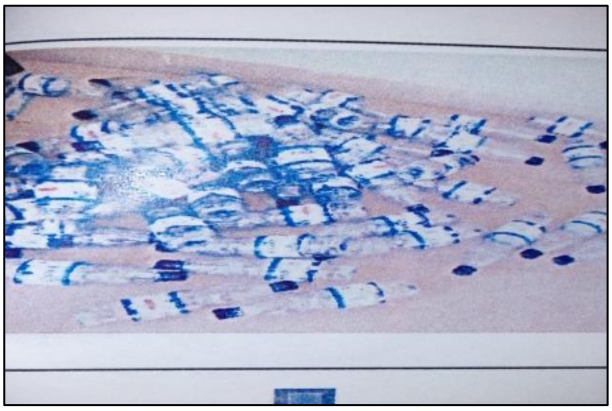

Plate 1 The Waste FARO Water Bottles.
Source: Self

Plate 2 The *Grewia mollis* Plant.
Source: Self

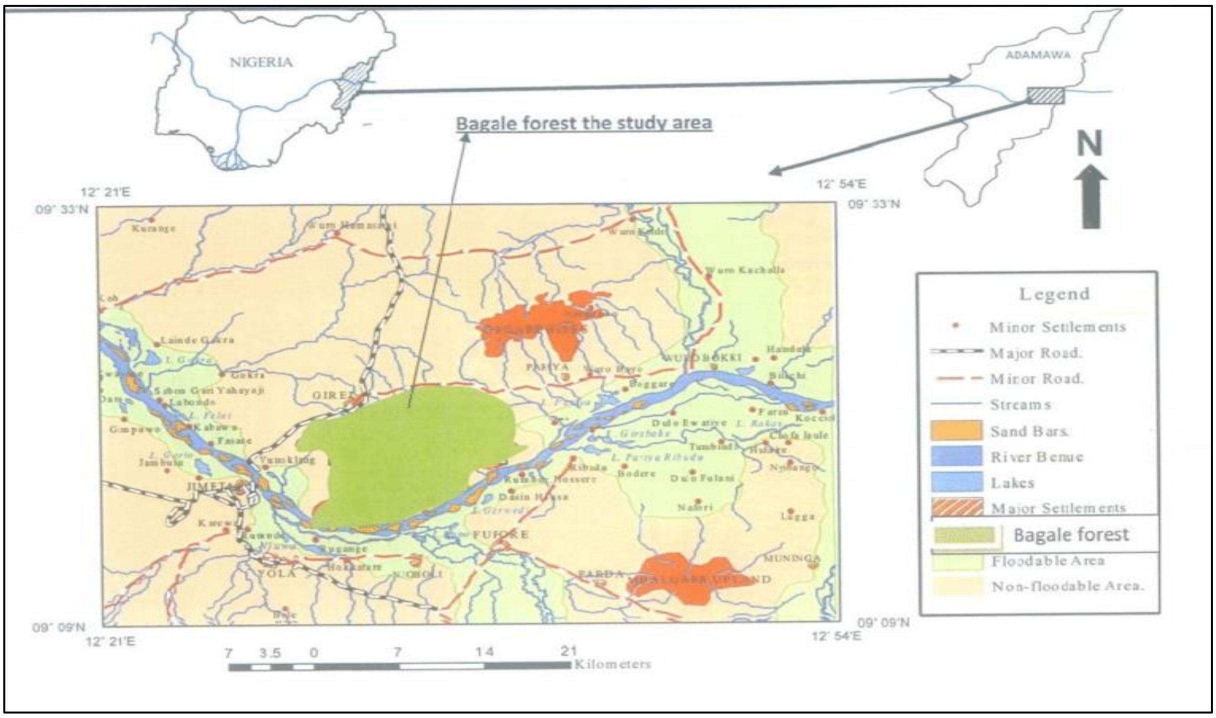

Plate 3 Map of Girei Local Government Area Showing the Study Area.
Source: [19]

➢ *Sample Preparation*

The WFWBs were crushed into minute particles (plate-4a) to aid the dissolution in solvent and the GMF was chemically extracted and processed by retting, scouring, bleaching, and mercerizing procedures (plate-4b) according to the standard methods adopted by [11, 20, 21].

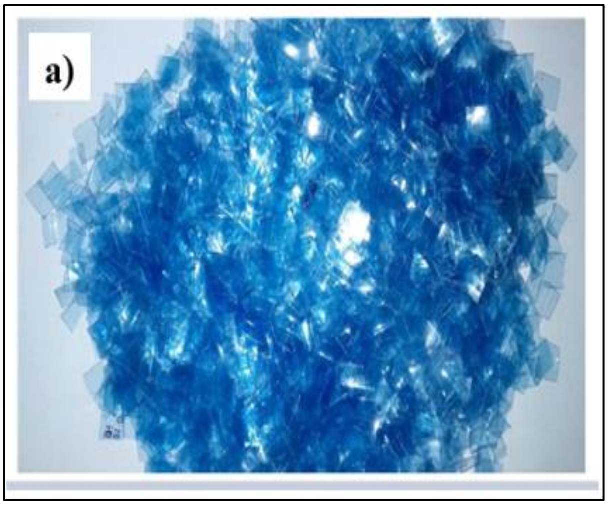

Plate 4a Pulverized Waste FARO Water Bottle.

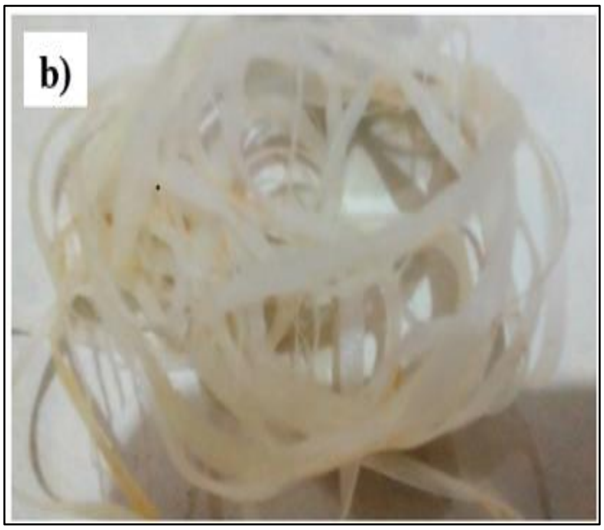

Plate 4b Chemically Processed *Grewia mollis* Fibre.

➢ *Preparation of Waste FARO Water Bottle Solution*

The solvent for dissolving WFWBs was prepared by melting phenol in an oil bath at 45^0C and mixing it with liquid 1, 1, 2, 2-tetrachloroethane in the ratio 60/40 w/w according to the standard procedure described by [11, 20, 22]. The pulverized WFWBs (plate-4a) were poured into a beaker containing phenol-1, 1, 2, 2-tetrachloroethane solution and heated to 100^0C and the mixture was then excited until the entire WFWBs melted, forming a viscous liquid (plate-5) according to the standard procedures used by [11, 20, 21].

Plate 5 Waste FARO Water Bottle Solution.

➢ *Fabrication of Grewia mollis Fibre Reinforced Waste FARO Water Bottle Composites*

Hand layup technique adopted by [11, 20, 21] was used in fabricating the composite. De-bonding material was introduced on the inner area of an aluminum mold accompanied by a pigmented gel coat to grant high quality surface finish. 20g of the crushed fibre was manually settled down in the mold randomly and the melted WFWB solution was poured and casted into the mold. The fabricated composites (plate-6) were allowed to dry to a structural panel for 1 hour at ambient temperature and were then removed from the mold and labeled and finally kept in an open space in the laboratory at room temperature for further use.

Plate 6 Fabricated *Grewia mollis* Fibre Reinforced Waste FARO Water Bottle Composites.

III. CHARACTERIZATION OF *GREWIA MOLLIS* FIBRE REINFORCED WASTE FARO WATER BOTTLE COMPOSITES

The physiochemical and the mechanical analyses for the treated and untreated GMF reinforced WFWBCs were investigated according to American Society for Testing and Material (ASTM) standards.

➢ *Moisture Absorption Analysis (ASTM D355)*

The analysis was conducted using a moisture analyzer device. The composites were partitioned into 20x10x3 (length x width x thickness) mm^3 parts, and were allow to saturate for up to 168hours at 25^0C in a coherent water bath. They were weighed both before and after been submerged in moisture and the proportion of absorbed moisture were then calculated and recorded in (Table I) using the formula provided by [11, 20, 21] as shown below:

$$\text{Absorption (\%)} = \frac{\text{Final weight} - \text{initial weight x100}}{\text{Final weight}} \quad (1)$$

Table 1 Moisture Absorption Behavior for the Treated and Untreated *Grewia mollis* Fibre Reinforced Waste FARO Water Bottle Composites.

Time (h)	**Untreated**	**5%NaOH**	**10%NaOH**	**15%NaOH**	**20%NaOH**	**25%NaOH**
24	28.20	21.50	21.00	20.00	19.05	18.50
48	29.10	23.00	22.72	21.45	20.02	18.90
72	29.80	23.91	23.75	22.49	20.89	18.94
96	30.30	24.26	24.07	23.00	20.99	18.98
120	30.40	24.26	24.07	23.00	21.06	18.99
144	30.40	24.26	24.07	23.00	21.06	18.99
168	30.40	24.26	24.07	23.00	21.06	18.99

➢ *Density Properties Analysis (ASTM D792)*

The analysis was carried out by weighing the composites using an electric balance and subsequently measuring their dimensions using Vernier caliper from which their volumes were then calculated and recorded in (Table II) using the equation employed by [23] as shown below;

$$Density\ (g\ cm3) = M\ (g)/V\ (cm3) \qquad (2)$$

Where; M= mass of the composite (g), V= volume of the composite (cm3).

Table 2 The Effect of Different Concentrations of NaOH on Density Properties of *Grewia mollis* Fibre Reinforced Waste FARO Water Bottle Composites.

Concentration of NaOH	**Density (gcm^{-3})**
5%	3.820
10%	3.930
15%	3.991
20%	4.0105
25%	4.0235

➢ *Tensile Strength Analysis (ASTM D638-99)*

The analysis was done using a Universal Hydraulic Digital Material Testing Machine (model: H50KS-0404, Hounsfield Series S, UK) with a cross-head speed of 10mm/min and a span distance of 50mm. The composites were sliced with dimensions of thickness (t) of 3mm and width (b) of 15mm and mounted on the machine and the tensile force was noted and recorded in (Table III) using the formula described by [11, 20, 21] as shown below:

$$\partial\square = \frac{p}{A} \qquad (3)$$

Where: $\partial\square$ = Tensile stress of the composite sample (MPa), p = Applied load (N), A = Width * thickness of composite sample (mm).

Table 3 The Effect of Tensile Strength on the Treated and Untreated *Grewia mollis* Fibre Reinforced Waste FARO Water Bottle Composites.

Fibre	**Tensile strength (MPa)**
Untreated	32.85
5%NaOH	35.25
10%NaOH	40.85
15%NaOH	44.05
20%NaOH	38.25
25%NaOH	34.06

➢ *Compressive Strength Analysis (ASTM D695-97)*

The **a**nalysis was performed using a Universal Hydraulic Digital Material Testing Machine (model: HUNG TA INSTRUMENT CO. LTD., Taiwan). The composites were carved into cuboid shapes with dimensions of thickness (t) of 3mm and width (b) of 15mm and placed on the machine and compressed. The compressive load was applied on the composites until failure occurs and the load was studied and recorded in (Table IV) using the formula adopted by [23] as depicted below:

$$\partial_c = \frac{p}{bxt} \qquad (4)$$

Where: ∂_c = compressive stress of the composite sample (MPa), p = load (N), b = width of the composite sample (mm), t = thickness of the composite sample (mm).

Table 4 The Effect of Compressive Strength on the Treated and Untreated *Grewia mollis* Fibre Reinforced Waste FARO Water Bottle Composites.

Fibre	**Compressive strength (MPa)**
Untreated	43.02
5%NaOH	47.20
10%NaOH	63.20
15%NaOH	67.50
20%NaOH	53.40
25%NaOH	51.00

IV. RESULTS AND DISCUSSION

➢ *Effect of Moisture Absorption on Grewia mollis Fibre Reinforced Waste FARO Water Bottle Composites*

The characteristics of fibre that was placed to treatment by various concentrations of NaOH were acquired together with that of the untreated fibre and were showed using descriptive statistics of percentage (%) scatter plot (Fig 1).

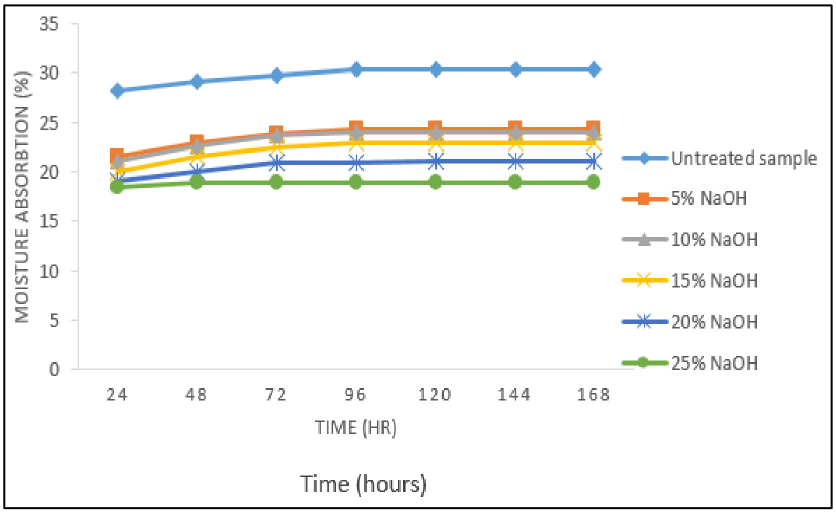

Fig 1 Effect of Moisture Absorption on *Grewia mollis* Fibre Reinforced Waste FARO Water Bottle Composites Against Time.

The absorption rate was very high at first 24 hours and constantly progressed up to 96 hours according to the analysis. This could be as a result of low cross-linking density between the fibre and the matrix [24]. A conspicuous equilibrium was reached at 96–168hours of soaking time as there was no meaningful change in percentage absorption for both the untreated and the treated composites within these periods. The absorption rate for the untreated composites was far higher than that of the treated ones as depicted in Fig 1. These remarks concur with the findings of [23, 25].

The higher absorption rate demonstrated by the untreated composites could be owing to the present of strongly polarized hydroxyl groups in natural fibre causing smooth interaction between the fibre and the moisture molecules and the low compatibility of the hydrophobic polymer matrix. Consequently, when subjected into moisture, it absorbed some volumes of moisture for first hours than the treated fibre, thus, resulting to a poor moisture resistance [26-29].

The lower absorption rate displayed by the treated composites could be due to greater extraction of lignin content of the fibre as the concentration of NaOH elevates gradually with time, which at higher concentration may damage the ultimate cell walls of the fibre and subsequently reducing the absorption behaviors of the fibre and strengthened the interfacial bonding between the fibre and the polymer matrix, resulting to a significant advancements in the physiochemical and the mechanical performances of the composites with higher concentration of NaOH [11, 20, 21, 23, 26, 30]. This account for a drastically slow increase in absorption rate with time for composites treated with 20 and 25%NaOH. Thus, the composite treated with 5% has the highest moisture content and the composite treated with 25% has the least. Consequently, as the concentrations of NaOH ascend moisture content falls. This displays that NaOH content and moisture content are indirectly proportional. Reports indicate that, mercerization tends to reduce moisture content [26].

- ➢ *Effect of Density Properties on Grewia mollis Fibre Reinforced Waste FARO Water Bottle Composites*

The effect of different concentrations of NaOH on density properties of GMF reinforced WFWBCs were presented using bar chart (Fig 2).

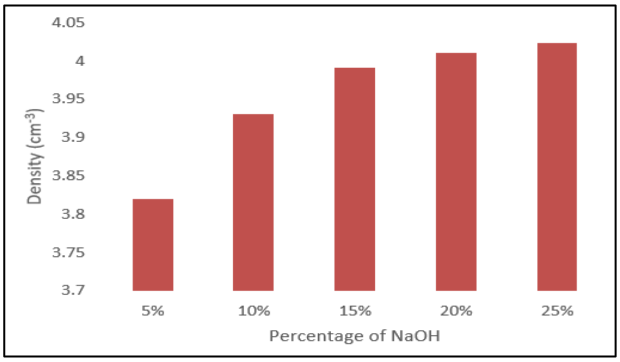

Fig 2 Comparison of Density Properties of *Grewia mollis* Fibre Reinforced Waste FARO Water Bottle Composites Prepared from Different Concentrations of NaOH.

The result revealed that the density properties of the composites increase as the concentration of NaOH increases from 5–25% and that the composite is denser at 20% and 25%NaOH which show highest values of 4.0105gcm^{-3} and 4.0235gcm^{-3} respectively than the other composites with 5-15%NaOH as presented in (Table 2). The composite treated with 25%NaOH has the highest density and that the composite treated with 5%NaOH, which is the lowest concentration, has the least as depicted in Fig II.

These observations have also been reported by [23]. This could mean that more solution of NaOH with much high concentration lead to disentangling of the fibre within the polymer matrix. As observed by Mylsamy and Rajendran [31] that much weight gain by plant fibre composites results in more moisture and water molecules becoming interlocked in the composites. In most cases, the density properties of natural fibres may probably differ according to the method of fibre extraction, age of the plant, moisture present in the fibre, surface treatment, soil condition in which the plant was grown, etc. [32].

Since density is affected by amount of fibre bonding, amount of void volume vis-à-vis high porosity has to do with compatibility of fibres with the polymer matrix during fabrication of the composites [33]. Fibre with low density suggest high porosity as in the case of GMF, the compatibility of the fibre with the polymer matrix was very weak, resulting to excess uptake of NaOH solution giving rise to high density as depicted in Fig-2.

Based on the fact that more solution of NaOH with high concentration causes high density while small solution of NaOH with low concentration causes low density. This shows that the composite treated with 5%NaOH will be preferred better than the composite treated with 25%NaOH for low weight applications. The implication of this is that the low weight composites could be less insensitive to applications where high weight variations are required, hence, have narrow range of applications where weight is considered as a parameter.

➢ *Effect of Tensile Strength (TS) on Grewia mollis Fibre Reinforced Waste FARO Water Bottle Composites*

The effect of TS on the treated GMF reinforced WFWBCs as the concentration of NaOH varies along with that of the untreated one were presented using bar chart (Fig 3).

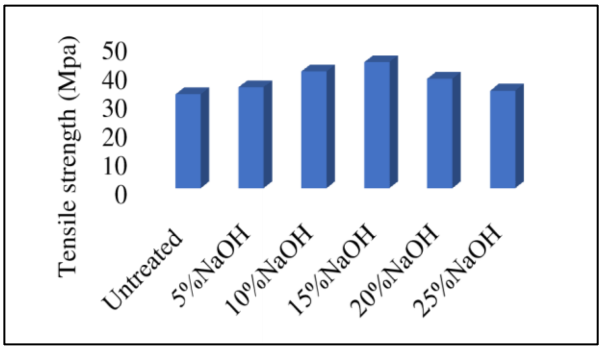

Fig 3 Tensile Strength Variation of *Grewia mollis* Fibre Reinforced Waste FARO Water Bottle Composites as the Concentration of NaOH Varies.

The result confirmed that TS elevates as the concentration of NaOH rises up to a threshold point of 15% and subsequently decline from 20–25% as noticed in this work and reported by [11, 20, 21]. This entails that composites treated with 10–15% present superior results than those treated with 20–25% as depicted in Fig III. This is because at minimum (%), the GMF is extremely condensed by the polymer matrix and there is little or no fibre contacting one another and that greater (%), above 15% will destroy its cell wall bringing about low bonding between the fibre and the polymer matrix leading to subsequent reduction in the mechanical performance of the composite [26].

There exist minor deviation in the manner as 15% becomes as the optimal with tensile value of 44.05MPa compared to untreated GMF reinforced WFWBs matrix with tensile value of 32.85MPa. Accordingly, on the frequency of %NaOH, threshold point of 15% had the apex set of mechanical performance. This finding coincides with the investigations of [11, 20, 21, 29]. This might be due to the fact that natural fibres are typically characterized by high moisture uptake and this scenario declines the adhesive attributes of fibre surface and weakens the interfacial bonding between the fibre and the polymer matrix consequently weakening the mechanical performance of the composites [11, 26].

➢ *Effect of Compressive Strength (CS) on Grewia Mollis Fibre Reinforced Waste FARO Water Bottle Composites*

The effect of CS on the treated GMF reinforced WFWBCs as the concentration of NaOH varies along with that of the untreated one were presented using bar chart (Fig 4).

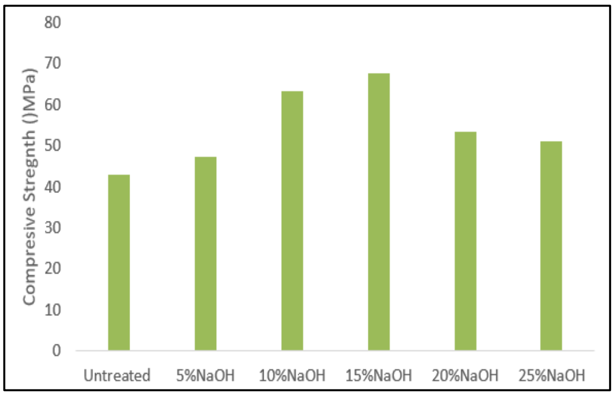

Fig 4 Compressive Strength Variation of *Grewia mollis* Fibre Reinforced Waste FARO Water Bottle Composites as the Concentration of NaOH Varies.

The analysis inferred that untreated composite gave poor CS with Compressive value of 43.02MPa compared to composites treated with 5–25%NaOH. This indicates that high strength is noticed in treated composites which elevate as the concentration of NaOH elevates up to a threshold point of 15% and gradually decline from 20–25% as recorded in this work and reported by [23].

This signifies that composites treated with 10–15% yielded apex results than those treated with 20–25% as depicted in Fig IV. This demonstrates that bonding between the fibre and the polymer matrix was very low at higher percentage (%) above 15%NaOH resulting to subsequence decline in the CS of the composites treated with 20-25%NaOH as established in the current study and same was presented in the work of [23]. There is slight disparity in the trend as 15% stands as the optimal with compressive value of 67.50MPa compared to the rest treated GMF reinforced WFWBs matrix with compressive values of (5%=47.20, 10%=63.20. 20%=53.40 and 25%=51.00) MPa, hence had the optimum set of mechanical performance. The outcome is in agreement with the report of [23, 31, 34]. This could be owing to the facts that alkali treatments remove the lignin and hemicellulose content of the fibre making it well bonded with the polymer matrix and improvement seen in their interfacial bonding which gives necessary strength [23, 35, 36].

V. CONCLUSION

The result confirmed that NaOH and H_2O_2 are suitable and reliable agents for retting, scouring, bleaching and mercerizing cellulose materials. Their suitability and reliability were ratified by the improved physiochemical and mechanical performances demonstrated by the treated GMF samples as likened to the untreated ones.

The composites were prosperously fabricated from *Grewia mollis* fibre and waste FARO water bottles. The result disclosed that chemical treatments significantly lower moisture content and elevates density properties of the fabricated composites there beyond improved their mechanical performance and the optimum improvements for both tensile and compressive strength were established at 15%.

This finding unequivocally showed that the fabricated *Grewia mollis* fibre reinforced waste FARO water bottle composites treated with 15%NaOH exhibited better resistance to moisture and marginally high density as well as good mechanical performance. In operating towards an ecologically friendly society, waste FARO water bottles can be transformed as matrix for *Grewia mollis* fibre composite fabrication which motivated the recyclability of waste in our environment. It can also propagate the quantity of natural fibre reinforced polymeric composites in the market and lower their prices.

The alkali treated fibre reinforced polymeric composites exhibited better characteristics than the untreated fibre reinforced polymeric composites based on the studied physicochemical and mechanical performances. Hence, could be sensitive to a wide range of applications, particularly where high tensile and compressive strength variations are crucial. Consequently, this study may infer a polymer composite derived from *Grewia mollis* fibre and waste FARO water bottles with potential features for structural application.

- *Conflict of Interest*

The authors declare no conflict of interest.

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