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DETERMINATION OF PHYTOCHEMICALS AND ELEMENTAL COMPOSITION OF KENAF NON-WOVEN FIBER USING PROXIMATE AND X-RAY FLUORESCENCE ANALYSIS

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ABSTRACT

The development of alternative inexpensive fillers to the viable commercially viable ones used in the manufacture of polymer composites has increased. Thus, information about kenaf fiber mat are needed and are very important. This study examined the proximate and elemental compositions of kenaf fiber and its possibility to serve as alternative filler. The kenaf fiber was prepared by drying the back at temperatures between 25 - 30 °C**.** The phyto-chemicals and elemental compositions of the kenaf fiber were valuated using standard methods and X-ray fluorescence. The results revealed that the percentage of the phytochemicals such as moisture content, volatile matter, fixed carbon, Nitrogen, crude protein, crude fat, crude fiber, carbohydrate, lignin, hemicellulose, cellulose and holocellulose were 6.56,12.13, 82.30, 1.83, 11.44, 9.44, 23.27, 43.72, 17.49, 14.34, 62. 60 and 76.94 % respectively. The inorganic minerals obtained were below 0.1 mg/g, except oxygen, phosphorus, sulphur, chlorine, potassium, calcium, magnesium, titanium and iron are 4.512, 5.012, 6.581, 12.419, 49.415, 0.441, 1.056, 0.1108 and 2.234 mg/g respectively. Based on the results, the fiber is safe for use as reinforcement in polymer composites.

Keywords*:* Phytochemicals, X-ray fluorescence, kenaf fiber, reinforcement, elemental composition

INTRODUCTION

Kenaf fibre is made from the bast (outer) and core (inner) fibers of the kenaf plant (*Hibiscus cannabinus L.*). Kenaf fibre is native to Africa and Asia and one of the most widely cultivated natural fibres. It is an herbaceous annual plant that is cultivated commercially in the United States in a variety of weather conditions for a variety of uses, including oil spill absorbent, animal feed and as food in Ghana. Kenaf can grow up to a height of 2.4 to 6 m at an average of 150 days [1]. Leaders in world kenaf production are India and China [2]. Agronomically, kenaf has advantages as regards to their resistance to climatic extremes, pests, and diseases [2]. Kenaf fiber can also be considered as a warm-season annual herbaceous plant that has great potential as a source of fiber, energy, and feedstock. Kenaf seeds have different potentials for various domestic and industrial uses. Kenaf fibre is a by-product of the kenaf industry. In the past, non-viable kenaf fiber components such as seeds and peels were discarded except if they can be utilized for animal feed. Researchers have suggested that kenaf seed oil is suitable for human consumption due to its unique fatty acid composition and antioxidant activity. It can be processed into cosmetic oil, cooking oil, bio-diesel and industrial lubricants. Oil produced by the kenaf plant is used for first-class cooking oil and margarine production [3, 4, 6]. Kenaf fibre is blessing to humanity due to its high carbon dioxide remediation rate and its capability to absorb large amount of carbon dioxide, nitrogen and phosphorus from the soil which were the major causes of greenhouse effect [4]. This made kenaf fiber significant to serve as standpoint of its environmental friendliness. Currently, kenaf fiber is viewed as an alternative material to replace the conventional or synthetic fibers as reinforcement in composites science and technology [2,4].

The low cost, no health risk, low density, high strength and modulus, and ease of the accessibility of kenaf fiber in numerous countries have made it befitting for use in composites manufacturing [2,5]. The seed is regarded as non-edible seed to humans but use for livestock feeds. Despite high nutrient values of kenaf seed, many are yet to tap the potentials and optimize its utilization in several countries of the world. The oxidative reactions in foods are the main cause of its deterioration. They are responsible for the nutritional value losses, as well as aroma, taste and texture degradation.

The kenaf fibre-based composite reinforce has not been explored so far. Thus, there is need for the creation of new alternative method that could be used for the utilization of using the kenaf peels (inner stem) as reinforcing filler in polymer composites for engineering applications for which may be safer, more economical, environmentally friendly preferentially and preferably from using commercially viable fillers sources. The studies on substituent fillers to the conventionally viable ones and other inorganic sources are very important. The high cost and sometimes unavailability of the conventional fillers coupled with the inefficiency of foreign exchange and poor quality of composites for engineering applications have rendered production of polymer composites an expensive venture in Nigeria [7].

In current years, studies have been conducted on the use of other nonconventional fillers especially the nonconventional fillers containing the phytochemicals and inorganic minerals, in order to replace the relatively more expensive conventional fillers [7]. However, no much literature data on the kenaf as reinforcing filler in composite formulation was found. The current study was aim to identify the phytochemicals and the elemental composition of kenaf fiber to serve as reinforcing agent on the mechanical properties of epoxy resin composites.

MATERIALS AND METHODS

Reagents used in this research were obtained in analytical grade and were sulfuric acid, 98% w/w (specific gravity 1.6338 at 20 °C) and calcium carbonate,

The materials used for the proximate analysis of the kenaf fiber analysis include Analytical Digital weighing balance, 0.000 g ± 0.001, Digital Oven, ~350^{o}C, Muffle furnace ~1200 ^{o}C, pH meter, range 1–14, and Digital Spectrophotometer (Multiparameter Photometer with COD HI83399, Hanna Instrument USA). Kenaf fiber was obtained at analytical grade from Malaysia in form of a fiber mat prepared at 20 -30 ^{o}C.

Determination of Ash content [5]

Ash was determined by burning the material in a muffle furnace in a porcelain crucible first at 400 °C for 30 minutes, then at 850 °C for 1 h 45 minutes and then gravimetrically estimated by the following equation.

$$\% \text{ AC} = \frac{\text{Final Weight after Ashing}}{\text{Intial weight before Ashing}} \times 100$$

Determination of Percentage Moisture Content of Samples (PMC) [5]

Moisture content of samples was determined based on mass loss after two hours at 105 °C under N_2 purge. Approximately 0.5 g of air-dried sample was weighed into a ceramic crucible. The samples were placed inside of an oven, which was initially purged with N_2 gas for ≥ 20 min at a flow rate of 3 L min^{-1}, to ensure removal of all oxygen. After the 2 h heating, the furnace was turned off and samples were transferred immediately to a desiccator, left to cool for one hour and then weighed.

$$\%MC = \frac{Wc - Dc}{Dc} \times 100$$

Where, Wc is the Air-dried weight of sample; Dc is the Oven dried weight of sample at103^{0}C MC = Moisture content.

Determination of Volatile Matter [7]

Volatile matter was determined by heating the oven dry samples under N_2 purge at 850 ^{0}C. During heating, the crucibles containing the sample were covered with ceramic lids, placed in a stainless-steel box inside of a muffle furnace. A N_2 purge line and thermocouple were inserted through the top of the furnace and down into the stainless-steel box through a small hole in the box cover. The box was purged with N_2 gas for about 5 min at a flow rate of 5 L min-1. After the initial purge, the N_2 flow rate was decreased to 3 L min-1, the furnace was set to the desired peak separation temperature, and turned on. The temperature inside of the stainless-steel box was measured every 60 s during the heating treatments. Once the temperature inside of the stainless-steel box reached 850 ^{o}C separation temperature, the furnace was switched off and furnace door opened. The N_2 purge inside the stainless-steel box was maintained (3 L min-1) during cool down (2–4 h), after which the crucibles were weighed.

Determination of Percentage Fixed Carbon of the Charcoal Samples (PFC)

The percentage fixed carbon, PFC was calculated by subtracting the sum of percentage volatile matter (PVM) and percentage ash content (PAC) from 100. The carbon content is usually estimated as a "difference", i.e., all the other constituents are deducted from 100 as percentages and the remainder is assumed to be the percentage of fixed carbon [8, 9, 10]. This was determined using; fixed carbon (% FC) = 100 % - (%VM + %AC)

Determination of Percentage Crude Fat

The percentage crude fat was determined according to the American Organization of Analytical Chemist method [6]. The Soxhlet extraction method was used to extract out the fat. The extracted fat was then calculated:

$$\% \text{ Fat} = \frac{\text{Weight loss} \times 100}{Initial\ weight\ of\ sample}$$

Determination of Crude Protein [5]as described by Shuaibu *et al.* [7]

Crude protein was determined by the Kjeldahl procedure. This consists of three techniques of analysis namely: Digestion, Distillation and Titration. Exactly 5 ml of the digested sample was taken, and then 3 ml of 40 % (w/v) NaOH was added. The mixture was steam distilled for 2 minutes into a 50 ml conical flask containing 10 ml of 2% Boric acid mixed indicator solution placed at the receiving tips of the condenser. This changed the colour from red to green showing that all the ammonia liberated had been trapped. Furthermore, digestion was done by taking

0.50 g of ground dried sample carefully into the kjeldahl digestion tubes to ensure that all samples got to the bottom of the tubes and was added to 10 ml of conc. H_2SO_4 which were set in the appropriate heater. The digestion was left for 2 hours after which a clear colourless solution was left in the tube. The digestion was cooled and carefully transferred into 100 ml volumetric flask and made to mark with distilled water. The sample obtained from distillation was then titrated against 0.01 N HCl. When the green colour turned to wine, it indicates that all Nitrogen trapped and Ammonium Borate have been removed as Ammonium chloride [7, 8, 10].

$$\% \text{ Nitrogen} = \frac{(\text{VS} - \text{VB}) \text{ NA x } 0.01401 \text{ x } 100}{W}$$

Where, VS = Volume of acid used in titration, VB = Volume of base, NA= Normality of acid and W = Mass of sample used.

Determination of Crude Fiber Determination [5]

Precisely 2 g of the initial weight of sample (W1) was accurately weighed into the fiber flask and 100 ml of 0.255 N H_2SO_4 was added. The mixture was heated under reflux with the heating mantle. The hot mixture was filtered through a fiber sieve cloth. The difference obtained was thrown off and the residue was returned to the flask to which 100 ml of 0.313 M NaOH was added and heated under reflux for another one hour. The mixture was filtered and 10 ml of acetone was added to dissolve any organic constituent. The residue was washed with 50 ml hot water twice and then transferred into the crucible. The crucible and the residue were oven dried at 105 °C overnight to drive off moisture. The oven dried crucible containing the residue was cooled in a desiccator and later weighed for ashing at 550 ^{0}C for four hours. The crucible containing white and grey ash (free of carbonaceous material) was cooled in a desiccator and weighed to obtain W2. The difference W1 – W2 gave the weight of fiber [7, 8, 10].

$$\% \text{ fiber} = \frac{W_1 - W_2}{W_t} \times 100$$

Where $W_1 - W_2$=residual weight of sample remaining and W_t= weight of sample at time t

Determination of % CARBOHYDRATE = 100 – (FAT + PROTEIN + FIBER + ASH + MOISTURE)

Determination of Hemicellulose

Exactly 1 g of dried fiber sample was transferred into a 250 mL Erlenmeyer flask. 150 mL of 0.500 mol/dm^3 NaOH was added. The mixture was boiled for 3 h 30 min with distilled water. It was filtered after cooling through vacuum filtration and washed until neutral pH. The residue was dried to a constant weight at 105 °C in a digital analytical laboratory oven. The difference between the sample weight before and after this treatment is the hemicellulose content (%w/w) of dry fiber sample [7, 8, 10].

Determination of Lignin

Exactly 5 g of dried fiber sample was weighed into a 1000 mL beaker and 50 mL of 72% H_2SO_4 was added. The sample was kept at room temperature for 2 h with carefully shaking at 30 min intervals to allow for complete hydrolysis. After the initial hydrolysis, 800 mL of distilled water was added. The second step of hydrolysis was made to occur in an autoclave for 1h at 120 °C. The slurry was then cooled at room temperature. Hydrolyzates were filtered through vacuum filtration using a filtering crucible. The acid insoluble lignin was determined by drying the residues at 105 ^{0}C and accounting for ash by incinerating the hydrolyzed samples at 575 °C in a muffle furnace. The acid soluble lignin fraction was determined by measuring the absorbance of the acid hydrolyzed samples at 320 nm. The lignin content was calculated as the summation of acid insoluble lignin and acid soluble lignin [13,18].

Determination of Cellulose

This was calculated as: Cellulose = 100% - (Hemicellulose + lignin + Ash + Alpha-cellulose)

Determination of Holocellulose estimation

Holocellulose is the total carbohydrate fraction (cellulose and hemicellulose) of the raw material as was estimated [7, 8, 10].

RESULTS AND DISCUSSION

Table 1: PROXIMATE PROPERTIES OF KENAF FIBRE

S/N	Parameter	Amount (%)
1	Ash	5.57
2	Moisture	6.56
3	Volatile Matter	12.13
4	Fixed Carbon	82.3
5	Nitrogen	1.83
6	Crude Protein	11.44
7	Fat	9.44
8	Crude Fiber	23.27
9	Carbohydrate	43.72
10	Lignin	17.49
11	Hemicellulose	14.34
12	Cellulose	62.60
13	Holocellulose	76.94

Table 2: Elemental Composition (mg/g) of Kenaf Fiber

S/N	Element	% Composition (mg/g)
1	O	4.512
2	Mg	1.056
3	Al	0.058
4	Si	1.152
5	P	5.012
6	S	6.581
7	Cl	12.419
8	K	49.415
9	Ca	0.441
10	Ti	0.118
11	V	0.019
12	Mn	0.032
13	Fe	2.234
14	Co	0.027
15	Ni	0.005
16	Cu	0.030
17	Zn	0.002
18	Zr	0.062
19	Ni	0.004
20	Ag	0.024
21	Ba	0.012
22	Ta	0.009
23	W	0.009

The investigation on preferential fillers to the conventional one and other inorganic sources are very significant. The high cost and sometimes unavailability of the conventional fillers coupled with the shortages of foreign exchange and poor quality of composites for outdoor applications have rendered blends and composites production an expensive venture in Nigeria [7, 10, 13]. The prices of the locally available polymer products are high most especially as the concentrates containing commercially viable filler are usually imported

The results in Table 1 depicted that kenaf fiber contained 5.57% ash content, moisture content 6.56%, 12.13% volatile matter, 82.30% fixed carbon, 1.83 % nitrogen content, 11.44 % crude protein, 9.44% crude fat, 23.27% crude fiber, 43.72% carbohydrate, 17.49% lignin, 14.34% hemicellulose, 62.60% and 76.94% holocellulose accordingly.

For the ash content, moisture content and volatile matter, similar results were reported [8, 9, 14], though their results were slightly lower than the ones obtained for this research. Moisture content of natural fibers has been reported to be within the range of 5-10%, while the one obtained for this research was on the lower side. The crude protein, crude fat, crude fiber and carbohydrate values reported in these studies were significantly higher than the reported proximate content of kKenaf fiber’s seed powder reported [10-12]. From these results, kenaf fiber stem can be considered as a good source carbohydrate and protein. The high fiber content of kenaf stem will help to improve mechanical, thermo-mechanical stiffness and stability properties of polymer materials for different engineering application [12,17]. It was discovered that the kenaf fiber contained 62.60% cellulose, 14.34% hemicellulose, 76.94 % holocellulose and 17.49% lignin recorded in this research in Table 1 were similar to the data reported [13-15].

The results in Table 2 show the minerals composition and their corresponding values of the kenaf fiber. Ti, V, Zr, Fe, Co, Cu, Mn, Mo and Zn were reported in this as trace elements with their corresponding values below 0.1 mg/g.

These heavy metals are required by polymer scientists and Engineers as additives for the manufacture of composites for outdoor applications [7, 15]. This is because, they, in high level are toxic and can damage the living healthy cells [3, 15]. Although, many of these heavy metals were not detected in kenaf seed, it follows therefore that kenaf fiber seed may not be deleterious when consumed by man or animals [3, 8, 9, 16].

CONCLUSION

The percentage composition of the phytochemicals comprising of moisture content, volatile matter, fixed carbon, Nitrogen, crude protein, crude fat, crude fiber, carbohydrate, while the proximate of the fiber composition that contain lignin, hemicellulose, cellulose and holocellulose have quantitatively been determined and shown to fall appropriately within the well-established literature of the fiber. The trace elements composition of the kenaf fiber were also determined using XRF.

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