

The impact of potassium permanganate (KMnO_4) treatment on the tensile strength of pineapple leaf fiber reinforced with tapioca-based bio resin

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Abstract

Purpose – The impact of potassium permanganate (KMnO_4) treatment on the tensile strength of an alkali-treated pineapple leaf fiber (PALF) reinforced with tapioca-based bio resin (cassava starch) was studied.

Design/methodology/approach – The PALF was exposed to sodium hydroxide (NaOH) treatment in varying concentrations of 2.0, 3.7, 4.5 and 5.5g prior to the fiber treatment with KMnO_4 . The treated and untreated PALFs were reinforced with tapioca-based bio resin. Subsequently, they were subjected to Fourier transform infrared (FTIR) and tensile test analysis.



Findings – The FTIR analysis of untreated PALF revealed the presence of O-H stretch, N-H stretch, C=O stretch, C=O stretch and H-C-H bond. The tensile test result confirmed the highest tensile strength of 35N from fiber that was reinforced with 32.5g of cassava starch and treated with 1.1g of KMnO_4 . In comparison, the lowest tensile strength of 15N was recorded for fiber reinforced with 32.5g of cassava starch without KMnO_4 treatment.

Originality/value – Based on the results, it could be deduced that despite the enhancement of bioresin (cassava starch) towards strength-impacting on the fibers, KMnO_4 treatment on PALF is very vital for improved tensile strength of the fiber when compared to untreated fibers. Hence, KMnO_4 treatment on alkali-treated natural fibers preceding reinforcement is imperative for bio-based fibers.

Keywords Pineapple leaf fiber, Tapioca-based bioresin, Potassium permanganate, Reinforcement, Chemical treatment, Tensile strength

Paper type Research paper

List of abbreviations

CO_2	Carbon dioxide
$\text{C}_2\text{H}_6\text{O}$	Acetone
FTIR	Fourier transform infrared
NaOH	Sodium hydroxide
KMnO_4	Potassium permanganate
PALF	Pineapple leaf fiber
TBR	Tapioca-based bioresin
UTS	Ultimate tensile strength

1. Introduction

Man-made polymer composite materials are widely used in industries to meet requirements such as lightweight and high strength (Atuanya, Government, Nwobi-Okoye, & Onukwuli, 2014; Atuanya, Onukwuli, & Aigbodion, 2014; Odera, Onukwuli, Ezeh, Menkiti, & Agu, 2021). The high increase in the rate of these man-made materials, waste disposal treatment, waste disposal services and incineration pollution are becoming a great challenge (Azeez & Onukwuli, 2017; Jayaramadu, Rajulu, & Guduri, 2010; Ray & Okamoto, 2003). Natural fibers are been introduced annually from renewable resources and are used to reinforce polymer matrix (Ezeh, Onukwuli, Odera, Ugonabo, & Okeke, 2020; Osoka, Onukwuli, & Kamalu, 2018; Odera, Onukwuli, & Aigbodion, 2018). They are beneficial to our environment due to their biodegradability and waste natural materials utilization (Azeez & Onukwuli, 2018; Government, Onukwuli, & Odera, 2018). Natural fibers have been recognized as a good potential reinforcement for engineering fiber composites due to been lightweight, non-toxic, high-specific modulus and are easy to process (Ray, Sarkar, Rana, & Bose, 2001; Mishra, Misra, Tripathy, Nayak, & Mohanty, 2001; Sherey, Boudenne, Ibos, Candaub, Joseph, & Thomas, 2008). They are also eco-friendly, relatively cheap, easy and safe handling, biodegradable, available in abundance and other peculiar properties (Wang, Panigrahi, Tabil, & Crerar, 2007; Li, Panigrahi, Tabil, & Crerar, 2004; Parsania, Sankhavara, Chopda, & Patel, 2020). Natural fibers absorb CO_2 while in their growth stage (Abdelmouleh, Boufif, Belgacem, & Dufresne, 2007; Government, Onukwuli, & Azeez, 2019; Ikezue, Okafor, & Onukwuli, 2019). There is a great improvement in flexural strength, tensile strength and impact strength of surface-modified fiber reinforced polymer composite (Girones, Lopez, Vilaseca, Bayer, Herrera-Franco, & Mutje, 2011; Dhanalakshmi, Ramadevi, & Basavaraju, 2015; Thakur & Thakur, 2014a). Pineapple leaf fiber (PALF) is one of the waste materials in the agricultural sector. It is widely grown in Malaysia as well as Asia. Commercially, pineapple fruits are very important, and the leaves are considered waste materials that are been used for the production of natural fiber. PALF has peculiar and excellent properties such as its high strength and low micro-fibrillar angle, which makes it desirable for several industrial and

domestic applications. This suggests the better performance of natural fibers such as PALF in comparison with the synthetic fibers and thus imperative to take advantage of the natural gifted fibers in exchange for synthetic fibers. Thorough research studies have been done by [Thakur and Thakur \(2014b, c\)](#) and [Thakur, Thakur and Gupta \(2014\)](#) on the effective utilization of different kinds of natural fibers and the effect of different processing conditions on cellulose fibers and polymer matrix. The studies revealed that the major challenge of natural fibers as a reinforcement material is improper contact of adherent surface and polymer matrix with a bad interaction load transformation from matrix to fiber. In order to improve the adhesion property of fibers, surface modification should be carried out using appropriate chemicals ([Thakur, Thakur, & Gupta, 2013](#); [Pappu et al., 2015](#)). The modifications can be achieved via alkaline treatment, grafting with malic anhydride copolymer, using a saline coupling agent, permanganate treatment and acetylation treatment ([Azeez, Onukwuli, Nwabanne, & Banigo, 2020](#); [Ezeh & Onukwuli, 2020](#); [Government et al., 2019](#); [Onukwuli & Ikezue, 2014](#)). It was recorded that single fiber tensile testing conducted on untreated and treated PALF showed the ultimate tensile strength (UTS). Young modulus and strain at break of the PALF sample indicate that the strain at break of the treated PALFs is less than 7.9% in comparison to that of the untreated PALF. The treated PALF has higher UTS greater than 630 MPa when compared to the untreated PALF with the highest UTS of 1620 Mpa recorded for treated PALF of 6% wt/wt NaOH ([Eric, Bernard, Elsie, Solomon, Johan, & Elvis, 2021](#)).

The use of potassium permanganate (KMnO_4) for this modification on a PALF reinforced with tapioca-based bio resin has not been studied. Consequently, this research focused on the impact of KMnO_4 treatment on an alkali-treated PALF reinforced with tapioca-based bio resin.

2. Materials and method

2.1 Materials

2.1.1 Pineapple leaf fiber (PALF) extraction. PALFs were extracted from pineapple leaves from a pineapple plantation in Ihiagwa Owerri Imo State, Nigeria. The leaves were processed using a manual method by water immersion for 24 hours. The surface was scraped to remove the outer layer with the aid of a broken plate. Fibers were pulled, cleaned and dried in sun for four days.

2.1.2 Tapioca bioresin (cassava starch). This was extracted from fresh cassava tubers and also prepared manually. Cassava starch was processed manually, and fresh cassava tubers were obtained from farmland in Ihiagwa Owerri west, Imo state. Fresh cassava tubers were peeled, rasped, cut into smaller sizes, soaked in water, ground and then sieved by using a chiffon material to separate the starch from the chaff. The starch was kept inside a bag that drained its water, and the dry cassava starch was obtained.

2.1.3 Chemicals (reagents) used. Sodium hydroxide (NaOH), KMnO_4 and acetone ($\text{C}_3\text{H}_6\text{O}$) were used for the permanganate treatment of fibers. The chemicals are of analytical grade.

2.1.4 Equipment. Electronic weighing balance, spatula/stirrer, distilled water, paper tape, gloves, water bath, beakers, sample foil and yarn textile testing machine.

2.2 Methods

2.2.1 Pineapple leaf fiber (PALF) characterization. Before and after the treatment, the fibers were examined using Fourier transform infrared (FTIR) spectroscopy. It was used to detect the functional groups in the fibers. FTIR spectroscopy assists in identifying the changes in the chemical compound of fibers before and after the chemical treatments ([Rouison, Sain, & Couturier, 2004](#)). The FTIR test was conducted using a Perkin Elmer using transmission method with Potassium bromide (KBr) pellets. The wave number swept was from 3500 to 1000 cm^{-1} .

2.2.2 Alkali pretreatment of fibers. Pineapple fibers of various weights, 1.4, 2.0, 2.5, 3.2 and 4.0 g obtained from Ihiagwa, Owerri, were immersed in varying concentrations of NaOH , 2.0, 3.7, 4.5 and 5.5g, respectively, which were dissolved in 500ml of distilled water. This was

performed at temperatures of 30, 45, 60 and 90°C at reaction times of 45, 60, 90 and 120 min, respectively. The fibers were washed with distilled water to remove any trace of NaOH. They were further dried for 48h, reweighed and carefully kept for future use.

2.2.3 Potassium permanganate treatment ($KMnO_4$). Permanganate is a compound that has permanganate group MnO_4^- in it. In this treatment, there is a formation of cellulose radical through MnO_3^- ion formation and there is an initiating graft copolymerization which the highly reactive Mn^{3+} ions are responsible for. In this study, after the alkali treatment of the fibers, the fibers were washed with water, sun-dried and reweighed. Some of the fibers were immersed in solutions of (0.5, 0.7, 0.9 and 1.1g) of $KmnO_4$, respectively, which were dissolved in 100 ml of acetone for 50 min at 30°C. The fibers were then rewashed with distilled water to remove the residues of $KmnO_4$. They were further air dried for 24–48 hours and reweighed after the treatment.

2.2.4 Reinforcing with tapioca-based bioresin (cassava starch). 32.5g of prepared cassava starch was used to reinforce 2.0g of both treated and untreated PALF. This was done by the immersion of 2.0g of fiber into the beaker of 32.5g of cassava starch and left for 24h. It was further dried for five days after immersion. Subsequently, they were weighed to know the level of absorption and kept for further use.

2.2.5 Tensile testing. Tensile testing involved subjecting the treated, untreated and reinforced fibers to a controlled tension until it failed. The Tensile strength of the PALF was obtained using the yarn textile testing machine shown in [Plate 1](#).

3. Results and discussion

3.1 FTIR analysis

The FTIR spectra of the fibers are presented in [Figures 1–4](#). The spectrum of each of the graphs shows various peaks in the absorbance versus wave number relationship FTIR spectrum is a graph of infrared light absorbance on the vertical versus wave number. The unit of the wave number is cm^{-1} . The peaks and their corresponding intensities represent the functional groups of the fiber ([Furniss, Hannaford, Smith, & Tatchell, 2009](#); [Eddy, Ita, Dodo, & Paul, 2012](#); [Skoog, West, Holler, & Crouch, 2004](#)). The FTIR analysis of untreated PALF revealed the presence of O-H stretch, N-H stretch, $C\equiv$ stretch, $C=O$ stretch and H-C-H bond. The analysis of permanganate treated fiber revealed the presence of Hydrogen-bonded O-H stretch, H-C-H asymmetric and symmetric stretch, $C\equiv C$ stretch, H-C-H bond and $N=O$ stretch. The FTIR analysis of untreated PALF reinforced with cassava starch resin showed the presence of Hydrogen-bonded -OH stretch, $C\equiv N$ stretch, $C\equiv C$ stretch, $N=O$ stretch and $N=O$ bond. The analysis of permanganate-treated pineapple fiber reinforced with cassava starch resin showed reveals the presence of $C\equiv$ stretch, C-H stretch, off $C=O$ and $\equiv C-H$ stretch.

3.2 Permanganate treatment

3.2.1 Effect of permanganate treatment on a pineapple leaf fiber at 30°C. The alkali pretreated fibers were immersed in solutions of varying concentrations of $KMnO_4$ (0.5, 0.7, 0.9 and 1.1g)

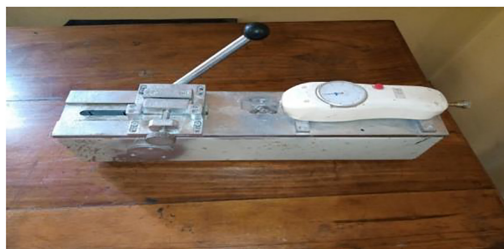


Plate 1.
Yarn textile testing machine

Figure 1.
FTIR of untreated
pineapple leaf fiber

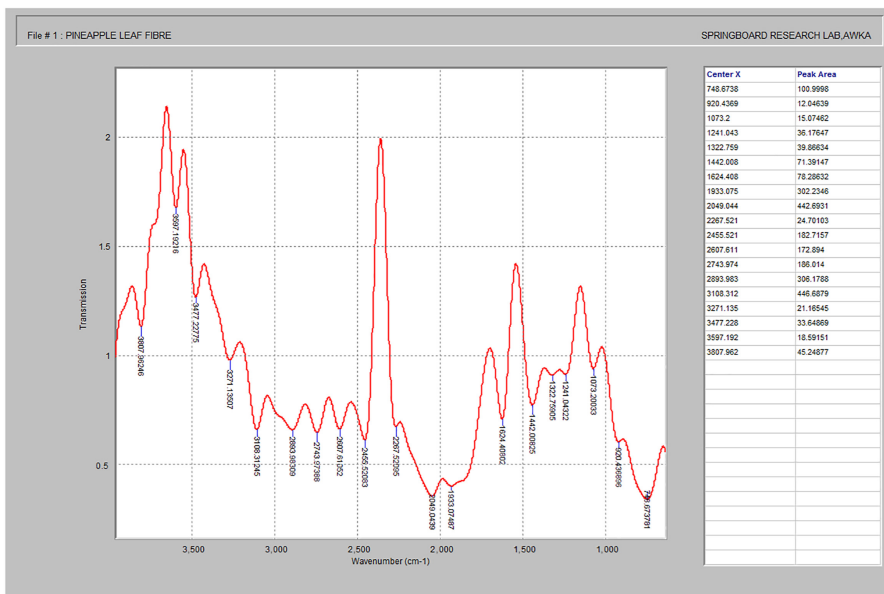
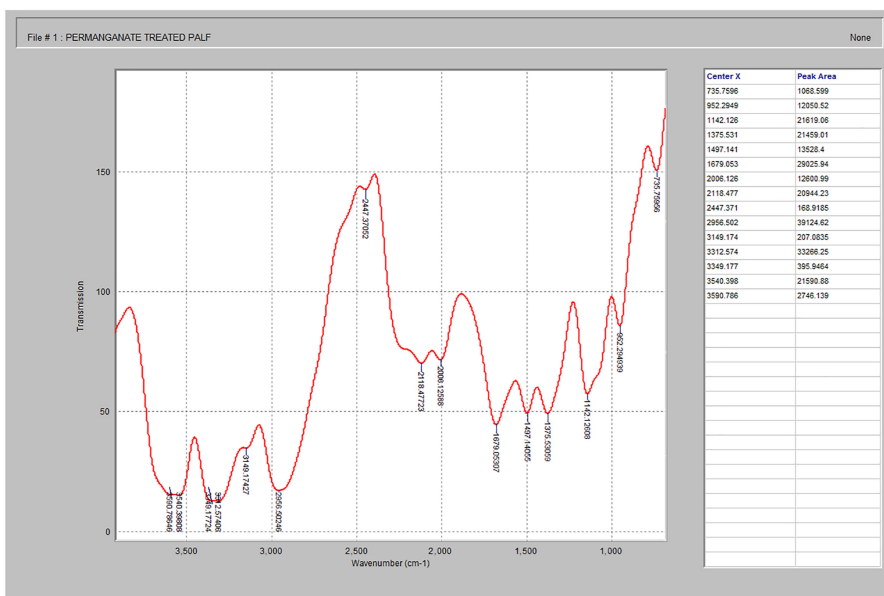


Figure 2.
FTIR of permanganate
treated pineapple
leaf fiber



that were dissolved in 100 ml of acetone each at 30°C. Different performances were observed. Distinct concentrations of KMnO_4 affected the fibers differently such that there were variations in the weight and strength. The loss in the weight of the treated fibers was seen to be KMnO_4 concentration dependent. The higher the concentration of KMnO_4 used in the

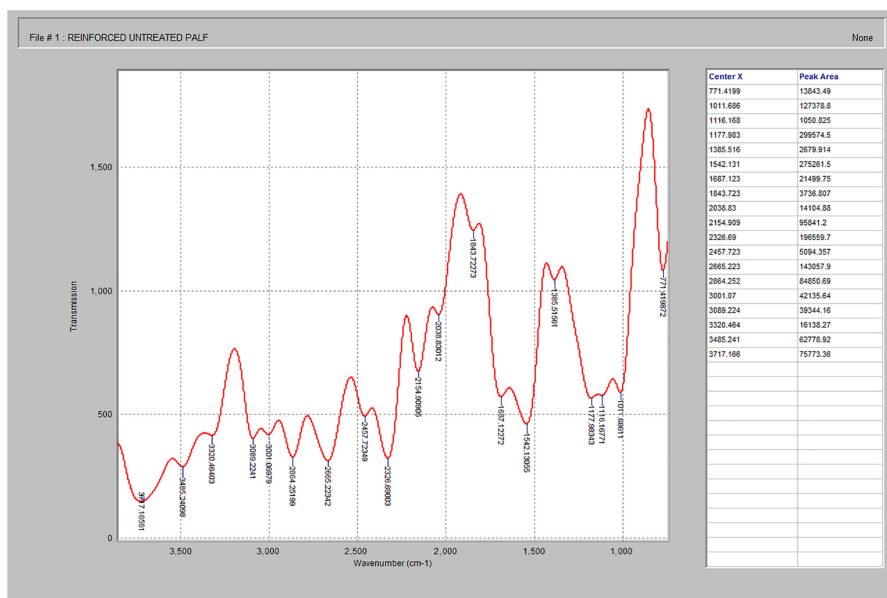


Figure 3. FTIR of untreated PALF reinforced with TBR

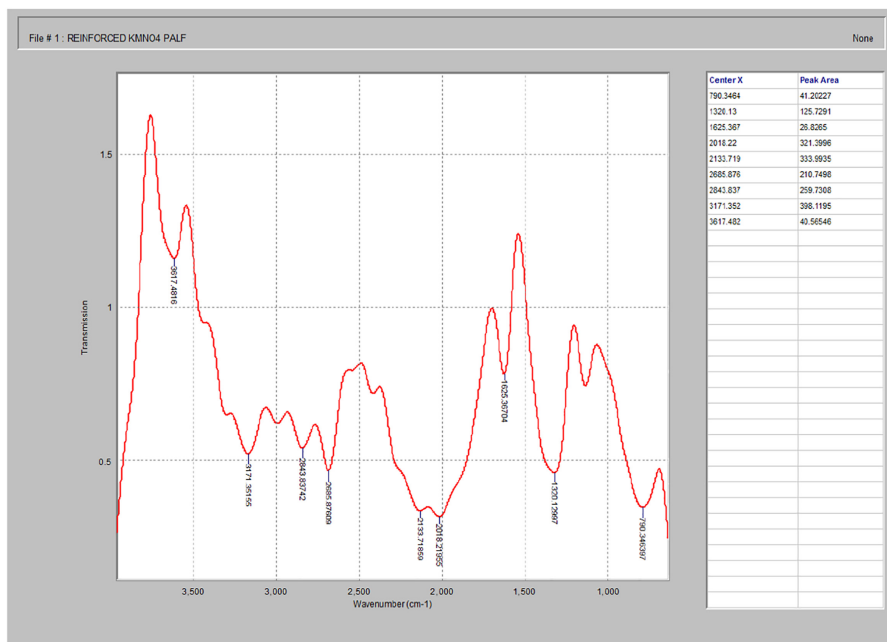


Figure 4. FTIR of permanganate treated PALF reinforced with TBR

Table 1.
Permanganate
treatment of pineapple
leaf fiber of different
samples at varying
concentrations

Sample	Conc. of KMnO ₄ (g)	Conc. of Acetone (ml)	Temp (°C)	Time (min)	Initial weight (g)	Final weight (g)	Weight loss (g)
A (1.4g)	0.5	100	30	50	1.4	1.23	0.17
	0.7	100	30	50	1.4	1.19	0.21
	0.9	100	30	50	1.4	1.10	0.30
	1.1	100	30	50	1.4	0.79	0.66
B (2.0g)	0.5	100	30	50	2.0	1.75	0.25
	0.7	100	30	50	2.0	1.37	0.63
	0.9	100	30	50	2.0	1.30	0.70
	1.1	100	30	50	2.0	1.27	0.73
C (2.5g)	0.5	100	30	50	2.5	2.05	0.45
	0.7	100	30	50	2.5	1.60	0.90
	0.9	100	30	50	2.5	1.57	0.93
	1.1	100	30	50	2.5	1.54	0.96
D (3.2g)	0.5	100	30	50	3.2	2.57	0.63
	0.7	100	30	50	3.2	2.22	0.96
	0.9	100	30	50	3.2	2.04	1.35
	1.1	100	30	50	3.2	1.84	1.16
E (4.0g)	0.5	100	30	50	4.0	3.09	0.91
	0.7	100	30	50	4.0	2.70	1.30
	0.9	100	30	50	4.0	2.68	1.32
	1.1	100	30	50	4.0	2.62	1.38

treatment of the PALF, the higher the fiber loses its weight and the higher the strength of the fiber. Increased weight losses of 0.66, 0.73, 0.96, 1.16 and 1.38g obtained from the fiber samples of 1.4, 2.0, 2.5, 3.2 and 4.0g, respectively, are from the highest concentration of 1.1g of KMnO₄. This infers that the highest weight loss was obtained from the highest concentration of 1.1g KMnO₄ when compared to the values recorded with the lower concentrations of KMnO₄ (0.5, 0.7 and 0.9g). Table 1 displays permanganate treatment on PALF of different samples at varying concentrations while Figure 5 shows the final weights (g) of the permanganate treated samples.

3.3 Reinforcing of PALF with TBR

An increase in the weight of the fibers after they were reinforced with bioresin was observed. The fiber with the lowest weight of 2.45g was not treated chemically with KMnO₄, hence a shred of evidence that treated fibers absorbed more reinforcement substance and thus more tensile strength was recorded for treated fibers. The highest tensile strength of 35N was obtained from the fiber that was reinforced with 32.5g of cassava starch and treated with 1.1g of KMnO₄, while the lowest tensile strength of 15N was obtained from fiber reinforced with

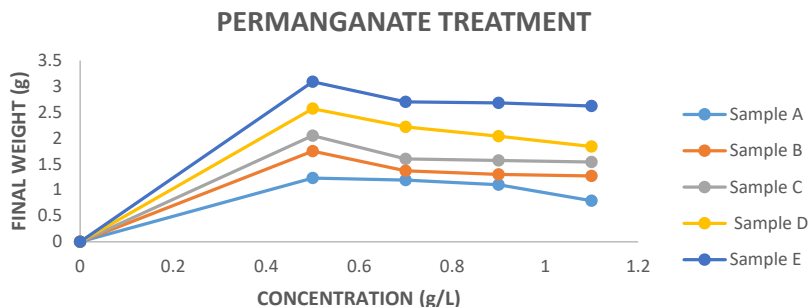


Figure 5.
Permanganate-treated
samples

32.5g of cassava starch but without KMnO_4 . This implies that despite the enhancement of bioresin (cassava starch) towards impacting strength on the fibers, chemical treatment on the fibers is very vital because it helps to induce strength on the fibers. Table 2 shows the change in mass and tensile strength for permanganate-treated PALF reinforced with TBR. The tensile strength of permanganate-treated PALF reinforced with TBR is depicted in Figure 6.

The tensile strength of pineapple leaf fiber

3.4 Tensile strength

After the analysis of permanganate-treated PALF using a yarn textile testing machine, an increase in concentration of the KMnO_4 used in the treatment was discovered to lead to increase in the strength of the fiber. All samples have their lowest strength (force at break) 12.5, 15 and 10N, respectively, for samples A, B and C at their lowest concentration of 0.5g/l KMnO_4 and their highest strength at the highest concentration of 1.1g/l except for sample B with the highest tensile strength of 22.5N at the concentration of 0.9 g/l of KMnO_4 after which the tensile strength decreased to 20N at 1.1g of KMnO_4 . The lowest strength (force at break) of 8N was obtained from the untreated samples of the three samples. This is proof that KMnO_4 treatment of the fiber enhanced the strength of the fiber. Table 3 and Figure 7 show the tensile strength of permanganate treated PALF with varying concentrations.

Sample	Conc. of TBR (g)	Weight before reinforcement (g)	Weight after reinforcement (g)	Weight gain (g)	Tensile strength (N)
Untreated	32.5	2.00	2.45	0.45	15
0.5g KMnO_4	32.5	1.75	2.89	1.14	25
0.7g KMnO_4	32.5	1.30	3.04	1.74	27.5
0.9g KMnO_4	32.5	1.37	2.70	1.33	20
1.1g KMnO_4	32.5	1.27	2.80	1.53	35

Table 2. Change in mass and tensile strength for permanganate treated PALF reinforced with TBR

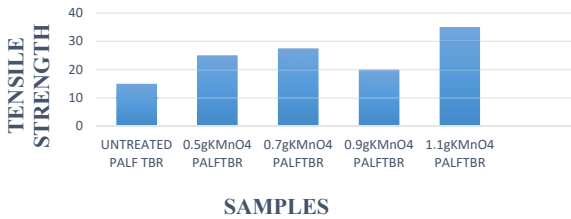
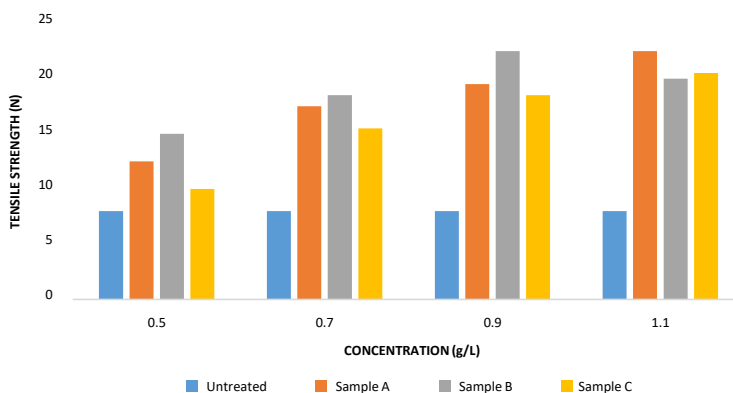


Figure 6. Tensile strength of permanganate-treated PALF reinforced with TBR

Conc. of KMnO_4 (g/l)	Sample A		Sample B		Sample C	
	Weight of fiber (g)	Force at break (N)	Weight of fiber (g)	Force at break (N)	Weight of fiber (g)	Force at break (N)
Untreated	1.40	8	3.20	8	4.00	8
0.5	1.40	12.5	3.20	15	4.00	10
0.7	1.40	17.5	3.20	18.5	4.00	15.5
0.9	1.40	19.5	3.20	22.5	4.00	18.5
1.1	1.40	22.5	3.20	20	4.00	20.5

Table 3. Tensile strength of permanganate treated pineapple leaf fiber with varying concentrations

Figure 7.
Tensile strength of permanganate treated PALF



4. Conclusion

The impact of chemical treatment (KMnO_4) on the tensile strength of PALF reinforced with cassava starch was studied with a yarn textile testing machine. Distinct concentrations of KMnO_4 affected the fibers differently such that there were variations in the weight and strength; thus, the loss in the weight and increase in the strength of the treated fibers was KMnO_4 concentration dependent. This was evidenced in the tensile strength result which showed maximum tensile strength of 35N from fiber treated with 1.1g/l of KMnO_4 and reinforced with 32.5g of cassava starch when compared to the minimum tensile strength of 15N from fiber without KMnO_4 treatment. Increase in the concentration of KMnO_4 in the treatment of the alkali-treated PALF led to increase in the strength of the fiber. Consequently, for improved tensile strength of alkali-treated PALF reinforced with tapioca-based bio resin, KMnO_4 treatment is crucial.

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