

Effect of alkali treatment on physical and mechanical properties of Cattail and Kenaf fibres

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ABSTRACT – REZUMAT

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NaOH treatment is widely used for natural fibre extraction. However, the different treatment conditions produce fibres with other characteristics. The present work aims to study the effect of alkali treatment conditions on Kenaf bast and Cattail stem fibres' properties. Two conditions at two different levels were selected. They are soaking time (2 and 4 hours) and alkali treatment temperature (80 and 120°C). Untreated Kenaf and Cattail fibres were used as control samples. The NaOH process occurred due to the decline in the fineness of Cattail and Kenaf fibres. SEM micrographs of treated fibres showed a clear surface with rectangular pits for Cattail fibres and dissociation of the technical fibre. Best tensile strength obtained for Kenaf fibres for 80°C and under 4 hours. However, a temperature of 120°C and a duration of 4 hours confirmed the best results in terms of lignin removal, proved by IR spectra. Also, X-ray diffractograms suggested that the crystallinity index increases with the highest conditions. The properties of Kenaf bast fibres are found to be superior to Cattail fibres. Characteristic ranges of Cattail and Kenaf fibres after alkaline treatment can be resumed respectively as the diameter of (205–496) and (66–162 µm), the linear density of (31–48) and (14–22 tex), tenacity of (6–8) and (13–17 cN/tex), elongation of (2.6–3.3) and (3–6.2%), lignin ratio of (20.7–23.7) and (15.13–22.6%), alpha-cellulose of (50.2–55.6) and (53–59%), and crystallinity of (43.9–58.6) and (55.4–66.3%). Findings showed that Kenaf bast fibres are found to be great resistant and thinner than Cattail fibres and compared to other fibres.

Keywords: Cattail fibre, Kenaf fibre, NaOH process, physical properties, mechanical properties

Efectul tratamentului alcalin asupra proprietăților fizice și mecanice ale fibrelor de papură și chenaf

Tratamentul cu NaOH este utilizat pe scară largă pentru extracția fibrelor naturale. Cu toate acestea, condițiile diferite de tratament produc fibre cu caracteristici diferite. Lucrarea de față își propune să studieze efectul condițiilor de tratare cu alcalii asupra proprietăților fibrelor liberiene de papură și chenaf. Au fost selectate două condiții la două niveluri diferite, și anume timpul de impregnare (la 2 și 4 ore) și temperatura tratamentului alcalin (80°C și 120°C). Fibrele de papură și chenaf netratate au fost utilizate ca probe de control. Procesul NaOH a avut ca rezultat o scădere a fineței fibrelor de papură și chenaf. Micrografiile SEM ale fibrelor tratate au evidențiat o suprafață clară cu caneluri dreptunghiulare pentru fibrele de papură și disocierea fibrei tehnice. Cea mai bună rezistență la tracțiune a fost obținută în cazul fibrelor chenaf la o temperatură de 80°C și o durată de până la 4 ore. Cu toate acestea, o temperatură de 120°C și o durată de 4 ore au confirmat cele mai bune rezultate în ceea ce privește îndepărtarea ligninei, dovedită cu ajutorul spectrelor IR. De asemenea, difractogramele de raze X au sugerat că indicele de cristalinitate crește la cele mai ridicate niveluri de temperatură. Proprietățile fibrelor chenaf sunt superioare fibrelor de papură. Intervalele caracteristice ale fibrelor de papură și chenaf după tratamentul alcalin pot fi reluate, și anume: diametrul de (205–496) și respectiv (66–162 µm), densitatea liniară de (31–48) și (14–22 tex), tenacitatea (6–8) și (13–17 cN/tex), alungirea de (2,6–3,3) și (3–6,2%), raportul de lignină de (20,7–23,7) și (15,13–22,6%), alfa-celuloză de (50,2–55,6) și (53–59%) și cristalinitatea de (43,9–58,6) și (55,4–66,3%). Rezultatele au arătat că fibrele de chenaf sunt foarte rezistente și mai subțiri decât fibrele de papură sau față de alte tipuri de fibre.

Cuvinte-cheie: fibră de papură, fibră chenaf, proces NaOH, proprietăți fizice, proprietăți mecanice

INTRODUCTION

Bio-composites reinforced with Natural Fibres (NFs) have increased widely. In addition, such composites can be used for various applications such as automotive, marine, military, and sports equipment, in industrial and home applications [1]. Moreover, NFs have many advantages especially since they are eco-friendly, non-hazardous, and degradable. NFs can be provided by plants, animals, and minerals. Considering NFs from plants, fibre quality depends

on the plant growth region (climate, soil, etc), the position of the fibre in the plant, variety, etc [2]. Besides, extraction methods and processing parameters also have an impact on the final properties of NFs [3]. Among the processes reported in the extraction of NFs, the alkali treatment is the most used method, due to its simplicity and cost-effectiveness [4]. Research on the extraction process using NaOH treatment confirms the enhancement of mechanical, physical, and thermal properties of composite mate-

rials reinforced with NFs [5]. Alkali treatment contributes to the removal of non-cellulosic compounds like lignin and hemicelluloses [6]. So, it reduces the amorphous region and increases the crystallinity index [7], resulting in the modification of mechanical characteristics, roughness, and thermal stability. In addition, it was proved that the NaOH process is efficient in getting the best quality fibre because of the disturbance of the hydrogen bond in the chemical structure which increases the roughness and eliminates the percentage of binding material like hemicellulose and lignin content [8].

Several efforts have been made to study many properties of the stem fibres [9]. Some fibres have not been enough developed and, at some times, few researchers have explored their various morphological, mechanical, physical, and chemical analyses [10]. Cattail stem fibres are considered one of these categories. Considering the no availability of data on the characterization of the effect of extraction parameters on the Cattail stem fibre quality, this study was undertaken to fill this gap. To explore all stem fibres and investigate their properties, especially for reinforcing bio-composite materials, it is essential to study these unknown characteristics of less-considered fibres like Cattail.

First, this study focuses on analysing the effect of NaOH alkali duration and temperature with a concentration of 20 g/l on Cattail fibres. Second, a comparison was carried out between the characteristics of extracted cattail stem fibre, a recently discovered plant, with another fibre widely discussed in the literature, which is the Kenaf bast fibre. In addition, there is no relative information related to the effect of the duration and temperature of alkali treatment on the properties of the Cattail stem fibres. The properties of fibres were carefully observed and compared with untreated raw fibres. Therefore, physical, chemical, and morphological characteristics will make foundations for future investigations on the valorisation of these fibres for potential sources in reinforcing bio-composites for different applications.

EXPERIMENTAL

Materials

Cattail plants (figure 1, a), also called Typha, were collected from the local river in Moknine, Tunisia. Kenaf (figure 1, b), also called Hibiscus Cannabinus,



Fig. 1. Plants of: a – Cattail; b – Kenaf

was cultivated in an experimental field in a semi-arid region in Tunisia.

The procedure of fibre extraction

Extraction of the Kenaf and Cattail Stem fibres was conducted at NaOH concentrations of 20 g/l at 80 and 120°C for 2 and 4 hours. After completion of the alkaline treatment, fibres were washed several times in hot water, and pH was neutralized by using dilute acetic acid, rinsed in water, and dried under ambient conditions. For the extraction, 5 g of Cattail or Kenaf was extracted using a Mathis LABOMAT and the liquor ratio was equal to 1/40.

So, five tests were conducted for each fibre, composed of 4 alkali treatment test conditions (1, 2, 3, 4) and a non-treated test (NT) as shown in table 1. The untreated test presented fibres manually extracted without NaOH for both Kenaf (KNT) and Cattail (CNT), used as control samples. Alkali treated tests for Cattail (C1, C2, C3, C4) and Kenaf (K1, K2, K3, K4) were codified as presented in table 1.

Characterization

SEM Analysis

The morphology (longitudinal and cross-section views) of Cattail and Kenaf fibres were examined using a scanning electron microscope (SEM) Hitachi S-2360N.

Physical, chemical and mechanical properties

To determine the diameter of Cattail and Kenaf fibres, an optic microscope (Leica) was used following the French standard NF G 07-004 (1983). Also, the linear density of Cattail and Kenaf fibres was measured using the gravimetric method using the ISO 1973 (1995) standard. Besides, Alpha-cellulose content and lignin fraction were measured using the TAPPI

Table 1

EXTRACTION CONDITIONS AND CODED TESTS FOR CATTAIL AND KENAF FIBRES					
Test	Extraction conditions			Code	
	NaOH (g/l)	Temperature (°C)	Duration (h)	Cattail	Kenaf
1	20	80	2	C1	K1
2	20	80	4	C2	K2
3	20	120	2	C3	K3
4	20	120	4	C4	K4
NT	-	-	-	CNT	KNT

standard method T 203 cm-09 and T222 om-11 respectively. Finally, mechanical properties were determined with an LLOYD dynamometer according to the NF G07-002, 50 tests were carried out to determine the maximum strength that fibre can support and the corresponding strength and elongation.

FTIR analysis

The functional groups present in Cattail and Kenaf fibres and their unique chemical bonds were found by Perkin Elmer Spectrum 100 FT-IR, connected to an ATR Mode. The sample was analysed in the wavelength range of 500–4500 cm^{-1} at 25°C.

X-Ray diffraction study

The amorphous and crystalline phase of the Cattail and Kenaf fibres were analyzed using X-ray diffraction X'P ERT Pro model (1967) by PANalytical with $\text{Cu K}\alpha$ ($\lambda = 1.5406 \text{ \AA}$) as a radiation source with an accelerating voltage of 40 kV. The crystallinity index (CI) was calculated using the following equation:

$$CI \% = 100 \times \frac{(AC)}{(AT)} \quad (1)$$

where AC presents areas of crystalline peaks and AT the total area of amorphous and crystalline peaks.

RESULTS AND DISCUSSION

Morphology of Cattail and Kenaf fibres

The effect of NaOH treatment conditions on Cattail and Kenaf fibres was discussed in figures 2 and 3 through SEM images. Impurities around the raw fibres are highly clear in figure 2 (CNT) and figure 3 (KNT) due to the existence of non-cellulosic compounds. Moreover, it was determined from the longitudinal of CNT and KNT, many rectangular indentations on the surface. This observation was also followed by other studies on lignocellulosic fibres like *Typha Australis* [11] and *Catalpa bignonioides* fibres [12]. When the fibre underwent treatment for 2 h at 80°C, defibrillation of both Kenaf and Cattail did not

occur but there was a removal of some parts of impurities. With raising the duration (2 h to 4 h), impurities are well eliminated for Cattail fibres, but it was still present in Kenaf fibres. Although some percentage of impurities was seen, defibrillation started to take place, as shown in figure 2 (C2) and figure 3 (K2). However, in the case of Cattail fibres SEM micrographs of longitudinal views in figure 2, show some rectangular pits on the surface of Cattail fibres which can be explained by the removal of fatty substances formed by Tyloses as reported by other researchers working on *Borassus* fruit fibres [13]. Destruction of these rectangular entities begins when accelerating temperature to 120°C as followed in figure 3 (C3). Moreover, figure 2 (C4) and figure 3 (K4) depict that the defibrillation process can be accelerated with both higher temperature and duration of alkali treatment (120°C and 4 h).

So, the main observation was that the obtained fibres were more and more clean and rougher with increasing NaOH conditions treatment. As it will be confirmed in the ATR-FTIR analysis reported in the next part, alkaline treatment dissolves majorly lignin, and hemicellulose. Therefore, defibrillation occurs when raising temperature and duration of the process and the ultimate fibres appear. This can be more revealed by the presence of some holes and grooves on the fibre surface which confirmed the dissociation of one fibre to smaller ones. Cross-section micrographs in figure 2 (C4) and figure 3 (K4), proved that Kenaf and Cattail fibres are formed by several single fibres called ultimate fibres linked together by non-cellulosic materials.

Physical properties of Cattail and Kenaf fibres

Variations in diameter and linear density were reported in figure 4. Raw materials show the highest diameter, due to the presence of impurities and non-cellulosic materials. Even after alkali treatment, Cattail fibres showed a big fibre diameter compared to raw

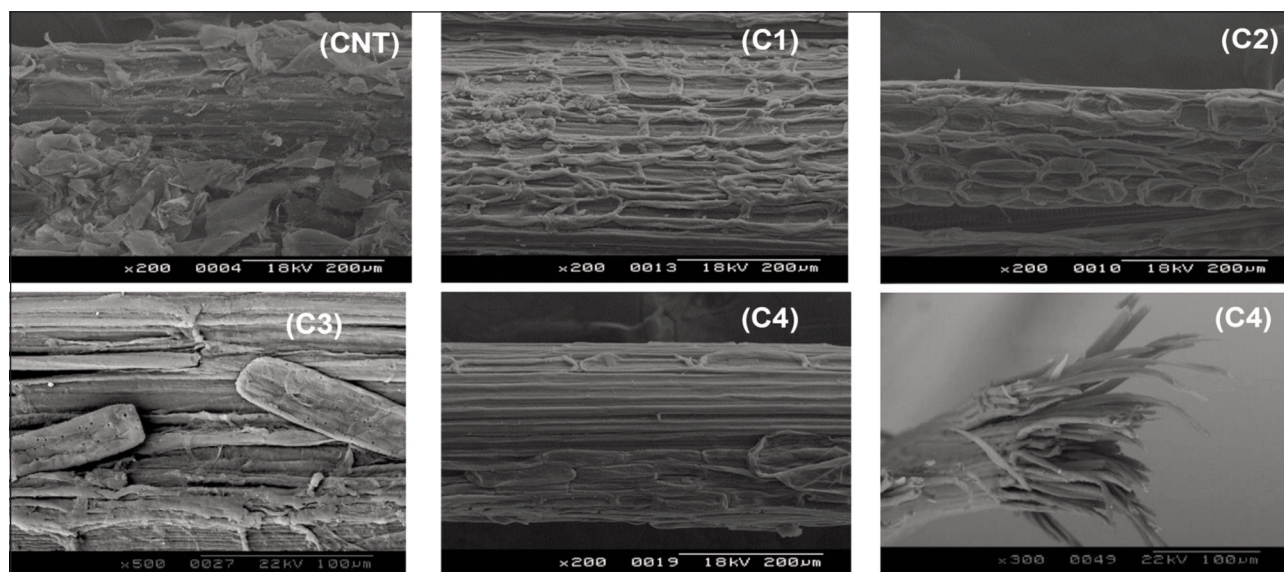


Fig. 2. SEM micrographs of a longitudinal view of Cattail fibres under different treatment conditions (CNT), (C1), (C2), (C3), (C4) and cross-section for (C4)

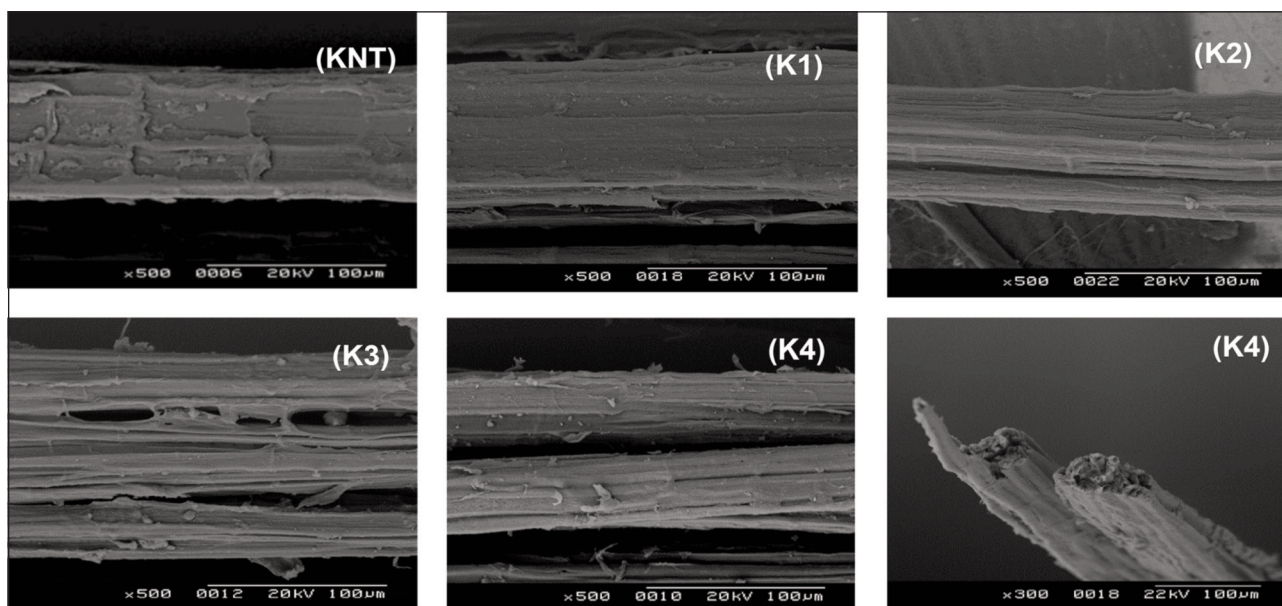


Fig. 3. SEM micrographs of a longitudinal view of Kenaf fibres under different treatment conditions (KNT), (K1), (K2), (K3), (K4), and cross-section for (K4)

Kenaf fibres. In addition, (KNT) established thinner fibres with 13 tex and 150 µm whereas Cattail NaOH treated fibres showed 31 tex and 205 µm for (C4). Due to sodium hydroxide, the fineness of obtained fibres is enhanced which confirms the removal of foreign compounds from both Cattail and Kenaf surface fibres. In fact, (KNT) and (CNT) have an average diameter of 145 and 520 µm respectively. After alkali treatment, the diameter decreased, and the best results were obtained after 4 hours at 120°C showing a reduction of 54 and 60% for (K4) and (C4) respectively. The decrease in fibre diameter can be explained by the NaOH treatment reaction.

Generally, kenaf and cattail fibres like other lignocellulosic fibres, presented technical fibre bundles, which are formed by the association of ultimate fibres as shown in the SEM cross-section in figure 2 (C4) and figure 3 (K4). After alkali treatment, the partial removal of non-cellulosic materials especially lignin as shown in the figure 6, favours the defibrillation of the technical fibre. This separation resulted in

bundles of fibre with reduced diameter [14]. Besides, linear density proved that the extraction process at 80°C for both Kenaf and Cattail showed fibres larger than the one extracted manually after 2 or 4 hours of treatment. As the diameter, the best linear density was reported for (K4) and (C4). This may be clarified by the dissociation of technical fibre to smaller ones by reduction of the fraction of binding materials.

Reduction in fibre diameter was reported due to the removal of non-cellulosic materials from the Kenaf and Cattail out layers. This investigation was reported also for Bamboo fibres [15]. The decrease in fibre diameter increases the length/diameter ratio which can improve the adhesion of Kenaf and Cattail fibres with polymer matrix and certainly would ameliorate the mechanical properties of resulted composite materials [16–19].

Mechanical properties of Cattail and Kenaf fibres

Variations in Tenacity and Elongation were reported in figure 5. Before alkali treatment, tenacity was equal to 5 and 14 cN/tex for CNT and KNT respectively.

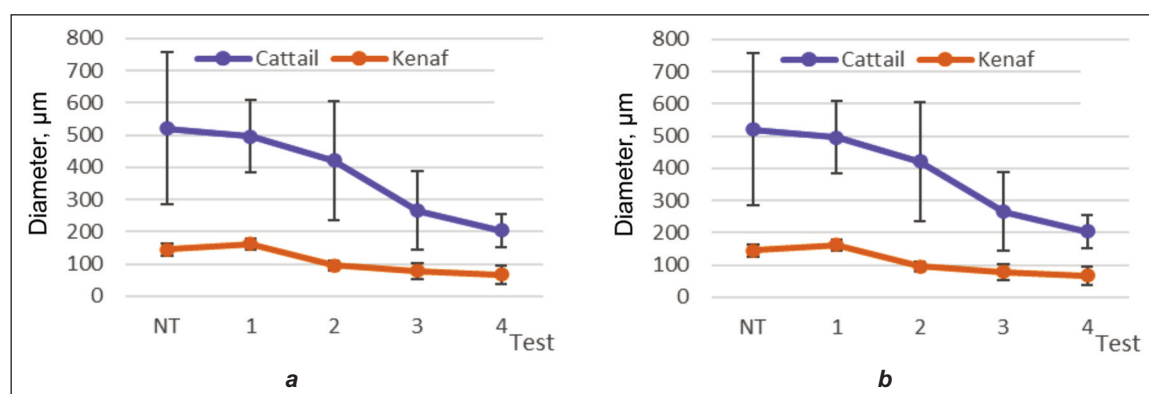


Fig. 4. Properties of Cattail and Kenaf fibres: a – variation of diameter; b – variation of linear density for different conditions

After the chemical process, the best improvement for cattail fibres was considered at 120°C for 4 hours. Sodium hydroxide treatment enhances the mechanical properties of NFs. In addition, due to the removal of microvoids and a decrease in non-cellulosic compounds, the stress transfer between ultimate fibres increases and tenacity improves [20]. However, the best results for Kenaf fibres were obtained at 80°C for 4 hours. When the Temperature increased to 120°C, although tenacity increased and presented the best results as the duration increased as well for Cattail fibres, the tenacity of K3 reduced after 2 hours compared to (K1) and (K2) and continued the reduction after 4 hours to reach 13 cN/tex for (K4). This may be attributed to the excessive treatment conditions which can damage kenaf strength. NaOH begins to integrate into internal molecules of Kenaf fibres when it reaches a higher temperature and soaking time [21]. This difference in mechanical behaviour may also be explained later by the difference in chemical composition between Kenaf and Cattail fibres presented by high lignin levels for Cattail fibres.

Considering the elongation, alkaline treatment, led to decreased elongation rates for Kenaf fibres. This phenomenon is very clear after the first condition of treatment with a reduction of 64% for (K1) compared to (KNT). When alkali duration and temperature increased, elongation rates were between 2.54% and 3.34%. These investigations were in contrast to those of Cattail fibres. The first condition (C1) shows an enhancement of 27% compared to (CNT). When boosting soaking time (4h) for Cattail fibres, elongation rises slightly to reach 6.2%. However, further increments in temperature and duration led to reduced elongation rates to reach 3.01% for (C4) like Kenaf fibres. The same results were reported in the case of Okra bast and corn husk fibres [22]. Removal of non-cellulosic compounds leads to smaller microfibril angles, which promotes easy reorientation of fibrils along the direction of tensile force. As a result, there is a better distribution of the load which enhances the tensile strength of fibres and decreases their elongation, especially for kenaf. Moreover, the elongation percentage was affected by lignin content. Some researchers suggested that an increase

in lignin fraction enhances the extension ability of natural fibres, which confirms the elongation decreases. This is supported by other research on cattail fibres showing that the fibres extracted from the leafiran variety of cattail (with 26% of lignin) presented higher elongation than kenaf fibre (with 17% of lignin) [23].

Chemical composition

Chemical constituents of the fibre, especially cellulose fraction affect the mechanical properties of the composite material reinforced with natural fibre. The concept of NaOH treatment was to eliminate non-cellulosic compounds like lignin, hemicellulose, wax, and pectin to obtain the best mechanical properties. Figure 6 depicted that before alkali treatment, the lignin ratio was highest for cattail fibres at about 30% compared to 22% for kenaf fibres. Best results were obtained for 120°C after 4 hours showing a reduction of 32% and 30% for (C4) and (K4) respectively. Alkali treatment confirmed modification of chemical constituents by enhancing alpha-cellulose content by 22 and 27%, the best results for (C4) and (K4) respectively. These results in turn would increase tensile strength in the using of these fibres for composite reinforcement. Removal of the lignin fraction increases the alpha-cellulose fraction.

ATR-FTIR analysis

Figure 7 illustrates the spectra for Cattail and Kenaf fibres with NaOH and without NaOH treatments. Overall, curves show that untreated and treated fibres have differences in terms of band intensity. Considering KNT and CNT fibres, FTIR spectra showed clear bands at 3353 cm^{-1} , 2902 cm^{-1} , 1726 cm^{-1} , 1625 cm^{-1} , 1417 cm^{-1} , 1316 cm^{-1} , 1230 cm^{-1} , 1025 cm^{-1} and 894 cm^{-1} . In addition, the first band at around 3353 cm^{-1} is related to hydroxyl OH stretching of alpha-cellulose. Besides, the second band at 2902 cm^{-1} is associated with C-H stretching jointed to CH_2 and CH attesting to the existence of cellulose, and hemicellulose. The band at 1726 cm^{-1} is attributed to pectin and hemicellulose and the band at 1625 cm^{-1} is connected to the presence of lignin. The next band was observed at 1417 cm^{-1} confirming the CH stretching of lignin. Another peak at

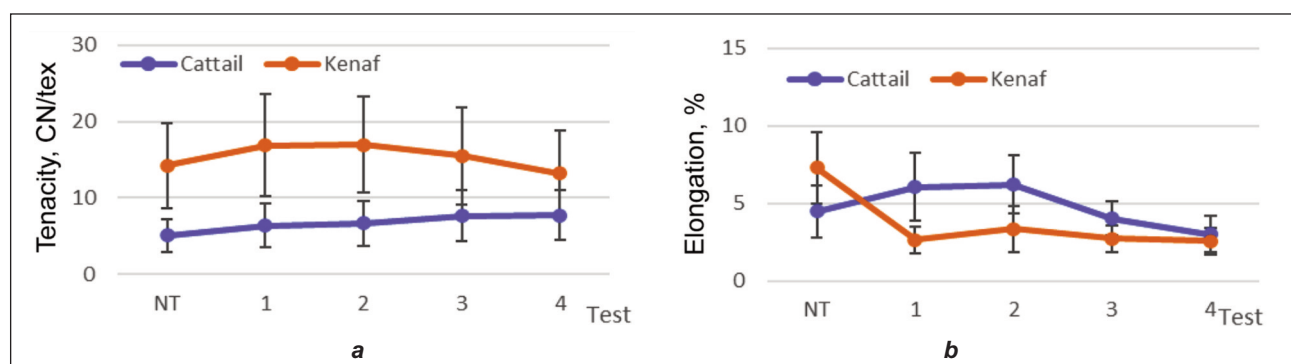


Fig. 5. Properties of Cattail and Kenaf fibres:
a – variation of tenacity; b – variation of elongation for different conditions

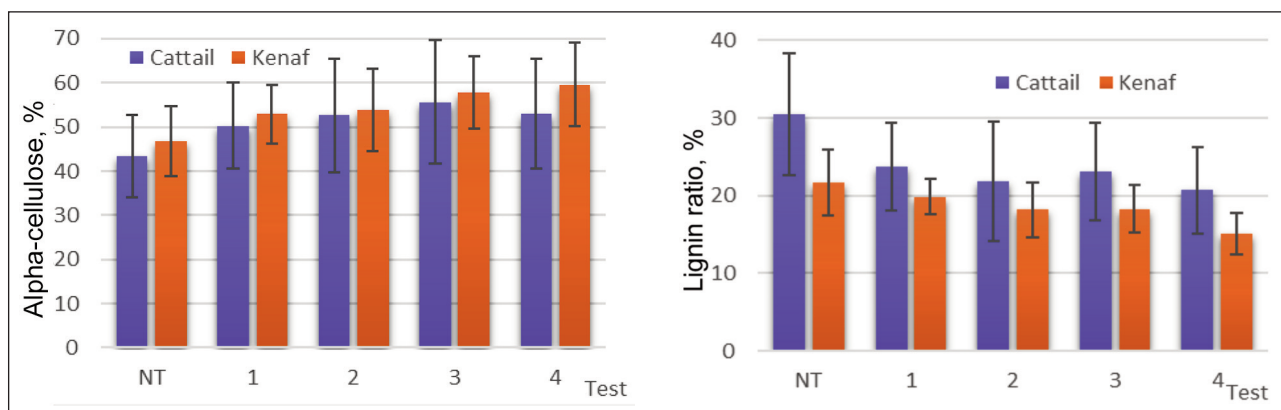


Fig. 6. Variation of Lignin ratio and alpha-cellulose for different conditions of Cattail and Kenaf fibres

1316 cm^{-1} jointed to the hemicellulose fraction was detected. Moreover, the spectra confirm the presence of lignin at 1230 cm^{-1} with vibrations of C=O and O-C-O groups. Another band at around 1025 cm^{-1} indicated cellulose by the asymmetric stretching of O-C-O ester groups. Finally, a band at 894 cm^{-1} was found for both (KNT) and (CNT) indicating cellulose existence by the C-OH group [24]. After the NaOH process, it was clear that chemical constituents and vibrations were affected majorly by the duration of the treatment. In addition, some bands were attenuated or dismissed especially for (K2), (K4), (C2), and (C4). A clear attenuation of the band around 1625 cm^{-1} was observed for (K1), (K2) and (K3). The same band disappeared for (C2), (C4), and (K4). Besides, the bands at 1417 cm^{-1} , 1316 cm^{-1} , and 1230 cm^{-1} were no longer detectable for (C2), (C4), and (K4). This confirms that the removal of some portion of lignin and hemicellulose was more affected by the duration of treatment. This can be assumed that a longer soaking time, will remove binding materials and increase cellulose, especially for cattail fibres.

X-Ray analysis

The X-ray diffractogram of cattail and Kenaf fibres was represented in figure 8, showing four main diffraction peaks for both Cattail and Kenaf at 2Theta angles of approximately 15°, 16°, 22,5° and 35° corresponding to (101) (101-), (002) and (040) lattice planes, respectively [14]. The crystallinity index of untreated and treated fibres was calculated using equation 1 and results are summarized in table 2. Overall, Kenaf fibres have higher crystallinity compared to Cattail fibres which was expected since they have lower lignin content (figure 6). Moreover, after alkaline treatment, crystallinity rises by 14% and 6% for (C1) and (K1) respectively. Besides, with further increase in NaOH conditions, crystallinity augments and becomes equal to 58.6% and 66.34% for (C4) and (K4) respectively like the crystallinity of other fibres, for example, hemp (56%), kenaf (62.9%), and kusha (65.18%) as reported by Ravindra et al. [25]. This may be explained by the removal of some portion of amorphous materials like lignin, which ameliorates the packing of cellulose.

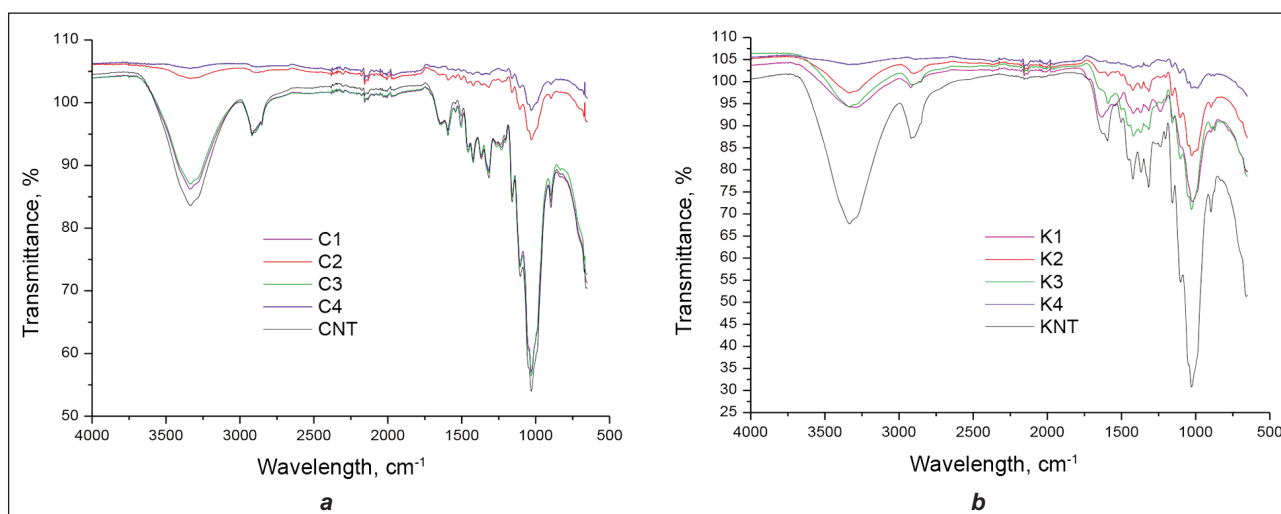


Fig. 7. ATR-FTIR Spectra analysis for: a – Cattail fibres; b – Kenaf fibres

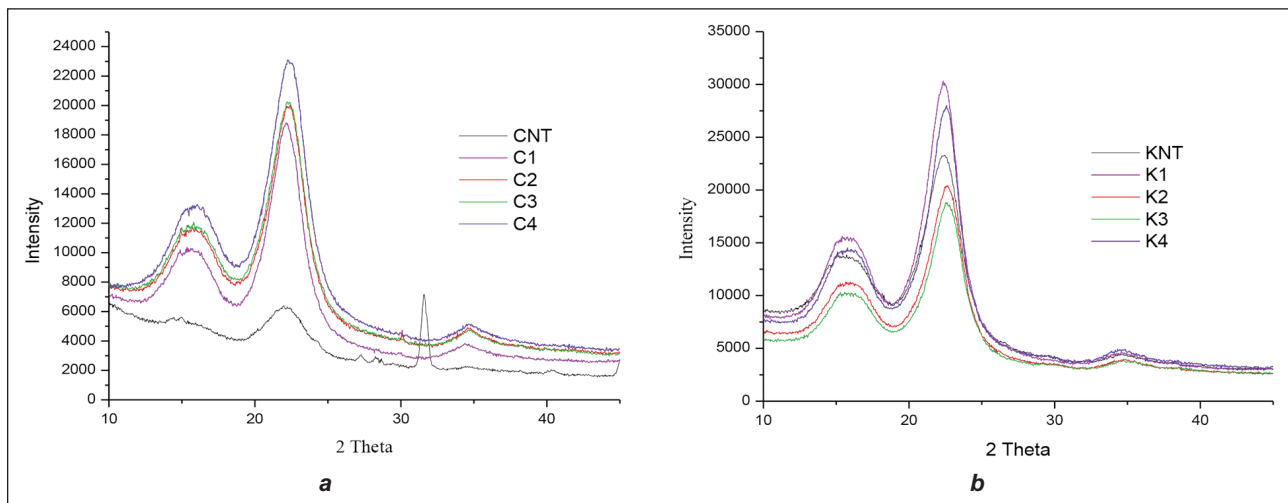


Fig. 8. X-ray diffraction pattern of: a – Cattail fibres; b – Kenaf fibres for different conditions

Table 2

CRYSTALLINITY INDEX OF CATTAIL AND KENAF FIBRES FOR THE DIFFERENT COMBINATIONS										
	CNT	KNT	C1	K1	C2	K2	C3	K3	C4	K4
Crystallinity (%)	38.54	52.42	43.86	55.39	53.25	60.3	56.5	59.4	58.6	66.34
CV (%)	12.4	10.96	15.46	10.48	14.78	12.45	16.45	13.14	17.82	11.45

CONCLUSION

Biodegradable lignocellulosic fibres are currently performed to reinforce composite materials thanks to their low cost, low density, and high performance. Research on novel fibres and the necessity to valorise the existent biomass is very interesting. In this study, Cattail and Kenaf fibres were extracted and characterized. Alkaline treatment led to the removal of amorphous compounds like lignin, pectin, and hemicellulose as reported in FT-IR analysis and chemical composition. The experimental work shows that diameter and linear density decreased after all treatment conditions and thinner properties were observed for Kenaf fibres. Mechanical properties confirmed better results after alkaline treatment and the best results were obtained for (C4) and (K3). X-ray findings confirmed enhancement in the crys-

tallinity index for both Kenaf and Cattail. From SEM micrographs, impurities were removed from the surface of fibres and the obtained fibres show a structure of composite fibre formed by several ultimate fibres. Even though the properties of Cattail fibres are found to be generally inferior to Kenaf fibre, this study suggests the possibility of introducing these fibres in the field of industry. Work is in progress to investigate the relationship between microstructure and mechanical properties. Also, the need to study the thermogravimetric behaviour to identify the thermal stability of the obtained fibres will be more needed to complete this study to make a reliable comparison between Kenaf and Cattail fibres quality. So, more research and work must be done to provide its characteristics to state the appropriate application like reinforcing bio composite, etc.

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