

Study of extraction and characterization of ultimate kenaf fibres

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ABSTRACT – REZUMAT

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This study proposed an extraction process of Tunisian kenaf fibres to obtain ultimate fibres with minimum aspect ratio, minimum retention capacity and high yield and high absorption capacity. The extraction process was performed by varying the treatment duration (120–180 min), the temperature (110–130°C), and the sodium concentration (10–40 g/l). After that, a factorial design, that has been built using statistical software Minitab.17, was followed to identify the optimum operating conditions. After the extraction process, kenaf fibres were used to produce dry-laid nonwoven fabrics. Results reveal that mixed treatment improves the absorption properties of fibres. To characterize these fibres, some properties were measured like morphological structure and absorption properties: absorption and retention capacity. The morphology of the cellulose fibres (length and, diameter distribution), obtained from the optimum process, was determined by measuring 300 fibres with «Leica» optical microscopy. Ultimate fibres extracted from kenaf had an absorption capacity of 12.5 g/g and a retention capacity of 0.65 g/g. Finally, the characteristics of the optimum ultimate kenaf were compared to those of other vegetable fibres.

Keywords: kenaf fibre, extraction, morphological structure, absorption capacity and retention capacity

Studiul extracției și caracterizării fibrelor finale de chenaf

Acest studiu a propus un proces de extracție a fibrelor de chenaf din Tunisia pentru obținerea fibrelor finale cu raport de aspect minim, capacitate minimă de retenție și randament ridicat și capacitate mare de absorbție. Procesul de extracție a fost realizat prin varierea duratei de tratament (120–180 min), a temperaturii (110–130°C) și a concentrației de sodiu (10–40 g/l). După aceea, a fost urmat un model factorial, care a fost construit folosind software-ul statistic Minitab.17, pentru a identifica condițiile optime de funcționare. După procesul de extracție, fibrele de chenaf au fost folosite pentru a produce nețesute prin fixare uscată. Rezultatele arată că tratamentul mixt îmbunătățește proprietățile de absorbție ale fibrelor. Pentru a caracteriza aceste fibre, au fost determinate unele proprietăți precum structura morfologică și proprietățile de absorbție: capacitatea de absorbție și retenție. Morfologia fibrelor celulozice (lungimea și distribuția diametrului), obținute în urma procesului optim, a fost determinată prin măsurarea a 300 de fibre prin microscopie optică „Leica”. Fibrele finale de chenaf extrase au avut o capacitate de absorbție de 12,5 g/g și o capacitate de retenție de 0,65 g/g. În cele din urmă, caracteristicile fibrelor de chenaf optime finale au fost comparate cu cele ale altor fibre vegetale.

Cuvinte-cheie: fibră de chenaf, extracție, structură morfologică, capacitatea de absorbție și capacitatea de retenție

INTRODUCTION

Improving natural fibre exploitation, particularly ligno-cellulosic fibres, has become one of the main tracks for sustainable manufacturing. Several recent research have focused on how raising extraction process efficiency while keeping into consideration the ecological concern. It has resulted in a renewed interest in cellulosic fibres [1, 2]. Many natural fibres, such as alfa, sisal, luffa, and kenaf, are of interest because they are environmentally friendly. In addition to their multiple uses, natural fibres have shown many advantageous properties such as lightness, resistance and flexibility which give them a wide perspective of application in the textile field. Twofold gains can be realized: these materials are recyclable and eco-friendly hence many advantages are taken in the automobile industry and medical applications. Many researches [3, 4] illustrated the application of agave fibres. Ben Marzoug and Saieb [5–7] presented the

utility of technical esparto fibres. Valcineide et al. have studied luffa fibres [8]. Thompson et al. have investigated henequen fibres, especially their absorbent properties [9]. According to their extraction methods, natural fibres have very different qualities. Indeed, one can obtain discontinuous and short fibres or continuous and long ones [10].

A multidisciplinary team of scientists, experts and farmers has been mobilized to plant kenaf in different regions of Tunisia. This allowed carrying out tests in a scientific way based on maximizing yield and reducing costs per hectare. In this work, we are interested in finding the appropriate method to extract Tunisian kenaf fibre directly from the stem and this is the difference and the novelty of our work. Indeed, the previous research works report that the extraction is carried out on the Liberian part previously extracted from the kenaf stem by retting. Extraction methods are presented in this work: enzymatic retting and

chemical extraction. The obtained fibres are characterized and a comparative study of their properties was carried out [15–19]. This work is aimed at developing an optimized extraction process of ultimate kenaf fibres from technical fibres obtained from a combined process using mechanical and chemical extraction.

MATERIALS AND METHODS

Materials

Kenaf (*Hibiscus cannabinus*), a herbaceous annual plant similar to jute, is quite popular in the Western world because of its eco-friendly nature. It has high biological efficacy and ecological adaptability. It can absorb CO₂ and NO₂ three to five times faster than forests and its deep roots can improve the soil. The plant has an ideal blend of long and short fibres for many paper and paperboard products (figure 1) [1].



Fig. 1. Kenaf stem

Technical fibre extraction

To extract fibres from the organic matrix, different methods can be used. These methods have a great influence on the fine structure of the obtained fibres [11]. In the current study, a mechanical method has been used to extract kenaf fibre. The harvested kenaf plant as a whole is processed in a mechanical fibre separator “horizontal opener machine”, making possible separation of the bast and core fibres possible. Technical fibres were not sufficiently separated. For this reason, and to ensure an effective separation, two passages in the “Shirley Analyser machine” were used (figure 2).

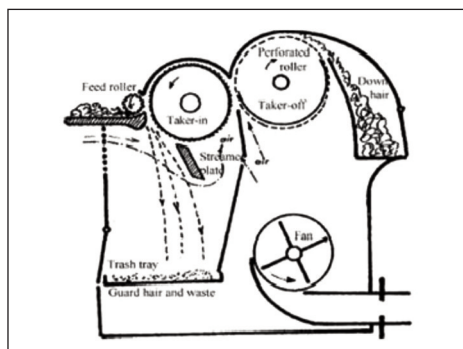


Fig. 2. Schematic of the Shirley Analyser

Ultimate fibre process

Long vegetal fibre extraction methods, such as linen, agave and kenaf, can be mechanical, chemical or biochemical. In this work, a chemical process was

used. This process preserves the fibre and is based on eliminating only non-cellulosic materials. Chemical extraction could be processed in different ways using, for example, sodium sulfate, bisulfite, kraft and so on.

In this study, a combined process was used. This process aimed to eliminate the non-cellulosic material using NaOH and bleaching process by the use of hydrogen peroxide. The extraction bath is as follows:

- 5 g dried fibres;
- Liquor ratio 1/40;
- 30 g/l of sodium hydroxide;
- 35 ml/l of hydrogen peroxide;
- 25 ml/l of stabilizer of hydrogen peroxide.

The treated fibres were thoroughly washed in warm water to remove dissolved substances. The resulting fibres were neutralized using a solution of acetic acid (10 ml/l), rinsed abundantly with water and dried under ambient conditions. Based on this process, several experiments were carried out to find the suitable extraction method for kenaf fibres. A series of preliminary experiments were conducted and they demonstrated that less than 120 min at 110°C and 20 g/l of soda concentration, the individual fibres were always stuck together and the extraction appeared to be impossible and more than the highest conditions, the extraction degraded the fibres and needed more time and energy. Therefore, the extraction process parameters were fixed as follows:

- the treatment duration D (min): from 120 to 180;
- temperature T (°C): from 110 to 130;
- the soda concentration C (g/L): from 20 to 40.

Hydrogen peroxide was added to the pulp bath for bleaching, with a concentration of 35 ml/l. A stabilizer (CHTT stabilizer A4) was also added to slow the rate of peroxide decomposition under alkaline conditions and combined with neutralized metal impurities that could catalyse the decomposition of H₂O₂ and induce fibre damage. The resulting pulp was washed, neutralized and finally dried at 105°C.

Methods

This study aims to evaluate the effect of extraction parameters on fibre properties. So, we measured some properties of kenaf fibre obtained. To characterize these fibres, some tests were made like morphological structure and absorption properties.

Extraction optimization

In this work, we used a composite Box-Behnken design to investigate the influence of extraction parameters (temperature, time and soda concentration) on the fibre properties, to determine the optimum operating conditions for the process and to establish statistical models for the prediction of fibre properties [12]. A Box-Behnken design is a three-level design in which all the design points are at the centre of the design and centered on the edges of the cube, equidistant from the centre. Key features of this design are as follows:

- allows efficient estimation of quadratic terms in a regression model;

Table 1

THE EXPERIMENTAL DESIGN USED				
Factor	Min	Centre	Max	Level
Treatment duration (mn)	120	150.00	180	3
Temperature (°C)	110	120.00	130	3
[NaOH] concentration (g/l)	20	30.00	40	3

- consists of fewer design points therefore, is less expensive to run than central composite designs.

So to better assess the effect of the temperature, the time and the soda concentration on the fibre properties, a statistical study was performed using Minitab 17. So ANOVA was used and diagrams of the main effects of these parameters and the interaction between them were drawn (figure 3) [12].

In this statistical study, the soda concentration, the duration of treatment and temperature are the input parameters. However, the yield, absorption, retention capacity, and L/D are the output parameters as presented in figure 3.

Desirability function

An experimental database was elaborated by varying extraction parameters. In this database, we used as input variables the temperature, treatment duration and soda concentration. The outputs were the satisfaction degrees. They were related to the extraction yield, Absorption, retention capacity and length/diameter ratio (L/D). We evaluated the satisfaction degree by using a mathematical function, in only one parameter that grouped these different degrees of satisfaction, and that permitted us to define a global desirability index. For each property influencing the global

satisfaction, we calculated the individual satisfaction degree and we affected a relative weight to indicate the property's importance. We used the desirability functions shown in figure 4, a and b in which we took into account the target "Y_{target}", and the importance of every property "Y_i" in the definition of global desirability.

In this study, we used two types of desirability functions "d_i": desirability function to maximize and to minimize. Thus, to maximize a property "Y_i", such as the yield and absorption capacity, the desirability function (shown in figure 4, a) had to be used, where d_i was calculated as follows:

$$d_i = 0 \text{ if } Y_i \leq Y_{min} \quad (1)$$

$$d_i = \left[\frac{Y_i - Y_{min}}{Y_{target} - Y_{min}} \right]^S \text{ if } Y_{min} \leq Y_i \leq Y_{target} \quad (2)$$

$$d_i = 1 \text{ if } Y_i \geq Y_{target} \quad (3)$$

To minimize a property "Y_i", such as the aspect ratio and retention capacity, the desirability function (shown in figure 4, b) had to be used, where d_i was calculated as follows:

$$d_i = 1 \text{ if } Y_i \leq Y_{target} \quad (1)$$

$$d_i = \left[\frac{Y_i - Y_{max}}{Y_{target} - Y_{max}} \right]^S \text{ if } Y_{target} \leq Y_i \leq Y_{max} \quad (2)$$

$$d_i = 0 \text{ if } Y_i \geq Y_{max} \quad (3)$$

For each property affecting the global desirability, we calculated the satisfaction degree "d_i" and we attributed a relative weight to indicate the property's importance. We grouped these different satisfaction degrees by using the Derringer and Suich desirability function [13] defined as follows:

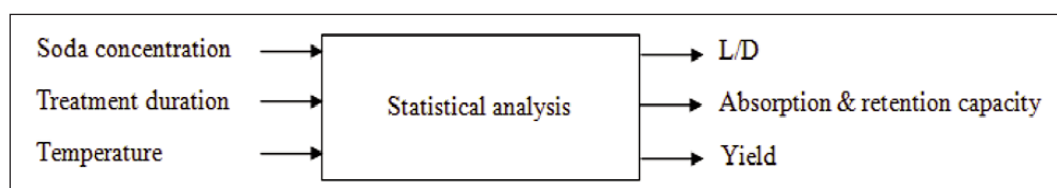


Fig. 3. Statical model

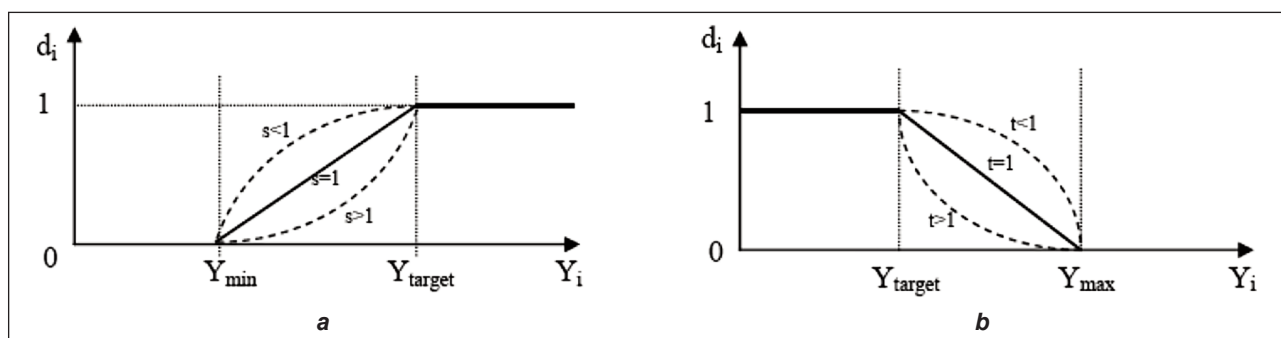


Fig. 4. Desirability function to: a – maximize; b – minimize

$$d_g = \sqrt[w]{d_1^{w_1} \times d_2^{w_2} \times \dots \times d_n^{w_n}} \quad (7)$$

where d_i is the individual property's desirability function Y_i , $i \in [1 \dots n]$, w_i – the weight of the property Y_i in the “dg” desirability function, w – the sum of w_i and n – the number of properties. The compromise between the properties (maximize yield and absorption capacity, minimize ratio aspect and retention capacity) was better when “dg” increased; it became “perfect” when “dg” was equal to 1. When the satisfaction degree “di” of the property Y_i was equal to 0, the response had a value outside of tolerance the function “dg” was equal to 0 and so the compromise was rejected.

RESULTS AND DISCUSSIONS

Morphological and physical characterization of technical fibre

Studies of morphologic characteristics can ensure a comparison between different extraction methods, give more information about the surface and evaluate a fibre diameter.

SEM results

A scanning electron microscopy (SEM) photograph of technical fibre illustrated that it was composed of individual cellulosic fibres bound together by lignin (figure 5).

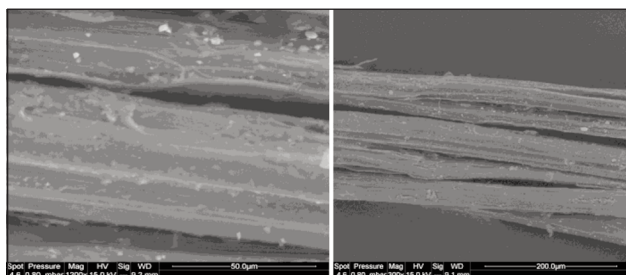


Fig. 5. SEM photographs of technical fibre

Diameter distribution

The results give us an average of 69,16 microns in diameter for crude fibre. Figure 6 represents the diameter distribution of the crude fibre, this distribution confirms the natural character of the fibre.

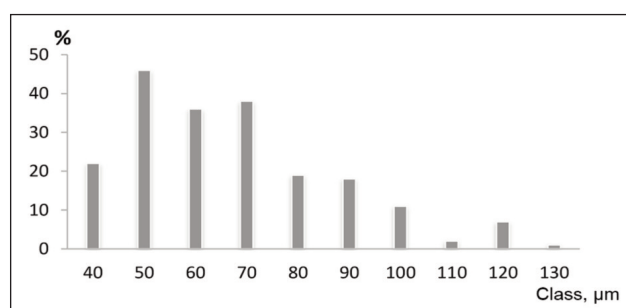


Fig. 6. Diameter distribution

The length distribution

The crude fibre presents an average length of 37 mm. Figure 7 presents the length distribution of the

crude fibre. This distribution is similar to all-natural fibre. In one batch of fibre length, there are different length classes of fibres. The variation of length compared to the average, was 51% which means that the batch of the raw fibres is not homogeneous and that the fibre is quite irregular.

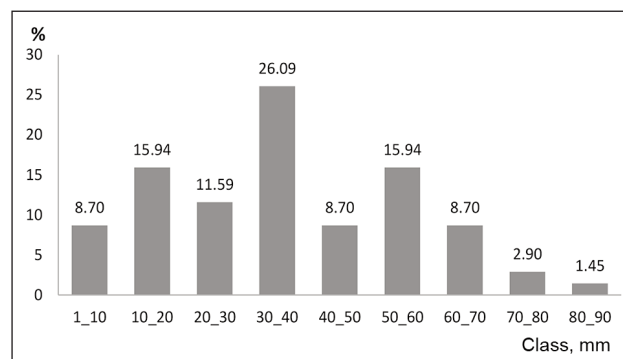


Fig. 7. Length distribution

Optimization of ultimate fibre extraction

The kenaf extracted fibres will be used for producing a wet-laid nonwoven textile used for insoles. For this reason, we optimize the extraction of fibre to have the appropriate characteristics of fibre for this use. Figure 8 presents the resulting ultimate fibres extracted from the technical Tunisian kenaf fibres.



Fig. 8. Kenaf fibres:
a – technical fibres; b – ultimate fibres

Statistical analysis

To evaluate the effect of extraction parameters on extraction yield, the diagrams of the main effect and the interaction diagrams were used.

It is observed, from figure 9, that the soda concentration and the temperature are the most influential parameters in the extraction yield. Figure 10 reveals that all parameters: Temperature (T), Treatment Duration (D) and soda concentration (C) are influential in aspect ratio. Through the exploitation of figure 11, it can be noted that all parameters are influencing the absorption capacity. The most influential parameter in retention capacity property is both the soda concentration and the duration. The temperature has less impact. To ensure the validity of the results obtained by the diagrams of the main effect or interaction diagrams, the ANOVA was used.

The results of this test are presented in table 2 where we notice:

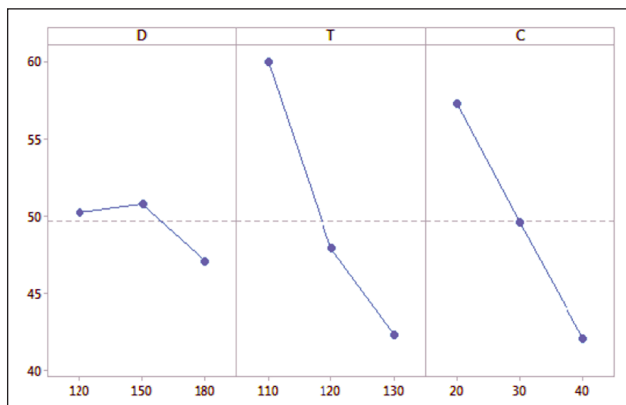


Fig. 9. Main effect diagram for extraction yield

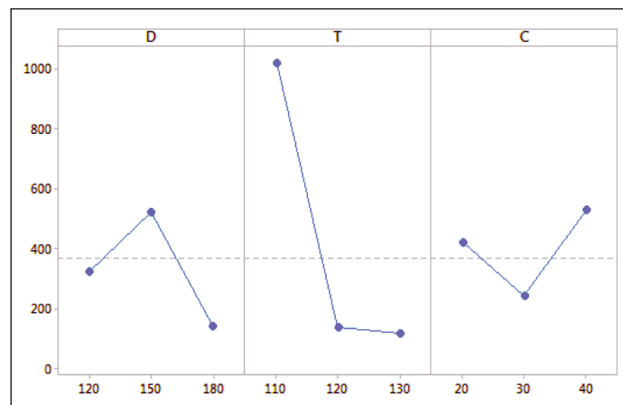


Fig. 10. Main effect diagram for L/D

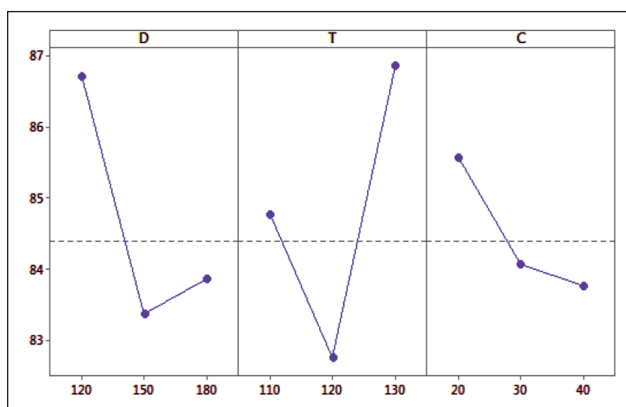


Fig. 11. Main effect diagram for absorption capacity

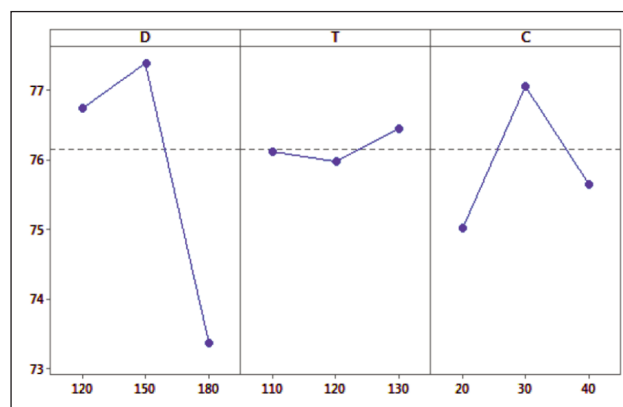


Fig. 12. Effect diagram for retention capacity

Table 2

ANOVA results					
Indicator	Parameter	DL	Sum of squares	F-value	p-value
Yield (%)	D	1	20.963	1.66	0.254
	T	1	626.757	49.73	0.001
	C	1	462.080	36.66	0.002
Aspect Ratio L/D	D	1	65418	0.50	0.511
	T	1	1622397	12.38	0.017
	C	1	24524	0.19	0.683
Absorption capacity (g/g)	D	1	16.289	23.80	0.005
	T	1	8.797	12.85	0.016
	C	1	6.5479	9.57	0.027
Retention capacity (g/g)	D	1	22.984	8.70	0.032
	T	1	0.219	0.08	0.785
	C	1	0.769	0.29	0.613

Note: DL – degree of freedom; p-value is the significance level for the hypothesis that the coefficient is zero; F-value – The F-value in an ANOVA is calculated as the variation between sample means/variation within the samples.

We conclude from table 2 and through the p value that all parameters affect all measured properties. The temperature, treatment duration and soda concentration are influential parameters, especially for absorption capacity. In this study, we choose to optimize the extraction process of kenaf fibre to obtain ultimate fibres with minimum aspect ratio, minimum

retention capacity, a high yield and absorption capacity. The coordinates of the optimum properties' values for the extraction parameters and properties obtained are prescribed in the following table.

This multiple-response optimization method allows having a compromise between the various answers or outputs. The quality of the solutions found is

EXTRACTION OPTIMISATION RESULTS					
Output	Yield (%)	Absorption capacity (g/g)	Retention capacity (g/g)	Aspect ratio (L/D)	Optimum extraction
Value «d _i »	64.58	12.48	0.65	199.95	-
Desirability «Dg» (%)	98.04	86.45	47.68	76.49	74.56

determined by the value of the global desirability «Dg», the closer this value is to 1 the better properties will be met. Through this optimization study based on the calculation of the individual desirability «d_i» of each output parameter and the combined desirability presenting the compromise between all the properties, the optimum conditions were set. From this study, we have chosen:

- [NaOH] = 20g/l;
- Temperature: 110°C;
- Duration: 180 min.

We choose these optimal values of temperature and duration of treatment the lowest for economic reasons. After choosing the optimum conditions for the extraction of fibres, we have to validate them. For this reason, we apply these conditions to extract fibres and we characterize them.

Characterization of the optimum process

Length and diameter of the fibre

The fibres' morphological analysis had a major utility. In fact, throughout the manufacture of the pulp, the cellulose fibres were subjected to physicochemical treatments with high intensity, and their morphology was modified. Therefore, it was important to limit the harmful effects of the processes on morphology to the profit of the positive effects. The measured pulp properties could be related to the fibres' morphological properties. For example, the more the fibres had a high length, the more the number of connections was important and the pulp mechanical resistance was significant.

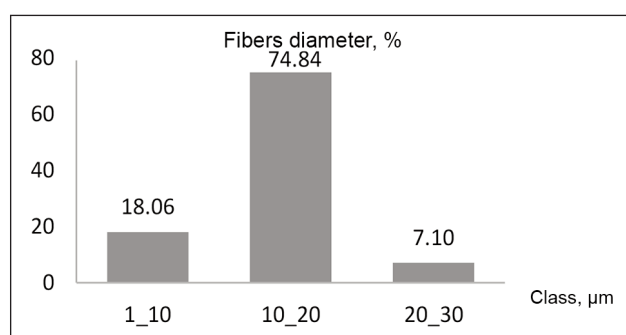


Fig. 13. Diameter distribution of Kenaf ultimate fibres

The results give us an average of 69.16 microns in diameter for crude fibre and 13.98 microns for chemically extracted fibre. This is due to the elimination of impurities stuck in the fibre. Figure 13 represents the diameter distribution of the ultimate fibre; this distribution confirms the natural character of the fibre.

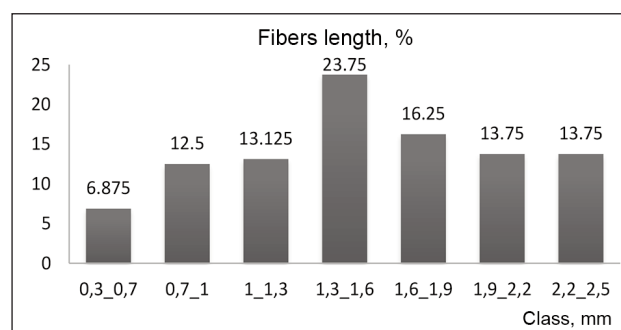


Fig. 14. Length distribution of kenaf ultimate fibre

The ultimate fibre presents an average length of 1.32 mm (figure 14).

SEM results

Studies of morphologic characteristics can ensure a comparison between different extraction methods, give more information about the surface and evaluate a fibre diameter. The longitudinal view of kenaf fibre (figure 15, a) shows that there are impurities accumulated on the surface of the mechanical extracted fibre. It is also lignin and pectin. These impurities were dissolved in figure 15, b by the chemical treatment which is why the treated fibre became thinner, and more flexible, and the ultimate fibres were separated.

The microstructure images of the kenaf fibre are presented in figure 15, a and b represent longitudinal and transversal views of the crude kenaf fibres. This figure shows that the kenaf fibre is covered with non-cellulose compounds. Figure 5 shows SEM images for fibres treated with enzymes which still have non-cellulosic materials on the surfaces of the fibre after extraction, this figure shows also an incomplete extraction. These deposits on the surface of the fibres are highly visible in the SEM images of the ultimate fibres and they are aligned parallel and always seem to be less stuck, the structure of the fibre shows that the surface of the fibre is clean and

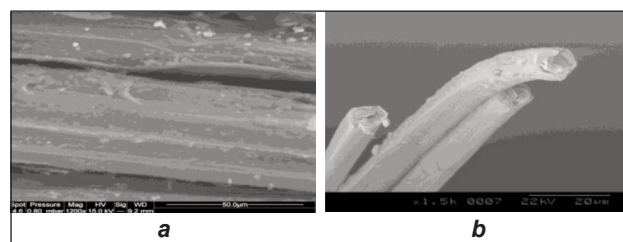


Fig. 15. Morphologic images: a – technical fibre; b – ultimate fibre

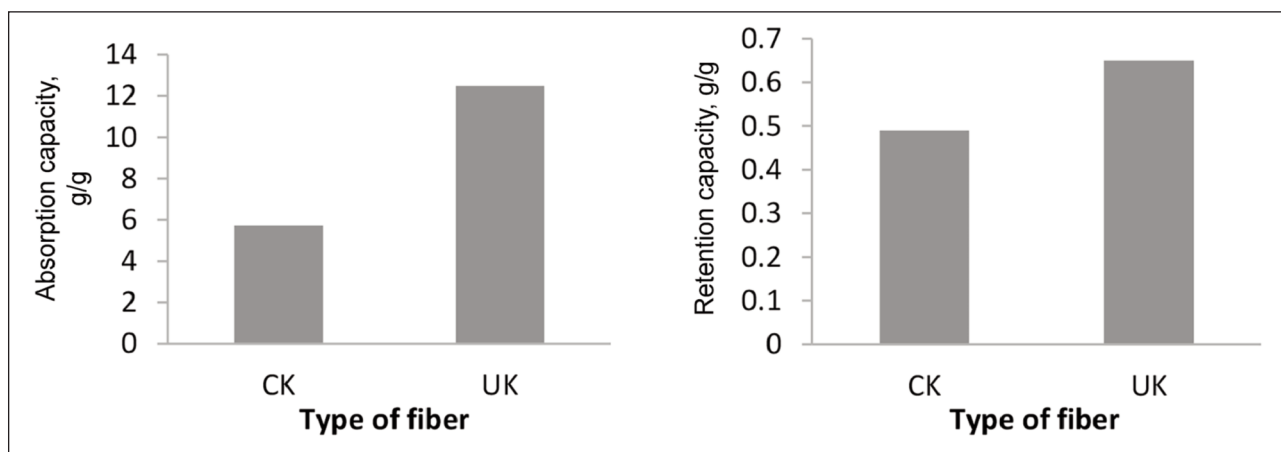


Fig. 16. Retention and absorption capacity of crude kenaf (CK) and ultimate Kenaf (UK) fibres

become smoother, the ultimate fibres are visible but not yet separated or deteriorated. The SEM images for chemically extracted fibres show that the fibres are degraded after prolonged exposure to high concentrations of sodium hydroxide. Although the ultimate fibres are visible, we can still see non-cellulosic substances covering the surface of the fibre and that is not completely removed. The transversal view of kenaf fibre shows that is composed of many fibre cells which are presented as aligned fibrils with materials cementing the fibres together. This material in the interfibrillar region is etched away as a result of the treatment done. The ultimate kenaf fibre has a circular section measuring approximately 12 microns in external diameter and 6,5 microns in internal diameter. We also note the presence of a very small lumen, which gives the fibre a thermal insulating power. So to conclude, vegetable fibres have generally a similar morphology and they can be simulated as a natural composite composed of ultimate fibre bundles of cellulose, linked together by gummy and waxy substances, constituting the matrix. The same result has been showed by other researches [16, 18, 19].

Absorption capacity and retention measurement

Absorption and retention capacities are linked to hydrophilic groups and amorphous zones. Figure 16 lists retention and absorption capacities for unmodified and modified kenaf fibres. The analysis of data shows that the treatment with NaOH/H₂O₂ yielded higher absorption and retention capacity values. The kenaf absorption capacity was approximately 12.5 g/g with a variation coefficient of 3%; this was higher than the alfa one (a value of 9.4 g/g) and the Agave one (a value of 10.5 g/g). This indicated that the wettability and penetrability of the material to liquid improved due to the removal of ligneous cement and the creation of supplementary hydrophilic sites consisting mainly of hydroxyl groups [14].

CONCLUSION

In other studies, we have chosen to work with a mixed extraction process that combines a NaOH extraction process and an oxygen peroxide bleaching process. We started by studying the kenaf fibre mechanically extracted, then we dealt with optimizing the extraction process to obtain ultimate fibres. A composite Box-Behnken design was used to identify the optimum operating conditions (temperature, treatment duration and soda concentration) for Tunisian kenaf fibre. The optimum extraction conditions were found by using 20g/l soda concentration, time =180 min and temperature = 110°C. In the second step, we investigated the control test (yield, morphological properties, absorption and retention capacity). Once extraction was achieved, characterization of ultimate kenaf fibres was done. The ultimate kenaf fibres obtained had a capacity absorption was about 12.5 g/g and an average diameter of 13.98 µm. The arithmetic length was about 1.32 mm. Tunisian kenaf fibres could potentially be utilized for many applications such as paper products including handicrafts, geotextiles, filters, packaging, baby diapers and composites.

The chemical and physical analysis of the kenaf fibres were determined and found suitable as textile-grade fibre for various applications. The moisture content of the fibres was found suitable which will further enhance the comfort properties of the fabric. The cellulose content of the fibre was found satisfactory. The increase in cellulose per cent will give higher strength to the fibre.

The morphological properties of kenaf bast fibres were found to be significantly affected by the extraction methods used in this work. In our case, we are interested in applying these fibres in the manufacturing of wet-laid nonwoven which will be put in insoles.

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