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Research Article

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Study of the Mechanical Properties of Hemp Fibers Treated with Supercritical CO_2 Fluid

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Abstract

The aim of this study is to analyze the efficiency of supercritical CO_2 ($scCO_2$) fluid technology for the pretreatment of hemp fiber materials. In this investigation, although the limited conditions studied here, the test results show that we have obtained a promising quality of hemp fibers after this specific pretreatment in presence of swelling agent. The $scCO_2$ treatment appeared to remove non cellulosic compounds from the fiber, thereby resulting in improvement in its appearance, and decrease in its linear density. We should continue this investigation through further studies, since it is an environmentally benign process.

Keywords

Supercritical fluid; Hemp fiber; Mechanical properties; Fineness; Tensile properties

Introduction

For the last two decades, natural cellulose-based fibers are increasingly gaining attention as their application is diversified into textile [1] composite and engineering end uses such as building materials [2]. They have started to be considered as alternatives to conventional man-made fibers in various applications such as transportation, construction and packaging. Some of them like hemp, flax, kenaf, jute, and sisal, which have been utilized as fiber sources since historical times, have become the focus of research attention once again [3].

Generally, Lignocellulosic agro-based materials show a composite structure which is constituted by an organic matrix reinforced by bundles of cellulose microfibers. The organic matrix is mainly composed of hemicelluloses, pectin, lignin, waxes, and proteins [4].

In our case, hemp fibers contain 70.2%-74.4% cellulose, 3.7%-5.7% lignin, 17.9%-22.4% hemicellulose, 0.9% pectin, and 0.8% wax [5]. They are among the most suitable fibers for use in various textile applications. However, their extraction is a very complicated process. Traditional treatments such as dew retting, tank retting and chemical methods have been used to separate hemp fiber bundles and remove non-cellulosic materials. Unfortunately, these traditional treatments have many disadvantages like environmental pollution, fiber degradation and long-term treatment [6].

Other possibilities for refining coarse fibre bundles are the steam explosion process [7] the ultrasonic technique [8] and the enzymatic treatment [9]. All of them are still in exploratory stage.

In this study, the $scCO_2$ fluid technique was employed in the pretreatment of hemp fibers because it is an environmentally benign process. It is found here that the non-cellulosic compounds around the fibers could be removed apparently by $scCO_2$ under appropriate conditions and in presence of swelling agent [10,11].

Materials and Test Methods

Materials

Hemp fibers were sourced by CDE (Chanvriers De l'Est) in Eastern France. They have been treated at 3 different conditions in this study using $scCO_2$ as shown in Table 1.

Test Methods

Pretreatment in $scCO_2$: All experiments were performed on a batch-type supercritical extractor. The pretreatment of the hemp fibers was conducted in a 10 cm^3 sample cartridge [12,13]. After heating the sample to a specific temperature, the $scCO_2$ was injected into the stainless-steel vessel via the high-pressure syringe pump to the desired pressure. The testing sample was then treated for a certain time at a constant temperature and pressure. In order to enhance the swelling effect of $scCO_2$ for the hemp fibers, a certain amount of swelling agent was added into the $scCO_2$ treating system. After the supercritical process, the sample was taken out after decompression and dried.

Measurments: After $scCO_2$ treatment, hemp fibre samples were cut into 55 mm in length to be used as specimens in tensile tests. Based on the textile standards, the specimens were conditioned at 20 ± 2°C and 65% ± 2% relative humidity for 48 hours before tests were started [14].

Fiber fineness: Fibre fineness is defined as linear density in terms of textile technology. The NF EN 13392 test method (13392 2001) has been used to calculate the fiber linear density as shown in the equation (1). Each fiber was first weighed using an electronic micro-weighting balance [15].

$$T\ (Tex)=m(g)/L(m).10^3 \quad (1)$$

With T: Fiber linear density (Tex)

m: Fiber weight (gram)

L: Fiber length (meter)

The fiber specimens were conditioned at 20 ± 2°C and 65% ± 2% relative humidity for 48 hours before every test [16].

Tensile properties: The tensile properties of hemp fibers were determined according to single fiber tensile test as described by ASTM

Table 1: Experimental conditions.

Samples	Time (min)	Temperature (°C)	Pressure (MPa)
Sample 1 (S_1)	60	100	25
Sample 2 (S_2)	60	120	25
Sample 3 (S_3)	60	150	25

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D3822 standard (ASTMD3822 2020) using MTS tensile Tester. For each test, the 55 mm long specimen was placed on a cardboard which had a window (35 mm) to define the clamping length of test specimen (Figure 1). Both the lower and upper ends (10 mm each) of fiber were glued to the cardboard [17]. Thus, the fiber was fixed across the cardboard window. The cardboard was fixed in the tensile testing machine, and was cut along the discontinuous lines as shown in Figure 1, so that the applied load could be transmitted through the fiber only. The load-elongation curve was then recorded for each specimen during the test.

Parameters generated from the measurements: Specific tensile strength, strain and the work of rupture were generated from the load-elongation curves (Saville 1999).

Strain: Elongations at break were read from the load-elongation curves and converted to maximum strain, ε_{max} (%) using equation 2.

$$\varepsilon_{max}(\%) = \frac{l - l_0}{l_0} \cdot 100 \quad (2)$$

Where:

ε_{max}(%) is maximum strain in %; l is elongation at break in mm; l_0 is initial length in mm (35 mm).

Specific tensile strength: The specific tensile strength was expressed as tenacity-strength related to fineness (load per unit fineness). Maximum loads were read from the load-elongation curves and specific tensile strength was determined using equation 3.

$$Tenacity\ (cN/Tex) = \frac{F_{max}(cN)}{Linear\ density\ (Tex)} \quad (3)$$

Where: F_{max} is the maximum load in cN.

Work of fracture: The work of fracture is determined as the area under the load elongation curve. It indicates the total energy required to break the fiber. Higher work of rupture means that the fiber is more durable. It is calculated using numerical integration as shown in equation 4.

$$W = \left[\left(\frac{F_1 + F_2}{2}\right)\Delta l + \left(\frac{F_2 + F_3}{2}\right)\Delta l + \ldots + \left(\frac{F_{n-1} + F_n}{2}\right)\Delta l\right] \quad (4)$$

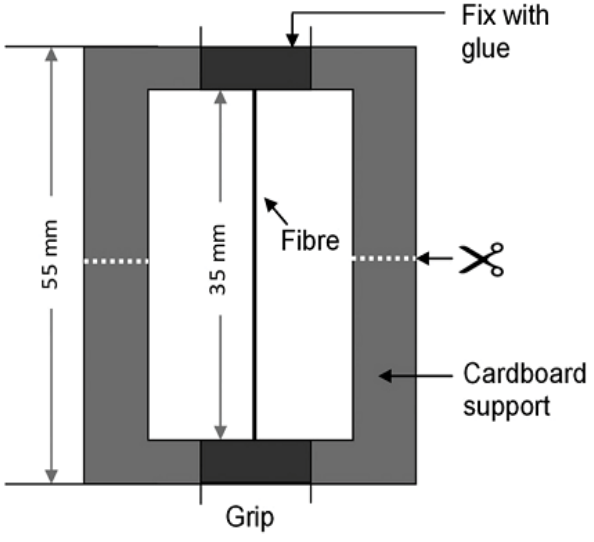

Figure 1: Set-up for tensile testing of single fiber.

Where:

W is work of rupture in mJ; F_1 is the first data point in N; F_2 is the second data point in N; and F_n is the n^{th} data point in N; Δl is elongation interval in mm.

Surface morphology analysis: Scanning electron microscopy (SEM) test was conducted to examine the surface morphology of untreated and treated hemp fibers [18]. The HITACHI model was used in this study for image acquisition at a voltage of 20 kV.

Fourier transform infrared spectroscopy analysis: The purpose of FTIR test was to observe the change of functional groups, which will help to confirm the removal of impurities of hemp fibers after $scCO_2$ treatment. The FTIR test was conducted using Bruker IFS 66/S spectrometer [19]. Hemp fibers were ground into fine particles for Fourrier transform infrared spectroscopy (FTIR) analysis.

Results and Discussions

Typical stress-strain curve

Stress–strain curves for treated hemp fibers are displayed in Figure 2. They are obtained by simple tensile test. Their forms are relatively similar.

Fineness

The mean value of the linear density of untreated hemp fibers was 16.50 Tex with a coefficient of variation of 21%. It seems that it follows a decreasing trend while increasing the temperature of the $scCO_2$ treatment process (Figure 3). For higher temperature 150°C, the linear density was 5.3 Tex with a coefficient of variation of 29.7%. That means that hemp fibers are finer after $scCO_2$ treatment, which

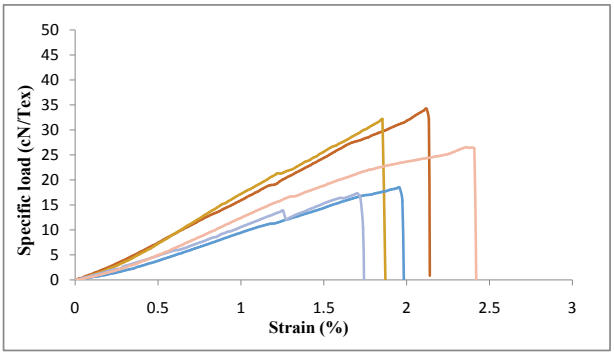

Figure 2: Example of load-elongation curves for hemp treated fibers.

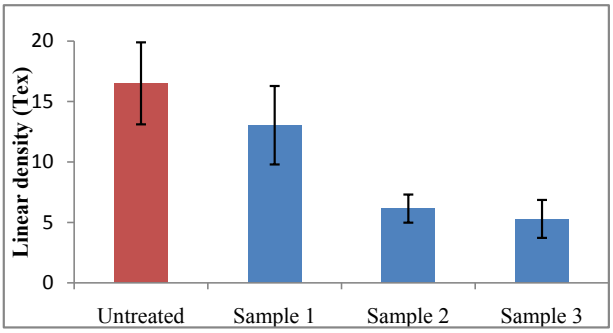

Figure 3: Hemp fiber fineness.

is very interesting result in terms of fiber quality [20]. SEM analysis (Figure 4) shows less impurities at the surface of treated fibers, which explain the efficiency of the treatment. The decrease of the weight per unit of length and consequently the linear density (Tex) of the treated fibers seems to be the result of the cleaning of the impurities such as lignin present around the hemp fibers.

Strain

Figure 4 shows strain values of untreated and treated hemp fibers. They vary from 0.79% for the untreated fibers to 2.5 % for the treated ones. These small values are attributed in general to the brittle aspect of this kind of fibers [21]. It seems that the strain follows an increasing trend with temperature. At higher temperature of the process, it seems that there are less impurities in the hemp fibers, which let them single and more extensible. These impurities such as lignin are like a matrix around fibers. For that, we think that untreated hemp fibers are stronger and less extensible.

Specific tensile strength

The specific tensile strength is displayed in Figure 5 for untreated and treated hemp fibers. It seems that it follows a decreasing trend while increasing the temperature of the treatment process (Figure 5). Its mean value was 44.23 cN/Tex with a coefficient of variation of 14.41% for the untreated hemp fibers. For high temperature 150°C, the specific tensile strength was 36 cN/Tex, 18.6% lower than the untreated hemp fiber [22]. This could be explained by the fact that higher temperature promotes the thermal expansion of hemp fibers, resulting in the increase of their free volume. Therefore, the CO_2 molecules can more easily diffuse into the hemp fibers and extract the impurities. It seems that untreated hemp fibers are stronger mainly because of the presence of the impurities such as lignin compound around the fiber.

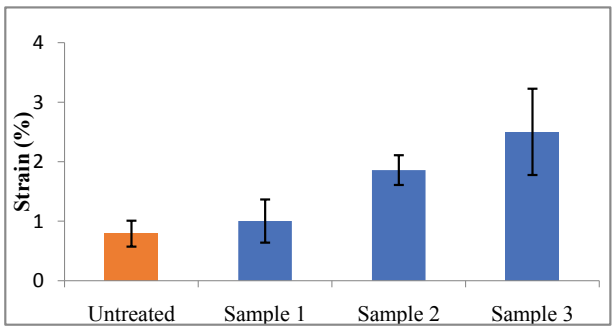

Figure 4: Strain behaviour.

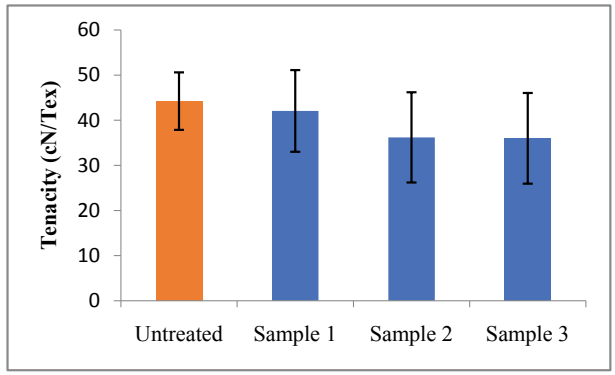

Figure 5: Tenacity behaviour.

Work of rupture

Figure 6 shows the work of fracture for raw and degummed hemp fibers. The effect of temperature on this work seemed to follow a decreasing trend (Figure 6) with the highest W being 0.27 mJ/Tex with a coefficient of variation of 21.92% for 100°C of the $scCO_2$ treatment (S_1). This trend was similar to that of the tenacity and was the reverse of the trend observed for strain.

Morphology analysis

The scanning electron microscopic (SEM) images of untreated and treated hemp fibers are displayed in Figure 7. As shown, the surface of untreated hemp fiber seemed to have impurities present on the surface. After treatment, fibers become apparently smoother and looked cleaner. The $scCO_2$ treatment was able to remove some of these impurities. The sample S_3, which was treated at 150°C, has less impurities at the surface compared to the others[23].

FTIR analysis

Figure 8 shows FTIR spectra for untreated and treated hemp fibers. The peak at 3325 cm^{-1} represents the O-H stretching vibration

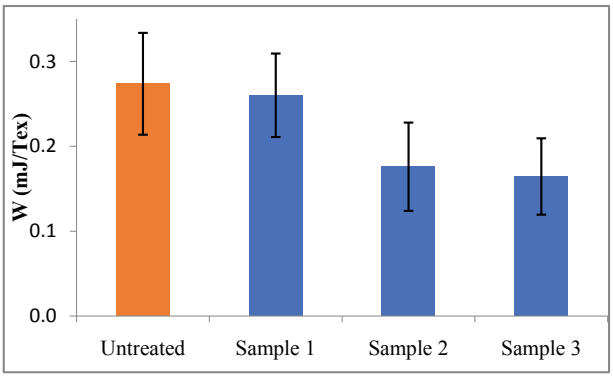

Figure 6: Work of fracture.

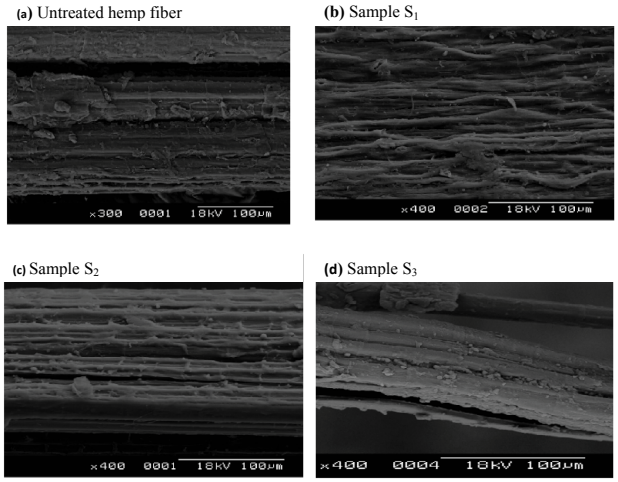

Figure 7: SEM images of hemp fibers before and after $scCO_2$ treatment.

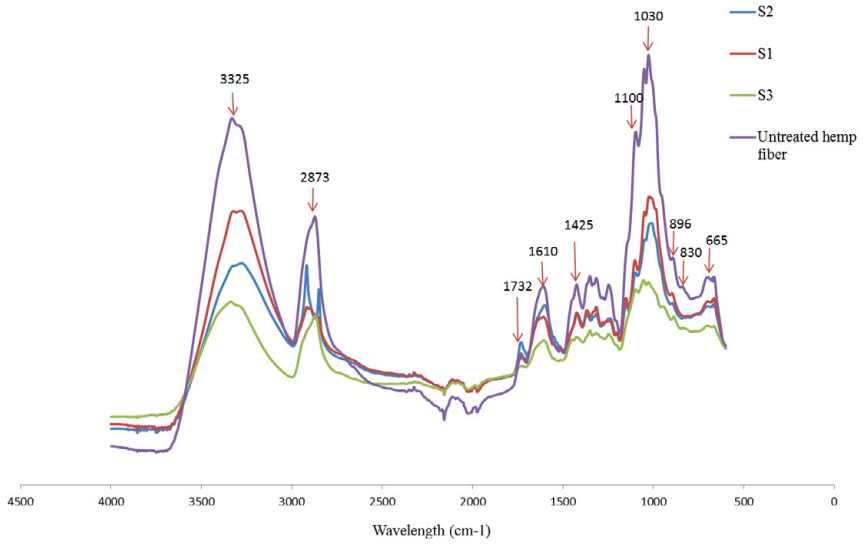

Figure 8: FTIR spectra of untreated and treated hemp fibers.

and hydrogen bond of hydroxyl groups assigned to intramolecular or intermolecular hydrogen bonding and free OH hydroxyl [24]. The peak at 2873 cm^{-1} represents C–H stretching vibration of methyl and methylene groups in cellulose and hemicellulose [24]. The peak around 1732 cm^{-1} is attributed to the presence of the carboxylic ester in pectin and wax. The reduction of its intensity is especially pronounced for the sample S3. This could be explained by the partial removal of these non-cellulosic components. The peak around 1610 cm^{-1} represents the C=O stretching in lignin [25-27]. The weakening of its intensity is due potentially to the partial degradation of lignin. The peak at 1425 cm^{-1} is associated to the aromatic ring of lignin, its decrease after treatment confirms the partial degradation of this component. The peak around 896 cm^{-1}, which represents the β-D glucosidic bond in cellulose remained unaffected after treatment [28].

Conclusion

This study deals with the application of $scCO_2$ fluid technology for the pretreatment of hemp fibers. It was found that the $scCO_2$ fluid could remove the impurities of the hemp fibers in presence of some swelling agent, especially at higher temperature. In comparison with the conventionally method where a large amount of strong alkaline solutions is employed, the $scCO_2$ fluid is a better way to eliminate non cellulosic compounds from the hemp fibers. We should continue this investigation in order to determine the best configuration in terms of temperature, pression and time for hemp pretreatment fibers.

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