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Impact of Surface Treatment of Flax Fibers on Tensile Mechanical Properties Accompanied by a Statistical Study

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Abstract: In this work, a surface treatment with sodium bicarbonate ($NaHCO_3$) at different concentrations of 4 and 8% is applied to the surface of the flax fiber for a period of 80 hours at room temperature. The purpose of this study is to observe the effect of different treatment processes on the flax fiber, i.e. on its mechanical properties such as: stress and strain at break and Young's modulus. A major test campaign of more than 240 tests is carried out. Due to the variability of the plant fibers, more than 80 samples were tested for each group at a gauge length (GL = 10 mm). The results of the quasi-static tensile tests have a large dispersion which makes it possible to measure the degree of variability in the stress and strain deformation and the Young's modulus of the fiber. This degree of variability has been studied by means and statistical tools such as the Weibull distribution at two.

Keywords: Natural fiber, flax fiber, Quasi-static traction, Weibull statistics.

1. Introduction

Underdeveloped countries such as Algeria or the progressive depletion of oil help to make the search for new sources of energy and raw materials indispensable. In addition, the problem of global warming encourages manufacturers to seek alternative solutions to fossil fuels and to put in place comprehensive strategies for sustainable development. Cellulosic fibers are an excellent solution because they come from renewable resources to replace traditional materials.

Thus, there is a strong interest in cellulosic fibers for the purpose of substituting mineral fillers (glass fibers) and for reinforcing polymer matrices in order to obtain a bio-composite. The most used cellulosic fibers in bio composites are: flax, hemp, kenaf, banana, palm and sisal [1-6]. To improve fiber / matrix adhesion ecological treatments are applied. These bio composites become feasible for vast fields of application and the automotive industry is the first applicant [7-10]. This sector is more and more interested in plant fibers in order to use renewable and perennial materials or in secondary structures in applications that respond to the concern for the preservation of the environment. [11-22]. All natural fibers are composed of Cellulose, hemicelluloses and Lignin, these elements have an impact on the mechanical propertiess [23-26].

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Research on the characterization of plant fibers has gained importance in recent years. Hafsi et al. [15] studied the possibility of incorporating date palm fibers to reinforce the concrete for use in hot areas. To do this, they chose date palm fiber which was added to gypsum samples in the form of cutting layers at different volume fractions, in order to determine the extent of their impact on the improvement of thermal performance. The results were very satisfactory, reaching a 16% improvement rate for gypsum samples reinforced with a single cut fiber form and 32% of the samples reinforced with layered fibers.

An experimental study presented by Saaidia et al. [16] on the mechano-physical properties accompanied by a statistical study of treated and untreated jute yarns. The mechanical properties namely the Young's modulus, the stress and the deformation at break had an increase at low concentrations of NaOH (2%) and at low immersion time (2h) of 5-34% for stress from (117.7 to 178.4 MPa) and from 5 to 25% for the Young's modulus (19.78 to 26.4 GPa) relative to the untreated fibers against mechanical properties at high concentrations of NaOH (5%, 10% and 25%).

A recent study presented by Beloudah et al. [17] characterizes a new Lygeum Spartum L (LS) natural cellulosic fiber to evaluate their potential use as reinforcement in composites. The LS stems were cut to a length of about 50-97 cm, washed with distilled water to remove contaminants and adhering dirt. After that, the stems were completely immersed in the container filled with fresh water and covered for a period of 15 days at room temperature to undergo microbial degradation. The fibers were subjected to a quasi-static tensile load with a constant speed of 1 mm / min at a measurement length of 40 mm. The average values of the tensile stress, the tensile strain and the Young's modulus of 30 samples were respectively found to be 280 MPa, 1.49 - 3.74% and 13.2 GPa.

The aim of this work is to study the quasi-static tensile mechanical behavior of more than 240 sample populations of untreated and treated $NaHCO_3$ flax fibers at different concentrations with an emergence time of 80 h. Next, the results obtained were analyzed by applying probabilistic and statistical approaches such as the Weibull distribution at two and three parameters using the Last square-LS method.

2. Experimental techniques

Natural flax fibers were emerged in a solution of $NaHCO_3$ (sodium bicarbonate) with different concentrations (4% and 8%) making a total of 240 fibers treated for a period of 80 hours at room temperature. Then, the fibers are emerged in the distilled water for 10 to 20 minutes to remove any impurity, and finally the fibers were dried in an oven at a temperature of 55 ° C for a period of 4 hours.

Before the quasi-static tensile test, the diameters of the flax fiber are measured at six locations all along the fiber. These measurements were taken using a ZEISS optical microscope equipped with a digitally controlled Moticam 2500 camera driven by a MoticImages Plus V2.0 image processing program. Six diameter measurements are thus obtained for each fiber and the diameter retained is the average of these six values. The average diameter measured is 18.68 ± 3.43 μm. The section of the fiber is considered circular. This area is calculated from the average diameter of the fiber.

The gauge length used in this work is therefore 10 mm. The mechanical properties (tensile strength, Young's modulus and ultimate strain) of the flax fiber are determined according to ASTM D 3822-07 using a measurement length (GL) of 10 mm. Due to the variability of natural fibers 80 samples are tested for each lot, in total more than 240 tests performed. The tests were conducted on a Zwick / Roell universal traction machine with a capacity of 2.5 kN. The tensile tests were carried out with a speed of 1 mm / min. The Young's modulus was calculated in the elastic part at 0.1-0.8% of the deformation value by determining the slope of the stress / strain curve. The mechanical properties namely the Young's modulus, the breaking stress and the breaking strain were calculated individually for each fiber tested. All the tests were carried out at an ambient temperature of 20 ° C. and a relative humidity of approximately 45%.

The surfaces of the untreated fibers were also examined using a JSM-5600 Scanning Electron Microscope (SEM) (Figure. 1b). The fibers were covered with a thin layer of gold to make it conductive.

3. Results and discussions

The flax fibers tested in static tension under the same conditions are grouped in three series of 80 specimens each, so a total of 240 fibers were used in this work. The figure 2 shows the typical curves in stress / deformation behavior of the untreated and treated tensile fiber unit fiber. It is obvious that there is a significant dispersion in the results, this is a character of natural fibers, hence the need for a statistical study.

It appears that the flax fiber and untreated tensile tested has the same pace (behavior). Indeed, this behavior is characterized by an elastic quasi-linear region until the abrupt rupture of the fiber. In other words the flax fiber shows a fragile behavior with a sudden drop in the load.

The results of the tensile tests of a single natural flax fiber are difficult to achieve because of the very small diameter of the fibers (of the order of 18 μm) and to be analyzed because of the great dispersion recorded. (figure 3). This dispersion can be mainly related to several factors Belaadi et al [6] and Silva et al. [18]: parameters / test conditions, characteristics of the center of the fiber and the measurement of its section. The test parameters that could influence the results can be: instrumentation accuracy, measurement length, strain rate, machine grip type, and machine compliance. Regarding the characteristics of the plant, the age, type of treatment (fiber extraction mechanism) as well as the microstructure of the fiber.

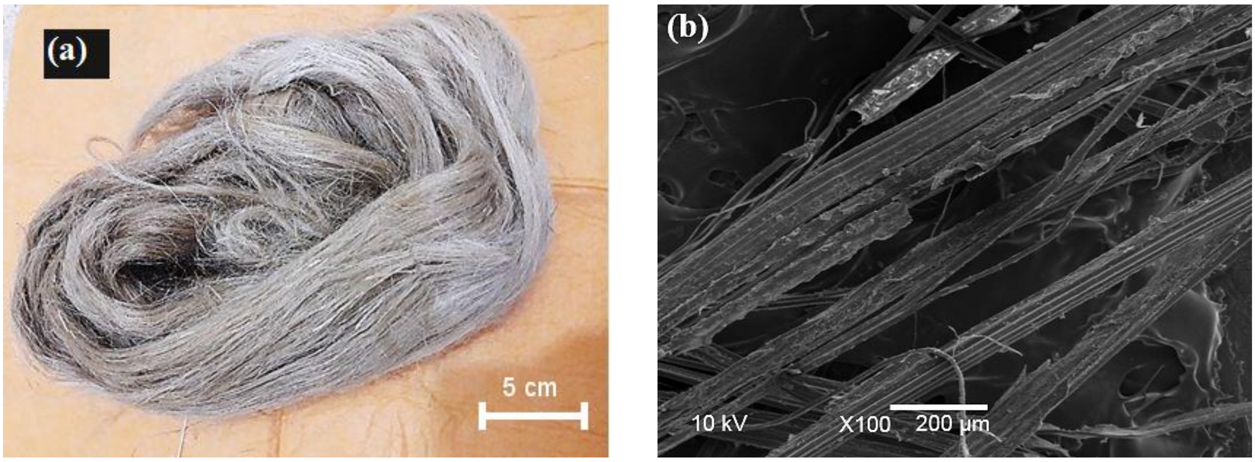

Fig1. Untreated flax fibers (a) sample used in this work and (b) Longitudinal SEM micrograph obtained by SEM.

The average values of the mechanical properties of the 240 validated specimens of the untreated flax fibers treated at the different concentrations of $NaHCO_3$ are given in Table 1. The reading of this table shows the rather significant dispersions of the values of the mechanical properties obtained, this shows although we have to deal with a very heterogeneous material and therefore a statistical treatment is necessary.

The effect of the bicarbonate treatment is mentioned in (Table 1) and the increase in mechanical properties strongly depends on the concentration of the chemical used ($NaHCO_3$) and the duration of the treatment [1-2,5]. By way of example, the untreated fiber has the average mechanical properties of the tensile stress and the Young's modulus respectively: 1045 MPa and 51.32 GPa. On the other hand, increases in the breaking stress of 22% and 40% are recorded respectively for treated fibers (Table 1), for a duration of 80 hours, in a $NaHCO_3$ solution having concentrations of 4 and 8%, then that the increases in Young's modulus are: 41 and 82%.

Table 1 allows us to point out that the treated flax fibers, for a duration of 80h, at 8% of $NaHCO_3$ has the best behavior allowing to have the average value of the highest stress (1473 MPa) this is in good concordance with Fiore et al. [5] for sisal fiber for the same duration of treatment which finds a stress value of 930 MPa with a concentration of 10%. While, the chemical treatments with $NaHCO_3$ have little influence on the deformation at break is the elastic behavior that is a characteristic of flax fiber.

The statistical processing of the results makes it possible to adjust the resistance data to a distribution law in order to estimate the average mechanical characteristics of the material. In general, the Weibull distribution or the lognormal distribution is the best candidates for the mechanical properties data. The two-parameter Weibull distribution was applied for the mechanical properties ie the tensile stress and the Young's modulus which showed a large dispersion of the results (Figure. 4), so the Weibull equation is as follows [4,16]:

$$F(\chi) = \exp\left[-\left(\frac{\chi}{\chi_0}\right)^{m_x}\right] \tag{1}$$

Where $F(\chi)$ is the probability of survival depending on the parameter χ, m is a dimensionless shape parameter directly related to the dispersions of the experimental data, and χ_0 is a local parameter representing an average value of the parameter χ (minimum life of stress, strain and modulus), [4, 16, 18, 27]. The value F is evaluated by a metric estimator (value of an average rank) [17-19]:

$$F_i = \frac{i}{n+1} \tag{2}$$

Where i is the rand of the $i^{\text{ème}}$ data point and n the number of points.

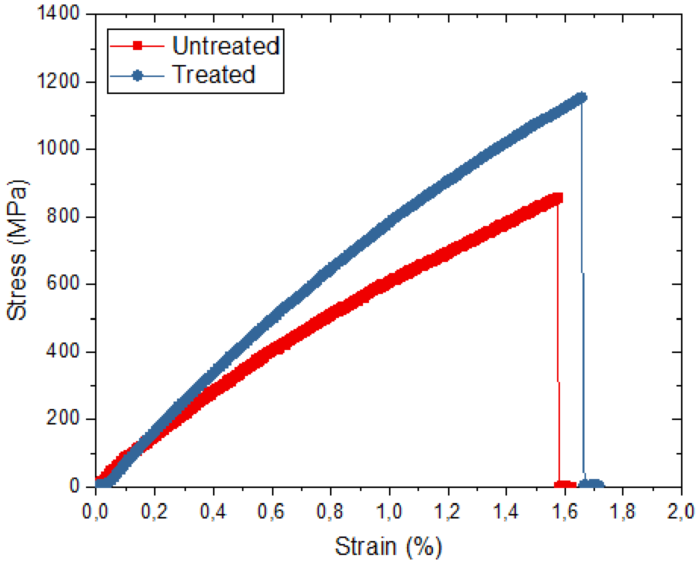

Fig2. Stress-strain curve of an untreated flax fiber compared with that treated.

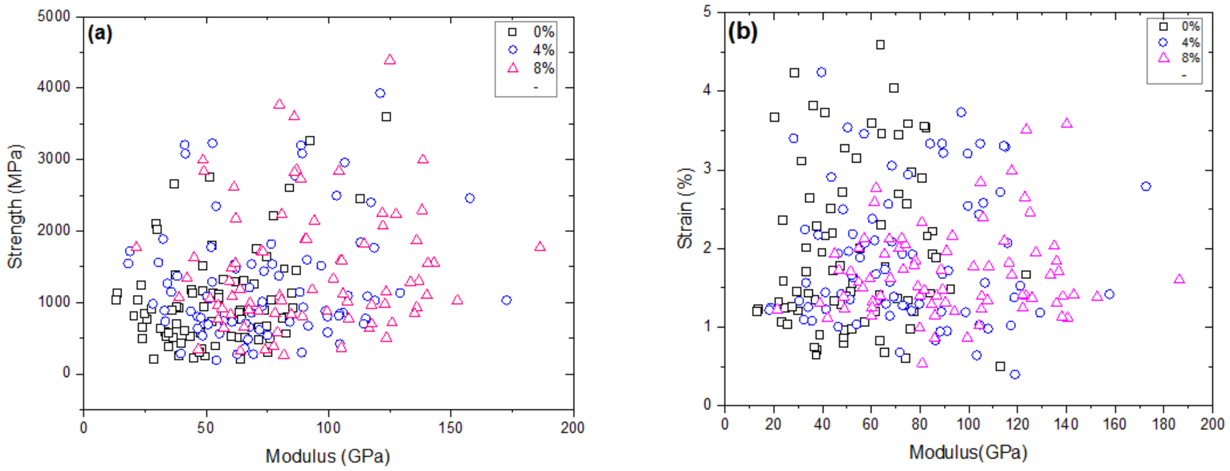

Fig3. Mechanical properties as a function of the Young's modulus of untreated flax fibers treated at different concentrations of $NaHCO_3$

The parametres m and χ_0 are obtained from a plot of a straight line of Weibull $\ln[\ln(1/1-F)]$ in terms of $\ln(\chi)$, therefore, a linear model is obtained. The two and three parameter Weibull distributions were applied for the mechanical properties namely the breaking stress and the Young's modulus which showed a large dispersion of the results (Figure 3 and Figure 4) using the statistical software MINITAB version 16.

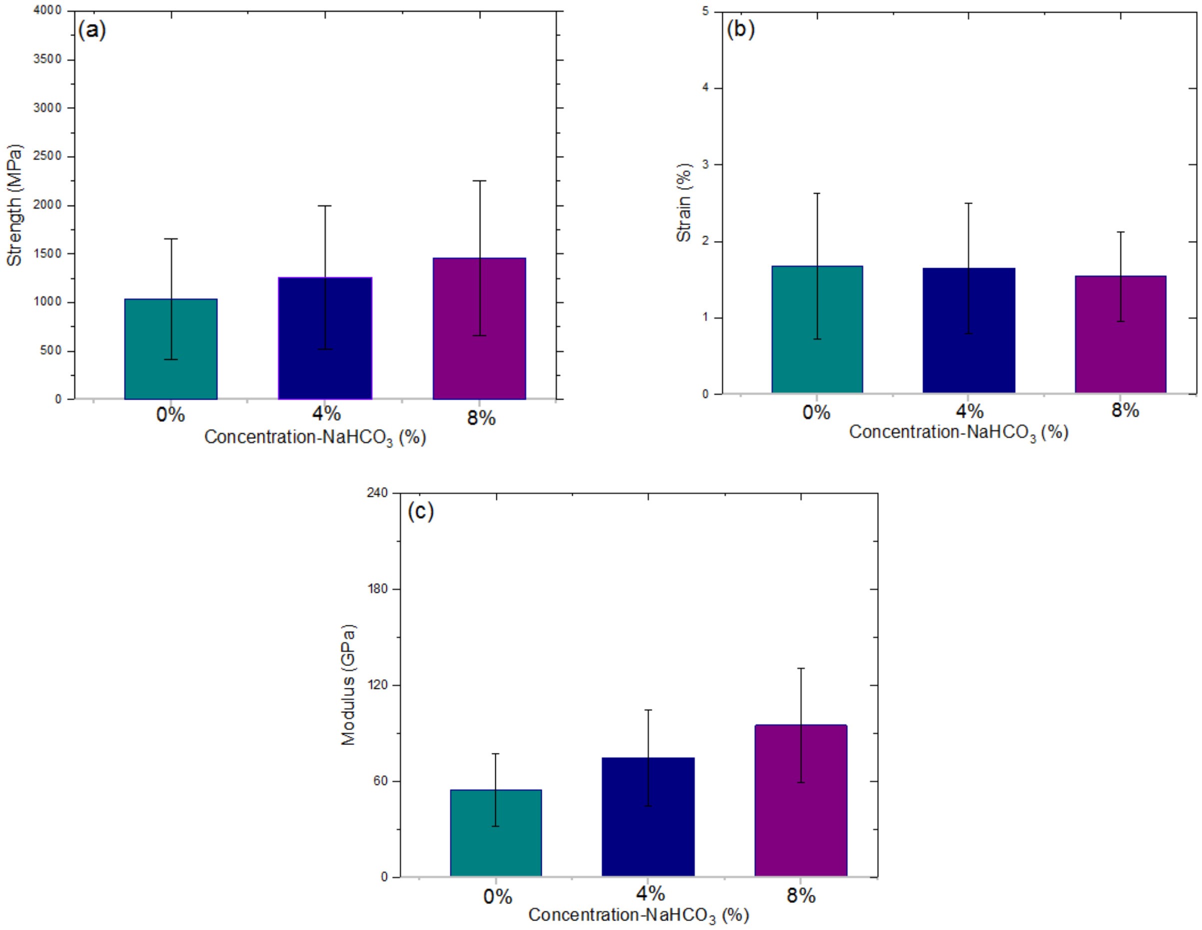

Fig4. Average tensile strengths (a), (b) strain and Young's modulus (b) for untreated, 4 and 8% w/w of sodium bicarbonate solution treated flax fibres.

Figure 5 shows the 2 and 3-parameter Weibull distribution for strain and strain and Young's modulus of untreated flax fibers treated at different concentrations of $NaHCO_3$. A two-parameter Weibull distribution provides a reasonable approximation of the experimental data for the three mechanical properties with respect to the three-parameter Weibull (Table 2). The Weibull shape and local parameters (which are the characteristic values of the distribution) for each mechanical property. The values obtained from the Weibull modulus of the breaking stress m_σ and the Young modulus m_E of treated and untreated flax fiber are presented in Table 2, for example for the untreated fiber the values are respectively: 2.02 and 2.56.

Table 1: Results of mechanical properties of untreated flax fibers treated at different concentrations of $NaHCO_3$ obtained in this work.

Concentrations	**Stress (MPa)**		**Strain (%)**		**Young Modulus (GPa)**	
	Mean	SD	Mean	SD	Mean	SD
0%	1045	701	1.75	1.01	51.32	28.55
4%	1278	825	1.68	0.87	72.65	31.57
8%	1473	869	1.61	0.56	93.64	35.02

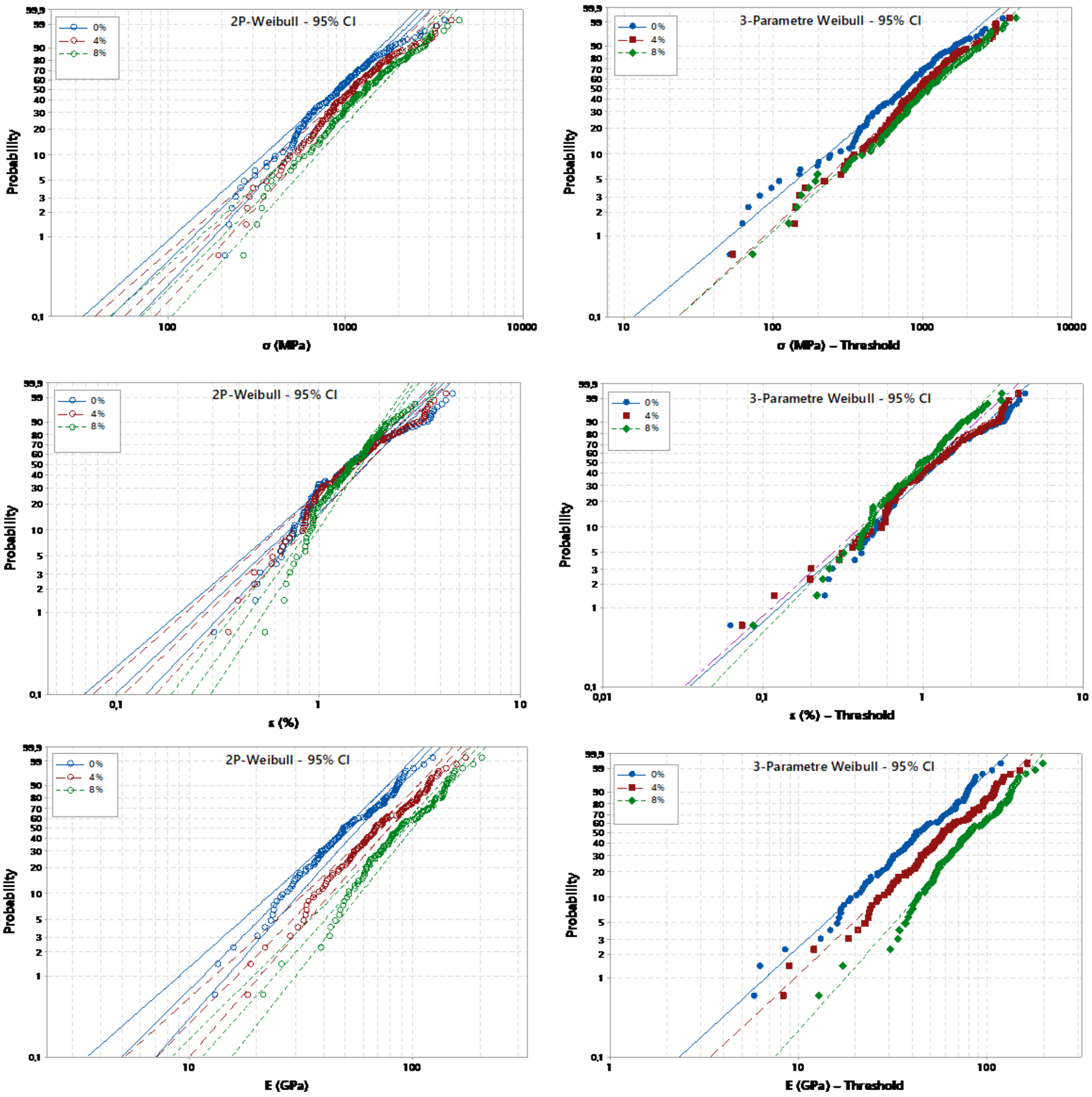

Fig5. Distribution of 2P and 3P-Weibull for mechanical properties of untreated and treated flax fibers at different concentrations of $NaHCO_3$.

Table 2: Values of the 2-parameter and 3-parameter Weibull distribution of untreated flax fibers treated at different concentrations of sodium bicarbonate.

	Stress (MPa)			Strain (%)			Young Modulus (GPa)		
	m_σ	σ_0	σ_u	m_ε	ε_0	ε_u	m_E	E_0	E_u
2-P Weibull									
0%	2.02	989		2.31	1.73		2.56	66	
4%	2.10	1153		2.39	1.88		2.72	84	
8%	2.12	1346		3.42	1.72		3.03	135	
3-P Weibull									
0%	1.52	950	134	1.74	1.45	0.21	2.25	58	6.91
4%	1.61	1041	122	1.71	1.72	0.23	2.32	71	8.97
8%	1.70	1210	173	2.09	1.33	0.38	2.74	98	8.59

4. Conclusions

The study conducted on the behavior of flax fibers treated with sodium bicarbonate ($NaHCO_3$) at different concentrations under quasi-static traction loading makes it possible to identify the following essential points:

- The results obtained show that the treatment of flax fiber surfaces with an 8% solution of $NaHCO_3$ concentration is the best treatment for this type of fiber. In other words, the chemical treatment of the fibers with $NaHCO_3$ for duration of 80 h with 8% concentration, allows a 40% and 82% increase in the breaking stress and the Young's modulus, respectively, with respect to the untreated fibers.

- The values of the Weibull modules of the flax fiber tested at a gauge length (GL) of 10 mm for the maximum stress and the Young's modulus are respectively equal to 2.02 and 2.56. While slightly higher values (m_σ = 2.12 and m_E = 3.03 respectively) are found for the treated fiber at a concentration of 8% $NaHCO_3$.

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