Effect of Chemical Treatment on Mechanical and Hygric Properties of an Innovative Clay-Sand Composite Reinforced with Juncus acutus Fibers

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Posted Date: 5 September 2024

doi: 10.20944/preprints202409.0430.v1

Keywords: Juncus acutus fibers; Alkali treatment; Composite; Compressive and flexural strengths; Moisture buffer capacity; Water-absorption capacity

Effect of Chemical Treatment on Mechanical and Hygric Properties of an Innovative Clay-Sand Composite Reinforced with Juncus Acutus Fibers

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Abstract: This work aims to investigate the impact of chemical treatment of Juncus acutus fibers on the hygric and mechanical performances of an innovative bio-sourced clay-sand-Juncus acutus fibers composite. This lightweight specimen has been produced from a mixture of 60 % natural clay and 40 % sand by mass, as a matrix, and reinforced with different amounts of Juncus fibers. The fibers were used as a partial replacement of sand in mixture by volume at 0 % (Control Specimen), 5 %, 10 %, and 20 %. In order to enhance the fiber-matrix interfacial bound, the raw fibers have undergone an NaOH alkali treatment with different concentrations of 1, and 2 wt. %. Morphological and mineralogical evaluation based on SEM micrographs and EDX analyses revealed that, the alkaline concentration of 1 wt. % is the optimized one for natural fiber surface modifications (removal of impurities without any surface damage). This optimal concentration is confirmed by tensile strength tests, showing that the treated fibers with this alkali solution presents the higher tensile stress value of 81.97 MPa, compared to those of treated fibers with 2 % NaOH (74.45 MPa), and the untreated fibers (70.24 MPa). However, mechanical test-results highlighted the benefit effect of the fiber alkali treatment on both the compressive and flexural strengths, particularly for fiber contents of 5 % and 10 %, which corresponds to a strengthening rate of 25 % and 30 %, respectively. Results also showed that the sample containing treated fibers exhibited a better moisture regulating property than that produced with untreated fibers. The specimens are classified as excellent hygric regulator based on their moisture buffer performance (MBV > 2 g/(m^2.%RH)), according to NORDTEST classification. Results also indicated that the capillary water absorption and the apparent moisture diffusivity of composites was lowered due to high fiber-matrix interfacial bound, after fiber treatment. Consequently, the composite with treated fibers is less diffusive compared to that with untreated fibers, and thus expected to be more durable in a humid environment. These results were confirmed by morphological analysis of fibers-matrix interfacial transition zone.

Keywords: juncus acutus fibers; alkali treatment; composite; compressive and flexural strengths; moisture buffer capacity; water-absorption capacity

1. Introduction

One of the major sectors that consume a significant amount of total energy in the world is the building sector [1,2]. This leads to a large amount of greenhouse gas emissions [3,4]. To meet this challenge, the academia and industry must develop visionary construction practices to provide innovative lightweight materials that meet the new requirements of users in terms of energy saving, environmental concerns and also hygrothermal comfort. Bio-based materials is proposed in last years as an attractive alternative, because they are made from renewable raw materials and allow carbon sequestration thanks to photosynthesis during their growth [5–7]. Add to the environmental benefits, the use of the vegetal fibers in construction materials is addressed to provide them some advantages such as, low density, insulating thermal capacity, recyclability, low cost manufacturing and the ability to regulate indoor humidity buildings [4,8–10]. On the other hand, the environmental negative

 doi:10.20944/preprints202409.0430.v1

impacts of conventional building sector resulted in part from the production phase of the binders (Portland cement, hydraulic lime, gypsum, etc…). Indeed, taking the example of Portland cement, the production of this binder is responsible for 5% to 8% of human kind's CO_2 gas emissions [11,12].

Therefore, the use of an alternative binder matrix can improve the environmental balance of the composite material while keeping satisfactory mechanical, hygric and thermal characteristics. In this context, natural clay is promising for low environmental impacts: the resource is available in large quantities, the energy required to extract, transform and produce materials from earth is extremely low and it is a recyclable material [13]. As an example, an earth-hemp mixture is estimated to be more than 20 times less costly in grey energy compared with hemp-lime, with a carbon impact more than 5 times lower [14]. In addition, earth material is highly performant from a hygric point of view, due to its capacity to regulate ambient relative humidity [13,15].

Although the mentioned advantages of the material composite reinforced with natural fibers, the incompatibility of raw fibers with the matrix, leads to a significant degradation in the physical, mechanical and hygric properties of the resulted composites [16,17]. This incompatibility is mainly due to impurities (hydroxyl groups) on the natural fiber's surfaces, which cause a weakening of the bond strength between the fiber and the matrix [18].

To overcome this issue, an appropriate physical or chemical pre-treatment of vegetable fibers prior its insertion in the matrix is required, in order to remove the impurities from fibers surfaces and thus enhance the fiber-matrix interfacial bonding [19–21]. This leads to a reduction of the volume of voids within the composite, that results in enhancement of its mechanical performances [22].

This present research is a continuation of our recently published work [8], that deals with the thermo-mechanical characterization of an innovative clay-sand composite reinforced with Juncus acutus fibers. During the elaboration of this lightweight sustainable composite, the Juncus fibers was incorporated into the clay-sand matrix in the natural state without any pre-treatment.

Therefore, the objective of this research is to study the effect of chemical treatment of Juncus acutus fibers on the mechanical and hygric performances of the proposed innovative clay-sand-Juncus acutus fibers composite. The natural fibers have undergone an alkali treatment with different concentrations of NaOH (1, and 2 wt. %), during an immersion duration of 1 hour, at laboratory conditions (25 °C, 50 % RH). The optimum NaOH concentration is selected on the basis of the morphological structure modifications and the tensile stress of the treated fibers. Then, the impact of fibers treatment by the optimal NaOH concentration on the hygro-mechanical properties of the composite material with different amounts of fibers was investigated. The hygric properties included the water absorption kinetics, the apparent moisture diffusivity, and the moisture buffer value (MBV). Whereas, the mechanical properties consisted of the comprehensive and the flexural strengths, the corresponding elastic modulus, and the ductility behavior. In addition, a morphological and mineralogical characterization of the fractured of composite with and without treated and untreated fibers was performed.

2. Materials and Experimental Methods

2.1. Materials

The Jancus acutus plant (sharp-pointed rush) is a renewable resource, grown spontaneously in abundance in many regions in Tunisia, essentially in the North-West ones. The shape of natural Juncus acutus plant is shown in Figure 1a. This plant is currently used for a variety of purposes, including as a source of fibers for making paper, baskets, and other woven products, and for medicinal purposes [25–27] The natural Juncus actus used in this study were collected from the region of El-Kef city, located in North-West of Tunisia.

Prior to use as specimen reinforcement, the natural stems have been dried under natural conditions for few days and crushed using a laboratory crusher into small fibers (Figure 1b and Figure 1c). They were then added at natural state or treated chemically as a partial replacement of sand containing in control specimen with proportions of 0.6:0.4:0.3 by weight of naturel clay, sand, and water respectively. The level replacements of fibers in the specimen ranged from 0 % (control specimen) to 20 % by volume.

The sand used in this study is mostly fine grained according to French standard NF P94-056 [28], with a maximum size of 1 mm. It exhibited 1443 kg/m^3 in apparent density and 1.32 in fineness modulus.

 doi:10.20944/preprints202409.0430.v1

Commercially available clay material, already used as a clay plaster inside the house, supplied by 'Argilus Industry' which is located in the West region of France, has been used as binding matrix to produce specimen based Juncus fibers. Before being used in the mixture, the clay was crushed and sieved with a square mesh of 0.6 mm. The bulk density of the crushed clay is 1380 kg/m^3 and the Atterberg Limits, including Liquid Limit (W_L), Plastic Limit (W_P)and Plasticity index (I_P) are 20.25 %, 17.08 % and 3.17 %, respectively.

The produced specimen consists of clay, sand, water, and treated Juncus fibers mixture, where four sample-compositions containing Juncus fiber content of 0 (control specimen), 5, 10, and 20 % vol. as natural sand replacement. The composition-mixes and the corresponding designations are reported in Table 1. The physico-chemical properties of the natural fibers and the material composite elaboration procedure has been reported in our previous publication [8].

2.2. Alkaline Treatment of Natural Fibers

The main objective of the chemical treatment is the ionization of the hydroxyl group by removing the hydrogen bonding from the fiber structure, according to the relationship below [18,29]. In this process, alkali solution can dissolve a certain amount of waxes, oily contents, lignin, and hemicellulose covering the external surface of fiber, which increased its roughness [30].

$$\text{Fiber-cell} - OH + NaOH \longrightarrow \text{Fiber cell-O-Na}^+ + H_2O + \text{impurities}$$

Prior to use as specimen reinforcement, alkali treatment of Juncus fibres was conducted by their immersion in Sodium hydroxide (NaOH) solutions at different concentrations of 1 and 2 wt. %, during 1h, in order to select the better treatment conditions. The companion fibers, taken as reference material, were immersed in water, for the same time duration. Afterwards, the alkalized fibers were filtered and washed several times with distilled water to remove any traces of alkali and impurities. Fibers were then dried in an oven at 60°C for 48h.

2.3. Morphological and Mechanical Characterization of Treated and Untreated Fibers

2.3.1. Morphological and Mineralogical Characterization

The Scanning Electron Microscopy (SEM) was used to examine the influence of chemical treatment on the surface morphology of Juncus acutus fibers. The micrograph analyses were performed using FEI Quanta 200 Scanning Electron Microscope, conducted at low vacuum scanning conditions, with an accelerating voltage ranging from 5 to 10 kV.

2.3.2. Fiber Tensile Strength-Test

The tensile strength and elasticity modulus of the treated and untreated Jacutus acutus stems were evaluated by performing uniaxial tensile-test of single stem, according to ASTM D3822 [31]. An universal TINIUS OLSEN H50KS testing machine was used, under a controlled displacement loading rate of 1 mm/min (Figure 2a).

A total of dried nine Juncus acutus stems (treated and untreated), measuring approximately 14 cm in length were selected for tensile tests. The ends of each stem were soaked up in epoxy resin and then sandwiched between two aluminum plates of (2 x 2 cm) in dimensions (Figure 2b). The aluminum plates fixed on either side of the stem were gripped to the upper and lower hydraulic clamps, as shown in Figure 2.b.

2.4. Mechanical Characterization of Material Composite Reinforced with Treated and Untreated Fibers

The mechanical characteristics of the hardened composite reinforced with different contents of treated and untreated fibers was examined through compressive and three-point bending-tests, according to the European Standard EN 196-1 [32]. The compressive tests were performed on cylindrical specimens (80x160 mm), using an electromechanical machine TINUS OLSEN H50KS model, with a maximum load capacity of 50KN under loading rate of 1.6 mm/min (Figure3a).

Three samples were tested for each composition-mix and the average-value of measurement data was reported. The value of the compressive strength σ_c and the ultimate strain, are given by the Eq. (1) and Eq (2):

 doi:10.20944/preprints202409.0430.v1

$$\sigma_c = \frac{F_{max}}{S} \quad (1)$$

$$\varepsilon = \frac{\Delta l}{l_0} \quad (2)$$

Where: σ_c (MPa) is the compressive strength, F_{max} (N) is the maximum load, S (mm^2) is the cross section of specimen, ε (mm/m) is the strain, Δl (mm) is the displacement, and l_0 (m) is the initial length of specimen.

The compressive modulus of elasticity (also called Young's modulus) was determined from the stress-strain diagram obtained from the compression test. Considering the linear portion of the curve where Hooke's law is valid, the young's module E_c, was calculated using Eq. (3):

$$E_c = \frac{\sigma}{\varepsilon} \quad (3)$$

The three-point bending-tests were performed on prismatic specimen (40 × 40 × 160 mm), using TINUS OLSEN H50KS model testing machine, according to the standards EN 196-1 [32]. The tests were conducted with a control deflection rate of 2 mm/min and a span-value of 210 mm in length (Figure3b). The flexural strength-values were calculated according to Eq. (4):

$$\sigma_f = \frac{3}{2}\frac{F.L}{b.d^2} \quad (4)$$

Where: σ_f (MPa) is the bending stress, F_f (N) is the maximum load, L, b and d (mm) are the span of specimen in three-point bending-test, the thickness, and the average width, respectively.

The flexural elastic modulus from flexural-test was calculated using the Eq. (5).

$$E_f = k.\frac{L^3}{4.b.d^3} \quad (5)$$

E_f (MPa) is the flexural modulus, k (N/mm) is the elastic stiffness of specimen, which corresponds to the slope of the linear portion of load-deflection curve.

2.5. Hygric Characterization of the Composite Specimens

2.5.1. Water Transport Properties Characterisation

The water absorption tests consisted of immersing the base of the dry testing specimens (prismatic specimens with dimensions of 4 x 4 x 16 cm^3 in liquid water bath on a perforated grid at a depth of 3 mm (Figure 4). The wet specimen mass in function of time was recorded gradually at varied time intervals of 30 s, 1 min, 2 min, 5 min, 10 min, 20 min, 1 h, and then every 2 h, through a digital balance placed near the water bath. The water absorption rate (also called the water uptake) was determined using the following Eq. (6) :

$$M_t = \frac{m_t - m_d}{m_d} \times 100 \quad (6)$$

Where m_d and m_t are the dry sample weight (before initiating the test) and the moist sample weight a time t, respectively.

The most important parameter for water absorption is the apparent diffusion coefficient (D_A) because it shows the ability of solvent molecules to penetrate inside the material structure [25–28]. This coefficient can be calculated according to Fick's law by using the following relation [29]:

$$D_A = \pi\left(\frac{h}{4M_{st}}\right)^2\left(\frac{dM_t}{d\sqrt{t}}\right)^2 \quad (7)$$

Where M_t (%) is the water absorption rate vs. time, given by Eq (6); M_{st} (%) is the saturation water absorption rate; D_A (m^2/s) is the apparent moisture diffusivity coefficient, h is the height of specimen; $dMt/d\sqrt{t}$ is the slope of the weight gain versus square root of time relation (in the linear region).

The side faces of the specimen were waterproofed using a heat-shrinkable plastic film, thus a unidirectional water transfer along the length of the specimen was only assured. So that, h in the formula should be the specimen length. This avoids to use a correction factor in the Eq. (7) considering the water penetrating through the specimen edges, as used by some authors [33–35].

 doi:10.20944/preprints202409.0430.v1

2.5.2. Moisture Buffer Capacity Evaluation

The study of the hygric behaviour of specimen has been performed by investigating its moisture buffer capacity, according to the experimental method addressed by the NORDTEST project [36]. The MBV relates the moisture uptake or release per surface area under the cyclic variation of relative humidity, which can be determined from Eq (7) [37].

$$\mathrm{MBV} = \frac{\Delta m}{S.(RH_{high} - RH_{low})} \quad (8)$$

where, MBV (g/m².%RH) is Moisture Buffer Value; Δm (g) is the uptake or release of moisture; A (m²) is the surface area of specimen ; RH_{high} and RH_{low} are high and low relative humidity levels (%).

For the MBV test, specimens measuring 70 x 70 x 40 mm in sizes were prepared and sealed on the five sides, before being stabilised in desiccator at 50 % RH, for 48 h. After stabilization, all samples were placed in a climatic chamber (BiaClimatic type CL2-25) and then subjected to cyclic changes in relative humidity with a corresponding duration of : 75 % RH during 8 h and 33 % RH during 16 h, at a constant temperature of 23 °C (Figure 7).

3. Results and Discussion

3.1. Effect of Alkaline Treatment on Surface Morphology and Tensile Strength of Juncus Acutus Fibers

The fibers surface morphology of a natural fiber and treated fiber with the different NaOH concentrations can be analyzed from SEM images shown in Figure 5 (a-d). As it can be seen, the natural fiber surface is coverage by some components that may be residual of lignin, hemicellulose, pectin and waxes which are the main reasons of the high sensitivity of fibers to water absorption, by consequence causing a weak adhesion fiber-matrix [18,38,39]. However, the treatment with 1% NaOH solution cleaned the surface by removing completely the impurities and waxy layers. This phenomenon was observed by many researchers [18,29,40]. It can be observed from the SEM images the apparition of some free spaces between the fibrils that will promote interlocking the bonding between the fiber and the matrix. For higher alkali concentrations (2%), one can observed a drastic surface damage causing their embrittlement, due to severity of the acid attack [41,42].

The average tensile stress-strain curves for natural and treated Juncus acutus single stem with sodium hydroxyl at concentration of 1% and 2% are depicted in Figure6. The results clearly indicated that the stem treated with 1% NaOH displayed the highest tensile strength (81.96 MPa) than those untreated (70.25 MPa) and treated with 2% NaOH (74.45 MPa). The increase in tensile strength, after treatment process, is possibly due to the removal of non-cellulosic parts and thus increasing the cellulose fractions, which protect the fibrous structure of the fiber against physical and chemical degradations [43].

3.2. Effect of Alkaline Treatment of Fibers on Compressive and Flexural Strengths of the Reinforced Composite Material

The typical stress-strain diagrams of specimens reinforced with treated fibers (CS0F, CS5TF, CS10TF, CS20TF) and with untreated fibers (CS0F, CS5UTF, CS10UTF, CS20UTF) are presented in Figure 7. The corresponding parameters values, including compressive strength, ultimate strain and elastic modulus are reported in Table 2. It can be observed that, the two sets of curves exhibit different trends. Indeed, the compressive strength of specimen reinforced with untreated fibers decreased regularly with increasing fibers volume. However, that reinforced with treated fibers increased to a maximum of 1.81 MPa when the specimen contains 5% of fibers, then decreased to a value 0.94 MPa (remains higher than that of control specimen) for the specimen with 20% fibers. The comparison of this maximum strength to that for specimen with the same volume of untreated fibers exhibits a strengthening rate of approximately 25%.

Otherwise, the comparison between the compressive strengths of specimen with treated fibers and that with untreated fibers, averaged over the entire fiber's levels range, revealed that the alkaline treatment of fibers (by the optimal solution) leads to a significant increase of the average compressive strength from 1.22 to 1.50 MPa. The correspondent strengthening rate is around 20%. This enhancement is attributed to the improvement of the bond between the fibers and the matrix pasta and the stiffness of the fibers, resulted from the removal of impurities and waxy layers covered raw

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fibers surface. The Alkali treatment of fibers efficiency on the improvement of bio-based materials compressive parameters was reported by Naiir et *al*. [18] for gypsum mortar reinforced with doum palm fibers and Kamaruddin et al. [44] in the case of cassava starch-palm wax blends reinforced with Cymbopogan citratus fibers.

According to our results, the treatment of fibers also affects the compressive elastic parameters. Indeed, the average ultimate compressive strain value is dropped from 10.31 mm/mm for specimen with untreated fibers to 9.52 mm/mm for that with treated fibers. The average values of the corresponding elastic modulus increased from 141.24 MPa to 178.54 MPa. One can concluded from these last results that, for the same volume of fibers, although specimen with treated fibers has a higher compressive strength, its ductility is decreased compared to that with untreated fibers. This is resulted from a shorter plastic phase and weaker strain capacity before failure, due to a reduction in the specific energy absorption. This compressive behavior is explained, one hand by the enhancement of the linking between the treated fibers and the clay-sand matrix, and on the other hand, by the enhancement of the fiber stiffness and the roughness of its surface.

It was interesting to noted that, during the compressive tests, the control specimen completely disintegrated, while the specimen reinforced with treated fibers, maintained its initial structure due to the bridging effect of fibers. This is confirmed the ductile behavior for reinforced specimen and the brittle behavior with sudden fracture for the control specimen. The change in elastic behavior of specimen from brittle to ductile has been reported by several authors when vegetable materials were used as additives in concrete based on different binder types [1,4,8,11,45,46].

The load-deflection diagrams of specimens with reinforced treated fibers (CS0F, CS5TF, CS10TF, CS20TF) and with untreated fibers (CS0F, CS5UTF, CS10UTF, CS20UTF) are displayed in Figure 8. The corresponding parameters values including flexural strength, ultimate deflection, elastic stiffness, elastic modulus, and flexural toughness are reported in Table 3. The increase of the untreated fibers volume in matrix pasta results in a regularly degradation of its flexural strength. In contrast, the increase of treated fibers volume in the matrix leads to an improvement in its flexural strength, as compared to the control specimen. Data reveal maximum strength of 0.87 MPa for reinforced composite with 10% treated fibers. This corresponds to a strengthening rate of about 25% and 30%, as compared to the control specimen and the composite with 10% untreated fibers, respectively. This specific behavior was observed by some authors, such as Zaid et *al*. (2021) when they studied the effect of treatment of Diss fibers on the physico-mechanical characteristics of Diss concrete based on alternative binder and Ajouguim et *al*. (2021) when they investigated the effect of Alfa fibers on the mechanical and thermal properties of compacted earth bricks.

Otherwise, based on the results reported in Table 3, the flexural strength, averaged over the entire fibers volume range, increased from 0.62 MPa for specimen with untreated fibers to 0.77 MPa for that with treated fibers. The correspond strengthening rate is around 20%. This enhancement could be attributed to the improvement of the links between the fibers and the matrix due to fibers surface roughness as well as the tensile strength of the fibers results in the treatment effect. These findings agree with those obtained by other research-works [18,30,47,48].

The flexural elastic parameters were also affected by the alkali treatment. Indeed, the average ultimate deflection value decreased from 0.81 mm for the specimen with untreated fibers to 0.24 mm for that with untreated fibers. The corresponding elastic modulus increased from 311 MPa to 950 MPa. It should be noted that, during the flexural tests, a typical bridging phenomenon was observed for the specimen reinforced with treated fibers, which results in delaying the cracks propagation and retaining the specimen structure [8].

3.3. SEM Micrographs and EDX Analysis

The Figure 9, shows SEM micrographs and EDX analysis of fractured CS0F, CS10UTF and CS10TF specimens. The SEM observations of the CS0F specimen (Figure 9a) display a homogeneous matrix, with high compactness without cracks. The grains of sand are well coated by the clay which resulted of a compactness material with higher mechanical strength compared to the CS10UTF and CS10TF specimens (Table 2 and Table 3). The most dominant elements in the EDX spectra are: C, O, Ca, Si, Fe, which are the main components of the clay matrix.

Figure 9-b shows internal microstructure of the CS10UTF specimen. One can note that, the addition of untreated fibers leads to poor adhesion between untreated fibers and the matrix. Figure

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9-c presents the internal microstructures of the CS10TF specimen. On can observed that, the treated fibers are well embedded with the matrix paste, which confirms the treatment efficiency. Besides, the voids were minimized compared to the CS10UTF specimen, and the mortar becomes denser, which is a major indicate of the better interfacial adhesion. X-ray analyses in the interfacial transition zone for the CS10TF and CS10UTF specimens reveal that the C, O and Si elements appear to be the most dominant in the EDX spectra as they are the main components of the lignocellulose fiber structures. Moreover, one can note that the alkali treatment leads particularly to a significant increase of the C element content from 14.6 % to 44.4%. This is possibly due to the removal of non-cellulosic parts from the fiber surface and thus increasing the cellulose fractions. In contrast, the Si reduced from 22% to 5.5%, which could be explained by the removal of the rough outer surface of the fiber containing this element after treatment.

It should be noted that the results derived from the SEM observations were in perfect agreement with those reported in other research works focused on the effect of fibers treatment on the morphological characteristics of reinforced composites based different types of vegetable fibers [49–51].

3.4. Effect of Alkaline Treatment of Fibers on Water Absorption Capacity and Moisture Diffusivity of the Reinforced Composite Material

The water absorption rate of specimens reinforced with different levels of untreated and treated fibers as a replacement of sand, as a function of the square root of time is given in Figure 10. One can noted that, water absorption increases with fibers content for specimens reinforced with treated or with untreated fibers. This behavior was attributed both to the hydrophilic character of fibers and to the segregation fiber phenomena resulting from fiber overloading. These two factors amplify the porosity that occurs during the drying period of the specimens. This finding agrees with that reported by several authors for bio-based materials [33,39,52–55]. For all levels of fibers, the water absorption at saturation point (also called saturation water uptake) for specimen with the treated fibers is lower than that for specimen with untreated fibers. The reduction percentage of the water absorption at saturation, averaged over the different levels of fibers, is around 25%. This significant reduction can be explained by the improvement of fiber-matrix interfacial adhesion, which reduced the available polar–OH groups in the system [54,56,57]. Moreover, the study showed a faster water absorption for specimen reinforced with untreated fiber. This can be attributed to the large numbers of polar –OH groups, which formed the hydrogen bonding between the free –OH groups of the cellulose and water molecules [56–59].

For the better understanding of the water absorption mechanisms in the composite reinforced with different levels of treated or untreated fibers, the corresponding moisture diffusion coefficients were estimated based on the Eq. (6). Table 4 summarizes the obtained results. One can easily observe that, the moisture diffusion coefficient for the composite with treated and untreated fibers, increase with increasing the fibers content. However, for a given level of fibers, the moisture diffusion coefficient for composite with treated fibers is lower than that with untreated fibers. This is attributed to the better adhesion between fibers and the matrix. The composite with retreated fibers is expected to be more durable in a humid environment. The similar effect of alkaline treatment was found in several work-researches: Lahouioui et al. [60] for a Date palm fibers reinforced sand-cement composite and El-Abbassi et *al*. [61] in the case of Alfa fibers reinforced polypropylene composite.

3.5. Effect of Alkaline Treatment of Fibers on Moisture Buffer Value (MBV) of the Reinforced Composite Material

An example of moisture uptake and release during the MBV test for CS20TF and CS20UTF specimens is shown in Figure 11. The ambient relative humidity in the climate chamber during the test is also reported in this Figure. Table 5 gives the MBV value in desorption, adsorption and average for the different formulations (with treated and untreated fibers) as well as their classification according to the Nordtest classification. It can be noted that the MBV values ranged from 2.00 to 2.23 g/(m².%RH) for specimen reinforced with treated fibers and from 1.82 to 2.18 g/(m².%RH) for that with untreated fibers. Consequently, specimens with treated fibers can be considered as better hygric regulators for hygrothermal comfort than that with untreated fibers. They can be classified as excellent hygric regulators, according to the NORDTEST project classification [36].

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It should be noted that our results fall generally well within the range of the published ones for composite materials. Indeed, the MBV values range from 1.94 to 2.24 g/(m².%RH) for hemp-lime composites [62], from 1.99 to 2.15 g/(m²%RH) for hemp-concrete composites [63,64] and from 2.07 to 2.28 g/(m².%RH) for hemp-clay composites [13]. However, the classical masonry materials, like brick and aerated concrete exhibited significantly lower values of MBV equal 0.39 and 1.05, respectively [65]. They are classified as limited and moderate hygric regulators respectively, according to NORDTEST classification.

4. Conclusions

The main objective of this work is the enhancement of the hygro-mechanical performances of an innovative bio-composite clay-sand Juncus fibers. The enhancement procedure adopted in this study was based on the alkali pre-treatment of raw fibers at different concentrations of NaOH (1%, 2% and 10%) under ambient conditions.

The experimental tests highlighted the flowing results:

1) Based on SEM images, it was found that the immersion of raw fibers in 1% NaOH solution for 1h, gives the optimal results, regarding to fiber surface modifications. This result is confirmed by tensile stress-strain tests showing that the fiber treated with 1% NaOH displayed the highest tensile strength (81.96 MPa), compared to those untreated (70.25 MPa) and treated with 2% NaOH (74.45 MPa).

2) Due to the treatment effect, the water absorption at saturation (saturation water uptake) of the composite material, averaged over the different fiber's levels, is reduced by about 25%. Besides, the composite water absorption is more slowly for composite reinforced with treated fibers. The treatment of fibers has also a benefit impact on the composite apparent moisture diffusion coefficient. The composite with treated fibers is expected to be more durable than that with untreated fibers in a humid environment.

3) The MBV values ranged from 2 to 2.23 g/(m².%RH) for composite reinforced with treated fibers and from 1.82 to 2.18 g/(m².%RH) for that reinforced with untreated fibers. Based on these results, formulations with treated fibers are more efficient hygric regulators for hygrothermal comfort compared to those with untreated fibers.

4) The treatment of fibers enhanced the mechanical properties of the resulting composite, for the different volumes of fibers. The compressive strength reached a maxima value of 1.81 MPa at 5% of fibers addition, while the flexural strength reached a maximum value of 0.87 when the composite contains 10% of fibers. This corresponds to a strengthening rate of about 25% and 30% respectively, when compared to specimens with the same volume of untreated fibers. Otherwise, the composite strengthening rate, averaged over the entire fibers levels range (from 0% to 20%) is about 20% for both the compressive and the flexural strengths. This is could be attributed to the enhancement of both the adhesion fiber-matrix and the stiffness and the roughness of the fibers surface, resulted from the treatment.

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