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The effect of chemical treatment on the mechanical and thermal properties of composite materials based on clay reinforced with sawdust

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Abstract. This study investigates the effect of the chemical treatment of sawdust on the mechanical and thermal properties of a clay composite reinforced with sawdust in variable mass proportions: 0.5, 1, 1.5, 2 and 2.5%, intended for use as brick in rural houses in desert areas. The sawdust was chemically treated with 5 wt% NaOH and 0.01 wt% KMnO₄ solutions. The mechanical test results of the reinforced composite with alkalitreated sawdust show an increase in flexural and compression strength, reaching a maximum value of 0.89 and 4.85 MPa, respectively. However, the thermal test results show a significant decrease in the thermal conductivity of the sawdust-reinforced composite. The best result recorded is that of untreated sawdust, which has a value of 0.37 (W.m⁻¹.K⁻¹) compared to the one reinforced with treated sawdust.

Keywords: sawdust / clay / thermal conductivity / mechanical properties / chemical treatment

Résumé. L'effet du traitement chimique sur les propriétés mécaniques et thermiques des matériaux composites à base d'argile renforcée par la sciure. Dans ce travail, nous avons étudié l'effet du traitement chimique de la sciure sur les propriétés mécaniques et thermiques d'un composite à base d'argile. Cette dernière a été renforcée avec la sciure de bois à des proportions massiques variables: 0.5, 1, 1.5, 2 et 2.5 % pour l'utiliser comme briques dans les maisons rurales situées en zones désertiques. La sciure a été traitée chimiquement avec les solutions de NaOH (5 wt%) et KMnO₄ (0.01 wt%). Les résultats des essais mécaniques du composite renforcé avec la sciure traitée aux alcalis ont montré une augmentation de la résistance de flexion et de compression, atteignant respectivement, une valeur maximale de 0,89 et 4,85 MPa. Bien que, le test thermique a montré une diminution notable de la conductivité thermique du composite renforcé de sciure de bois et le meilleur résultat a été enregistré avec la sciure traitée.

Mots clés: sciure / argile / conductivité thermique / propriétés mécaniques / traitement chimique

1 Introduction

In recent decades, scientists and researchers have paid great attention to the use of natural building materials to reduce the environmental pollution caused by cement manufacturing, which is a potential risk to human health and the environment [1–4]. The characteristics of natural materials are such that they are affordable, locally available, environmentally friendly, recyclable and offer good thermal insulation [5]. However, adobe bricks do not have the same mechanical strength as concrete or even fired bricks [6]. Several studies have been carried out in strengthening clay materials with natural fibers. The latter are known for their lightness, biodegradability and environmental friendliness [7–12]. Koadri et al. [13] studied the effect of alkali treatment of palm fibers on the mechanical properties of the composite and found that the flexural and compressive strength of treated fibers composites improved compared to untreated fiber

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 Table 1. Chemical compositions of the lime.

Compositions	CaO	MgO	$\mathrm{Fe_2O_3}$	Al_2O_3	SiO_2	SO_3	Na_2O	$\rm CO_2$	$CaCO_3$
Mass $(\%)$	>83.3	< 0.5	$<\!2$	$<\!1.5$	$<\! 2.5$	$<\! 2.5$	< 4.7 – 0.5	< 5	< 10

composite. The results show that the increase is respectively about 57% and 60%, respectively, compared to untreated fibers composites.

Fertikh et al. [14] used Diss fibers to reinforce different matrices, in particular cement, lime and clay. The results show that the Diss fiber-reinforced clay-lime combination performs better than the cement matrix. Djohore et al. [15] manufactured a composite made of coconut fiber-reinforced clay that was treated with a potassium hydroxide solution to make clay materials that are stabilized with cement. The manufactured mortars consist of clays reinforced with 8% cement and fibers of various weight fractions (0.2, 0.4, 0.6 and 0.8%). This study was carried out to verify the influence of these fibers on the properties of the materials. The results indicate that the addition of the above-mentioned fibers improves the mechanical performance of clay mortars reinforced with 8% cement.

Laaroussi et al. [16] investigated the thermal properties of a sample prepared with clay brick mixtures. Similarly, Meukam et al. [17] studied the mechanical and thermal characteristics of stabilized soil bricks for use as a building material. Elhamdouni et al. [18] examined the Alfa fibers effect on the thermal characteristics of clay-based materials. The authors mixed clay (chosen as the base material) with different percentages of Alfa fibers (0.5%, 1%, 2%, 3%, 4%). The results obtained show that increasing the percentages of Alfa fibers improves the thermal properties of this mixture.

Taallah [19] studied the effect of the use of date palm fibers on the mechanical properties and behavior of BTC. The results indicate that there is a slight improvement in the dry compressive strength of the blocks with 0.05% fiber, 8% cement and 10 MPa compaction pressure. In the case of quicklime, the improvement concerns the dry tensile strength of blocks with 0.05% fibers and 8% and 12% lime. However, the presence of the fibers has a positive effect on the mechanical behavior of the composite by increasing its ductility compared to the brittle behavior of the matrix alone.

Ajouguim et al. [20] used fibers extracted from the Moroccan Alfa plant, as a reinforcement of compacted earth bricks. Alfa fibers were modified by alkali and hydrothermal treatments respectively. The results show that the mechanical properties (flexural and compressive strength) of the compacted earth bricks increase with the addition of natural fibers. The compacted earth bricks with 1 wt% of alkali treated Alfa fibers show better mechanical properties with low thermal conductivity. To this end, this present work proposes to prepare a sawdust fiber composite by treating the surface of the fibers with alkali and KMnO₄. In addition, the morphology and surface chemistry of the fiber was studied by XRD, FTIR for the untreated and treated fiber, also by SEM of the composites, and correlated with the above-mentioned studies to evaluate its mechanical and thermal properties.

Table 2. Chemical composition of the mixing water.

Parameters	Experimental values	Standard values [22]	
Conductivity (μ S.cm ⁻¹)	1320	_	
Total Dissolved Solids (TDS)	660	_	
Salinity (%)	0.6	0.6	
Temperature (°C)	22.1	_	
Potential Hydrogen (pH)	8.30	6.5 - 9.5	
Turbidity (NTU)	0.240	5	
$Ca^{2+} (mg . L^{-1})$	200.4	_	
${ m Mg}^{2+}~({ m mg.L}^{-1})$	24.31	150	
$\mathrm{Cl}^{-} (\mathrm{mg.L}^{-1})$	141.8	_	
HCO_{3}^{-}	157.99	_	
$\mathrm{SO_4}^{-2}$	165.95	250	
NO_3^-	4.64	50	
PO_4^{3-}	0.25	0.5	
NO_2^-	0.00	2	
${ m NH_4}^+$	0.12	0.2	
K^+	1.3	_	
Fe^{2+}	0.05	0.3	

2 Materials and methods

2.1 Lime

The lime used in this study is produced in the Ghardaia region (southern Algeria). Table 1 lists the chemical compositions of this material [21].

2.2 Water

The water used in this study is tap water from the Civil Engineering Laboratory of the University of Laghouat, Algeria. The water analysis was carried out at the laboratory of the Algerian water company (*Algérienne des Eaux* – ADE), a public company, in the Laghouat district. Table 2 shows the results obtained

2.3 Clay preparation procedures

In this study, 2/1 type clay (SiO₂/Al₂O₃ ratio = 2.58) was used. It originates from Bou-Saâda region, a district located 69 km southwest of M'sila (north-central Algeria). To prepare the clay, the following steps were completed:

- grinding of the sample rocks into small pieces with a mortar;
- the material was dried in an oven at 105 °C for 24 h;
- the clay pieces were crushed in a grinder;
- the resulting material was sieved.



Fig. 1. Treated and untreated sawdust.

2.4 Chemical treatments of sawdust

2.4.1 NaOH treatment

Sawdust was used as a reinforcement and was obtained from a carpentry workshop. The sawdust went through different stages to be processed. First, it was dried in ambient air and then placed in the oven for 24 h at a temperature of 80 °C. Then, the sawdust was treated with a 5% (wt) NaOH solution for 24 h at room temperature; the ratio sawdust/solution used was 1:15 (w/v). Traces of NaOH were neutralized with a 2% sulfuric acid solution of distilled water for 10 min. Next, the sample was subjected to successive washes with distilled water. Finally, the sawdust was dried in an air oven at 105 °C for 5 h.

2.4.2 KMnO₄ treatment

The sawdust was treated for 3 min with $\text{KMnO}_4 \text{ at } 0.1 \text{ g.L}^{-1}$ which was dissolved in an acetone solution of density 20 °C (density = 0.791, purity = 99.5%). It was then rinsed with distilled water to remove excess potassium permanganate. Finally, the treated sawdust was dried in ambient air. Figure 1 shows the sample obtained.

2.5 Preparation of composites

Several steps were performed to prepare the desired composite. First, the sawdust with different percentages (0.5, 1, 1.5, 2 and 2.5 wt%) was placed in a kneader ("Controls" model 65-L0012/E, made in France) with a quantity of clay and lime (12 wt%). The process of adding lime to the composition was done to stabilize the composite to minimize the fragility of the clay. Before hydrating the composite, the components were carefully blended to obtain a homogeneous mixture. Finally, tap water was added to the mixture as indicated in equation (1):

$$\frac{E_a}{A} = 0.5,\tag{1}$$

where E_a and A are respectively the quantities of water and clay in grams.

Then the mixture was kneaded until a homogeneous mixture was obtained. The latter was poured into prismatic molds (dimension: $4 \times 4 \times 16 \text{ cm}^3$). Next, the molds were placed in a shock device at 60 shocks per second. This operation was carried out to distribute the mixture in a compatible manner in the molds and to eliminate air bubbles. Then, the mixture was deconfined and stored in a damp room (20 °C, RH = 95%) for 28 days. Afterwards, it was dried in a ventilated oven at 60 °C until a constant mass was reached before being tested. It should be pointed out that the manufactured composite consists of sawdust structures randomly mixed with clay. Figure 2 illustrates the different steps of the preparation procedures. Table 3 shows the coding of the different fibers used in the mixture.

2.6 Measuring apparatuses

2.6.1 Particle's size by laser diffraction

Laser diffraction is a technique used to determine the size and distribution of particles in powders to predict the behavior of the powder in a given process. This technique is based on the interactions between a set of particles and laser radiation. The particle size of our material was determined using the Granulometer Mastersizer model 2000 (a device manufactured in the UK with a particle size distribution of $0.02-2000 \,\mu\text{m}$).

2.6.2 Chemical composition

The chemical composition of the clay was identified using X-ray Fluorescence Spectrometry (Phillips PW 2400 type, manufactured in France).

2.6.3 X-ray diffractometry test

The degree of crystallinity and the orientation of crystallites can have a direct effect on the modulus and properties of fibers in general, which is why the evaluation of the fibers used in this study was a necessity. To achieve the above objectives, we evaluated the crystallinity of sawdust by X-ray Diffractometry using the High Score Diffractometer (X' Pert Pro PW3209, Panalytical, made in



Fig. 2. Composite preparation steps.

France). Equatorial diffraction patterns (2Θ) were recorded by 10° to 40° with Cu-Ka radiation at 40 kV and 20 mA. These procedures were adopted to determine the physical structure of the sawdust as a function of the crystallinity index (CrI (%)). In this regard, Segal et al. [23] developed an empirical method for estimating the degree of crystallinity of cellulose (Eq. (2)) that can be described as:

$$\operatorname{CrI}(\%) = (I_{002} - I_{am})/I_{002} \times 100,$$
 (2)

where I_{002} represents the intensity of the highest peak of cellulose I at an angle of about 2 Θ to 22.7°; I_{am} indicates the intensity ascribed to the amorphous cellulose given at an angle of approximately 2 Θ to 18.9°.

2.6.4 Fourier transform infrared (FTIR) test

The FTIR test was carried out to identify the particle nexus of the models used. The IR spectra were documented on a Fourier Transform Spectrometer of the JASCO FTIR-4200 model (France). In addition, the absorption spectrum was identified from 4000 to 400 cm^{-1} . The FTIR spectra are realized by mixing samples with KBr and preparing typical plates using a laboratory press.

2.6.5 Scanning electron microscopy (SEM) test

The SEM test is performed to check the structure of any sample with an electron beam for analysis. The test, in this case, was performed using a TESCAN VEGA3 SBU EAST PROBE type probe (France). This apparatus is controlled by a computer working in topography mode (secondary electrons) and in composition mode (backscattered electrons). The SEM images were obtained by the secondary electron imaging method with a beam acceleration voltage of 15 kV.

2.6.6 Mechanical tests

The three-point bending and density tests were performed according to EN 196-1 standard on the prepared samples. This operation was carried out using a mechanical testing device (Controls type, Italy) with a maximum capacity of 100 KN, in a closed compression circle measured by the PC.

 Table 3. List of abbreviations used.

Materials/ Composites	Label
Cl	Clay
S	Sawdust
US	Untreated sawdust
STA	Sawdust treated with alkaline (NaOH)
STK	Sawdust treated with $\rm KMnO_4$
US/Cl	Clay reinforced with untreated sawdust
STA/Cl	Clay reinforced with sawdust treated with alkaline (NaOH)
STK/Cl	Clay reinforced with sawdust treated with $\rm KMnO_4$

2.6.7 Thermal tests

A Hot Disk TPS 2500 was used to measure the thermal conductivity of the materials used in this study. The thermal test allows the sample to be studied without violating the sample size requirements. The materials were measured in accordance with the ISO 22007-2 standard. The hot disc apparatus is centered on the use of a sensor that includes a resistance working by Joule effect. It should be noted that the thermal conductivity of the material can be deduced from the electrical resistance of the sensor. Thus, the sensor is a temperature monitor, as it helps to give precise information about the studied material. The hot disc is sealed between two pieces of samples under investigation. This idea is clearly demonstrated in equation (4) [24]:

$$\Delta T(\tau) = \frac{P_0}{\pi \frac{3}{2} a \lambda} D(\tau), \qquad (3)$$

$$\tau = \frac{\sqrt{\kappa t}}{a},\tag{4}$$

$$\lambda = \kappa \cdot \rho \cdot c, \tag{5}$$



Fig. 3. Clay particle analysis.

where:

- $-\Delta T(\tau)$: the average temperature;
- $-\tau$: a dimensionless parameter that is called the *characteristic time ratio*:
- $-\lambda$: the thermal conductivity of the material (W.m⁻¹). $K^{-1});$
- κ : thermal diffusivity (mm².s⁻¹); ρ .c: the specific heat (MJ.m⁻³.K⁻¹);
- $D(\tau)$: the dimensionless time function;
- P_0 : the output power of the Hot Disk sensor (W.m⁻²);

 $-\alpha$: the radius of the disc.

3 Results and discussion

3.1 Particle size analysis of clay

Figure 3 illustrates the distribution of clay samples as a function of diameter in µm. The results show that the clay in question is composed of almost 4% silt and 76% sand. Thus, the clay used in this experiment is sandy-silty.

3.2 Basic chemical composition of clay

Table 4 presents the results of the basic chemical analysis of the clay. The analysis reveals that silica and alumina are the main oxides in the sample studied. Their percentages are respectively 35.21% and 13.62%. Thus, the clay is an aluminosilicate. On the other hand, the calcium ratio is 15.53%, i.e., the material studied is rich in calcite (CaCO₃). It is necessary to specify that the alumina/silica fraction gives information on the permeability of the material to humidity. When the ratio is massive, the permeability is significant [25]. In our case, the ratio is low $A1_2O_3/$ $SiO_2 = 0.38$. It can therefore be deduced that this low value is analogous to the humidity percentage [26]. Moreover, the ratio $SiO_2/Al_2O_3 = 2.58$ (maximum substitution of Si^{4+} by Al^{3+}) is lower than the standard bentonite value of 2.7. The results obtained indicate the existence of small amounts of quartz in the clay ratio [27]. Thus, the overall composition of other oxides (such as Fe_2O_3 , MgO, K_2O , and Na_2O) reaches a percentage of 13. It can therefore be concluded that the examined clay is not pure [28].

3.3 X-ray diffractions

3.3.1 Clay

The spectral analysis of the X-ray diffraction of the clay (Fig. 4) indicates that the material consists of the following components: quartz (SiO_2) , calcite $(CaCO_3)$ and dolomite. More importantly, the presence of quartz reaches its maximum when compared to that of calcite. We can deduce that the mineralogical composition of the clay fraction of our material contains 35.21% guartz and 15.53% calcite as the main crusts in our sample.

3.3.2 Sawdust

Figure 5 shows the X-ray diffractograms of the treated and untreated fibers. As can be seen, all samples show the characteristic peak of cellulose I, which corresponds to crystallographic planes 002 [29]. The results indicate that the crystallization index of sawdust treated with NaOH and KMnO₄ increased compared to untreated sawdust. The crystallinity index of untreated sawdust reached the value of 17.89%, while for sawdust treated with NaOH and $KMnO_4$, the values are 45.96% and 39.19% respectively (Tab. 5). This can be justified by the fact that the use of NaOH and KMnO₄ can reduce amorphous hemicellulose and lignin with impurities such as wax and oils and rearrange the crystalline regions so that the fiber has a more crystalline nature [30–32]. Therefore, it can be noted that the degree of cellulose is higher in treated samples due to a decrease in hemicellulose during the treatment process [33]. The alkaline treatment also affects the crystalline properties by transforming cellulose I into cellulose II [34]. On the other hand, the crystallinity index of sawdust treated with KMnO₄ decreased compared to sawdust treated with NaOH. This is probably due to the increase in the amorphous cellulose content during KMnO₄ treatment [35].

3.4 Infrared spectroscopy analysis

3.4.1 Clay

Figure 6 shows the FTIR spectra of the studied clay. The band extending from 3200 to $3800 \,\mathrm{cm}^{-1}$, located at $3614.0 \,\mathrm{cm}^{-1}$, corresponds to the elongation vibrations of the internal OH groups. The band at $1801 \,\mathrm{cm}^{-1}$ corresponds to the elongation vibrations of the carbonyl groups (C=O) [34]. On the other hand, the band at 1440 cm⁻¹ can be attributed to the CH_3 elongation vibration [33]. The band of $1024 \,\mathrm{cm}^{-1}$ corresponds to the elongation vibration of the siloxane groups -(Si-O)-[36]. The band of 784 cm⁻ can be attributed to the deformation vibrations of Si-O-Al [36]. Finally, the two bands positioned between $867 \text{ and } 710 \text{ cm}^{-1} \text{ correspond to the valence vibrations of}$ the corresponding CO bond $(CaCO_3)$ [37].

3.4.2 Sawdust

Figure 7 shows the spectra of untreated and treated sawdust. The band of approximately $3453 \,\mathrm{cm}^{-1}$ is caused by the O-H stretching vibration which shows the increased intensity during treatment. This indicates an increase in

Table 4. Chemical compositions of the clar

Components	SiO_2	Al_2O_3	$\mathrm{Fe}_{2}\mathrm{O}_{3}$	CaO	MgO	SO_3	K_2O	Na ₂ O	LOI (Loss on ignition)
Percentage (%)	35.21	13.62	5.64	15.53	3.41	0.35	3.85	0.10	22.20



Fig. 5. X-ray diffractions patterns of US (untreated sawdust), STA(sawdust treated with alkaline NaOH), STK(sawdust treated with $KMnO_4$).

the oxygen content of the sawdust. While the band at 2920 cm^{-1} corresponds to the stretching vibration of the C-H bonds of the -CH and $-\text{CH}_2$ group of the cellulose, lignin and hemicellulose segments [38]. The band at 1640 cm^{-1} is credited to the carbonyl groups C=O (1744 cm^{-1}) which represent hemicelluloses. These, however, disappear during sawdust processing due to the significant extraction of hemicellulose from the sawdust during processing. The 1640 cm^{-1} band is credited to the stretching and vibration of the H-OH bond of the water [36]. Nevertheless, the 1420 cm^{-1} band is credited to the deformation vibrations of the C-H bonds of the aromatic rings. But the 1264 cm^{-1} band is related to the deformation of the acetyl groups (xylans) of lignin. Finally, the 1030 cm^{-1} band is affected by an elongation of the C-O of cellulose [38–40].

Table 5. Crystallinity Index (CrI %) of untreated and treated sawdust.

Materials	US	STA	STK
I ₀₀₂	95	372	324
I_{am}	78	201	197
CrI~(%)	17.89	45.96	39.19







Fig. 7. FTIR spectra of untreated and treated sawdust.

3.4.3 Composites

Figure 8 illustrates the spectra of composites reinforced with treated and untreated sawdust. The band of approximately $3705-3428 \text{ cm}^{-1}$ is caused by the O-H stretching vibration which resembles the intensity in-



Fig. 8. FTIR spectra of composites: US/Cl, STA/Cl, and STK/Cl.

creased during treatment. However, this indicates the increase in oxygen in the sawdust. This increase could be attributed to cleavage of the sensitive bonds caused by the treatment [41]. The band of about 2513 cm^{-1} personifies the vibration valence (CH) of the cellulose which increases with treatment. In addition, it should be noted that the 1441 cm^{-1} bands converge towards the CH₃ elongation vibration. Finally, the 1024 cm^{-1} band represents the valence vibrations of the CO and COC bonds of cellulose [42].

3.5 SEM analysis

Figure 9 shows the SEM micrographs of the composite reinforced with treated and untreated sawdust. Figure 9a, based on the results obtained by XRD, indicates the presence of carbonates and quartz in the sample, so that the carbonates (calcite) appear as clear accumulations and the quartz as small grains [43,44]. Based on Figure 9a, we can also note the emergence of cavities of different sizes. leading to the brittleness of the clay matrix. On the other hand, we can obtain a homogeneous microstructure with small cavities and fewer cracks if the clav is reinforced with untreated sawdust, as shown in Figure 9b. In addition, as shown in Figure 9c, the alkali-treated sawdust composite shows a remarkable decrease in cavities, where the majority of the pores are welded and the material becomes more compact. The alkali treatment dissolves the wax layers on the outside of the sawdust, removing most of the lignin and pectin, resulting in a rougher surface.

The same observation has been made by other researchers who have suggested that this roughness can be attributed to the removal of hemicellulose, lignin and cellulosic components after treatment, which facilitates mechanical bonding between the sawdust and the clay matrix [45–47]. In addition, the same observation was made based on Figure 9d where a substantial reduction of cavities is strongly detected. This, however, contributed to the adhesion between the clay matrix and the sawdust treated with potassium permanganate. Consequently, the permanganate treatment has a positive effect on the improvement of the mechanical properties of the composite under study because it sculpts the surface of the fibers and makes them rougher due to the interaction of permanganate with the lignin components, and it may also reduce the hydrophilic nature of the sawdust [48–51].

3.6 Mechanical tests

3.6.1 Effect of untreated sawdust on mechanical properties

It is generally assumed that the addition of fibers to materials would affect their properties and behavior. In this experiment, sawdust was added to clay; Table 6 lists the average values of the compression and bending results. It should be noted that the sample set was based on different ratios with a minimum of three samples for each proportion. The results show that the addition of sawdust increases the compressive strength from 4.3 to 4.70 MPa (approximately 9%). Similarly, the addition of sawdust increases flexural strength by 0.77 to 0.84 MPa. The increase in compressive strength is related to the homogeneity of the microstructure due to the presence of fewer pores, as demonstrated by the SEM tests (Figs. 9c and 9d). In addition, the addition of sawdust at 2.5%indicates a significant decrease in flexural and compressive strength (0.80 MPa and 4.65 MPa) as the composite brittles. Therefore, the optimum percentage of sawdust required to produce a high-strength clay matrix with significant strength is 2%.

3.6.2 Effect of treated sawdust on the mechanical properties

Figures 10 and 11 shows the mechanical strength of the samples studied with treated/untreated sawdust. The results obtained show an improvement in the mechanical characteristics after the treatment. The maximum bending strength values are 0.89 MPa and 0.88 MPa for the alkalitreated sawdust composite and the permanganate-treated sawdust composite, respectively. In addition, the treated sawdust composite had the highest compressive strength (4.87 MPa) and (4.82 MPa) than the untreated composite (4.70 MPa). Consequently, the mechanical strength could be improved and modified when the material is treated. However, this could be justified by the improved adhesion between the sawdust and the mineral matrix [19]. This behavior is primarily related to the effects of chemical treatment of the material. This treatment, however, eradicates amorphous materials on the outer surface of the sawdust and dissolves part of the lignin and pectin. Consequently, it helps to produce a correct and rough surface that improves the interfacial adhesion of the matrix and reinforcements [52,53].

3.7 Thermal properties

Table 7 lists the thermal properties of the studied composites. The results confirm that the thermal conductivity of the composites reinforced with untreated sawdust $(0.37 \text{ W.m}^{-1}.\text{K}^{-1})$ is lower than that of raw clay $(0.44 \text{ W.m}^{-1}.\text{K}^{-1})$. In addition, it can be noticed that the thermal conductivity of the treated sawdust composite increases with different ratios to record 0, $41 \text{ W.m}^{-1}.\text{K}^{-1}$ for the NaOH and 0, $40 \text{ W.m}^{-1}.\text{K}^{-1}$ for the KMnO₄. Therefore, the



Fig. 9. SEM micrographs: (a) clay (Cl); (b) US/Cl composite; (c) STA/Cl composite; (d) STK/Cl composite.

$\begin{array}{c} \text{Composition} \\ \text{Clay} + \text{Lime 12 (wt) \%} \end{array}$	Sawdust ratio (%)	Flexural strength (MPa)	Compressive strength (MPa)	
	0	0.77	4.3	
	0.5	0.78	4.59	
	1	0.80	4.60	
US/Cl	1.5	0.82	4.63	
	2	0.84	4.70	
	2.5	0.80	4.65	

Table 6. The results of the mechanical tests of the prepared mixtures.

thermal conductivity of the treated sawdust changes when it increases by about 11% (NaOH) and 8% (KMnO₄) compared to the untreated sawdust composite. This is due to the chemical treatment which improves interfacial adhesion as it leads to a decrease in voids, thus facilitating heat transfer [54]. It can be seen that the addition of the untreated sawdust to the clay matrix resulted in a decrease in the thermal conductivity of the composite since it generates porosity and air bubbles. This last point is important because it reduces the thermal conductivity [55,56], which improves the thermal insulation of the composite. The same observation was confirmed by H. Chaib et al. [57], the authors reported that the addition of date palm to the clay matrix leads to a decrease in thermal conductivity.

4 Conclusion

This study was conducted to evaluate the mechanical and thermal properties of the sawdust-reinforced matrix-clay composite. A composite was prepared from clay originated from Bou-Saâda region (south-eastern Algeria) and mixed with sawdust at mass contents varying from 0.5 to 2.5% with a 0.5% step. The results indicate that the addition of



Fig. 10. Addition effect of treated and untreated sawdust on the flexural strength of the studied composite.



Fig. 11. Addition effect of treated and untreated sawdust on the compressive strength of the studied composite.

Table 7.	Thermal	properties	of composites.	

Samples	$\begin{array}{l} {\rm Thermal\ conductivity} \\ \lambda({\rm W.m}^{-1}.{\rm K}^{-1}) \end{array}$	Thermal diffusivity $E (mm^2.s^{-1})$	Specific heat C (MJ.m ^{-3} .K ^{-1})
Cl	0.44	0.39	1.12
US/Cl	0.37	0.42	0.89
STA/Cl	0.41	0.50	0.82
STK/Cl	0.40	0.43	0.93

sawdust in the clay matrix allows to reduce cavities and to obtain a more homogeneous structure thanks to the adhesion between the matrix and the sawdust. This nevertheless contributes to the physical, mechanical and thermal properties of the sample studied.

Furthermore, the results show that the chemical treatment of the sawdust improves the mechanical properties (flexural and compressive strength) of the composite for several reasons, including: non-propagation of cracks, good adhesion of the sawdust in the matrix and the presence of cellulose in the sawdust due to its good flexural strength. In addition, the composite reinforced with the untreated sawdust shows a significant decrease in thermal conductivity. It can therefore be concluded that these composites can be used as an alternative to cement in construction or as thermal insulation. Additional research is underway to improve the durability and strength of these composites in the future.

References

- 1. C.D. Atis, O. Karahan, Properties of steel fiber reinforced fly ash concrete, Construct. Build. Mater. 23, 392 (2009)
- 2. M. Tlijania, A. Gouadriab, R. Benyounesc, J.F. Durastantid, A. Mazioude, Study and Optimization of Palm Wood Mechanical Properties by Alkalization of the Natural Fiber, Int. J. Sci.: Basic Appl. Res. 11, 30 (2013)

- 3. E. Simard, Les matériaux de construction résidentielle une perspective durable: analyse comparative, Université de Sherbrooke, Québec, Canada, 2009, 9 p.
- 4. M.R. Khelifa, Formulation et caractérisation d'éco-bétons renforcés aux fibres d'alfa pour des bâtiments verts et durables, Thèse de doctorat, Cergy-Pontoise, 2017
- 5. D. Silveira, H. Varum, A. Costa, T. Martins, H. Pereira, J. Almeida, Mechanical properties of adobe bricks in ancient constructions, Construct. Build. Mater. 28, 36 (2012)
- 6. S. Yetgin, Ö. Çavdar, A. Çavdar, The effects of the fiber contents on the mechanic properties of the adobes, Construct. Build. Mater. 22, 222 (2008)
- 7. R. Bodîrlău, I. Spiridon, C.A. Teacă, Influence of components ratio upon mechanical properties of wood/thermoplastic polymer composites, Cell. Chem. Technol. 43, 205 (2009)
- 8. N. Benmansoura, B. Agoudjila, A. Gherablia, A. Karechea, A. Boudenne, Thermal and mechanical performance of natural mortar reinforced with date palm fibers for use as insulating materials inbuilding, Energy Build. 81, 98 (2014)
- 9. P. Lertwattanaruk, A. Suntijitto, Properties of natural fiber cement materials containing coconut coir and oil palm fibers for residential building applications, Construct. Build. Mater. 94, 664 (2015)
- 10. B. Mazhoud, C. Florence, P. Sylvie, L. Christophe, Mechanical properties of hemp-clay and hemp stabilized clay composites, Construct. Build. Mater. 155, 1126 (2017)
- 11. M. Rokbi, H. Osmani, A. Imad, N. Benseddig, Effect of chimical treatement on flexure Properties of natural fiberreinforced polyster composite, Proc. Eng. 10, 2092 (2011)

- B.F. Yousif, A. Shalwan, C.W. Chin, K.C. Ming, Flexural properties of treated and untreated kenaf/epoxy composites, Materials & Design 40, 378 (2012)
- 13. Z. Koadri, A. Benyahia, N. Deghfel, K. Belmokre, B. Nouibat, A. Redjem, Étude de l'effet du temps de traitement alcalin de fibre palmier sur le comportement mécanique des matériaux à base d'argile rouge de la région de M'sila, Matériaux & Technique **107**, 404 (2019)
- 14. S. Fertihk, M. Merzoud, M. Bounadja, A. Ghania, A. Benazzouk, Caractéristiques mécaniques des composites à base de fibres de diss dans différentes matrices, in: Journées de l'AUGC et IBPSA, France, 2012
- 15. A. Djohore, S.A. Djomo, M.O. Boffoue, E. Emeruwa, Effet de l'addition de fibre de coco traitées à la potasse sur les propriétés mécaniques des matériaux de construction à base d'argile-ciment, Eur. Sci. J. 14, 104 (2018)
- N. Laaroussi, A. Cherki, M. Garoum, A. Khabbazi, A. Feiz, Thermal properties of a sample prepared using mixtures of clay bricks, Conf.: Sustain. Build. Technol. Energy Proc. 42, 337 (2013)
- P. Meukam, Valorisation des briques de terre stabilisées en vue de l'isolation thermique de bâtiments, Thèses de doctorat, Université de Yaoundé I, 2004
- Y. Elhamdouni, A. Khabbazi, C. Benayad, A. Dadi, O.I. Ahmid, Effect of fiber alfa on thermophysical characteristics of a material based on clay, Energy Proc. 74, 718 (2015)
- B. Taallah, Étude du comportement physico-mécanique du bloc de terre comprimée avec fibres, Thèse de doctorat, Université de Mohamed KhiderBiskra, Algérie, 2014
- S. Ajouguim, S. Talibi, C. Djelal-Dantec, et al., Effect of alfa fibers on the mechanical and thermal properties of compacted earth bricks, Mater. Today: Proc. 1, (2020)
- 21. A. Layeb, A. Halboui, Caractérisations physico-mécaniques d'un bloc de terre stabilisé avec la chaux à base des fibres (déchet de pneu uses-verre), Mémoire de fin d'étude, Université Ziane Achour, Algérie, 2017, 5 p.
- 22. Catalogage à la source bibliothèque de l'OMS, Directives de qualité pour l'eau de boisson, 2^e éd. (vol. 2), Genève, 2000, pp. 222–327
- 23. L. Segal, J.J. Creely, A.E. Martin, C.M. Conrad, An empirical method for estimating the degree of crystallinity of native cellulose using the X-ray diffractometer, Text. Res. J. 29, 786 (1959)
- Y. He, Rapid thermal conductivity measurement with a hot disk sensor Part 1.Theoretical considerations, Therm. Chim. Acta. 436, 122 (2005)
- I. Jarraya, S. Fourmentin, M. Benzina, Adsorption de Cov par un matériau argileux tunisien organo-Modifié, J. Soc. Chim. Tunis. 12, 139 (2010)
- A. Qlihaa, S. Dhimini, F. Merahaka, N. Hajjaji, A. Srhiri, Caractérisation physico-chimique d'une argile Marocaine, J. Mater. Environ. Sci. 7, 1741 (2016)
- 27. B. Sorgha, P. Bressollier, B. Guel, et al., Étude des propriétés mécaniques des géomateriaux argileux associant la décoction de parkiabiglobosa (néré), C. R. Chim. 1, (2016)
- H. Sadki, K. Ziat, M. Saidi, Adsorption d'un colorant cationique d'un milieu aqueux sur argile locale activée, J. Mater. Environ. 5, 2060 (2014)
- B.A.P. Ass, M.N. Belgacem, E. Frollini, Mercerized linters cellulose: Characterization and acetylation in N,N-dimethylacetamide/lithium chloride, Carbohydr. Polym. 63, 19 (2006)

- V.P. Kommula, K. Obi Reddy, M. Shukla, T. Marwala, in: International Conference on Advances in Marine, Industrial and Mechanical Engineering (ICAMIME'2014), Johannesburg (South Africa), 2014
- K. Obi Reddy, C. Uma Maheswari, M. Shukla, J.I. Song, A. VaradaRajulu, Tensile and structural characterization of alkali treated Borassus fruit fine fibers, Compos. Part B: Eng. 44, 433 (2013)
- B. Wang, L. Tabil, S. Panigrahi, Effects of chemical treatments on mechanical and physical properties of flax fiber-reinforced composites, Sci. Eng. Compos. Mater. 15, 43 (2008)
- 33. A. Ayse, S. Mohini, Isolation and characterization of nanofibers from agricultural residues–Wheat straw and soy hulls, Bioresour. Technol. 99, 1664 (2008)
- 34. W. Liu, A.K. Mohanty, L. Drzal et al., Effects of alkali treatment on the structure, morphology and thermal properties of native grass fibers as reinforcements for polymer matrix composites, J Mater Sci. 39, 1051 (2004)
- 35. A. Lahsini, J. Bentama, A. Addaou, M. Rafiq, Caractérisation physico-chimique et étude de frittage d'une argile destinée à l'élaboration de membranes de filtration tangentielle, J. Chim. Phys. 95, 1001 (1998)
- M. Gourouza, A. Zanguina, I. Natatou, A. Boos, Characterization of a mixed clay Niger, Rev. CAMES Sci. Struct. Mat. 1, 29 (2013)
- 37. A. Arfane, A. Salhi, M. El Krati, et al., Étude cinétique et thermodynamique de l'adsorption des colorants Red 195 et Bleu de méthylène en milieu aqueux sur cendres volantes et les mâchefers, J. Mater. Environ. Sci. 5, 1927 (2014)
- 38. R. Harrechie, Élaboration et caractérisation des composites à matrice polymère et à renforts végétal et nanométrique lamellaire, Thèse de magister, Génie des polymères, Université Frahat Abbas, Setifl, Algérie, 2015, 68 p.
- 39. C. Amandine, G. Olivier, J. Frédéric, F. Sylvain, Utilisation de la spectrométrie infrarouge pour une quantification rapide du taux d'humidité dans des fibres végétales, Revue des composites et des matériaux avancés 24, 81 (2014)
- S. Benyoucef, D. Harrache, Caractérisation de la microstructure de sciure de bois de pin sylvestre, J. Mater. Environ. 6, 765 (2015)
- V. Tserki, N.E. Zafeiopoulos, F. Simon, C. Panayiotou, A study of the effect of acetylation and propyonylation surface treatments on natural fibers, Compos. Part A. 66, 1110 (2005)
- 42. A. Elabed, Réactivité thermique et cinétique de dégradation du bois d'arganier application à l'élaboration de charbon actif par activations chimique à l'acide phosphorique, Thèse de doctorat de l'Université Mohammed V-Agdal, Rabat, Maroc, 2007, 142 p.
- 43. A. Arib, A. Sarhiri, R. Moussa, T. Remmal, M. Gomina, Caractéristiques structurales et mécaniques de céramiques à base d'argiles : influence de la source de feldspath, C. R. Chim. 10, 502 (2007)
- 44. H. Chemani, Nouvelle formulation d'éléments de construction, bioclimatiques par ajout d'eau rejetée d'abattoir, in: 23^e Congrès Français de Mécanique, 2017
- 45. A. Benyahia, A. Merrouche, Effect of Chemical Surface Modifications on the Properties of Alfa Fiber-Polyester Composites, Polym.-Plastics Technol. Eng. 53, 403 (2014)
- 46. P. Ramadevi, S. Dhanalakshmi, C.V. Srinivasa, B. Basavaraju, Effect of alkali treatment on water absorption of single cellulosic abaca fiber. BioResour. 7, 3515 (2012)

- 47. J. Rout, M. Misra, S.S. Tripathy, S.K. Nayak, A.K. Mohanty, The influence of fibre treatment on the performance of coir-polyester composites. Compos. Sci. Technol. 61, 1303 (2001)
- 48. A. Benyahia, A. Redjem, Z. Rahmouni, A. Merrouche, Study of the mechanical properties of a composite material: Alfa fibers/unsaturated polyester, Rev. Română de Mater. / Roman. J. Mater. 46, 25 (2016)
- 49. S. Dhanalakshmi, P. Ramadevi, G.R. Raghu Patel, et al., Effect of fiber loading and surface modification ontensile behaviour of natural areca composites, Ciencia-e-Tecnica Vitivinicola 29, 99 (2014)
- P. Ramadevi, S. Dhanalakshmi, B. Basavaraju, V.B. Pramod, Study of the effect of chemical treatments on the tensile behavior of abaca fiber reinforcedpolypropylene composites. J. Adv. Chem. 10, 2803 (2014)
- M.M. Rahaman, A.K. Mallik, M.A. Khan, Influences ofvarious surface treatments on the mechanical anddegradable properties of photografted palm fibers, J. Appl. Polym. Sci. 105, 3077 (2007)

- 52. Y. Cao, S. Shibata, I. Fukumoto, Mechanical properties of biodegradable composites reinforced with Bagasse fiber before and after alkali treatments, Compos. PartA: Appl. Sci. Manuf. 37, 423 (2006)
- D. Sedan, C. Pagnoux, A. Smith, T. Chotard, Mechanical properties of hemp fiber reinforced cement: Influence of the fiber/matrix interaction. J. Eur. Ceram. Soc. 28, 183 (2008)
- A.G. Kulkarni, P.K. Rohatgi, Electrical resistivity and dielectric strength of plant fibers, J. Mater. Sci. 16, 1719 (1981)
- G. Kelling, S. Kapur, N. Sakarya, E. Akça, C. Karaman, B. Sakarya, Basaltic tephra: potential new resource for ceramic industry, Br. Ceram. Trans. 3, 129 (2000)
- 56. K. Lui, L. Lu, F. Wang, W. Liang, Theoretical and experimental study on multi-phase model of thermal conductivity for fiber reinforced concrete, Construct. Build. Mater. 148, 465 (2017)
- 57. H. Chaib, A. Kriker, A. Mekhermeche, Contribution to the study of the thermomechanical properties of the adobe (case of the south of Algeria Ouargla), Int. J. Civil Eng. Technol. (IJCIET) 3, 24 (2020)

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