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Study of thermal and mechanical performance of cement matrix materials reinforced by nanofibers cellulosic of date palm waste

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Abstract

Recently, the importance of environmental issues has made scholars to focus on the implementation of bio-degradable resources in different applications. This project aims to determine the technical feasibility of introducing raw palm waste into cement to develop a lightweight construction nanocomposite material and efficient thermal insulation. Furthermore, the characterization of extracted nanofibers cellulose was carried out through Fourier transform infrared spectroscopy (FT-IR), scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS) analysis of the date palm waste were investigated to characterize the microstructure and the chemical composition of the samples. Moreover, the characterization carried out by X-ray diffraction (XRD) analysis, confirmed the control of the micro and nanoscale dimensions of the fibrils. On the other hand, in nanocomposites, the results experimentally revealed a significant decrease in the coefficient of average thermal conductivity (λ) in the range of 0.0732 and 0.168 $W.m^{-1}.K^{-1}$; with the increase in the mass fraction of nanofibers induces a gradual in the cement matrix (3%, 5%, 10% and 15%). In addition, the nanocomposite demonstrated a good adhesion state of the nanofiber/matrix interface and acceptable mechanical properties with a maximum compressive and flexural strength of 4.54 MPa and 2.62 MPa respectively, which correspond to material standards range used in non-load-bearing masonry defined by (ASTM C109 / C 109-95). These thermal and mechanical performances are competitive with those of other insulating bio-based materials available on the market.

Keywords: Nanofiber cellulose of date palm (NFC); structural and morphological characterization; biocomposite; thermal conductivity; mechanical strength.

1. Introduction

Today, some countries produce agricultural waste that is not well valued. Algeria is one of the countries in North Africa that has a large amount of waste from unvalued palm trees. So forth, the palm tree forests of the Algerian oases are also highly regarded for their recreational, aesthetic, spiritual, and natural values. The palm (Phoenix dactylifera L.) is considered one of the oldest tree species. For the past 7,000 years, the plant has been vital to the society, environment, and economy, especially in the Middle East and North Africa in countries like Saudi Arabia, Egypt, UAE, and Algeria [1].These by-products are commonly treated as rubbish, but recently they are used as thermal insulation materials in buildings [2,3,4–5].Therefore, plant fibers have attracted the attention of a large industrial market aimed at sustainable development by producing new products that are green and sustainable [6]. The selection depends on the desired characteristics of the final composite, engineered for such special application [7]. Plant fibers are promising and can be potential competitive to glass fibers because of their availability worldwide. Many researchers dealt with the use of palm trees by-products in construction materials due to their high thermal insulation properties. Different fibers can be extracted from date palm plants. These fibers are part of the petiole, rachis [8], bunch [9], fibrillium [10], leaves [11], palm leaflets, stalk fruit, date palm stone, palm coir [12], thorns, spathe and pedicels [13], leaf stalk, leaf sheath [14]. Several studies were conducted on long fibers alone or as reinforcements in composites. Natural fiber is currently used for strengthening purposes in industrial applications, including automotive, packaging, and structural components where there is no need for a high loading capacity [15]. The impact of petiole and rachis of palm trees on thermal conductivity, compressive and flexural strengths of gypsum based composite materials has been analyzed by [2]. The effect of the same fibers on thermal conductivity and compressive strength of cement based composite materials has been investigated by [3]. Mechanical properties of reinforced concrete with Algerian DPF have been evaluated by [16]. The effect of the addition of Algerian date palm fibers on thermal and mechanical properties of plaster concrete has been studied by [17,18]. The advent of nanomaterials promises a new range of tailored products that can intervene strategically in the cementitious system through a combination of physical and chemical means. Recently, we believe that the application of nanotechnology in cement and concrete remains limited on a commercial level, particularly given their relative cost differential [19,20-21].

All of these researchers have conducted experimental investigation on thermal insulation as well as on mechanical properties of composite construction materials incorporating various date palm fibers. Very interesting results were found leading to the conclusion that, adequate addition of date palm fibers to commonly used construction materials, such as mortar, significantly enhance their thermal insulation properties while their mechanical strengths are still acceptable. The objective of this study is to develop an energy efficient composite material that consists of cement matrix materials reinforced with date palm nanofibers. The bio-composite material is experimentally characterized in terms of thermal conductivity, as well as compressive and flexural strengths.

2. Materials and methods

2.1. Materials and samples preparation

The cellulosic nanofibers used are derived from the recovery of date palm waste from Toulgua province of Biskra, Algeria. Their preparation requires physical treatment described in figure1.

- *Extracting cellulose nanofibers from Date palm waste by ball milling*

Ball milling is an environmental-friendly and low-cost method, which uses friction, collision, shear or other mechanical actions to modify the crystalline structure of materials, such as alloys and polymers [22]. In consideration the compactness and hardness of date palm waste, the refinement and miniaturization of granulometry requires the handling of several types of grinders at different stages of grinding, in order to reduce the granulometry of the grains into particles and then in fine and ultrafine powders. Crushing is done by a planetary micro-shredder at ambient temperature for 8 hours with a speed of 400 rot/min, cycle 11. The time of stay of the material is about 8 hours. During high-energy grinding and each collision, the powder grains are refined and ground below the border of microns.

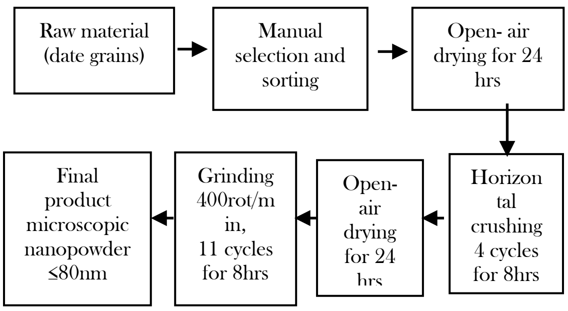

Figure 1. Diagram showing the stages in the preparation of date grain powder.

- *Cement*

The matrix used is cement (Portland CEM I 42.5 N) supplied by the cement factory of Hamma Bouziane, Constantine, Algeria, complies with Algerian standards NA 422 and NA 44. The chemical characteristics of this matrix are carried on Table 1.

Table1: The chemical components of Date Palm Waste and Portland Cement analyzed by energy dispersive spectroscopy (EDS)

Component	Cement	Date palm waste
	Percentage (Wt. %)	Percentage (Wt. %)
C	6.62	50.17
O	39.58	44.52
Na	0.52	0.12
Mg	0.99	0.16
Al	2.97	0.24
Si	9.05	0.60
S	1.05	0.18
K	0.66	0.49
Ca	36.72	2.89
Ti	0.14	0.00
Fe	1.70	0.20
Cl	0.00	0.28
P	0.00	0.15

- *Sand*

CEN standardized sand (ISO standardized sand) is a clean, natural and siliceous sand, notably in its finest fractions. The grains are generally isometric and rounded. The silica content is at least equal to 98%. The granulometric composition determined by sieving is in line with the requirements of EN 196-1 standards must be between the limits defined in the table 2.

Table 2: Granulometric composition of CEN reference sand (ISO standardized sand)

Square mesh dimensions (mm)	Cumulative refusal on sieve (%)
0.08	99 ± 1
0.16	87 ± 5
0.50	67 ± 5
1.00	33 ± 5
1.60	7 ± 5
2.00	0

2.2. Protocol of manufacturing the pellets (NFD) and Cement Matrix

The nano composite samples were prepared with a doping ration to cellulosic nanofibers of date nucleus opted and selected respectively (3% - 5% - 10% - 15%) relative to the mass of the cement matrix (1.gm). This composite is weighed in a precision scale (kern) and then kneaded to ensure its homogeneity with a vibration mixer, afterwards formulated dry in monolith with the help of a pelletizer, their final dimension is in the order of: (Diam - 10mm - Eps 1mm).

3. Characterization of the materials

Three microstructure analyses were conducted: X-ray diffraction (XRD) analysis, Scanning Electron Microscopy (SEM) and Fourier Transformation- Infra Red (FT-IR) analysis.

3.1. X-ray diffraction (XRD) analysis

X-ray diffraction has been used to determine the crystallinity of date nucleus after different treatments. The milled powder sample was placed and leveled on the sample holder to obtain uniform exposure to X-rays. The samples were analyzed using an X-ray diffractometer (The X'Pert PRO PANalytical diffractometer) at room temperature (RT) with a source of monochromatic CuKα radiation (= 0.1539 nm) in step-by-step scanning mode with an angle of 2θ between 10 ° C to 100 ° C range with a step of 0, 04 and a scanning time of 5.0 min.

3.2. Fourier transform infrared (FT-IR) spectroscopy analysis

FT-IR is a rapid analytical technique ideal for the initial classification of organic residues into groups with broadly comparable chemical composition. The infrared device is used to identify organic molecules by allowing the conservation of the fingerprint. The infrared spectroscopic method is based on interaction (electromagnetic radiation - matter) by Attenuation Reflection (A.T.R) or by Transmission. The samples are exposed directly to IR spectroscopy analysis. The infrared absorption spectra of date palm nanofibers are measured before and after the addition of (NFC) to the chemical composition of Portland cement, using a JASCO FT / IR-6300 Fourier Transform Infrared Spectrometer. The conditions used were 64 scans at a resolution of 4 cm^{-1} measured between 4000 and 600 cm^{-1}.

3.3. Scanning electron microscopy (SEM)/energy dispersive spectroscopy (EDS) analysis

The observations by scanning electron microscopy were characterized by scanning electron microscopy (SEM) in the environmental mode (ESEM). The waste and stabilized / solidified material samples (date palm nanofibers and Portland cement) are analyzed in solid state (pastille) in order to expose the maximum of the surface layer to the incident electrons. (See Figure 5). Energy dispersive spectroscopy (EDS) was used to analyze the chemical composition of date palm nanofibers and the Portland cement surface by using QUANTA200 FEI.

3.4. Thermal testing

Six pastilles (NFC0, NFC3, NFC5, NFC10, NFC15 and NFC100), of the same mass (1gm) composed of Portland cement, are prepared with the addition of date palm nanofiber mass of 0%,3%, 5%, 10%, 15% and 100%, respectively. The thermal conductivity of the six samples shown in Figure 2 is determined using a thermal conductivity instrument for automated testing. The measurements of the thermal conductivity of nano composites are carried out experimentally with the assistance of the WL 372 apparatus. It includes a linear and radial experimental assembly. These pastilles are placed in the middle of a cylinder with a heating element and other refrigerants connected with sensors record the temperatures at all pertinent points.

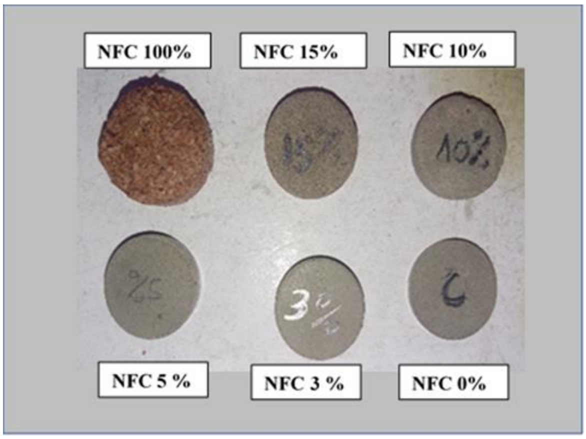

Figure 2. Doping ration to date palm nucleus cellulosic nanofibers with a cement matrix.

3.5. Mechanical testing

Cement pastes were prepared at a constant water / cement ratio (w / c) of 0.50 with five different NFC fractions (0%, 5%, 10% and 15%). The NFC fraction was calculated based on the weight of the NFCs relative to the weight of the cement particles. Then, the cement particles and the NFC suspension were added to a mixer and kneaded at a speed of 500 rpm for 120 s. After, the mixture was stopped for 15 s to scrape the wall and the bottom of the bowl, followed by another 120 s of mixing at 600 rpm. Next, the fresh cement pastes were poured into prismatic alloy steel molds of (40 × 40 × 160) mm3 and vibrated mechanically in a shock table for two minutes 120 shocks in order to eliminate air bubbles and reduce the voids. The cement samples were sealed at 20 ± 1 ° C and 50% relative humidity for hardening. After 24 ± 1-hour aging, the prismatic samples were removed from the mold and returned to the environment. The measurements of the compression and flexural strength of prism samples are carried out experimentally with the assistance of MATEST testing machines with dual measuring range.

4. Results and discussions

4.1. X-ray diffraction (XRD) analysis

We report in Fig.3. the XRD of the constituents of the date nucleus nanoparticle. We observe a weak broadening of the diffraction peak which is due to submicronic grains size of the component of the date kernel powder. The peaks of the XRD spectrum have been fitted by Gaussian functions. Using the Scherrer formula, the grain size was situated in the same range of 3.4–6.3 nm. We notice (fig.3.) nine peaks situated, respectively, at 2θ= 15.82 °, 18.04 °, 20.05 °, 23.29 °, 25.10 °, 26.56 °, 29.92 °, 33.11° and 38.62°. These summits correspond of the typesetting of the date nucleus nano powder such as SiO2, $(C_6H_{10}O_5)$ n, $CaCO_3$ and P2O5 (JCPD card N° 46–1045, 47–2462, 23–1302, and 41–1475). According to the table, no peak for the other constituents of the date nucleus, which have a weight less than 1%, have been detected.

Table 3: Size and strain of date constituents

N°	2θ	D [Å]	Crystallite size [nm]	Lattice strain [%]	Constituents of date palm
1	15,82	5,60	3,8	6,818	P_2O_5
2	18,04	4,91	5,3	0,254	SiO_2
3	20,05	4,42	5,2	4,019	SiO_2, $(C_6H_{10}O5)_n$
4	23,29	3,81	5,2	3,447	SiO_2, $(C_6H_{10}O_5)_n$, P_2O_5
5	25,10	3,53	6,3	0,050	SiO_2
5	26,56	3.32	3.4	0.051	SiO_2, $(C_6H_{10}O_5)_n$, $CaCO_3$
7	29,92	2,70	15	0,152	$(C_6H_{10}O_5)$ n
8	33,11	2,33	5,3	2,390	SiO_2
9	38,62	2,14	5,4	2,027	P_2O_5, $CaCO_3$

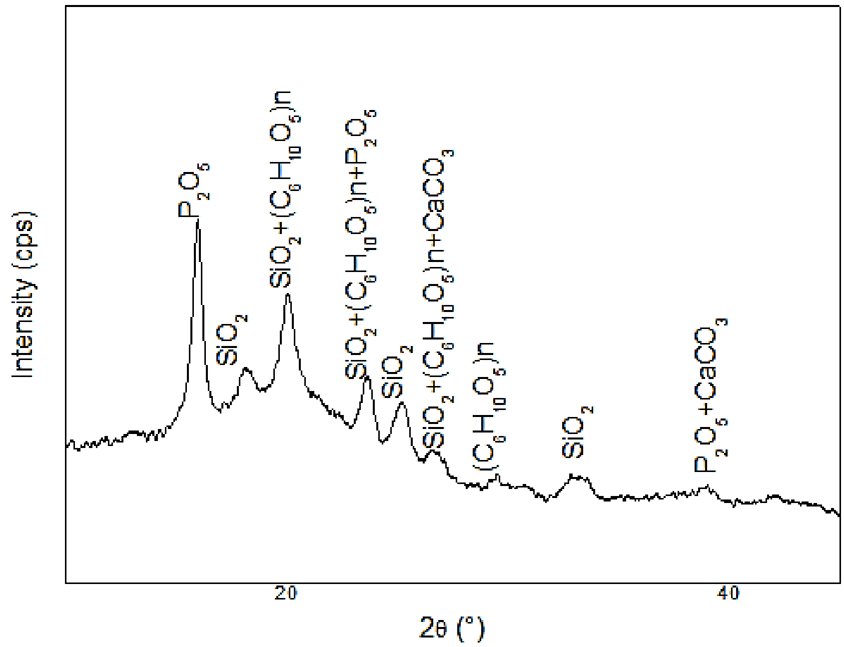

Figure 3. XRD of constituents of the date nucleus Nanopowder.

4.2. Fourier transform infrared (FT-IR) spectroscopy analysis

Figures 4. show results of FTIR spectra obtained for date palm nanofiber cellulose, after ball milling at different time intervals. The appearance of the three curved lines shows a similarity of the spectra of cellulose fiber and of all the nanofibers, thus indicating that their chemical compositions were similar. All samples had two main regions of absorbance, at low (500–1,800 cm^{-1}) and at high wavenumbers (2,700–3,500 cm^{-1}), in agreement with the report by [23]. The peaks in the 3300-3450 cm^{-1}, 2850-3000 cm^{-1}, 1470-1365 cm^{-1} and 1200-950 cm^{-1} regions are attributed to O–H stretching and bending vibrations, respectively, of hydrogen bonded hydroxy (OH) groups of cellulose [24].

The peaks at 2855-2950 cm^{-1} are assigned to the aliphatic saturated C–H stretching vibration alkyl (CH) groups of cellulose, hemicellulose and lignin [25].The peaks in the region 1415-1485 cm^{-1} are due to various lignin components [26] and C–H deformation of cellulose and lignin [27]. The presence of the peaks at 1710-1745 cm^{-1} and 1000 – 1300 cm^{-1} in date palm nanofiber cellulose is associated with the C = O stretching vibration of the acetyl and ironic ester groups. These groups are known to be present in pectin, hemicellulose, and/or an ester linkage of carboxylic group also known to be present in lignin and hemicellulose. The peaks at 1731-1750 cm^{-1} (C=O stretching of hemicellulose and lignin) and 1575-1620cm^{-1} (C=C aromatic skeletal vibration of lignin) do not appear in the cellulose fiber and all nanofibers. Peaks observed at wavenumber 3225-3610 cm^{-1}, 1485-1525 cm^{-1}, 995-1095 cm^{-1} and 740 -840 cm^{-1} are assigned to the aromatic alcohol. All the results from FT-IR analysis indicate no distinguish able difference occurred during ball milling in the spectra of cellulose nanofibers and cellulose fibers, retain their original molecular structure.

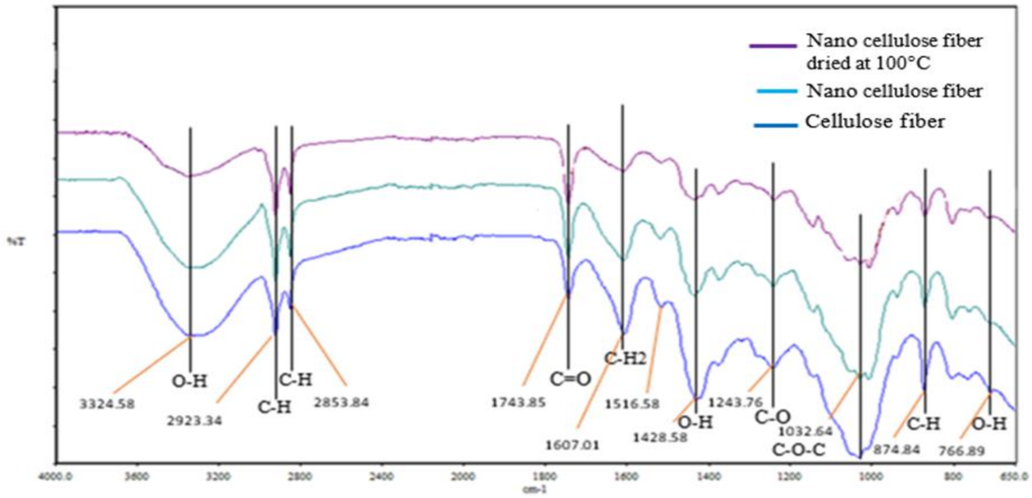

Figure 4. Comparison between FT-IR spectrums of different samples.

4.3. Scanning electron microscopy (SEM)/energy dispersive spectroscopy (EDS) analysis

Figure 5(a). shows the SEM image of the constituents of the date nucleus powders. We observe different aggregates formed by a coalescence of the constituents of the date nucleus nanocrystals. Energy dispersive spectroscopy (EDS) was carried out to investigate the composition of constituents of the date nucleus fig 5(b). The obtained results regarding the constituents of the date nucleus component powder revealed a carbon enriched composition with about 58.76 at %.

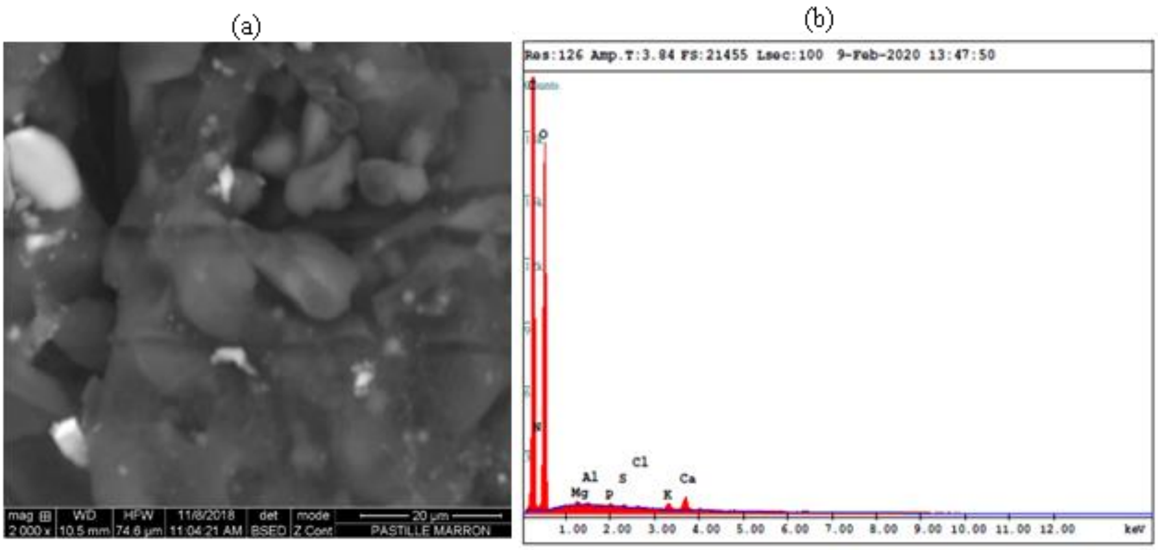

Figure 5. (a) SEM image of constituents of the date nucleus powder (b) EDS specter of date palm nanofiber.

The elemental composition of raw date palm nanofibers was determined from the peak areas and is summarized as shown in Table 4. Data represent the mean value of three independent quantification EDS spectrums tests. It was found that carbon and oxygen were the only consistent components in these materials. Other minor constituents in trace that were obtained from the analyses included sodium, magnesium, aluminum, silicon, phosphorus, sulphur, chlorine, potassium, titanium, iron and calcium.

Table 4: Chemical composition constituents of the date nucleus powder

Element	Weight %	Atom%	Intensity
C	50.17	58.76	1692.38
O	44.52	39.14	948.63
Na	0.12	0.08	11.41
Mg	0.16	0.09	22.87
Al	0.24	0.12	43.83
Si	0.60	0.30	133.42
P	0.15	0.07	33.09
S	0.18	0.08	44.26
Cl	0.28	0.11	65.06
K	0.49	0.18	106.60
Ca	2.89	1.02	590.63
Fe	0.20	0.05	19.06
Total	100.00	100.00	/

4.4. Thermal testing

Thermal conductivity is one of the most important properties of building materials. Fig 6 shows the influence of the incorporation of date palm nucleus nanofibers on thermal conductivity of the composite material. The measurement relative mean error of all the samples is about 2.4 %. One can see from Fig.6 that thermal conductivity decreases as the nanofiber content increases. The thermal conductivity of the $NFC_{(100)}$ pastille of natural nanofibers measured is very low 0.042 $W.m^{-1}.\ K^{-1}$. A similar result was reported by [28]. Compared to the $NFC_{(0)}$ pellet made of Portland cement which is 0.186 $W.m^{-1}.\ K^{-1}$. The thermal conductivity of the nano-composite $NFC_{(0)}$ goes from 0.186 $W.m^{-1}.\ K^{-1}$ at 0.168 $W.m^{-1}.\ K^{-1}$ for NFC3 (3%) by mass of nanofibers and 0.0732 $W.m^{-1}.\ K^{-1}$ for 15% of fibers (λ) decreases when the concentration of nanofibers increases.

There is a decreasing evolution which can be explained by the increase in concentration induced by a generation of more and more pores responsible for the decrease in (λ) which follows an almost linear regression as a function of the porosity of the composite. In addition, the increase in thermal conductivity with density and porosity has an always verified dependence on the materials of the mineral matrix and vegetable fibers according to [29] whose matrix is the basis of Portland cement. Density reduction can be considered as one of the advantages of using natural fibers mixed with cement, in fact the cements obtained are much lighter. A similar result was reported by [30,31] who studied the effect of the addition of wood chips and wood wool on the thermal properties of concrete also concluded that the addition of natural fibers improves the insulation characteristics of concretes.

It is important to note that the thermal conductivity of the $NFC_{(100)}$ ranges from (0.042-0.048 $W.m^{-1}.\ K^{-1}$) at about 29°C, which is a representative temperature for the building's insulating material. This thermal conductivity, is lower than date palm leaf base (petiole) (0.083 $W.m^{-1}.\ K^{-1}$) [25], rice straw (0.051-0.053 $W.m^{-1}.\ K^{-1}$) [32], and coconut fiber (0.057 $W.m^{-1}.\ K^{-1}$) [33]. It can be concluded that the reduction in thermal conductivity of the CNF cement matrix composite material is very interesting, in particularly for a relatively high-volume content of date palm nanofibers. Similar behaviour has been observed by several authors for composite materials reinforced with plant fibers [3,31].

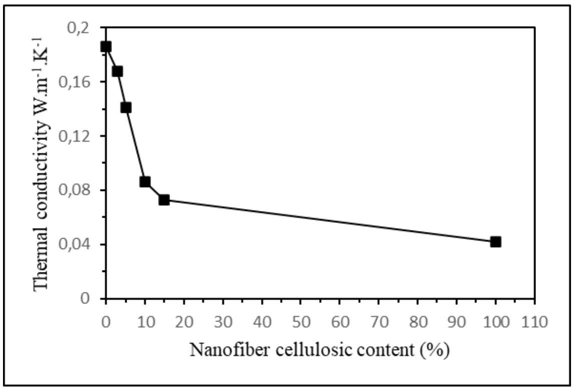

Figure 6. Evolution of thermal conductivity as a function of the content of Nanofibers cellulosic.

4.5. Mechanical testing

4.5.1. Compressive strength

Building materials are usually loaded with compression. For the composite studied, the influence of nanofiber concentration on compression resistance for different flood ages is shown in Figure 7, demonstrating that compression resistance significantly decreases with increased nanofiber concentration. There is a considerable loss (from 42.34 to 2.08 MPa) in mechanical resistance when the concentration of nanofibers increases from 0% to 15%. compared to the "control cement prism", due in part to the influence of the quantity of cement which is replaced by nanofibers. This drop is due to the porosity of the composite induced by the intra and extra nanofiber vacuum. The obtained results are in concordance with the literature [4,34]. The incorporation of cellulosic nanofibers into a mineral matrix makes the mortar less sensitive to flooding. Consequently, in the presence of NFC in large quantities, the compactness of concrete decreases causing the drop in resistance, according to spectroscopy "EDS" obtained for nanofibers which determined the presence of Zn is an element liable to delay hydration.

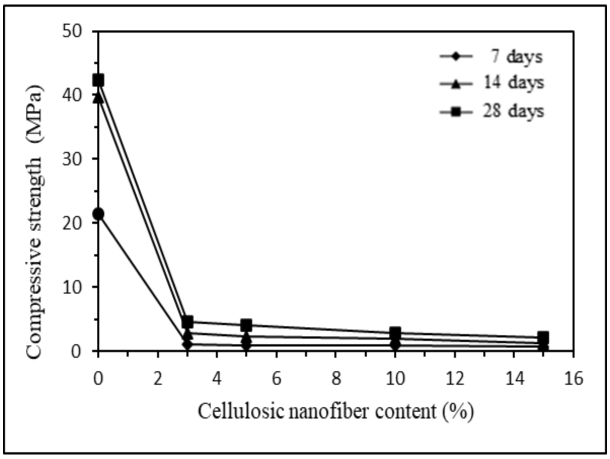

Figure 7. Compressive strength as a function of nanofiber content for different ages.

4.5.2. Flexural strength

The flexural strength evolution of the composite materials after the test is presented in figure 8. The figure shows that the 0% dosage of nanofibers "witness cement prism" revealed better resistance to compression and bending traction. Appending nanofibers from the date cores for a dosage of 3%, 5%, 10%, and 15%, in the various cases of cure (7,14, and 28) days, the pace of the curve indicates a significantly decrease in flexural strength compared to the "control cement prism". Indeed, addition of nanofibers supports the creation of pores within the samples. This behavior was reported by other researchers dealing with plaster and mortar reinforced with vegetal fiber [2, 35]. The composites have 3% by weight of the sample, which can improve flexural strength. Thus, we can confirm that the cement composite based on date core nanofiber exhibits an acceptable flexural strength performance.

The low sensitivity to the cure can be elucidated by the fact that nanofibers in favorable ratios in the cement matrix, would continue to moisturize the particles even in a dry environment. Differently in case the fibers are in minute amounts, not only would they not moisturize the particles but would create a network of open pores outside that would allow the material to be desiccated. In fact, the mechanical properties (compression and flexural strength) of the Cement / date palm nanofibers composite studied in this research show that this new biocomposite can be used as a light concrete compression resistance above the limit of (2MPa) defined by ASTM C 109C/ 109-95.

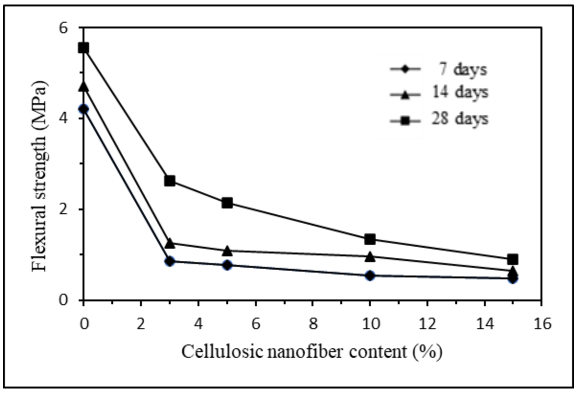

Figure 8. Flexural tensile strength as a function of nanofiber content for different ages.

5. Conclusion

The results of this study demonstrate the promise of using date palm biomass waste as raw material to produce nanofiber cellulosic. Ball milling for 24 hours successfully isolated nanofibers cellulose (NFC) with maximum crystallinity. Furthermore, the characterization of extracted nanofibers cellulose was carried out through Fourier transform infra-red spectroscopy (FT-IR), and scanning electron microscopy (SEM). The experimental results of X-ray diffraction (XRD) revealed high crystallinity of the cellulose nanofibers and showed that the average thickness was within the range 3.4–6.3 nm.

This experimental study analyzes the use of nanofiber cellulosic from date palm as reinforcement in cement matrix. The purpose is to evaluate the possibility of using the new biocomposite (FNC) as insulating material to reduce the heat loss in buildings. In addition, the thermal behavior study shows that increasing the concentration of NFC in the cement matrix decreases the thermal conductivity of composites. It is interesting to note that the influence of NFC pores on thermal properties is as evident as the NFC concentration. Then, it was experimentally demonstrated that the addition of 15% of NFC produced a composite with λ = 0.073 W.m-1. K-1. These values are close to the thermal conductivity ranges for several insulating materials. Consequently, the lowest values of thermal conductivity of Cement / NFC are given with 15% of NFC loading.

As results, the compressive strength and flexural strength for whole biocomposites samples based on cement matrix decrease with the increasing of nanofibers content. The NFC_3 based cement materials present the higher compressive strength and flexural strength than the other samples at different dosages such as NFC_5, NFC_{10}, and NFC_{15}. In addition, cement / NFC composites have good mechanical properties (resistance to compression and bending) compared to the other materials presented in the literature. Therefore, it must be kept in mind that using 5 % of NFC in cement matrix allows obtaining a composite with good thermal and mechanical properties.

The date palm waste nanofibers have a very low thermal conductivity of 0.042 $W.m^{-1}.\ K^{-1}$ which can be used as insulating material on the walls of buildings. The addition of nanofibers to the cement matrix improves the thermal resistance of the nanocomposite and in the otherwise reduces its mechanical resistance.

Thermal analyses tests of the nanofibers indicate that nanofiber can be used as a thermal insulation material since they are biodegradable and have positive environmental and economic impacts. The date palm waste is a good candidate for the development of efficient and safe insulating materials when compared to the other natural materials.

We consider that the availability and low cost of NFC waste may be a good opportunity for a serious proposal of sustainable materials for thermal insulation of buildings. At a time of sustainable development, the cellulosic nanofibers used in the manufacture of this material are recovering, thus promoting this approach and managing and valuing some of the waste produced by citizens.

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