

A natural ancestral saltwater treatment to modify the technological properties of date palms

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Photo 1.
Palm Groves in the region of Djérid in South Tunisia.
Photo M. Elaieb.

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RÉSUMÉ

Un traitement ancestral avec de l'eau salée pour modifier les propriétés technologiques des dattiers

Les plantations de palmiers ont une grande importance socio-économique et écologique en Tunisie. Actuellement, elles contiennent près de trois millions d'arbres, qui assurent d'importants approvisionnements en bois pour l'artisanat et l'industrie du meuble. Autrefois, le palmier dattier était également utilisé comme matériau de construction. Ses faibles propriétés technologiques ont été améliorées par l'immersion dans l'eau salée naturelle des troncs de palmiers fraîchement coupés. C'était une pratique ancestrale dans la zone du Maghreb, mais elle a disparu maintenant, et les informations sur les différents paramètres impliqués dans ce type de processus sont rares. L'objectif de cette étude est d'évaluer les propriétés technologiques principales du bois de *Phoenix dactylifera* L. quand il est conservé dans l'eau salée. Des échantillons de bois prélevés dans le sud de la Tunisie sur deux cultivars communs de palmier dattier (*Kentichi* et *Deglet Noor*) âgés de 40 à 50 ans, non traités et conservés par salage dans le lac Chot Djérid, ont été utilisés pour ces expériences. Les densités, les propriétés mécaniques, la résistance aux champignons lignivores et aux termites et la composition chimique des échantillons de bois non traités (témoin) et traités avec l'eau salée ont été déterminées. Les résultats ont montré une augmentation significative de la densité du bois de palmier séché à l'air après immersion dans l'eau salée. Le traitement avec l'eau salée a aussi amélioré le module de rupture du bois de palmier phœnix perpendiculairement et parallèlement aux fibres. Le bois non traité et le bois traité avec de l'eau salée, semblaient être tous deux sensibles aux attaques de termites, mais le bois de palmier traité semblait être plus toxique pour les termites. Les teneurs en composés extractibles, en lignine et en cellulose étaient légèrement plus abondantes dans les échantillons témoins, alors que les hémicelluloses étaient plus abondantes dans les échantillons traités. Les analyses minéralogiques ont permis également d'expliquer l'amélioration de la performance du bois de palmier après ce processus de conservation naturelle.

Mots clés : bois de dattier, modification chimique, traitement dans l'eau salée, résistance à la putréfaction et aux termites, propriétés mécaniques, Tunisie.

ABSTRACT

A natural ancestral saltwater treatment to modify the technological properties of date palms

Palm plantations are of great socio-economic and ecological importance in Tunisia. They currently contain some three million trees that produce significant supplies of wood for the craft and furniture industries. In the past, date palm wood was also used as structural material. Its poor technological properties were improved by immersing in natural saltwater the freshly felled palm tree trunks. This was an ancestral practice in the Maghreb area, but it has now disappeared and information on the different parameters involved in this kind of process is scarce. The aim of this study was to assess the main technological properties of *Phoenix dactylifera* L. wood when preserved by salting. Wood samples collected in southern Tunisia from two common date palm cultivars (*Kentichi* and *Deglet Noor*) ranging from 40 to 50 years in age, untreated and preserved by salting in the Chot Djérid lake, were used for the experiments. The densities, mechanical properties, resistance to fungus and termites and chemical compositions of the untreated (control) and saltwater-treated palm wood samples were determined. The results showed a significant increase in the air-dried density of palm wood after immersion in saltwater. Saltwater treatment also greatly improved the Modulus of Rupture of palm wood perpendicular and parallel to the fibres. Both untreated and saltwater-treated palm wood appeared to be sensitive to termite attacks, but the treated palm wood seemed to be more toxic to the termites. Extractives, lignin and cellulose contents were slightly more abundant in the control samples, while hemicelluloses were more abundant in the treated samples. The analyses of mineral composition also provided some explanation of the improvement in palm wood performance after this natural preservation process.

Keywords: palm wood, chemical modification, saltwater treatment, decay and termite resistance, mechanical properties, Tunisia.

RESUMEN

Tratamiento natural ancestral con agua salada para modificar las propiedades tecnológicas de las palmeras datileras

Las plantaciones de palmeras tienen una gran importancia socioeconómica y ecológica en Túnez. Actualmente abarcan unos tres millones de árboles que suministran provisiones significativas de madera para las industrias de cestería y mobiliario. En el pasado, la madera de las palmeras datileras se utilizaba como material estructural. Sus mediocres propiedades tecnológicas se mejoraban sumergiendo los troncos de las palmeras recientemente talados. Era una práctica ancestral de la zona del Magreb, pero actualmente ha desaparecido y la información de los diferentes parámetros implicados en este tipo de procesos es escasa. El objeto de este estudio es evaluar las propiedades tecnológicas principales de la madera de *Phoenix dactylifera* L. cuando se preserva mediante agua salada. Para los experimentos se utilizaron muestras de madera recogidas en el sur de Túnez de las dos variedades comunes de palmera datilera (*Kentichi* y *Deglet Noor*) de entre 40 y 50 años de edad, unas sin tratar y las otras conservadas en el agua salada del lago Chot Djérid. Se determinaron las densidades, las propiedades mecánicas, la resistencia a los hongos y a las termitas y la composición química de las muestras de madera de palmera no tratada (control) y tratada con agua salada. Los resultados mostraron un incremento significativo en la densidad de la madera de palmera secada al aire después de la inmersión en agua salada. El tratamiento también mejoró en gran medida el módulo de ruptura de la madera de palmera, perpendicular y paralelo a las fibras. Tanto la madera de palmera no tratada como la tratada con agua salada parecen sensibles a los ataques de termitas, aunque la madera tratada parece ser más tóxica para las termitas. Los contenidos de extracto de lignina y celulosa fueron ligeramente superiores en las muestras de control, mientras que las hemicelulosas son más abundantes en la muestra tratada. Los análisis de la composición mineral también proporcionan alguna explicación de la mejora en el rendimiento de la madera de palmera después de este proceso de conservación natural.

Palabras clave: madera de palmera, modificación química, tratamiento en agua salada, resistencia a los hongos y a las termitas, propiedades mecánicas, Túnez.

Introduction

Nowadays, the Tunisian primary wood-processing industry consumes more than 872,000 m³ of wood raw materials, with only 119,000 m³ supplied by local forest resources. In addition, several natural lignocellulosic fibers, as those from palm trees, are available around the country but they remain poorly exploited. The date palm (*Phoenix dactylifera* L.) is a tropical and subtropical tree that belongs to the palmae (*Arecaceae*) family and plays a very important role on both the socio-economic and ecological levels, mainly in southern Tunisia (Bendahou *et al.*, 2007). There are three million trees in Tunisian palm plantations, represented on an area of 22,500 ha (Rhouma, 1993), ensuring a potential significant wood production (figure 1).

The Palms are also used in programs to control silting and they are extremely useful to prevent desertification by creating a microclimate, which limits long-term degradation of ecologically weak environments (Bendahou *et al.*, 2007).

Date palm (*Phoenix dactylifera* L.), one of the most important non-wood renewable resources in Maghreb area, which is mainly used for its fruits, fibers and as a construction material (Nasser, 2014), could be also considered as a raw material for energy production (Nasser *et al.*, 2016), for the pyrolysis characteristics of the seeds (Babiker *et al.*, 2013), in briquettes production (Eissa *et al.*, 2013), and for biochars production (Usman *et al.*, 2016). The residual by-products from date palm production are used in various industries as raw materials for many applications such as lumber (El-Mously, 1997), particleboards and oriented strand boards (Hegazy and Ahmed, 2015), pulp and paper (Khiari *et al.*, 2011), wood-cement composites (Nasser and Al-Mefarrej, 2011) and wood-plastic composites (Abu-Sharkh and Hamid, 2004).

For several centuries, palm stems have been used as a material for the construction and manufacturing throughout the Maghreb area. The stipe, also called palm tree convenience, serves as a structure and carpentry in the construction of houses in the oasis in the Southern Tunisia, in the manufacture of portals or to cover the roofs (figure 2).

In past years, the wood producers used fresh palm wood, initially despite its very low natural durability and bad physical and mechanical quality. They finally treated it naturally by salt waters of the Lake of Chot Djérid (figure 3). Such a treatment consists in depositing the trunk of the palm tree freshly slaughtered in the Chot for a period ranging from 1 to 2 years (depending on the species).

After this stay, the appearance, colour, and the wood technological properties change. The woody material becomes more dense, durable and resistant. The resulting material is used in outdoor construction, without any other industrial or expensive process. This ancestral practice, mainly used in the Maghreb area, has disappeared and information on the different parameters involved in this kind of process (durations, dimensions of the trunks, etc.) is scarce. The improvement of living conditions has greatly influenced the population, whom replaced the palm wood construction by the concrete construction and thus the ancestral know

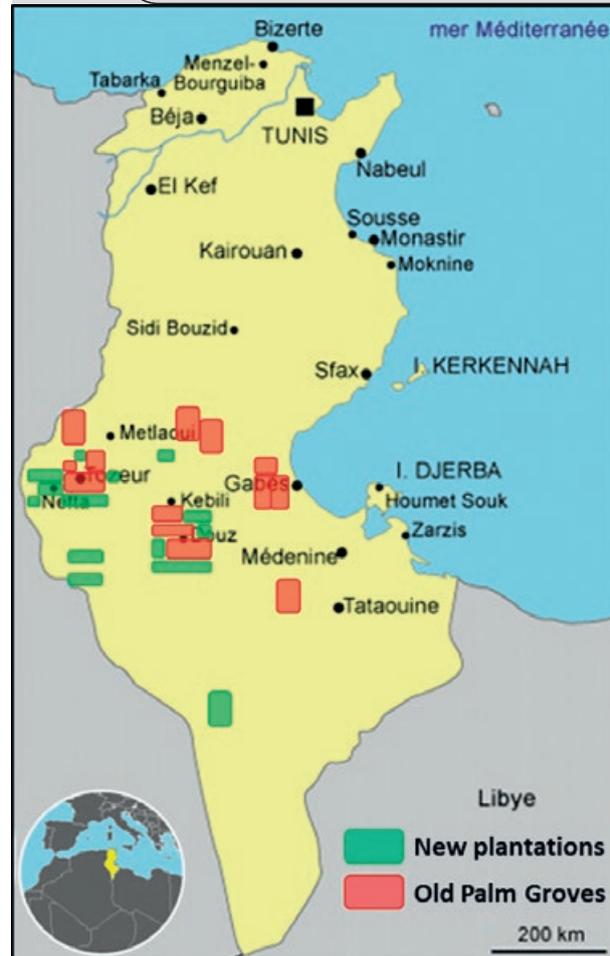


Figure 1. Repartition of the new and old Palm Groves in Tunisia
 Adapted from Rouma (1993).



Figure 2. Date Palm stems, treated naturally by salt waters of the Lake of Chot Djérid used as structural materials in the region of Djérid in Tunisia.



Figure 3.
Exposition of date palm tree trunk, freshly slaughtered, into the salt waters cycles into the Lake of Chot Djérid.

how was abandoned. This wood treatment process is not well known and what should be sought is unclear. Wood submerging in fresh water for prolonged periods can lead to extensive bacterial attack and marked increase in permeability (Ellwood and Ecklund, 1959). On another hand, while repeated saltwater immersions alternated with drying steps could result in tracheid separation and bursting in the wood surface (Johnson *et al.*, 1992). The effects of this damage on durability are less clear and need to be supported by substantial experimental data. It remains a lack of knowledge about wood properties after a long time of salt water exposure, primarily because marine borers rapidly destroy such materials in many cases (Zabel and Morrell, 1992). Luckily, the absence of such organisms into the Lake Chot Djérid makes possible a study of the effects of prolonged salt exposure on durability.

The objective of this work was to test date palm wood from the Southern Tunisia for resistance to fungal decay, termite resistance and for the chemical composition to get information on the origin of its particular properties.

Materials and methods

To assess the main technological qualities of palm wood preserved by salting while trying to understand what kinds of chemical modifications (impregnation of mineral compounds) come into play in the improvement of the treated wood quality by this natural and eco-friendly process, densities (air-dried, water saturated, basic), mechanical properties, decay and termites resistance tests were performed on native and water salt treated palm woods. Additional analyses conducted with an ICP-OES 700 Agilent device, evaluating the mineral compositions between native and salt-treated palm woods, give us some explanation ways about the performance improvement of wood after such a natural preservation process.

Palm wood samples selection

Two defect-free trees of common date palm cultivars (*Kentichi* and *Deglet Noor*) with ages ranging from 40 to 50-years and two samples (boards) preserved by salting in the Chot Djérid (*Kentichi* and *Deglet Noor*) taken from about 50 years old structure, were used for the experiments. The visual aspects of the selected palm wood samples are presented in figure 4. All palms were harvested at the Regional Center of Research on Oasis Agriculture - Tozeur / Degache - Southern Tunisia (figure 3). The site has the following characteristics: temperatures ranging between 10 °C in winter and 41 °C in summer, 50 mm annual rainfall and a calcareous soil. We can observe that after saltwater immersion, the both palm woods seem to be more dark, heavy, dense and homogeneous than the respective native palm wood samples.

Structural polysaccharides composition

Extractives contents

Woods of each sample were ground and passed through different sieves to obtain particles ranging between 0.2 and 0.5 mm. This sawdust was successively washed in a Soxhlet extractor with a toluene/ethanol (2:1, v/v) mixture (6 h), followed by ethanol (6 h) and dried at 103 °C for 48 h.

Holocellulose content

500 mg of extracted sawdust were placed in a 100 mL flask containing 30 mL of distilled water and heated at 75 °C. Acetic acid (0.1 mL) and 15% aqueous sodium chlorite (2 mL) were then added each hour for 7 h. The mixture was filtered on a Büchner funnel and the residue washed with water, soxhlet extracted for 2 h with ethanol and dried at 103 °C to a constant mass.

Hemicellulose and α -cellulose contents

The dried holocellulose prepared as above was placed into a 250 mL glass beaker covered with a glass cover containing 10 mL of 17.5% NaOH solution and maintained at 20 °C in a water bath. This mixture was agitated with caution with a rod glass until the holocellulose was soaked with the NaOH solution. A quantity of 5 mL of 17.5% NaOH solution were then added every 5 min during 15 min and the mixture left for 30 min. The mixture was then diluted with 33 mL of distilled water and kept for 1 h before filtration on a Büchner funnel. The crude α -cellulose residue was successively washed with 100 mL of 8.3% NaOH solution, 100 mL of distilled water, 15 mL of 10% acetic acid solution and finally 250 mL distilled water. The residual α -cellulose was dried at 103 °C to a constant weight and the hemicellulose content calculated as follows:

Hemicellulose (%) = Holocellulose (%) – α -Cellulose (%) (equation 1)

Klason lignin content

500 mg of sawdust were mixed with 72% H₂SO₄ (10 mL) for 4 h at room temperature. The mixture was then diluted with 240 mL of distilled water, heated under reflux for 4 h and filtered. The residue of Klason lignin thus obtained was washed with hot water and dried at 103 °C to a constant weight.

Physical properties

Different physical and mechanical tests were performed:

- Concerning control samples, a wooden disk 50 mm in thickness was cut at 1.40 m from the ground for each selected tree. To avoid errors during sampling, extreme cases such as excessively knotty trees or the presence of reaction wood or slope grain were taken discarded (ISO 4471, 1982). From each disk, 12 samples of 3 cm width from bark to bark were cut. These samples were then cut into strip 2 cm thick.

- For salt-treated date palm woods, similar samples were taken from previously collected boards.

Basic density (D_b), Air-dried density, after conditioning in a climatic room at 20 ± 2 °C and 65 ± 5% relative humidity (RH), and water-saturated (after drying in an oven at 103 °C) density (D_{m12} , D_s) (ISO 13061-2, 2014), of the wood samples were determined using wood specimens of 20 × 20 × 10 mm (along the grain). The densities were determined by the gravimetric method (Haygreen and Bowyer, 1996).

$$D_b = \frac{m_o}{V_h} \text{ (equation 2) ; } D_s = \frac{m_h}{V_h} \text{ (equation 3) ;}$$

$$D_{m12} = \frac{m_{12}}{V_{12}} \text{ (equation 4).}$$

Where m_h is the humid mass of the initial sample; m_o and m_{12} are the oven-dried and air-dried weight of the sample (g), respectively.

D_b is the basic density of wood (g.cm⁻³), D_s is the water-saturated density of wood (g.cm⁻³) and D_{m12} is the air-dried density of wood (g.cm⁻³). V_h is the green volume of the specimen (cm³), V_o is the oven-dried volume of the sample and V_{12} is the air-dried volume of wood sample.

Mechanical properties

To perform mechanical resistance tests, three point bending and compression tests were carried out for each of the selected wood tree samples, and Modulus Of Rupture (MOR) results were compared. An INSTRON 4467 Universal Mechanical Test Machine was used for the measurements. Samples were conditioned in a climate-controlled room with 65% RH and at 22 °C for the time required to stabilize the samples weights.

Bending test

Three point static bending tests were carried out according to the EN 408 (2010). The sample size was 300 × 20 mm × 20 mm³ (L × R × T). The moving head speed and span length were 0.09 mm.s⁻¹ and 260 mm, respectively. The load deformation data obtained were analyzed to determine the Modulus Of Rupture (MOR). The tests were replicated on 20 samples from each selected Palm tree.

Compression strength parallel to grain

Compression tests were carried out according to the EN 408 (2010). Deviating from the norm, a reduced specimen size of 30 × 20 × 20 mm³ (L × R × T) was used. The moving head speed was 0.09 mm.s⁻¹ to ensure wood sample rupture within 1.5 to 2 min. The load deformation data obtained were analyzed to determine the Modulus Of Rupture (MOR). An amount of 20 specimens per selected tree were tested.

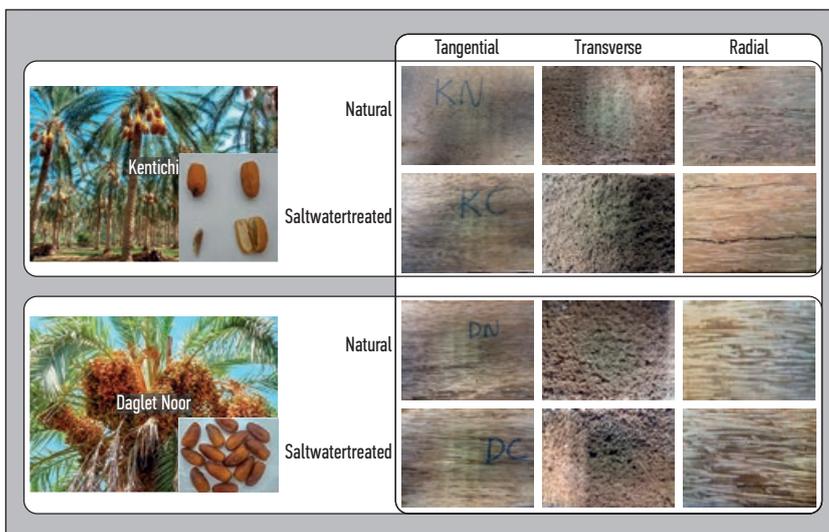


Figure 4. Appearance of natural and salt-treated palm wood samples, *Kentichi* and *Deglet Noor* varieties.

Fungal resistance tests

Laboratory decay tests using specimens with surrounding dimensions of 30 x 10 x 5 mm³ (L x R x T) were performed to examine the fungal resistance of the different date palm woods according to their salty water treatment. Beech (*Fagus sylvatica*) and pine sapwood (*Pinus sylvestris*) samples with the same dimensions have also been tested against fungi as controls. Sterile culture medium (65 mL), prepared from malt extract (40 g) and agar (20 g) in distilled water (1 L), was placed in a glass bottle of 72 cL volume, closed with Carded cotton inoculated with two small piece of mycelium of a freshly grown culture of three brown-rots [*Coniophora puteana* (CP) (BAM ebw. 15); *Poria placenta* (PP) (FPRL 280) and *Gloeophyllum trabeum* (GT) (BAM Ebw. 109)]; and one white-rot [*Coriolus versicolor* (CV) (CTB 863 A)].

The inoculated Petri dishes were incubated for 2 weeks at 27°C and 70% RH until malt extract agar was covered by mycelium. Date palm woods and control specimens were supported on sterile inert mesh to prevent contact with culture medium. All test samples previously oven dried at 103°C to constant weight (m_0) were γ -ray-sterilized and three specimens and one control sample were placed in each test device under sterile conditions.

All modalities were triplicated and the incubation was carried out for 8 weeks at 22 °C under controlled humidity conditions of 70% RH in a climatic chamber. At the end of the test exposure, mycelia were removed and all samples were oven-dried to constant mass at 103 °C (m_1). The extent of the fungal attack was determined based on the percentage of Weight Loss (WL) which was expressed as a percentage of the initial oven-dried weight of wood samples according to the following the equation 5:

$$WL (\%) = \frac{m_0 - m_1}{m_0} \times 100 \text{ (equation 5).}$$



Figure 5. Screening termites test devices, with adjustments to EN 117 standard guidelines, for beech and pine wood controls and date palm woods.

Termite resistance tests

Palm wood samples were exposed to termites (*Reticulitermes flavipes*, ex. *santonensis*) in non-choice screening tests. Beech (*Fagus sylvatica*) and pine sapwood (*Pinus sylvestris*) samples of dimensions 30 x 10 x 5 mm³ (L x R x T) have also been tested against termite as controls. Prior to the test, each sample and control samples (of the same dimensions) were dried at 103 °C in order to obtain its anhydrous initial weight (m_2), and then re-conditioned at 20 °C, 65% RH until constant weight. For each set of palm and control woods, three replicates were tested for their resistance towards termites. Each specimen was placed in the middle of a 9 cm diameter Petri dish containing 40 g of Fontainebleau wet sand (4 volume of sand / 1 volume of deionized water). A total of 50 termite workers, one nymph and one soldier were then introduced into each test device. These test devices were placed in a dark climatic chamber kept at 27 °C and > 75% RH for 4 weeks. Observations were carried out on a weekly basis in order to add water and check termite behavior (figure 5).

At the end of the exposure, the samples were removed, cleaned of sand and termite survivors were counted. Sample degradations were given a visual rating according to the criteria of EN 117 (2013) (the criteria being adjusted to the sample size). Then, the samples were dried at 103 °C to obtain their anhydrous weight after termite exposure (m_3) and their Mass losses (ML%) were calculated according to the equation 6:

$$ML (\%) = \frac{m_2 - m_3}{m_2} \times 100 \text{ (equation 6).}$$

Elementary composition of woods

The aim of the elementary composition analysis is to determine the major compounds of samples (Ca, K, Mg, Si, C, etc.) contained in salt water treated palm woods and to better understand the chemical process reaction involving the improvement of salt water treated palm wood properties.

Preparation of samples: After drying at 65 °C, wood samples were grounded (particles < 0.5 mm) in order to homogenize samples and allow representative aliquots for analysis (\approx 500 mg). Ground organic samples progressively absorb surrounding humidity. To correct and formulate results on dry material basis, residual moisture is quantified by drying 1 g of sample at 105 °C until constant weight.

Inorganic elements quantification (B, Na, Mg, Si, P, K, Ca, Mn, Fe, Cu, Zn, Pb): Samples were burned at 500 °C to eliminate organic matter (water, carbon and nitrogen oxides). Minerals, which remain in the ashes as oxides and carbonates, were acidly dissolved (HCl). However,

insoluble silicates from the persistent silicon naturally present in plants would retain micro-nutrients by chemical adsorption. They are removed by hydrofluoric acid (HF) addition after a second calcination.

Silica concentrations were directly determined during mineralization by ashes weighing before and after its elimination by HF addition.

Trace element concentrations were determined using an inductively coupled plasma mass spectrometer (iCAP™ Q ThermoScientific). For each element analysis, method was checked using certified reference material (SLRS 6 and strawberry leaves LGC7162). The measurement uncertainty was less than 10%.

Chlorine: Chloride ions have been extracted from samples by nitric acid solution (0.3 N), then analyzed through an Alliance Evolution II colorimeter according to Zall colorimetric method based on brown coloration of thiocyanate ferric complex detection.

Total carbon and nitrogen: determinations were performed by dry combustion (Dumas method) on an Elementar VarioMacroCube analyzer. Analysis method was checked using certified reference material (Orchard Leaves Standard 502-055).

pH determination

The pH measurement of the both native and saltwater treated palm woods was performed by the hot water extraction process. Ten grams (10 g) of grinded palm wood sample was placed in an 80 mL beaker with 200 mL of distilled water. The mixture was boiled during 10 minutes and then cool down to ambient temperature, decanted and finally the pH of the liquid was measured.

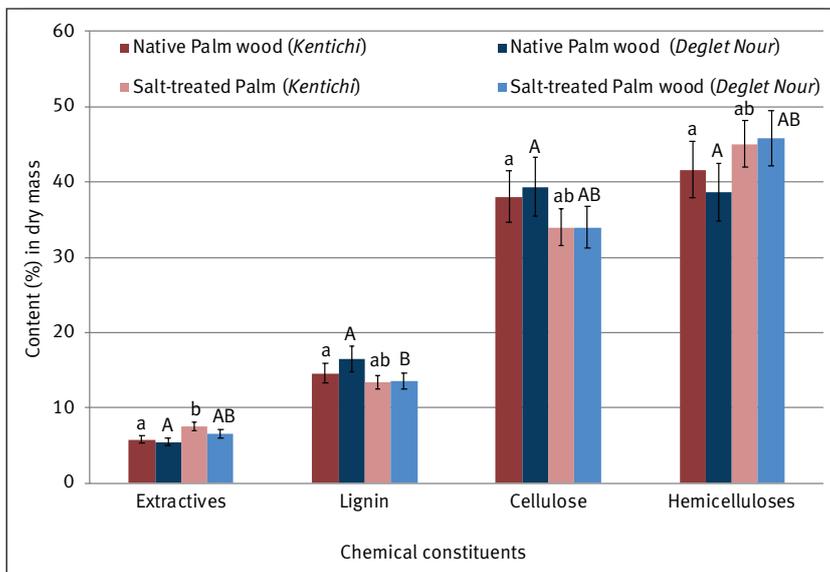


Figure 6. Contents of the different structural polymers of *Kentichi* and *Deglet Noor* varieties palm woods.

Statistical analysis

Statistical analyses (one-way analysis of variance) using Fisher test and the JMP 10.0.2 program (SAS Institute Inc., Cary, NC, USA) have been performed. The effects of natural salt water treatment on the wood structural polysaccharides and elementary compositions, densities, mechanical properties, decay and termites resistances were evaluated using ANOVA and Duncan's comparison test. Such analysis allows to class results into several categories from a to b for *Kentichi* variety and from A to B concerning *Deglet Noor* variety. Systems which are not connected by the same letter are largely different at the 5% level.

Results

Structural polysaccharides composition

Chemical composition detailed on figure 6 shows that hemicellulose, cellulose, lignin and extractive contents of the both studied native *Phoenix dactylifera* L. species ranged from 34.8 to 45.8%, from 34.2 to 43.3%, from 13.1 to 18.2%, and from 4.9 to 6.4%, respectively. So the studied palm wood species have amounts of hemicelluloses, lignin and cellulose similar to those encountered in softwood and hardwood (Amirou *et al.*, 2014).

These results are consistent with those from other studies conducted on the chemical composition of Morocco, Egyptian and Tunisian date palm woods which found holocellulose contents ranged from 12.0 to 74.8%, lignin contents ranged from 14.0 to 27.2% and extractive contents comprised between 4.0 and 12.8% (El Morsy, 1980; Khristova *et al.*, 2005; Bendahou *et al.*, 2007; Khiari *et al.*, 2010).

According to the figure 6, lignin and cellulose tend to be more abundant in the control samples, whereas hemicelluloses and extractive contents are more abundant in the natural salt-treated palm wood samples and that for the both palm wood varieties (*Kentichi* and *Deglet Noor*). According to the statistical analysis, except for the extractive content increase for *Kentichi* and Lignin content decrease for *Deglet Noor* cultivar, these results are not really statistically different. This chemical composition modification between native and treated samples could be explained by the chemical reaction caused by several minerals contained in the salt water as chlorine (Cl), sodium (Na) and sulfur (S) during wood exposure to such a water Guellala *et al.* (2010).

Physical properties

Air-dried Density (D_{m12}) is commonly used to compare different woods. Basic Density (D_b), water saturated Density (D_s) and Air-dried Density (D_{m12}) have been measured on each palm wood sample. Average values of these different densities are presented in table I.

Table I.Average values of Basic, Water-saturated and Air-dried densities of *Kentichi* and *Deglet Noor* palm woods.

Palm wood samples	Densities (%)					
	Basic Density (D_b)	SD*	Water-saturated Density (D_s)	SD*	Air-dried Density (D_{m12})	SD*
Native Palm wood (<i>Kentichi</i>)	0,210 ^a	0,13	0,829 ^a	0,21	0,217 ^a	0,12
Native Palm wood wood (<i>Deglet Noor</i>)	0,207 ^A	0,04	0,889 ^A	0,31	0,215 ^A	0,08
Salt-treated Palm (<i>Kentichi</i>)	0,227 ^a	0,06	0,913 ^{ab}	0,12	0,440 ^b	0,08
Salt-treated Palm wood (<i>Deglet Noor</i>)	0,243 ^{AB}	0,02	1,057 ^{AB}	0,35	0,361 ^B	0,02

* Standard Deviation on X replicates (refer to the respective part within Materials and Methods chapter).

Table II.Average values of Modulus of Rupture (MOR) in bending and compression strength of *Kentichi* and *Deglet Noor* palm woods.

Palm wood samples	Bending strength (MOR)		Compression strength (MOR)	
	Average values (MPa)	SD*	Average values (MPa)	SD*
Native Palm wood (<i>Kentichi</i>)	11,1 ^a	2,9	3,3 ^a	1,04
Native Palm wood wood (<i>Deglet Noor</i>)	15,8 ^A	4,13	5,9 ^A	1,17
Salt-treated Palm (<i>Kentichi</i>)	20,7 ^b	4,81	10,3 ^b	3,01
Salt-treated Palm wood (<i>Deglet Noor</i>)	30,2 ^B	6,26	14,3 ^B	1,56

* Standard Deviation on X replicates (refer to the respective part within Materials and Methods chapter).

The results obtained through this study are in agreement with those from Almi *et al.* (2015) who found that native Algerian date palm wood has a Basic Density ranged from 0.160 to 0.635 and a Water-saturated Density ranged from 0.838 to 0.995, according to the position of the wood in the tree (trunk, rachis, petiole, etc.).

Moreover, the Air dried and Water-saturated densities increase for the both palm wood species after their immersion in salt water (table I). This densification of salt-treated palm woods is attributed to the impregnation of some mineral compounds into the wood during its immersion into the Lake Chot Djérid.

Mechanical properties

The mechanical test results of each palm wood sample conditioned at 20°C and 65% RH (Relative Humidity) are shown in table II. Modulus Of Rupture (MOR) reflects the maximum capacity in bending or in compression and is proportional to the maximum moment borne by a sample. Results of flexural strength showed that Modulus Of Rupture (MOR) was ranged from 11.1 to 15.8 MPa for native Palm wood and that it was improved to 20.7-30.2 MPa by natural

saltwater treatment. Similar trend was observed concerning the flexural strength highlighting compression strength (MOR) improvement from 3.3 - 5.9 MPa to 10.3 - 14.3 MPa after saltwater immersion (table II). No result about natural saltwater treated palm woods is available in the literature, but the mechanical properties obtained on the native *Phoenix dactylifera* L. woods through this study are consistent with those issued from previous works: i) Amirou *et al.* (2014) and Keramat Jahromi *et al.* (2008) found date palm wood compression strength of 2.338 MPa and 4.458 MPa, respectively; ii) Keramat Jahromi *et al.* (2008) found that flexural strength of date palm trunk was around 7.166 MPa.

Decay resistance towards fungi

The effect of natural salt-water treatment on *Kentichi* and *Deglet Noor* Tunisian date palm wood species was studied on the wood durability towards both fungi. The tests were performed without accelerated ageing according to the methods described in the EN 350 standard and the results are exposed in figure 7. The median weight loss on the two native Palm wood specimens was 25.4% for *Coriolus versicolor*, 18.1% for *Coniophora puteana*, 17.0% for *Poria*

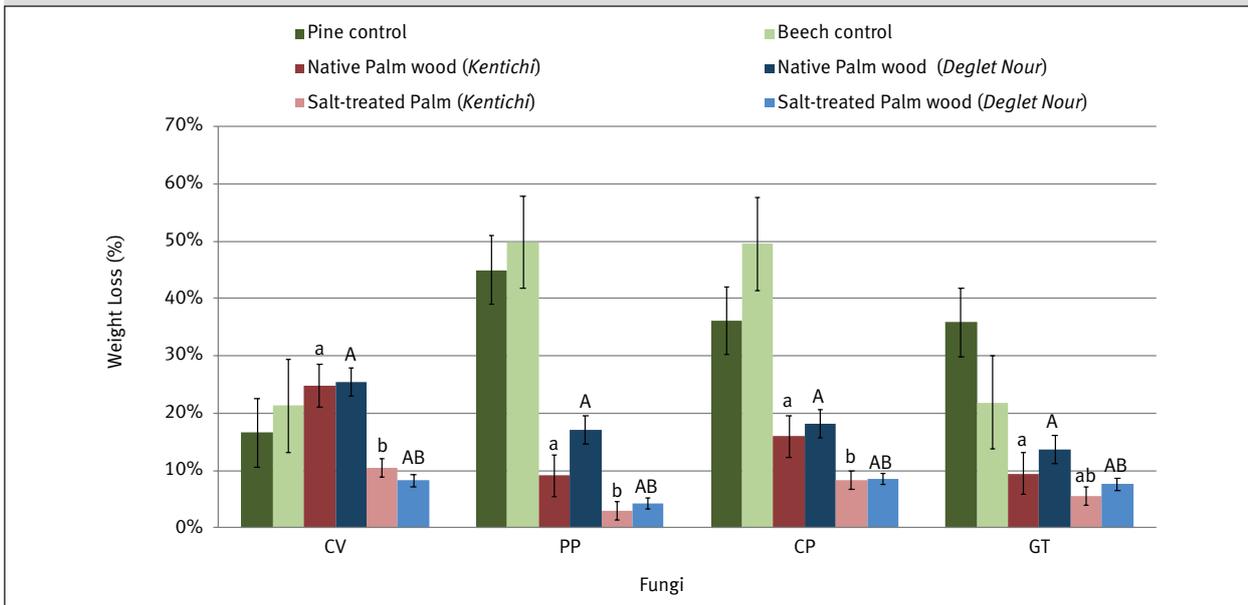


Figure 7. Decay resistance of native and natural saltwater-treated *Kentichi* and *Deglet Nour* varieties palm woods. CV: *Coriolus versicolor*; PP: *Poria placenta*; CP: *Coniophora puteana*; GT: *Gloeophyllum trabeum*.

placenta and 13.7% for *Gloeophyllum trabeum*. The weight loss of the controls allow the test validation. Similar results have been found by a previous study showing that white-rot fungi caused significantly more weight loss than brown-rot fungi on *Phoenix dactylifera* L. (Adaskaveg *et al.*, 1991).

According to the results highlighted in figure 7 and results from the statistical analysis, it is difficult to evaluate the significance of the degradation level difference caused by brown rot and white rot fungi.

It appears clearly that natural salt-water treatment of Palm wood allows decreasing of the weight loss due to fungal degradation. The median weight loss on the two modified Palm wood specimens was 9.4% for *Coriolus versicolor*, 8.5% for *Coniophora puteana*, 4.2% for *Poria placenta* and 7.6% for *Gloeophyllum trabeum*. The weight loss of the controls allow the test validation. According to EN 350 (2016), *Djérid* and *Deglet Nour* Tunisian date palm wood could be classified as moderately durable ($10 < WL \leq 15$) against *Gloeophyllum trabeum* and slightly durable ($15 < WL \leq 30$) towards *Coriolus versicolor*, *Coniophora puteana*, and *Poria placenta*. Natural saltwater treatment improves the decay resistance of the two tested palm wood classifying it as durable ($5 < WL \leq 10$) towards *Coriolus versicolor*, *Coniophora puteana* and *Gloeophyllum trabeum*, and as very durable ($WL \leq 5$) against *Poria placenta*.

The improvement of decay resistance of modified palm woods is probably due to their higher extractive contents than those of native wood and their impregnation of some mineral compounds coming from their salt-water exposition (Gómez *et al.*, 2016).

According to the statistical analysis and except the weight loss caused by *Gloeophyllum trabeum* fungi on native and treated *Kentichi* variety, other treated palm wood

samples had markedly lower weight losses than did the respective native blocks, although visual examination showed that fungal growth and damage were considerably reduced on the treated blocks. This added durability might be useful for preventing the development of decay in service, although it is difficult to assess the benefits of such salt loadings when the risk of leaching is high. Applying an additional oil-based treatment to the wood might delay leaching loss and maintain the protection (Schneider *et al.*, 1997).

Termite resistance

The termite resistance test is valid as the *Pinus sylvestris* control samples present a strong attack (ML = 22.0%, visual rating 4) and above 50% of the termite workers remain alive at the end of the test (table III). After 4 weeks of native *Kentichi* and *Deglet Nour* Tunisian date palm woods exposure to termites, the mean survival rate of the insects was 20.0% and 21.3% respectively, and termites had got through all samples causing tunneling and heavy degradations (ML = 41.4% and ML = 46.5%, respectively).

Native Tunisian date palm woods are classified as sensible towards termites. However, salt-water treatment allows to obtain a lower mass loss due to termites degradation (ML = 30.6% for *Kentichi* and ML = 32.6% for *Deglet Nour*, (table III) and a higher termite mortality (termite survival rates are 6.0% for *Kentichi* and 8.7% for *Deglet Nour*). As a conclusion, the modified *Kentichi* and *Deglet Nour* Tunisian date palm woods are also classified as sensible towards termites. Natural salt-water treatment does not allow to improve significantly the termite resistance of the two date palm wood species in order obtain a better visual rating than 4.

Table III.

Median values of Mass Loss (%) due to termite degradation, termite survival rates (%) and visual rating according to EN 117 (2013).

Palm wood samples	Mass Loss (%)	SD* (%)	Survival rates (%)	SD* (%)	Visual Rating According to EN 117
Native Palm wood (<i>Kentichi</i>)	40.4 ^a	0.1	20.0 ^a	4.0	4
Native Palm wood wood (<i>Deglet Noor</i>)	46.5 ^A	0.1	21.3 ^A	3.1	4
Salt-treated Palm (<i>Kentichi</i>)	30.6 ^b	0.1	6.0 ^b	5.3	4
Salt-treated Palm wood (<i>Deglet Noor</i>)	32.6 ^B	0.1	8.7 ^B	1.5	4
Beech control	26.6	0.0	63.3	9.4	4
Pine control	22.0	0.0	70.0	11.1	4

* Standard Deviation on X replicates (refer to the respective part within Materials and Methods chapter).

Table IV.

Dried Matter (DM), Silica, Chlorine and Total Carbon contents of Native and salt-treated *Kentichi* and *Deglet Noor* varieties palm woods.

Palm wood samples	Dried residual Matter DM (%)*	Silica SiO ₂ (%)*	Carbon Total C (%)*
Native Palm wood (<i>Kentichi</i>)	93,4 ^a	<LD	48,3 ^a
Native Palm wood (<i>Deglet Noor</i>)	92,1 ^A	<LD	47,6 ^A
Salt-treated Palm (<i>Kentichi</i>)	91,1 ^b	<LD	43,8 ^b
Salt-treated Palm wood (<i>Deglet Noor</i>)	91,8 ^{AB}	<LD	45,4 ^B

* Results expressed relative to the dry matter.

Discussion

Mineral composition of Water from the Lake Chot Djérid

A previous study conducted by Guellala *et al.* (2010) highlighted that the water of the Lake Chot Djérid around Tozeur area contains between 2 to 3 g/L of salt components, mainly composed by SO₄ (≈ 53%), Na (≈ 20%), Cl (≈ 12%), Ca (≈ 10%), HCO₃ (≈ 3%), K (≈ 1%) and Mg (≈ 1%).

Elementary composition of native and salt-treated date palm woods

The elementary composition analyses of the native and salt-treated palm wood samples were determined to explain the improvement of the properties of palm woods treated by salt water immersion process. Table IV shows Dried Matter, SiO₂ and Total Carbon contents of native and treated palm woods and highlight the variation of wood material due to its natural salt water exposure. We can clearly observe that the variations of Total Carbon and Dried Matter contents of

the both palm wood species are slightly decreasing before and after their salt water immersion, whereas their composition in Silica (SiO₂) increases. These results are similar than those obtain by Schneider *et al.* (1997), who showed higher contents of Silica within Douglas fir wood immersed into salt water than those of respective native wood. The relatively decreases of Dried Matter and Total Carbon contents of palm wood after its salt water immersion could be explain by its lower content of Lignin and Cellulose. Indeed, Lignin and Cellulose contain higher content of Carbon than those of hemicellulose (Wertz, 2010).

According to literature, Date palm trunk wood is mainly composed by carbon (76%) and oxygen (20%) (Khiari *et al.*, 2010). Date palm wood raw materials has also an important quantity of ashes (Khiari *et al.*, 2010), mainly constituted by Ca, Cl, K, S, and Na. In addition, the absolute amount of silicon is very low (Khrstova *et al.*, 2005). This composition can be attributed to the chemical composition of the soil environment in which the plants are growing (Khiari *et al.*, 2010). Other minor constituents in trace are also present in native date palm wood included Mg and Si (Agoudjil *et al.*, 2011).

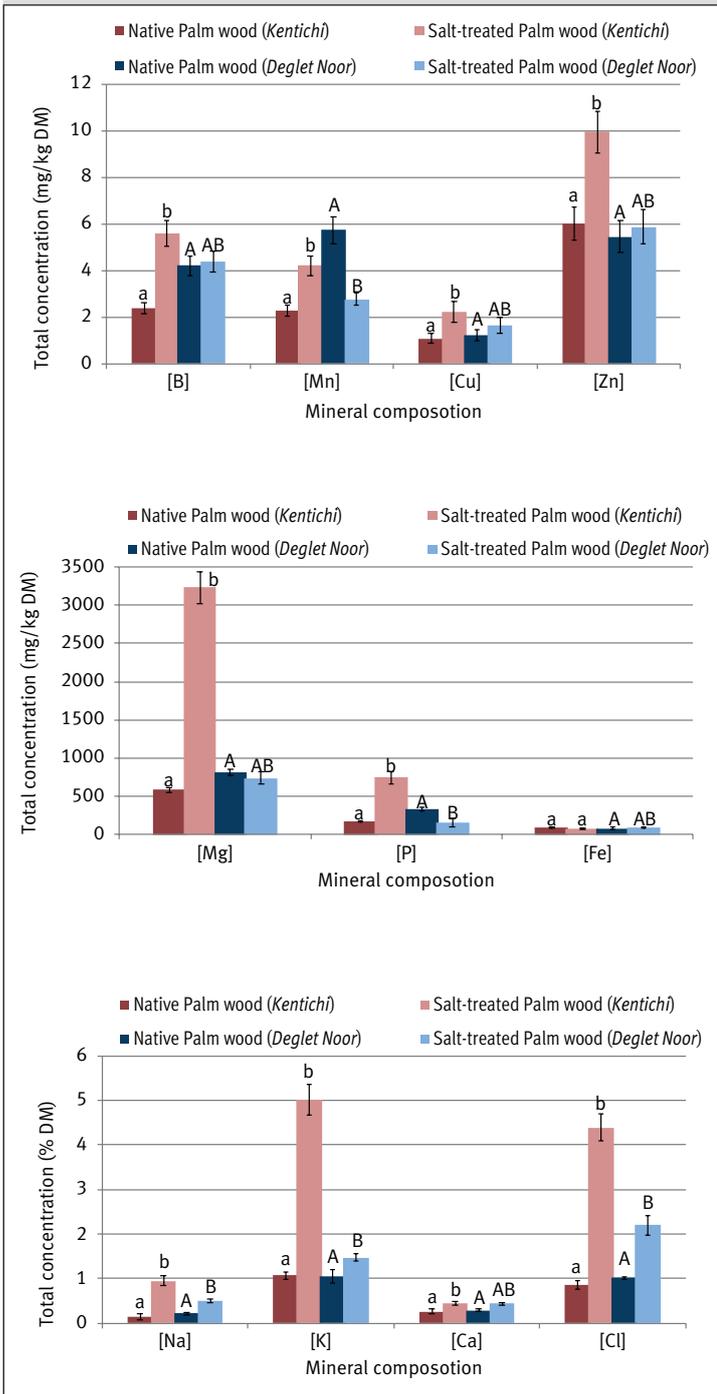


Figure 8. Mineral composition of native and natural saltwater-treated *Kentichi* and *Deglet Noor* varieties palm woods. Uncertainties correspond to the error accepted for validation of the CRM (10%).

Our results from colorimetric and ICP-MS analyses presented in figure 8 are in agreement with these statements. According to previous studies, it results that the main elements constituents of palm wood are Na, K, Ca, Mg and Cl (figure 8) and Si (table IV). Only traces of others minor mineral elements were identified in native and treated palm wood samples as B, Mn, Fe, Cu and Zn. To be noted that the detection limits did not allow us to measure lead contents.

On another hand, the total concentration of major and trace elements mainly increased after wood immersion in salt water and the increase is more important for *Kentichi* than those of *Deglet Noor* cultivars. This increase of the global wood mineral content after saltwater immersion undergoes to an increase of ashes in the treated palm woods impacting their durability properties.

Mineral composition of Salt-Treated palm wood and its influence on impact on wood decay resistance

According to the previous typical salt water composition, Schneider *et al.* (1997) showed, by Energy-dispersive X-ray spectroscopy (EDXA), that Douglas fir wood immersed into such a water has a higher content of chlorine (Cl), sodium (Na), potassium (K), and sulfur (S) than the respective native wood. Scanning Electron Microscope (SEM) examination highlighted also the present and crystal deposits into the wood of aluminum (Al), calcium (Ca), chlorine (Cl), magnesium (Mg) and silicon (Si) (Schneider *et al.*, 1997). Wood exposed to salt water appeared to contain high levels of sodium chloride (NaCl) and also a small amount of potassium chloride (KCl). These results are in perfect agreement of elementary composition of the woods studied in the present work.

As shown in table V, the high content of mineral salts into the salt-treated wood samples increases the alkalinity and the pH of wood and then could inhibit the mycelium growth on the treated samples (Bozkurt *et al.*, 1993). The increase of pH of date palm wood after saltwater exposure is statistically higher for *Kentichi* (from 3.6 to 5.7) than those of *Deglet Noor* (from 4.5 to 4.6) species.

Several studies highlighted the importance of pH on fungi activity: i) Highley (1976) found the cellulose and hemicellulases of brown-rot fungi to be more tolerant of low pH and more sensitive to high pH than those of white-rot fungi; ii) medium acidity was shown to exert a strong influence on the rate of lignin degradation by white-rot fungus, little degradation occurred below pH 3 or above pH 5 (Kirk *et al.*, 1978); and iii) while decaying wood, brown rot fungi create a low pH, primarily by oxalic acid production; however, unlike white-rot fungi, which also produce oxalic acid, the brown-rots are unable to metabolize this product (Takao, 1965). The pH statement could explain the fact that salt-treated date palm wood is more resistant against Brown Rot [*Coniophora puteana* (CP), *Poria placenta* (PP) and *Gloeophyllum trabeum* (GT)] than White Rot [*Coriolus versicolor* (CV)].

In addition, Schneider *et al.* (1997) noted that salt leaching from the salt-treated wood blocks during the test killed fungi on the feeder strips. Their EDX (Energy Dispersive X-Ray Analysis) examination showed elevated levels of sulfur and a strong odor of sulfur was emitted from fresh samples, the source of this element could not be determined. Sulfur appeared to be resistant to leaching (Schneider *et al.*, 1997). This salt leaching shows that the permanency of soluble salts is only satisfactory in the indoor where the material is not exposed to water. As expected, Catelli *et al.* (2016)

Table V.
pH values of Native and salt-treated *Kentichi*
and *Deglet Noor* varieties palm woods.

Palm wood samples	pH (determined by hot water extraction process)
Native Palm wood (<i>Kentichi</i>)	3,6 ^a
Native Palm wood wood (<i>Deglet Noor</i>)	4,5 ^A
Salt-treated Palm (<i>Kentichi</i>)	5,7 ^b
Salt-treated Palm wood (<i>Deglet Noor</i>)	4,6 ^B

proved that the salts were washed out by precipitation from the outdoor areas. This added durability might be useful for preventing the development of decay in service, although it is difficult to assess the benefits of such salt loadings when the risk of leaching is high. Applying an additional oil-based treatment to the wood might retard leaching loss and maintain the protection (Schneider *et al.*, 1997).

Mineral composition of Salt-Treated palm wood and its influence on impact on wood termite resistance

Termite resistance of salt-treated palm wood compared to those of native samples is less obvious than its decay resistance improvement. However, the termite mortality is higher for treated than those of native wood sample. This result can be explained by the followings:

- The high proportion of Silica contained into the treated palm wood influence termite damage and plays a significant role in its termite resistance (Dhawan *et al.*, 2007).
- High ash content reduces the food value of wood for termites because it is not absorbed in the gut body and passes through (Hapukotuwa and Grace, 2011).
- Some minerals in wood can have a toxic effect on the pests or disturb their physiology (Doi *et al.*, 1998; Kneip *et al.*, 2007). Termite damage was in relation to the chemicals of the wood samples and Subekti *et al.* (2015) showed that NaCl content present into wood sample have a high impact on termite mortality.

Conclusion

Native Tunisian *Kentichi* and *Deglet Noor* date palm woods showed a low durability: moderately durable against *Gloeophyllum trabeum* and slightly durable towards *Coriolus versicolor*, *Coniophora puteana*, and *Poria placenta* attacks, and were classified “sensible” against termites. Results showed that natural saltwater treatment in the Lake Chot Cherid of both date palm woods allowed to improve the wood decay resistance until the classes “durable” towards *Coriolus versicolor*, *Coniophora puteana* and *Gloeophyllum trabeum*, and “very durable” against *Poria placenta*. In addition, ICP-MS analyses allow understanding better the saltwater modification process of wood. However, this ancestral preservation process did not allow improving significantly the date palm wood

resistance against termites. In addition, results showed a significant increase of the air-dried density of palm wood samples which increases after the wood salt water immersion. Salt water treatment allows to improve greatly the palm wood MOR in bending and paralleling to the fibers.

In conclusion, all qualification testing showed that the saltwater treatment performed on Tunisian date palm woods (*Kentichi* and *Deglet Noor* cultivars) conferred to the wood material a good technological value. However, this process is still largely unknown and needs to be more controlled and optimized. In addition, the added durability might be useful for preventing the development of decay in service, although it is

difficult to assess the benefits of such salt loadings when the risk of leaching is high. Applying an additional oil-based treatment to the wood might retard leaching loss and maintain the protection. In this sense, it could be interesting now to think about a new industrial process based on this natural and ancestral method to valorize Tunisian date palm sector and commercialized new date palm wood materials.

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