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## DISPERSION AND STABILIZATION OF CERAMIC POWDERS IN THE PRESENCE OF WOOD FIBERS TREATED WITH AN ANIONIC SURFACTANT ADJUVANT: SODIUM LAURETH SULFATE

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### ABSTRACT

Wood nanofibers from munwiserie waste have been used as a partial replacement of cement for cement paste at a content of up to 0.5% by weight of cement. The effect of the nanofibrillated cellulose content on the porosity, the compressive strength and the degree of hydration of the cement was investigated. Results have shown an improvement in the compressive strength by more than 40% with 4% of added fiber cellulose. The chemical modification of nanofibers wood by grafting of chains alkyls in their surfaces can reduce the quantity of water absorbed by the sample. The addition of an anionic additive: Sodium Laureth Sulfate (SLS) in the water of mixing improves of more the surface of samples by minimizing the size of pores by emulsion effect, from where the absorption of water reduces. The alkaline treatment of fibers before being injected in the dough of cement has a very remarkable effect on the mechanical behavior of composites. The degree of hydration of the cement has increased with the cellulose content containing nanofibrils. The analyze revealed that the presence of nanofibrillated cellulose favored the hydration of the cement by producing more calcium silicate gel and portlandite, probably the main reason for this improvement in compressive strength.

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## INTRODUCTION

The use of the vegetables fibers in the strengthening of building materials is a relatively former domain, noting as example the impregnation of hair of horse in mortars of filler or the manufacturing of the blocks of raw earth strengthened by the straw. In cement based composites, the cellulose fiber had been used as pulp, short fiber, long fiber or fabrics (Ferreira, 2014; Olivito *et al.*, 2014). However, the used traditional methods obeyed no technological development and no scientific analysis that from 1970, when the first works were made to replace asbestos fibers in the prefabricated elements profiled in fibrocement (Sellami Asma, 2015). Reis (Reis, 2005) studied the possibility of substituting the synthetic fibers by natural fibers such as the coconut, the sugar cane, the bagasse. He showed that the epoxy strengthening of concretes of polymer by the fiber of coconut allows obtaining a light increase of the properties in the flexion of the composite contrary to two others types of fibers.

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The effect of surface treatment of the cellulose fibers with various coupling agents to enhance the durability of fiber and fibre-matrix interaction was reported in several studies. In order to stabilize the vegetable fiber, three classes of treatment can be mentioned in the bibliography: thermal, physical and chemical. Govin (2004) used a so-called "roasting" method for fiber processing. This technique therefore consists in heating the wood fibers under an inert atmosphere up to 280 ° C. for a relatively long period of time in order to modify its structure. This process has been able to limit the degradation of the fibers, so it has kept its mechanical properties to a minimum. Chemical treatment is the most studied worldwide, it consists of replacing the hydroxyl groups, highly hydrophilic and responsible for the swelling of the wood, by hydrophobic groups (Toledo Filho, 2000). Several studies have shown the importance of treating plant fibers with chemical solutions to increase the adhesion between the fiber and the matrix and consequently to improve the mechanical properties. Toledo *et al.* (Baley *et al.*, 2002) immersed sisal and coco in three solutions of different pH values; first they treated the fibers in water at pH = 8.3 (weakly basic medium), then in a solution of

lime Ca (OH) 2 at pH = 12 and finally in a solution of NaOH soda at pH = 11. The purpose of this type of treatment is to determine the impact of pH and the effect of the presence of calcium ions on the outer wall of plant fibers after immersion. In previous studies, our group has shown that the adsorption of surfactant on bleached cellulose fibers greatly enhances the aptitude of the substrate to uptake dissolved organic compounds from aqueous media (Aloulou *et al.*, 2004A; Aloulou, 2004b; Aloulou *et al.*, 2004c; Aloulou *et al.*, 2006; Alila *et al.*, 2007; Alila *et al.*, 2005). The improved solute adsorption was ascribed to the accumulation of the organic solutes within the aggregated domains formed by the self-assembly of surfactant monomers at the cellulose/water interface. Then, a chemical grafting of hydrocarbon structures that mimics the aggregated domains generated by the adsorbed surfactant molecules was accomplished (Marrakchi *et al.*, 2011). The aptitude of the ensued modified substrate to uptake dissolved organic solute in aqueous media has been investigated in a batch and in continuous operations. In this condition, the main environmental advantages such as biodegradability and renewal reduce or cease to exist. The aim of this study was to evaluate the effect of nanofiber treatment with cellulose acetate, and polymer from renewable resource, on the properties of cellulose nanofiber and cement based composite. Water absorption and mechanical tests were carried out on sisal fiber. Pull-out tests were carried out to evaluate the interface bond. For characterization of composites reinforced with 4% of treated cellulose nanofibers (Lf = 40 mm) flexural test were carried out. In this work, we continue our research regarding the potential use of modified cellulose substrate as a new composite material with cement matrix strengthened by vegetables nanofibers.

## MATERIALS AND METHODS

**Wood nanofiber:** The cellulose fibers are dispersed in water to form a suspension of 1 to 2% by weight. This suspension is passed through a high pressure homogenizer. This treatment involves subjecting the fibers to mechanical shear under high pressure, during which the fibers will be disintegrated and broken into nanofibrils more or less individual and disengaged.

**Adjuvant:** The additive used in our work is the SLS: (Sodium Laureth Sulfate), it possesses amphiphilic molecules (possess at the same time a hydrophilic group and a hydrophobic group). The hydrophilic part is the one which absorbs the water, while the hydrophobic part is an non polar part intended to attract lipids (Oil and fats), his chemical structure is represented on the face.

**Cement matrix:** To build the cement matrix, the proportion, by weight, of 0.5: 0.1: 0.4: 1: 0.35 (cement: silica: fly ash: sand: water) was used. The mixture were produced in a mechanical mixer of 20 dm<sup>3</sup>, according to following procedure (Ardanuy *et al.*, 2015): 30 seconds at low speed to add the cement products, 30 seconds at low speed to add the sand, 90 seconds stop to scrape the walls of the bowl, 60 seconds at high speed.

**FTIR analysis:** The Fourier transform infrared (FTIR) spectroscopy was used to analyze the composition change of the treated sisal. FTIR spectrum was obtained for cellulose fibers using a Perkin Elmer spectrometer.

**Water absorption test:** For the water absorption measurements, the specimens were withdrawn from the waters, wiped dry to remove the surface moisture, and then

weighted using an electronic balance accurate to 10<sup>-4</sup> g to monitor the mass during with time t. The moisture content, M(t) absorbed by each specimen is calculated from its weight before, w<sub>0</sub> and after, wt absorption as follows:  $Abs = 100(w_t - w_0)/w_0$ .

**Mechanical characterization: Compression resistance:** Several theoretical and experimental studies showed that the existence of fibers in a concrete influences slightly the compression resistance. In certain cases, we have a light increase of the ultimate constraint, in others a light decrease. This decrease can be caused by a bad compactness due to an excess of fibers, or a bad composition. The test of compression is led on test tubes of cylindrical form of diameter F = 3.5cm and of height H = 7cm. The principle of this try consists in putting the test tube under an increasing load until the break. Once the side surface of the test tube is rectified; the latter must be centered on the portico of didactic universal try with which its axis must be confused with the axis of hydraulic jack. The breaking load is the maximum load registered during the try. The compression resistance is the report enters the breaking load F<sub>c</sub> registered during the try and the transverse section of the test tube S. The compression resistance is thus calculated as follows:  $R_c = F_c / S$ . With:  $S = \pi \times \Phi^2 / 4$ , R<sub>c</sub> is directly obtained in MPa if F<sub>c</sub> is expressed in Mega Newton (MN) and S m<sup>2</sup>.

**Contact angle:** The measure of the angle of contact is a technique bound to the capacity of a liquid in Spread out on a surface by wet ability. The principle of this characterization thus consists to measure the angle enter the tangent of the profile of a drop of the liquid put down on the substratum, and the surface of the substratum. She allows measuring the energy of surface of the liquid or of Solid. In our work, we used the method of the angle of contact to deduct Hydrophilic or hydrophobic character of the surface of the sample. In our case, we used the water, polar solvent, as liquid of measure of angle of contact, this liquid allows to deduct the hydrophobic character (short angle, low energy of surface) or hydrophilic (small angle, big energy of surface) from the surface.

**Method of elaboration of composites studied:**

After the homogenization of the fresh material, this one is set up in molds. 24 hours later, samples were turned out and left to continue their ambient air drying during 10 days. The preparation of samples was realized with various rates of fibers (0,2; 0,4; 0,6; 0,8 and 1 %) with regard to compared with the total mass.

## RESULTS AND DISCUSSION

### Analysis by Infrared spectroscopy FTIR

The specter IR of wood nanofibers handled by the soda and the wood transplanted by the anhydride of acetic acid possesses certain modifications. Indeed, one of the main effects of alkaline treatment by the soda is the disappearance of the peak centered in 1735cm<sup>-1</sup> corresponds to the groupings carbonyls C=O, characteristic of hémicelluloses. Indeed, hémicelluloses is probably dissolved by the soda, which is a basic environment (Figure 1A). According to the obtained specters, we distinguish the peak corresponds to the groupings C=O is more intense for the wood modified by the anhydride of acetic acid, what reveals that we managed to transplant short chains alkyls of form (CH<sub>3</sub>-CH<sub>2</sub>-C=O) on wood fibers. Besides, we notice that the OH band some transplanted wood is less intense

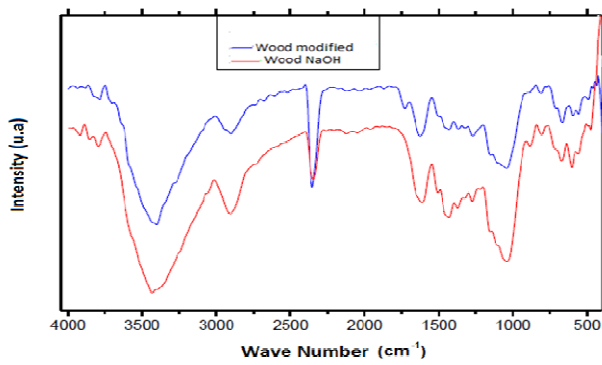


Figure 1-A. IR spectrum of 4000  $\text{cm}^{-1}$  to 500  $\text{cm}^{-1}$  of the fibers treated with soda and grafted fibers

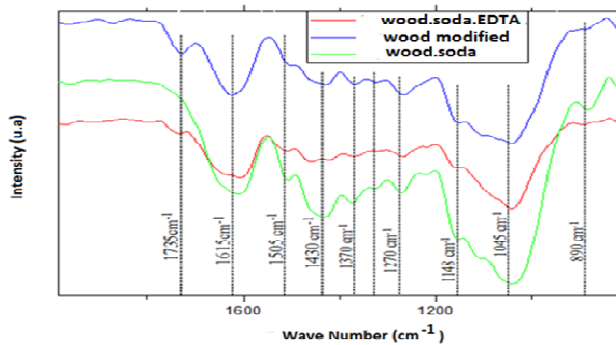


Figure 1-B. IR spectrum of 4000  $\text{cm}^{-1}$  to 500  $\text{cm}^{-1}$  of the fibers treated with soda, EDTA and grafted fibers

with regard to that some wood brute, indicating that a relatively important proportion of the functions OH some untreated wood was consumed by esterification with anhydrides. The band in 1620  $\text{cm}^{-1}$  is associated with the quantity of water absorbed by the wood and can vary with the nature of samples. So, peaks situated between 1120-900  $\text{cm}^{-1}$  correspond to the vibrations of valence of the groupings C-O and C-C and seem to remain unchanged. What shows that the modification summer realized on the surface while keeping the motives for polymers. By basing on previous studies, we notice that both bands, absorbing in 1610  $\text{cm}^{-1}$  and 1505  $\text{cm}^{-1}$  partners in the symmetric strains of the connections C=C of the aromatic cycles, are generally attributed to the lignin. After the alkaline treatment, the intensity of these bands decreases slightly, and that can be showed of a partial elimination of lignin. The characteristic bands of pectin are situated in the zone between 1750  $\text{cm}^{-1}$  and 1230  $\text{cm}^{-1}$ , certain studies identify these esterified pectin have 3 characteristic peaks in 1748, 1445 and 1234  $\text{cm}^{-1}$ , these peaks are in fact difficult to separate signals relative to the other constituents (Figure 1B).

The NMR spectra of fiber wood and fiber wood modified are shown in Fig. 1C. In the spectrum of fiber wood (Fig. 1Ca), all signals, i.e. those at 104.7 ppm (C-1), 89.8 ppm (C-4 of crystalline cellulose), 74.7 ppm (C-5), 72 ppm (C-2 and C-3), and 69.5 ppm (C-6 of crystalline cellulose) (Liu *et al.*, 2010), are attributed to six carbon atoms of the glucose unit. However, there is no signal of C-4 and C-6 of amorphous cellulose in the spectrum, suggesting the complete disruption of the cellulose amorphous structure during the acid hydrolysis of cotton. Notably, two more intense signals appear in the spectrum of fiber wood modified (Fig. 1Cb) in addition to those of fiber wood, due to carbon atoms of carboxylic groups C-7 at 173.8 ppm and methylene carbon in the acyl moiety at 20-40 ppm.

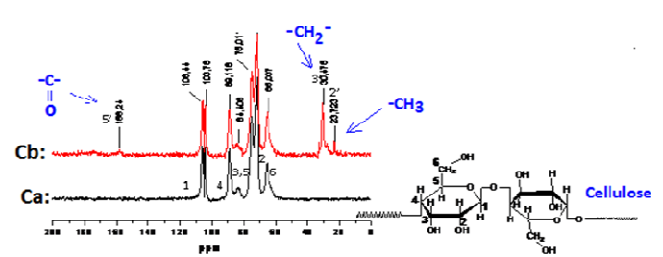


Figure 1-C. CP-MAS  $^{13}\text{C}$ NMR spectra of cellulose fibers and the modified after esterification

### Recovery of pores

The hydration of cement favors the liberation of a very important quantity of the ions calcium, the addition of a low concentration by adjuvant anionic (anionic surfactant SDBS) can assure the electrostatic neutralization. If we add a higher concentration of this additive, we shall have in this case an excess of the molecules of SLS in the mixture. By arriving at the CMC (critical micellar concentration), the molecules of the additive group together by forming micelles responsible for the superficial cover of complex  $\text{Ca}^{2+}$ /pectin, and as a result of recovery of pores in composites.

### Morphological characterization of nanofiber wood

Nanofiber wood (NFW) product from wood was characterized by scanning electron field spectroscopy and atomic force microscopy. Microscopic observations reveal a nanometric diameter of NFW. All nanofibers produced have a width that varies from a few nanometers to a maximum of 30 nm and a length of a few microns. The NFW extracted from the different straws by high pressure homogenization treatment have a uniform distribution of the width. These observations confirm the results obtained above. On the other hand the obtaining of nanofibrils whose diameter is around 5 nm confirms a perfect fibrillation which brings us to the ultimate constituents of the cellulose fibril (Figure 2).

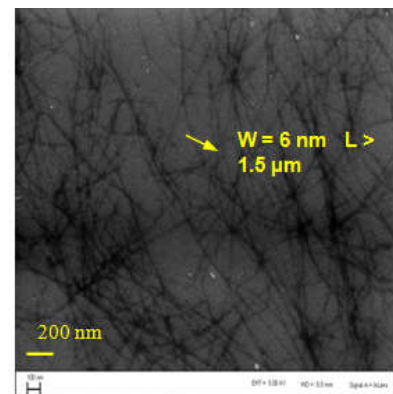
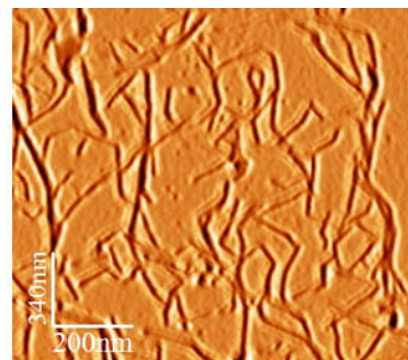
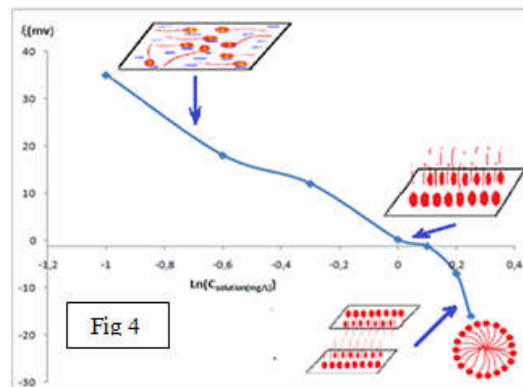


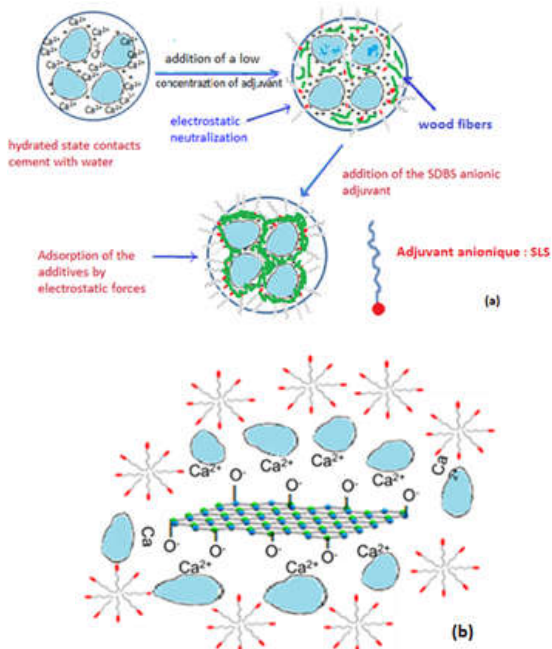
Figure 2. Morphologie of nanofiber wood

**Electrokinetic study of the fibrous suspensions handled by an additive in the presence of a cement matrix**

Every surfactant possesses a certain concentration molar called "critical micellar concentration": (CMC), from which it will be capable of forming micelles grouping together on the superficial part of the composite. This operation gives birth to a phenomenon of emulsion (fig3a-b). According to the curve of the potential Zeta, we notice that the addition of the additive in the manufacturing of composites, pass by several stages. Indeed, for a low concentration of the SDBS  $\sim (0,1\text{mmol} / \text{L} )$ , the potential Zeta is of the order of 35 mV, this value results from the ionization of calcium during the hydration of cement. The more the concentration by adjuvant increases, the more the potential Zeta decreases, this relation informs us about the role of the molecules of the anionic additive in the neutralization of the ions  $\text{Ca}^{2+}$ . By arriving at a certain concentration of the additive  $\sim(1.1\text{mmol} / \text{L} )$ , the total neutralization was made and we have negative values of the potential Zeta. These show that the molecules of the additive are in excess, where from the formation of micelles from the CMC (Figure 4).

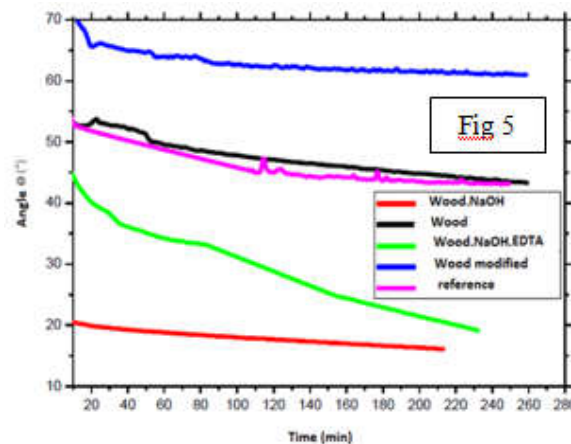


**Figure 4. variation of the zeta potential as a function of the concentration of adjuvant SDBS**



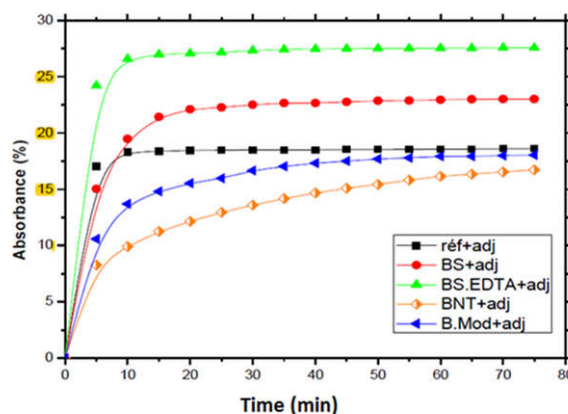
**Figure 3-a: state of the environment of cement grains: emulsion (wood fiber modified /cement / SLS); b: formation of micelles around wood fibers/cement**

We made appeal to this technique to analyze the aspect of the surfaces of samples, and see the effect of the various treatments processings of wood fibers on the absorbance in water. Indeed, measures of the angles of contact of drop of water on samples were realized by means of a camera CCD which allows registering, in a speed of images/s 30, the aspect of a calibrated drop of water. Then, powerful software of image processing allows to analyze the outline of the drop and to determine with a big precision the angle of contact with the plane surface. The evolution of the angles of contact for the various samples is presented on the Figure 5. The analysis shows that the grafting of the hydrocarbon chains the surface of fibers makes tip over their characters of surface of a hydrophilic state very marked in a hydrophobic state. By analyzing the result concerning the evolution of the angles of contact according to time, we distinguish a clear difference at the level of the variation of these angles.

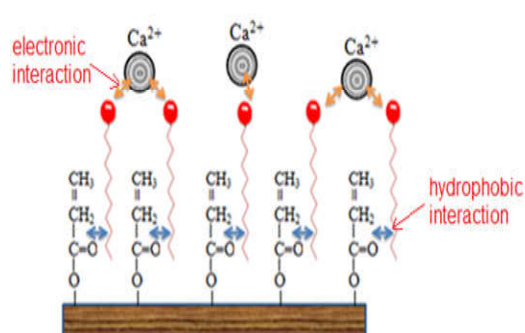


**Figure 5. variation of the contact angle as a function of the fiber treatment**

In fact, the alkaline treatment processing of fibers by the soda increases the hydrophilic aspect, and that reveals of the lo angle of contact registered for this sample, which remained stable near 20°. The same thing for fibers handled by the soda / EDTA the angle of contact of which varies 45° at the beginning. From the obtained curves, we notice that the wood modified by grafting of hydrocarbon chain possesses the highest angle (70°), it suggests that the evolution of character of surface is bound to the presence of chains alkyls which are going to paper the surface while adopting a perpendicular configuration on the surface to that this.



**Figure 6 a. effect of the adjuvant on water absorbance**

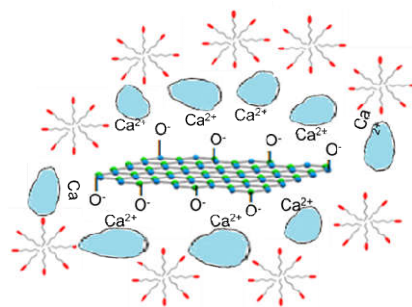


**Figure 6 b. Effect of the adjuvant (SDBS) on the interface fiber / matrix**

**Absorbance in water: Effect of the adjuvant:** In this part, we added an anionic additive which is the SDBS (dodecyl benzene sulfonates of sodium), to study his effect on the surface of samples and as a consequence on the absorbance in water. The realized measures showed that the contribution of the additive is very remarkable in the reduction of the quantity of absorbed water (Figure 6 a-b). The decrease of the rate of water absorbed by test tubes containing modified fibers and gross fibers about 4 % and 11 % respectively with regard to the reference, highlights the role of the additive in the superficial packaging of fibers. This result can be interpreted by the fact that the molecules of the used additive work as a super-plasticizing which covers the surface of fibers preventing as a consequence their contact with the water. The molecules of the additive play the role of an intermediate element between the ions of calcium stemming from the hydration of the cement on one hand, and the alkyls chains transplanted on the surface of fibers on the other hand. They are capable of getting a significant number of  $\text{Ca}^{2+}$  by their hydrophilic grouping through electrostatic connections, leaving their hydrophobic part bound, by connections of hydrophobic type, on the hydrocarbon chains. The decrease of the rate of absorption thus results from the reduction of pores within the made samples. What justifies the superficial shine of these test tubes when one we use the additive.

### Biographical notes

Fadhel Aloulou is an assistant teacher in Department of physics chemistry of the University de Sousse (ESSHTS). He has a PhD in the Inorganic Chemistry from Faculty of Sciences of Sfax, Tunisia (LMSE Laboratory). His research activities include chemical modification of cellulose and lignocellulosic materials, the synthesis of functional polymer for colloidal chemistry and emulsion polymerisation, and the exploitation of chemically modified cellulose fibres as reusable adsorbent for dissolved organic pollutants. He has published more than 40 papers in international journals dealing with polymer science and physical colloidal chemistry. Before landing in Sousse (University of Sousse): LabEM-LR11ES34: Laboratory of Energy and Materials, University of Sousse, Tunisia. His current professional interests are on cellulose renewable resources and the properties of macromolecular surfaces and interfaces. He has published different papers on the interaction of cellulose fibres with cationic and anionic surfactant. Sabrina Alila is an Associate Professor in Department of physics chemistry of the University de Sousse (ESSHTS). She has a PhD in the Inorganic Chemistry from Faculty of Sciences of Sfax, Tunisia (LMSE Laboratory) Her research in the LMSE Laboratory at the Department of University de Sfax, Tunisia.



Her research topic is concerned with surface chemical modification of cellulose fibres to enhance their absorption capacity towards dissolved organic pollutants, including pesticides and herbicides. She has published different papers on the interaction of cellulose fibres with cationic surfactant. Seffen Mongi is a Professor of Higher Education in Department of physics chemistry of the University de Sousse (ESSHTS). (26 years of experience in teaching and research at LabEM-LR11ES34: Laboratory of Energy and Materials, University of Sousse, Tunisia. «Experience of a wide range of subjects:» mineral and organic chemistry, atomistic, chemical thermodynamics, corrosion, metallurgy, energy materials. Local coordinator of the Euro-Mediterranean project: FP7 FP4BATIW Coordinator of the Tunisian-Moroccan bilateral project: 29 / TM / 13

### Conclusion

The works presented in this study join the objective to understand better the physic-chemical and mechanical phenomena, involved during the association of the wood with some white cement. The best knowledge of the interaction of these two materials allows an improvement of the obtained composite. Used wood fibers were considered as composites, they are chemically characterized by several components, of which the cellulose which assures our composites the best mechanical performances. However, the presence of the other elements as the sugars and the wax, can affect their resistances. For that purpose, we realized various types of treatments, to modify the surface of fibers and increase their collision with the cement.

The second part summer realized on the elaboration of samples by injecting wood fibers treated in the dough of cement, during which, we used an anionic additive. The intervention of the latter was very remarkable in the improvement of the surface of composites and reduction of the absorbance in water. Through this work, we were able to implement cement-based test tubes white and fibers lignocellulosique (wood). The challenge topped was the treatment processing of these fibers to improve the membership (support) fibers / matrix, and as a consequence make them more compatible with such an organic matrix (cement). One of the adults concerns concerning our subject, is the absorbance in water, which imposes on the material of strong dimensional variations, and which as a consequence can act negatively on their durabilities. That is why, we proposed the idea to substitute the groupings OH for the surface of fibers, by short alkyls chains grafting with the aim of reducing the quantity of water absorbed by fibers. Analyses realized on our samples showed the efficiency of the grafting of the hydrocarbon chains on the surface of fibers, in the reduction of the rate absorbed in water with regard to (compared with) that some only cement.

This characteristic was furthermore improved by adding to the composite to the fresh state a quantity of an anionic additive. The chemical treatments processing made on fibers seem to improve the membership between the matrix and its reinforcement. The composite containing fibers preprocessed in a solution of soda possesses the constraint in the the highest compression. These gains can be not only awarded to the increase of the interfacial connection white wooden / cement, but also very probably in the increase of the resistance of fibers.

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