

Effect of Chemicals Treatments on the Morphological, Mechanical, Thermal and Water Uptake Properties of Polyvinyl Chloride/ Palm Fibers Composites

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<https://doi.org/10.18280/rcma.290101>

Received: 9 September 2018

Accepted: 11 December 2018

Keywords:

poly (vinyl chloride), palm fibers, acetylation, alkali, mechanical, thermal, water absorption

ABSTRACT

The use of natural resources in composite materials becomes more frequently, as they are low-cost and lightweight materials. In addition, industrial trends tend towards Eco-products, hence the importance of integrating natural products that are recyclable and easily degradable. Aim of this study is prepared polymer composites of polyvinyl chloride (PVC) using palm fibers at different loading (10 and 30 % by weight). Improving the interfacial adhesion between matrix- filler interfaces using chemical modification of date palm fibers (DPF) by two types of treatments (acetylation, alkali). The effect of chemical treatment and fiber content on morphological, thermal, mechanical and water absorption properties of composites have been studied. It was found that the use of treated fibers in PVC composites improves the mechanical properties and decomposition temperature, and reduce water absorption of the composites.

1. INTRODUCTION

Composite materials are widely used all over the world in the manufacture of building materials, automobiles, naval parts and others. Composite materials consist of two constituents namely a matrix and reinforcement [1]. Synthetic materials have good mechanical properties and durability, but due to the environmental awareness, these materials have some criticisms because they are not easily biodegradable [2]. For Thus, efforts are being made to invent environmentally friendly composite materials to replace synthetic reinforcements such as glass, carbon, aramid fibers, etc., using natural cellulosic fibers like jute [3], coconut [4], sisal [5-7], bamboo [8-9], kenaf [10], sugar cane and rice [11-12]. The date palm (*Phoenix dactylifera* L) other source of cellulosic fibers with attractive morphology properties, has a fibrous structure can be extracted from different parts of plant such as: leaf fibers in the peduncle, bast fibers in the stem, wood fibers in the trunk and surface fibers around the trunk [13]. In Algeria, date palm is particularly abundant in Saharan oasis (more than 18.6 million date palms). The residual waste from these plants are incinerated or thrown every year after the harvest, generating more than 200,000 tonnes of waste causing major inconvenience in nature [14].

The effectiveness of composite materials depends to their ability to transfer stresses from the continuous phase (polymer matrix) to the dispersed phase (fillers). They are obtained when there is a good adhesion between surface of polymer and fibers with well dispersed in the matrix [15]. It is well known that the natural fillers are hydrophilic materials creates difficulties in achieving adhesion with polymer chains; generates voids around the fibers in composites, resulting in

higher water uptake [16]. For thus, the treatment of natural fillers and some additives were used in the composite to improve charge-matrix compatibility, such as such as alkali treatment or mercerization, silanization, acetylation, benzylation, acrylation, maleated coupling agents, isocyanates, permanganate treatment and plasma treatment [17-20].

Sodium hydroxide (Alkali treatment) is commonly used in Modification of the fiber, which decrease hydrogen bindings in cellulose and remove hydroxyl groups that tend to bind to water molecules to reducing the ability of fibers to absorb moisture [21]. In addition, acetylation is one of the most studied reactions for lignocellulosic materials. The principle of the method consists in reacting the hydroxyl groups (-OH) of the fibers components, i.e. lignin, hemicelluloses, and those of amorphous cellulose with acetyl groups (CH₃CO⁻) [22]. A reduction of about 50% in moisture absorption for acetylated jute fibers and up to 65% for acetylated pine fibers with increasing in shear strength of acetylated fiber composites has been reported in the literature [23-25].

Polyvinyl chloride (PVC) is among of many polymers that have been used as matrix in composites material, it is a thermoplastic material widely used in the fields of automotive, housing and construction, due to their high mechanical strength, and corrosion resistance. In addition, it is easy to manufacture and suitable for engineering applications for its hardness and flexibility [26]. However, PVC is not biodegradable; it is a contaminant material due to the release of harmful substances into the atmosphere, such as hydrogen chloride and dioxins, during processing or decomposition [27-28]. The combination of PVC and natural fibers is an interesting because of the ecological friendliness of natural

fibers.

The aim of this paper is to compare the impact of chemical treatment on the mechanical, water uptake and thermal properties of a palm date fiber-reinforced polyvinyl chloride (PVC) composites. The chemical treatments used in this work, alkali NaOH, and acetylation, have been chosen because they exhibit a different interaction mechanism with both fibers and polymer matrix. The properties of composites prepared with chemically-treated fibers will be compared to results obtained from untreated fibers composite as well as neat polymer.

2. MATERIAL AND METHODS

2.1 Materials

The polymer used as matrix was polyvinyl chloride (PVC) also containing plasticizer, stabilizer, and lubricant provided by the national unity of cable industry, located in Biskra southeastern Algeria. The physical characteristics of the polymer are listed in Table 1.

Table 1. Properties of polyvinyl chloride (PVC)

Polymerization degree	1250–1450
Density (g/cm ³)	0.43-0.50
Melt flow index at 200°C (g/s)	≥10
Moisture content (%)	≤0.3
Sulphate ash (%)	≤0.1%

The leaves of the date palm were collected from local agricultural resources (Biskra region, Algeria). They were washed several times with water to remove impurities and dried in the open air for 24 hours. After this, they are ground and sieved and stored in polyethylene bags for further compounding. Sodium hydroxide, ethanol, acetic acid, acetic anhydride, sulphuric acid and hydrochloric acid, were collected from reactive grade from Sigma-Aldrich, were used for the fiber surface treatments.

2.2 Chemical treatments of palm fibers

Palm fibers were subjected to the following surface treatments, in order to improve their interfacial adhesion with PVC matrix.

2.2.1 Alkali treatment

Palm fibers were treated with an aqueous solution of NaOH (5wt%). The fibers were immersed in the solution during 2 h at 50 °C temperature and then washed several times with distilled water containing acetic acid in order to neutralize the excess sodium hydroxide. After they are washed with distilled water until pH value of 7 was attained. The palm fibers were dried at 80 °C for 24 h in a vacuum oven.

2.2.2 Acetylation treatment

Palm fibers are immersed in acetic acid solution at 25°C for 45 min, and then they are decanted and immersed in a solution of acetic anhydride with a few drops of sulfuric acid for 2 hours at 50 °C. The fibers are filtered and washed with distilled water to remove excess acetic acid, followed by drying in an oven at 80°C for 24 hours.

2.3 Preparation of PVC composites

PVC powder and the various additives were placed in a mixer at a speed of 3000 rpm at 70 °C below the glass transition temperature of pure PVC powder. The different formulations obtained were used to prepare films by the calendaring process at 160 °C in a "SCHWABENTHAN polymix 200 p" calender. After the addition of the matrix the palm fiber was added as soon as the polymer had reached a steady plastifying state. Before mixing, the fibers were dried in a vacuum oven at 75 °C for 24 h to prevent the formation of porous products by water evaporation during the composite preparation. The fiber content in the composites was 0, 10 and 30 wt %. The molten mix was transferred to a preheated press at 160 °C under a pressure of 300 bar in a hydrolic press "SWHWABENTHAN polystat 300s" for 5 min molder for specimen fabrication.

2.4 Characterization

2.4.1 FTIR analysis

Fourier transform infrared spectroscopy (FTIR) spectra were recorded on SHIMADZU 8400S spectrometer to analysis the possible chemical bonding existing in the untreated and treated palm fibers using KBr pellets containing 1% of fibers finely ground at a range of 4000 – 400 cm⁻¹.

2.4.2 Morphological analysis

The morphology of the virgin PVC and the composites were studied using a Quanta FEG250 scanning electron microscope (SEM) operating at 15 kV. The specimens were carried out under liquid nitrogen. They were coated with a 50-100 μm layer of gold to avoid sample charging under the electron beam.

2.4.3 Tensile tests

The different composite samples PVC/treated and untreated palm fibers were subjected to tensile tests according to ASTM D638, using Zwick / Roell Z50 testing machine at 1 mm/min crosshead speed preventing the viscoelastic effect. Five measurements were conducted for each sample, and the results were averaged to obtain a mean value.

2.4.4 Hardness test

Shore hardness A of the samples was evaluated by using a hardness tester type Zwick/Roell. Samples were placed on a horizontal surface. Tester was kept in vertical position and pressed on the specimen so that the presser foot was parallel to specimen. Five readings at different points were noted, and average value is reported according to ISO 7619-1:2010.

2.4.5 Thermal analysis

Thermogravimetric analysis (TGA) was performed to determine weight loss as a function of temperature using SDT Q600 instrument. A sample of initial mass of 5–30 mg was introduced into a platinum crucible, the sample mass (TGA) variation was then measured as a function of temperature (or time) under an inert nitrogen atmosphere from 20°C to 700 °C, at a heating rate of 10 °C/min.

2.4.6 Water absorption

Water absorption of PVC/palm fiber composites was carried out according to ASTM D570-98 standard test method. The PVC/palm fiber composites were cut into rectangular shape (20 x 20 x 2 mm³) and then immersed in a distill water

bath and then the specimens were removed periodically from water, and the increase in their weight was measured on a balance with a precision of 0.0001 g over a period of 600 h. The percentage gain at any time (W_t) was calculated according to Eq (1);

$$W_t(\%) = \frac{W_w - W_0}{W_0} \times 100 \quad (1)$$

where, W_0 and W_w denote the initial weight of material and weight of material after exposure to water absorption, respectively.

3. RESULTS AND DISCUSSION

3.1 FTIR results

Figure 1 shows the IR spectra of untreated and treated date palm fibers. Untreated fibers (Figure 1.a) shows characteristics peaks; the broad peak at 3418 cm^{-1} which is related to the stretching of OH groups in celluloses and lignins [29]. The peaks at 2919 cm^{-1} , 2849 cm^{-1} respectively attributed to symmetric and asymmetric elongation vibrations of the CH-bonds of the $-\text{CH}_2$ group of the cellulose and lignin segments [30]. However, the strong and sharp peak at 1732 cm^{-1} attributed to $>\text{C}=\text{O}$ stretching vibration of hemicellulose [31]. The bands at 1622 and 1651 cm^{-1} are due to the water absorbed by the untreated palm fiber because of its pronounced hydrophilic character [32]. A small peak at 1513 cm^{-1} corresponds to the vibration of the benzene ring of lignin [33]. The peaks at 1460 , 1435 and 1383 cm^{-1} are due to the deformation vibrations of the $-\text{CH}_2$ groups and CH_3 , contained in lignin structures and hemicellulose. Peak at 1229 cm^{-1} exhibits the elongation vibration of the C-O bond of lignin. The small peak found at 1159 cm^{-1} corresponds to the vibration of C-O-C responsible for β -glucosidic bonds in cellulose and hemicellulose [34]. The elongation band of the C-H bond of lignin appears clearly by the peak 1107 cm^{-1} .

In acetylated fibers spectrum (Figure 1.b) there is a reduction on the intensities of the bands assigned to the substitution of the hydroxyl groups by the acetyl groups mainly those at 3418 cm^{-1} [35]. The absorbance in the region 1737 and 1229 cm^{-1} is due to the esterification of the hydroxyl groups resulting in increased stretching vibration of the carbonyl group (C=O). The spectrum of unmodified fiber shows an absorption peak at 1383 cm^{-1} attributed to the $-\text{C}-\text{H}$ bending vibration. After esterification, the added contribution of the acetyl ($-\text{C}-\text{CH}_3$) stretching vibration intensifies this absorption peak [36-37].

The acetylation reaction of cellulosic fibers with acetic anhydride has been confirmed by the FTIR analysis, this reaction is carried out as follows:

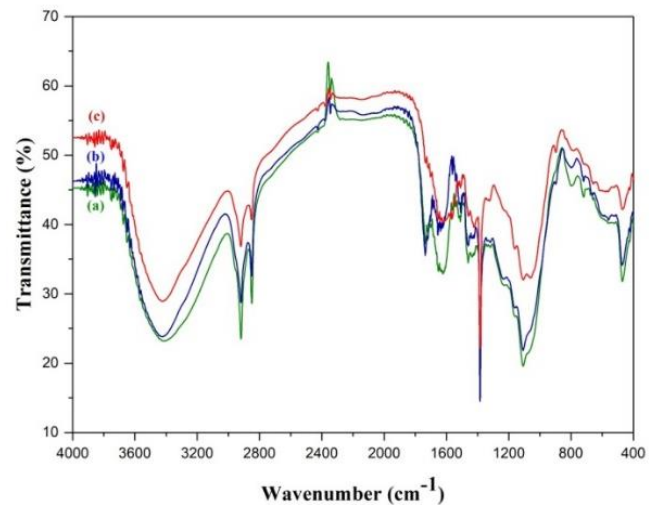
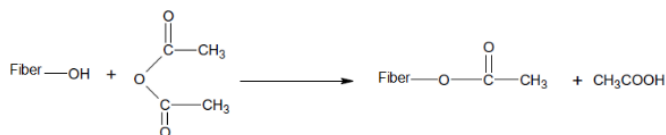


Figure 1. FTIR spectra of (a) untreated (b) acetylated and (c) alkali treated palm fibers

Alkali fibers spectrum (Figure 1. c), have the lowest amplitude, confirming that the chemical treatment reduces the effect and number of OH links. The intensity of the peaks located at 2920 and 2851 cm^{-1} decreased sharply, these two peaks correspond to the vibration of the C-H, C-H₂ and C-H₃ linkages of the lignins [38]. The alkaline treatment induces the partial disappearance of hemicellulose and lignin, confirmed by the disappearance of the band which characterizes the carbonyl group observed at 1735 cm^{-1} . a decrease in the peak intensity at 1421 cm^{-1} corresponding to the deformation of the $-\text{CH}_2$ group of lignin with the disappearance of the peak at 1460 cm^{-1} due to the aromatic rings of lignin is observed. It can also be seen that the intensity of the peak attributed to the vibration of the benzene ring of lignin between 1577 and 1507 cm^{-1} has decreased [39, 40]. The reaction is carried out as follows.



3.2 Characterization of PVC composites

3.2.1 Scanning electron microscopy

The effect of chemical treatments of the palm fibers surface on the interfacial adhesion of PVC/ palm fiber composites was studied by examining the fracture surfaces of composites with SEM. The SEM micrographs of the fracture surface of untreated and treated composites containing 30 phr of fibers can be seen in Figure 2a, b, c, and d. Examination of the fracture surface of the virgin polyvinyl chloride matrix (Figure 2.a) shows a very smooth and very homogeneous surface. Moreover, for untreated composites PVC/UPF (Figure 2.b) indicates that there are voids between the fibers and the matrix, which proves the incompatibility of the two phases because of the poor interfacial adhesion and the difference in the energies of the free surface of the hydrophilic palm fiber and the hydrophobic polymer. This facilitates the loosening of the fiber during the fracture [41].

The use of treated fibers with polymer chains (Figure 2.c, d), reduce the detachment and absence of aggregates of fibers. The empty spaces are smaller, or have disappeared most of the time. The use of the alkaline treatment also removes waxes from the surface of the fibers and promotes good chemical bonding between the fibers and the matrix especially when the

acetylation treatment is used. This improvement leads to the good wettability between the fiber and the polymer matrix, which results in a better dispersion of the fibers and in the reduction of the energy and the polarity of the charge. All these observations are consistent with the results of the mechanical tests [22, 42- 44].

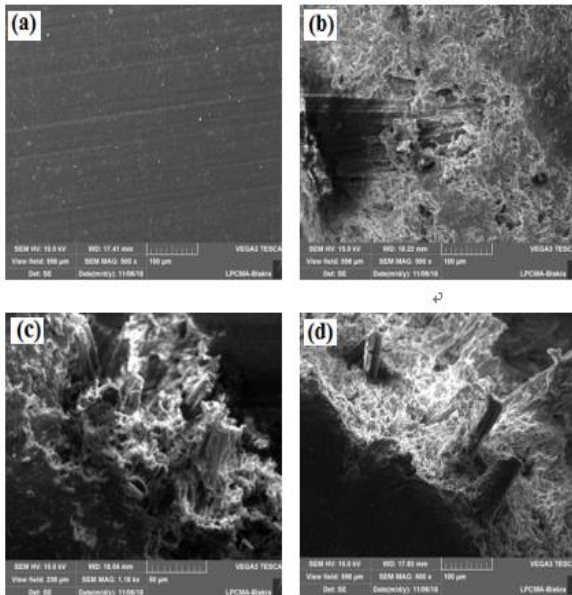


Figure 2. SEM micrographs of PVC composites with; (a) neat PVC, (b) untreated fibers, (c) acetylated and (d) alkali treated palm fibers

3.2.2 Mechanical characterization

The mechanical properties of thermoplastic matrix composites such as PVC depend on several factors, such as the dispersion of fibers, the amount of air stored in the composite during mixing [45]. However, according to Georgopoulos et al [46], it should be noted that the fiber-matrix interface plays an essential role in ensuring that each element constituting the composite contributes to the final property of the material.

The tensile strength of PVC composites reinforced with untreated palm fibers decreased with increasing of loading ratio compared to the neat PVC (Figure 3). These results are in agreement with those found by Yao et al [47], they recorded a decrease in the tensile strength of the composites with the increase of the loading rate, thus increases the interfacial air and weakens the interactions between the charge and the matrix. For the composites reinforced with alkalited and acetylated fibers we noted improvement in tensile strength of composites compared to untreated composites. This increase due to the better dispersion of the filler and in particular to the reinforcement of the interfacial bonds between the PVC and the treated palm fibers [25, 48]. The maximum tensile strength is 8.42 MPa achieved for the composites reinforced by acetylated fibers at 10wt% of the fiber rate.

The evolution of the elongation at break of the treated and untreated composites as a function of the palm fiber content (Figure 4) characterized by a significant decrease in elongation at break for composites reinforced with untreated fiber (PVC/UPF), this decrease due to the hydrophilic nature of the untreated fibers that absorb more moisture and cause swelling in composites which causes the embrittlement of the material. On the other hand to the increasing volume occupied by the fibers, leads to defects in the material and reduces interchain interactions [49].

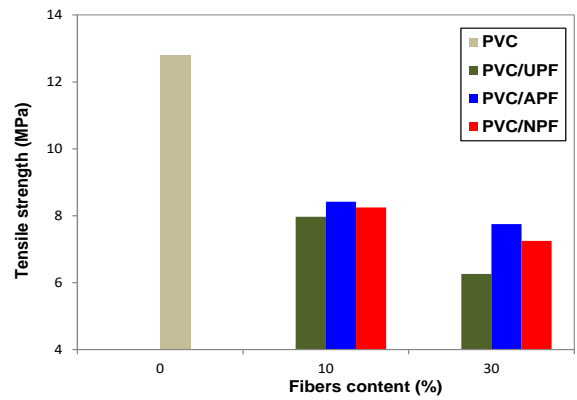


Figure 3. Tensile strength of neat PVC and composites with treated and untreated fibers

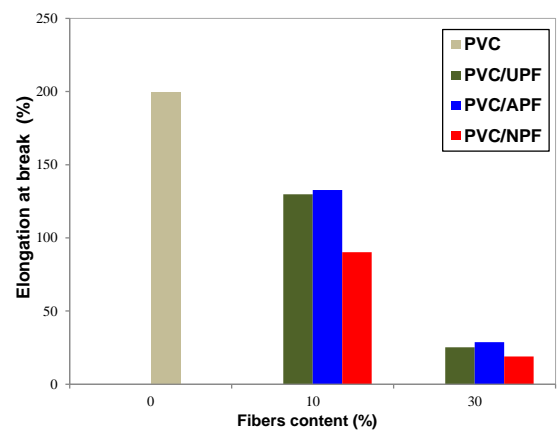


Figure 4. Elongation at break of neat PVC and composites with treated and untreated fibers

The addition of acetylated palm fibers, allows a certain increase in elongation at break compared to the composites made with the untreated fibers, estimated at 131.7 and 28.8 % for the formulations at 10 and 30wt% respectively. This improvement is attributed to the good dispersion of fibers which lends some flexibility to the material. Moreover, it is noted that the addition of the alkalited fibers in PVC matrix (PVC/NTF) does not bring any improvement in the deformation; such a result is confirmed by Nedjma et al. [50].

3.2.3 Shore hardness

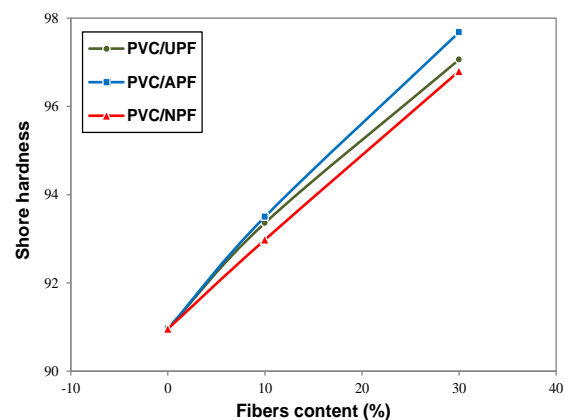


Figure 5. Shore hardness of neat PVC and composites with treated and untreated fibers

Shore hardness of PVC composites with untreated palm fibers (Figure 5), increase with the increasing of fibers. These results indicate the decrease in flexibility and the increase in stiffness [51]. For acetylated composites (PVC/APF) recorded increasing in Shore hardness. This is interpreted by the good dispersion of the fiber in the matrix with the decrease of the voids and a higher fiber-matrix interfacial adhesion [52]. The lower values for alkali treated composites (PVC/NPF) compared to untreated fibers are explained by the removal of hard constituents in palm fibers after treatments, hence the decrease in fiber hardness.

3.2.4 Thermal stability

The thermal stability of neat PVC and PVC/ palm date fiber composites was investigated with thermal analysis, and the results are shown on the TG thermograms of Figure 6.a and the DTG thermograms of Figure 6.b. The TG thermograms are identical for PVC matrix and the different composites. Several important thermal parameters are derived from these data, such as onset decomposition temperature, temperature and maximum speed, and residue rate. These properties are summarized in Table 2.

From the results obtained, it is noted that the onset decomposition temperature of PVC is slightly higher than those of PVC/UPF, PVC/APF, and PVC/NPF. Decrease of the degradation temperature with the addition of untreated fibers (PVC/UPF) explained by the poor adhesion between fibers and polymer chains which increase the friction between these chains with the rise in temperature causing the release of the hydrogen chloride gas [53]. For composites with acetylated fibers PVC/APF, there is an increase in the onset of degradation temperature over untreated fiber composites; this may be related to the increase of hydrogen bonds in acetylated fibers which requires significant break energies [35, 54].

Date palm fibers treated with sodium hydroxide caused the decrease of the onset decomposition temperature for composites; it is 228.1 °C, for PVC/NPF₃₀ formulations compared to that of untreated fiber composites PVC/UPF₃₀ which is 229 °C. This result probably due to the polarity of the fiber which has decreased in the case of mercerization in an alkaline solution, elimination of part of the lignin (more or less hydrophobic element and therefore more or less polar is eliminated). El Mechtali et al. [55] proceeded with the alkaline treatment of the almond shell particles and found a decrease in the temperature of the beginning of decomposition which they attribute to the decrease of the lignin content.

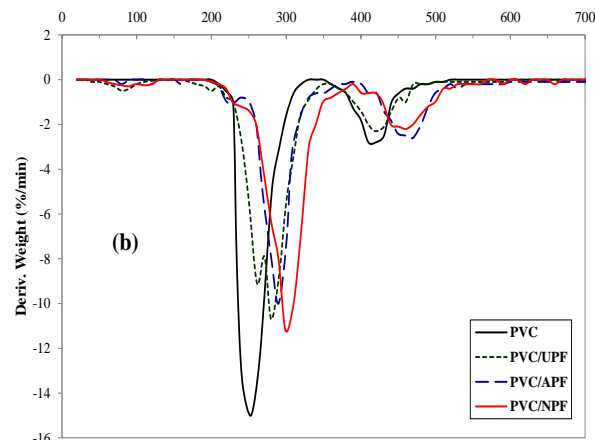
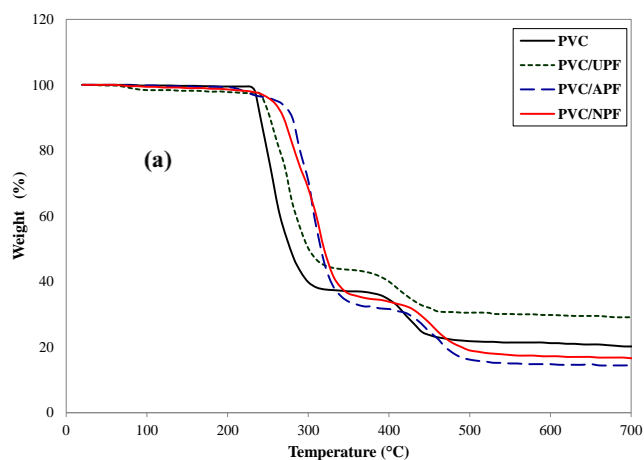


Figure 6. Thermograms of weight loss (a), and derivative weight loss (b) of neat PVC and composites with 30 wt% of treated and untreated fibers.

Composites samples exhibit a more stable thermal behavior than PVC after the onset decomposition temperature. This is due to a better intrinsic thermal stability of the cellulose fibers. Indeed, we can clearly see a decrease in the maximum decomposition rate of composites compared to that of PVC and we record a shift of temperature and maximum speed to the highest values. The results show that the introduction of the fiber into the PVC reduces the decomposition rate a sign of a better thermal behavior. The results are in agreement with the work of Ge et al. [56].

The DTG thermogram of untreated fiber composites, we notice the appearance of peaks around 80-120 °C which corresponds to the evaporation of the water physically adsorbed on the surface of the particles of the date palm fiber for untreated composites. This phenomenon can be attributed to the hydrophilic nature of untreated fibers that have the ability to absorb more water molecules. On the other hand, in the same temperature range, the polyvinyl chloride does not record any peak, which is the signature of the hydrophobic character of the PVC matrix.

Table 2. Thermal degradation and derivative weight loose of each thermal degradation stage with 30 wt% of fiber

Sample designation	PV C	PVC/UP F	PVC/AP F	PVC/NP F
Onset decomposition temperature (°C)	234	229.0	231.3	228.1
First Stage T _{max} (°C)	253	280.0	289.7	299.2
First Stage V _{max} (%/min)	15.0	10.7	12.6	11.2
Second Stage T _{max} (°C)	410	419.0	469.7	460.2
Second Stage V _{max} (%/min)	2.8	2.3	2.6	2.2
Residual rate (%)	20.0	29.0	14.2	16.6

3.2.5 Water absorption

The evolution of the water absorption of polyvinyl chloride composites with 10 and 30% of untreated fibers (Figure 7), mark an increase in the rate of water uptake with the incorporation of fibers in the matrix. Indeed, this is due to

hydrophilic nature of palm fibers, where, hydrogen bonds formed between water molecules and -OH groups present in cellulose and hemicellulose [57].

Acetylated composites (PVC/APF) show lower absorption compared to untreated composites. This indicates that the acetylation treatment improves the dispersion of the fibers, where by the acetyl groups make the surface of the fiber more hydrophobic. The reactive hydroxyl groups are the minor constituents of the fiber, i.e. lignin and hemicelluloses, and of amorphous cellulose [58].

Composites reinforced with alkali fibers (PVC/NPF) absorb more than untreated composites. This increase in absorption can be attributed to the removal of lignin which is hydrophobic, conversion of cellulose structure and improvement of interfacial adhesion [59]. Although there are others have found that this treatment decreases the water absorption that expressed by the strong adhesion in their composites.

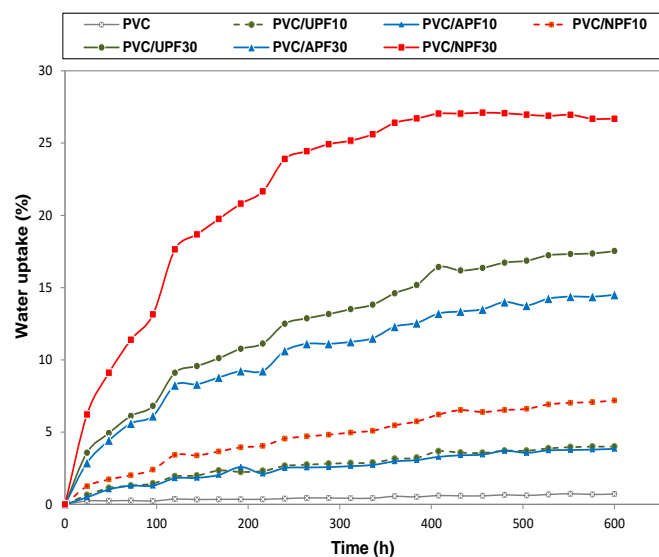


Figure 7. Water absorption of neat PVC and composites with treated and untreated fibers

4. CONCLUSIONS

In this paper, date palm fibers reinforced PVC composites with and without treatments were compounded and pressed into test samples with a loading rate of 10 and 30 wt%. The mechanical, thermal properties and water uptake of composites were compared and analyzed against neat PVC. The results show a change in structural of the fibers after treatments by the reduction of hydroxyl group band intensity of cellulose. The alkaline treatment leads to the partial disappearance of hemicellulose and lignin. The use of acetylated fibers in PVC composites improves the mechanical properties of the composite due to the chemical bonding between the fibers surface and the polymeric matrix.

The evaluation of the thermal properties of the composites shows the decomposition temperature of composites increase from 229 °C (untreated composites) to 231.3 °C and 228.1°C (acetylated and alkali composites, respectively), with fibers loading of 30 wt%. Water uptake test shows increase the absorption with the fiber content and the immersion time. The acetylated composites show the lowest rate of water absorption.

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NOMENCLATURE

PVC	Polyvinyl chloride
DPF	Date palm fibers
PVC/UPF	Polyvinyl chloride composite with untreated fibers
PVC/APF	Polyvinyl chloride composite with acetylated fibers
PVC/NPF	Polyvinyl chloride composite with acetylated fibers