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Effect of Advanced Chemical Treatments on the Tensile and Bending Properties of Date Palm Composites

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ABSTRACT

The contemporary focus on lignocellulosic waste valorization has triggered a transformative shift towards the utilization of biocomposite materials. This study endeavours to determine the impact of advanced chemical treatments on the properties (mechanical and physical) of composites based on date palm fibres (DPF). The investigation involves the application of soda (NaOH) and silane coupling agent (SCA) treatments, followed by IR spectroscopy analysis. The experimental framework includes the incorporation of 5 and 25% of treated and untreated fibres into polyvinyl chloride (PVC) composites, which are subsequently subjected to both tensile and 3-point bending deformations. Notably, the findings reveal a substantial improvement in mechanical properties when comparing Young's modulus of untreated fibres with those treated with SCA and NaOH with 7.2%, 75.37% and 37.04% in 5% filler content to the neat matrix. Similarly, in 25% filler content FTS composites showed 156.77%, 141.17% and 96.66% improvement respectively, similar results were found in bending modulus. This outcome confirms the potential of incorporating chemically treated date palm fibres to enhance the mechanical performance of composite materials. The advancements achieved through SCA and NaOH treatments present opportunities for the development of biocomposites with superior mechanical attributes. Such enhanced materials hold promise for a myriad of industrial applications, marking a significant stride towards sustainable and innovative solutions in the realm of lignocellulosic waste valorisation.

1. INTRODUCTION

Composite materials are emerging as a compelling alternative to traditional metallic materials, owing to their favourable characteristics such as chemical resistance, fatigue resilience, and adaptability. These materials give designers a great deal of versatility since they allow them to optimize both the strength and stiffness for each specific application.

Natural fibres have gained increasing attention as reinforcements for composite materials, supplanting traditional choices such as aramid (AF), carbon (CF), and glass (GF) are the most extensively used. However, there has been a significant push in recent decades to reduce their use in favour of natural fibres. Growing environmental concerns drive this shift, as natural fibres offer abundant, renewable, and biodegradable alternatives with low processing energy [1-3]. In addition, they are adequately durable, lightweight, and nonabrasive [4-6]. Compared to synthetic fibres, their specific weight is approximately half that of GF, and their tensile modulus is comparable to AF [4, 5, 7]. In this context, it is not surprising to see numerous studies using hemp [8-12], cotton [13, 14], pineapple leaf [15-19], ramie [20-22], bamboo [23-25], sisal [26-31], jute [32, 33] and date palm fibres [34-46].

In terms of matrices, enormous environmental pressure exists to select thermoplastic resins due to their recyclable nature. However, their economic and technical advantages, such as simple production, very long shelf life, high tensile strength, and enhanced moisture resistance, improve their appeal [47]. Combining natural reinforcements and thermoplastic matrices led to wood plastic composites (WPC) over the past four decades. There are many types of WPC on the market, made from polyvinyl chloride (PVC), polyethene (PE), polystyrene (PS), polypropylene (PP), polylactic acid (PLA), and acrylonitrile-butadiene-styrene (ABS). They can be used for both non-structural and structural purposes. PVC has seen the most rapid growth. However, in emerging nations,

wood resources are decreasing, but forest wastes and other types of natural residues can provide new sources to substitute wood products.

In a sustainable setting, the mechanical properties of wood plastic composites (WPC) depend a lot on the presence of chemicals that are not made from cells, like lignin, waxes, pectin, hemicellulose, and others. These components exert a notable influence on the interfacial adhesion matrix and reinforcement. Therefore, achieving improved interfacial resistance becomes imperative, which relies on achieving compatibility between the matrix and reinforcement, as well as optimizing the surface characteristics of the reinforcement material. Various surface treatment methods are available in the current literature to address this requirement, encompassing mechanical coupling, molecular entanglement, and chemical and electrostatic bonding. Among these, chemical treatments utilizing substances like alkali and silane offer a simple and cost-effective approach to modifying the chemical composition, surface structure, and overall composition of natural fibres. These treatments effectively remove non-cellulosic chemicals, enhance phase compatibility, and contribute to augmenting fibre surface roughness and thermal stability [48].

Benchouia et al. [49] explored the potential of date palm petiole fibers (DPP), expanded polystyrene waste (EPS), or their combination as aggregates to develop lightweight gypsum plaster hybrid bio-composites with enhanced mechanical and thermophysical properties for construction applications. Experimental formulations with varying DPP content from 0 to 15%wt with or without EPS were analyzed. Results indicate that while the addition of EPS or DPP adversely affects mechanical properties, it improves thermal insulation, reducing thermal conductivity and bulk density compared to neat gypsum plaster.

Moreover, Benchouia et al. [50] study focuses on developing polystyrene/date palm fibre composites (PS-DPF). Different fibre treatments were evaluated in varying concentrations (untreated, alkalinization, benzoylation). The composites exhibited improved mechanical properties with tensile strength ranging from 14 to 27 MPa and flexural strength from 31 to 44 MPa, along with satisfactory thermal stability and low thermal conductivity (0.118-0.141 W/m.K). The incorporation of fibres reduced bulk density and showed promise in thermal insulation, potentially reducing thermal conductivity by up to 50%.

Another study of Malti et al. [51] focused on reducing environmental impact by utilizing date palm leaflets (DPL) and expanded polystyrene waste to manufacture composite panels via hot compression process. Three composites were examined with varying weight fractions of DPL (70-80 wt.%) and expanded polystyrene (20-30 wt.%). Physical and mechanical tests revealed low,panel density (414-511 Kg/m^3) and improved maximum stress and flexural modulus up to 6.18 MPa and 13.5 MPa respectively, comparable to medium density fibreboard (MDF) and Durian peel and coconut coir (DPCC) perticleboards. However, the panels exhibited high water absorption (77-95%), limiting their use in moist conditions.

The study of Masri et al. [52] used a various combinations of reinforcement sizes and fiber-to-matrix weight ratios were tested to evaluate the properties of the leaflets polystyrene composites (LPC). Physical, mechanical, thermal and morphological analyses were conducted, including bulk density measurement, flexural modulus testing, maximum stress evaluation, thermal conductivity assessment, and SEM for fiber-matrix interface observation. The LPC exhibited good adhesion at the interface, acceptable mechanical properties (flexural modulus up to 0.78 GPa, maximum stress up to 2.84 MPa), and a density comparable to typical materials like wood and other WPCs. Thermal characterization revealed an average thermal conductivity suitable for thermal insulation applications (0.11-0.16 W/m.K).

The work of Eslami-Farsani [53] examines the impact of incorporating date palm fibres (DPF) and treating them with sodium hydroxide on the mechanical properties of polypropylene (PP) and EPDM polymer composites. Maleic anhydride grafted polypropylene (MAPP) is used as a compatibilizer to enhance the compatibility of DPF-filled PP/EPDM composites. Results demonstrate that adding fibres enhances tensile and bending strength but reduces impact strength. Moreover, composites with treated fibres exhibit superior mechanical properties compared to those with untreated fibres. SEM analysis of fracture surfaces indicates that MAPP and treated fibres enhance the interaction between the fibre and matrix components.

Similarly, the work of Chihaoui et al. [54] assesses the viability of using date palm wastes as reinforcement in PP composites. Fibres were extracted using mechanical defibration and subjected to three treatments: mechanical, chemical (NaOH), and enzymatic (xylanases and pectinases). Characterization involved chemical composition, morphology, and SEM analysis of the fibres. PP composites reinforced with date palm fibres were evaluated for stifness at macro and micro mechanical levels. Incorporating 40 and 60 wt% of enzymaticalyy treated date palm fibres enhanced PP's Young's modulus by 205% and 308% respectively. These treated fibres showed potential to replace glass fibres in composites, exhibiting similar stiffening effects. The intrinsic Young's modulus of fibres ranged from 16 to 24 GPa based on treatment method. Micromechanical analysis assessed the reinforcement efficiency, considering fibre length and orientation's contribution to the composite's Young's modulus.

To sum up, the review paper of Awad et al. [55] revealed that the incorporation of DPF in polymeric matrices enhance the mechanical and physical properties up to 60% compared to other natural fibres.

By the next few pargraphs, we shift the focus to examine the literature of date palm fibres reinforcement within PVC matrices which is the specific core of this work.

Armroune et al. [56] conducted a study on the treatment of date palm fibres with NaOH, resulting in a remarkable 178% increase in ultimate stress and a 167% increase in tensile modulus compared to untreated fibres. Furthermore, this treatment led to a reduction in the quantity of O-H bonds, consequently enhancing interfacial strength due to the reduced hydrophilicity of the fibres in comparison to untreated ones. Mohanty et al. [57] did a different study and found that treating date palm leaflets with acrylic acid increased their crystallinity index and tensile strength by 116% and 25%, respectively. Abdal-Hay et al. [58] investigated the effects of alkali treatment on the tensile characteristics of date palm fibres, revealing a 57% increase in ultimate tensile strength with the use of a NaOH solution. More recently, Bezazi et al. [59] delved into the impact of alkali treatment on the tensile response of date palm fibres (specifically rachis), highlighting the immersion duration and NaOH concentration as crucial parameters for optimizing properties. The authors identified

the optimal treatment condition as 3% NaOH for 8 hours, resulting in a remarkable 95% increase in tensile strength compared to untreated fibres. This improvement was attributed to the cleansing effect of alkali treatment, which removed a significant proportion of various compounds (including hemicelluloses, lignin, and wax), along with impurities, thereby facilitating the formation of pores on the fibre surface, particularly related to the removal of silica. Moreover, the higher crystallinity index (Cr.I.), the crystallite size (CS), and the degree of crystallinity (Xc) underscored the significant influence of crystallinity on the tensile properties of the fibres. Additionally, the authors noted a substantial enhancement in the thermal resistance of the fibres following alkali treatment [59].

Regarding composites, Shanmugam and Thiruchitrambalam [60] explored the impact of alkali treatment on unidirectional continuous Palmyra Palm Leaf Stalk Fiber/jute hybrid polyester composites. Their findings revealed enhanced storage and loss modulus, accompanied by a shift in Tan δ peaks towards higher temperatures with reduced peak height. Notably, the superior mechanical properties of these composites were attributed to the improved interfacial bonding facilitated by the alkali treatment. Similarly, studies conducted by Govardhan and Rao [61] on unidirectional Roystonea regia fiber with epoxy composites, and by Bachtiar et al. [62] on sugar palm fiber-reinforced epoxy composites, also demonstrated favorable outcomes for the mechanical performance of their composites following alkali treatment.

Ali et al. [63] discovered that treating Date Stones (DSs) with acetic anhydride chemically before heating them made the interactions between the DSs and PVC stronger, which improved the filler distribution in the matrix. However, this treatment resulted in a proportional decrease in material ductility (elongation at break) with increasing filler content, as well as a similar trend in strength at break. Nonetheless, incorporating heat and chemically treated fibres into PVC showed slight improvements in these properties.

Boussehel et al. [64] investigated the impact of chemical modifications on the final properties of PVC/palm fibre composites. While the addition of palm fibres improved the composites' mechanical properties, it also increased their water absorption. The treatments that were looked at, sodium hydroxide and sodium chlorite, were the most effective at improving the adhesion fibres and matrix.

Maou et al. [65] examined the shape, strength, and temperature of PVC/LDPE (low-density polyethylene) composites made stronger with DPF (leaf). They observed that the addition of fibres increased the mechanical properties, with the optimum performance achieved at a 30% fibre content. Furthermore, fibre addition contributed to the improved thermal stability of the composites.

Awad et al. [66] investigated the influence of DPF geometry on the properties of recycled PVC composites. Their findings revealed that the incorporation of fibres enhanced the composites' mechanical capabilities, with smaller-diameter fibres yielding the best results. Additionally, composites containing smaller fibres exhibited greater thermal stability.

Maou et al. [67] explored various chemical treatments for DPF and their impact on the properties of PVC/HDPE composites. Their study revealed that different treatments exerted diverse effects on the properties of the composites, with some enhancing mechanical capabilities while others improving thermal stability. Notably, maleic anhydride and peroxide treatments emerged as the most effective in enhancing both the thermal and mechanical properties of the composites.

In summary, the literature suggests that incorporating DPF into PVC composites can enhance their mechanical and thermal properties, albeit potentially increasing water absorption. However, the application-specific requirements dictate the choice of the most suitable chemical treatment for optimizing the final properties of the composites.

The objective of this study is to investigate the tensile and bending behaviour of PVC composites reinforced with date palm leaf powder. To the best of the authors' knowledge, no prior research has been conducted on the mechanical behaviour of PVC composites reinforced with this form of reinforcement. This research is particularly relevant in the current context due to the widespread use of PVC [68], coupled with Algeria's abundance of over 20 million palm trees [2]. Being the fourth largest producer of dates globally, Algeria generates more than 800,000 tons of date palm waste annually [69, 70]. In light of this socio-economic context, this study aims to evaluate the flexural and tensile properties of PVC composites reinforced with date palm fibre powder, with the goal of exploring industrial applications for this bio-reinforcement. To achieve this objective, two cost-effective and efficient treatments, namely mercerization and silane coupling agent treatment, were selected for investigation.

2. METHODOLOGY AND EQUIPEMENTS

The polyvinyl chloride (PVC) used in this research was provided by the ENICAB society in Biskra-Algeria. This company frequently uses PVC as an insulation material in the outer sheath of electrical cables.

Figure1. Preparation protocol of date palm leaf powder

Figure 2. RETSH AS 200 vibratory sieve shaker

Date palm leaflets (Phoenix dactylifera L) were collected in the Biskra region of Algeria and subjected to several treatments before being used as a filler. The leaflets were first cut between 1 and 2 cm before being washed twice, first in cold distilled water and then in hot distilled water, to remove any remaining impurities. Cold water, maintained at temperatures ranging from 10 to 20°C, was utilized to eliminate loose dirt and debris, whereas hot water washes, carried out at temperatures around 50°C, served to eliminate

bacteria and other microorganisms (Figure 1). Subsequently, the washed leaflets were air-dried and then underwent crushing and sieving processes using BOMANN KSW 6504 CB crusher into a fine powder between 50 and 100 µm using RETSH AS 200 vibratory sieve shaker with 3 mm/g amplitude and 99 second time interval (Figure 2).

2.1 Chemical treatments

Two types of treatments were applied:

First, a mixture of 2% wt. NaOH was prepared and mixed with 100 g of leaflet powder for 1 h. Then, the powder was rinsed with distilled water (DW) with 0.01 mol/L of acetic acid. Finally, a simple rinsing using distilled water alone was done. The obtained powder was named (FTA).

The second treatment used [3-(Methacryloyloxy) propyl] trimethoxysilane (Silane A174), also known as Silane a174. To avoid polymerization of the silane into polysiloxane, 1% wt. Silane A174 was dissolved in distilled water, and then acetic acid was added to reach pH = 2.9-4. After 15 min of silane hydrolysis, the NaOH treated fibres were placed in this mixture for 1 h at ambient temperature. Finally, the powder was rinsed with DW until pH = 7. The obtained powder was named (FTS). For both treated powders a drying step (80°C for 24 h) was applied to produce the final samples.

2.2 Composites elaboration

Table 1 illustrates the different formulas. Carefully prepared components were placed in an oven set to 80°C for one hour after being weighed out. The preparation of composites was carried out using the two-roll mill calendar manufactured by SCHWABENTHAN, specifically the "polymix 200 P" model. The optimal processing temperature for PVC was determined to be 160°C-165°C. Mixing was conducted for a duration of 10 min using a spatula until polymer fusion was achieved, followed by the subsequent incorporation of the fibre.

The composites were finally molded with a Schwabenthan Polystat 300S hydraulic press at 160-165°C and 300 bars for 10 min. A preheating step was carried out to avoid air bubbles and to degas before the final pressure was applied. In this case, 2 mm thick plates were obtained and then cut into tensile and bending specimens according to ISO 527-4 and ASTM D790, respectively.

Table 1. Composites formulation (wt %)

	PVC	FNT	FTA	FTS
PVC	100	-	-	-
PVC/FNT	95	5	-	-
	75	25	-	-
PVC/FTA	95	-	5	-
	75	-	25	-
PVC/FTS	95	-	-	5
	75	-	-	25

2.3 Fourier transform infrared spectroscopy analysis

A SHIMADZU FTIR-8400S was used and the samples were made into small 0.2 g of potassium bromide (KBr) pellets containing 0.001 g of either treated or untreated powder with a scanning range between 400 and 4000 cm^{-1} with a resolution of 2 cm^{-1}.

The degree of the C-H stretching vibration at -2900 cm^{-1} served as an indicator of the fiber's overall organic material content. To further characterize the fibers, ratios were computed based on the intensities of bands at 2900, 1595, and 1105 cm^{-1}, representing overall organic content, lignin content, and cellulose content, respectively. Employing two ratios instead of a single one was deemed more dependable in discerning between various fiber types.

The following ratios were then calculated:

$$R_1 = I_{1595}/I_{1105}; R_2 = I_{1595}/I_{2900}$$

where, R_1 represents the lignin content relative to the overall organic material, while the ratio R_2 represents the lignin content relative to cellulose.

2.4 Tensile test

The tensile test involves subjecting a "H2" sample to the jaws of a dynamometer. The mechanical properties of each sample up to rupture were measured following ISO 527 on a tensile machine Zwick/Roell Z050. The deformation rate was maintained at 250 mm/min. Three samples were tested for each variety.

2.5 Bending test

Flexural testing was performed on an INSTRON 5969 under the ASTM D790 standard. Since the obtained plates are of 2 mm thickness, the specimen were cut into 127 mm × 12.7 mm × 2 mm samples. Three samples were tested for each variety.

3. RESULTS

3.1 FTIR spectroscopy

The infrared spectra of untreated leaflets powder (FNT), soda (FTA), and silane coupling agent (FTS) treated presented in Figure 3. The peak at 3406 cm^{-1} is related to all the hydroxyls (primary and secondary), which have a slight decrease in intensity after modification. This is explained by converting some primary alcohols into a carboxylic acid. The peak at 709 cm^{-1}, which indicates the group's deformation (-OH), further supports the modification process. The bands centered around 2920 and 2854 cm^{-1}, which reflect the symmetrical and asymmetrical elongation vibrations of the C-H bonds of the -CH_2 group of the cellulose and lignin segments, are also decreasing. This reduction suggests alterations in the molecular structure of the fibers following treatment. The peaks corresponding to the C=O carbonyl groups of lignin (1735, 1238 cm^{-1}) were no longer observed after alkaline treatment (FTA). This is explained by the partial hydrolysis of hemicellulose in an alkaline medium, characterized by the rupture of C-O-C bonds between two monomers. Peak intensity at 1509 cm^{-1} confirms the decrease of C=C groups. Although some lignin peaks persist in the treated fiber spectrum, indicating the presence of residual lignin, mercerization effectively removes waxy epidermal tissue, sticky pectin, and hemicellulose binding fiber bundles. Eq. (1) describes the reaction between cellulose and soda:

$$\text{Cellulose-OH} + \text{NaOH} \rightarrow \text{Cellulose-O}^- \text{Na}^+ + \text{H}_2\text{O} + \text{impurities} \quad (1)$$

Regarding the spectrum of leaflet powder treated with the silane coupling agent (FTS), similar peaks as observed for FTA are recorded. However, new groups attributed to Si-groups (O-cellulose or Si-O-Si) appear in the region between 1050 and 1100 cm^{-1}. This confirms the substitution of OH groups of hemicelluloses by silanol groups via silane hydrolysis and condensation reactions. The signature of the silane grafting reaction on the powder indicates that intermolecular condensation occurred between adjacent silanol groups. The peak at 807 cm^{-1} represents Si-O from the reaction between the silane and the powder. All the results are in agreement with Abdelmouleh et al. [71]. The silanization reaction of date palm fibres with silane as a modifying agent was confirmed by the FTIR spectroscopic analysis and the reaction mechanisms are presented in Figure 4.

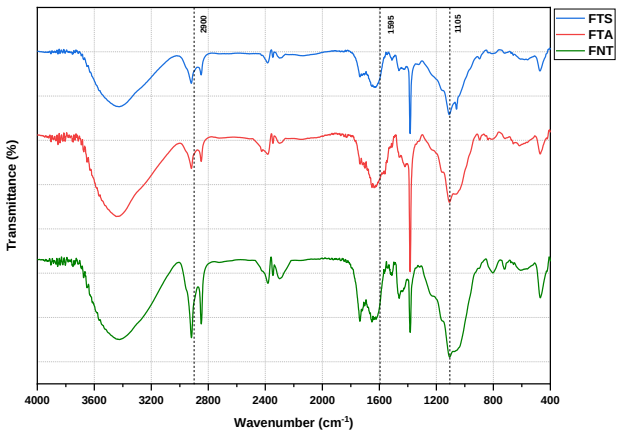

Figure 3. FTIR spectra of palm fibers untreated (FNT), and treated with: sodium hydroxide (FTA), silane coupling agent (FTS)

Table 2 represents the average values of the band intensity ratios $R_1(I_{1595}/ I_{1105})$ and $R_2(I_{1595}/ I_{2900})$. The R_1 and R_2 ratios found for untreated fibers (FNT), fibers that had been treated with NaOH (FTA), and fibers that had been treated with silane (FTS) show that the chemicals in the fibers changed after treatment. Untreated fibers exhibited relatively high R_1 and R_2 ratios (R_1=2.146, R_2=0.975), suggesting significant lignin content compared to overall organic material and cellulose, respectively. The R_2 ratio, which compares the lignin content to cellulose, another major component of plant fibers, is also elevated, suggesting that lignin makes up a significant portion of the fiber's composition compared to cellulose. After NaOH treatment, both R_1 and R_2 ratios decreased, indicating a reduction in lignin content, likely due to lignin removal during treatment. Since lignin is more soluble in NaOH than cellulose, the treatment selectively removes lignin, leading to a decrease in its proportion relative to both the overall organic material and cellulose in the fibers. This process is important because lignin can hinder the adhesion between fibers and polymer matrices in composite materials, so reducing its content can improve the overall performance of the fibers in composite applications.

On the other hand, silane treatment caused small increases in both ratios. Silane treatments involve applying a coupling agent to the fiber surface to enhance adhesion between the fibers and polymer matrices in composites. While the silane treatment itself does not directly affect lignin content, the increase in ratios may indicate interactions between the silane molecules and other components of the fiber, such as hemicellulose or extractives. These interactions could modify the fiber's surface chemistry, leading to a slight increase in the apparent lignin content relative to both the overall organic material and cellulose.

Table 2. Average values of the band intensity ratios $R_1(I_{1595}/ I_{1105})$ and $R_2(I_{1595}/ I_{2900})$ for utreated and trated palm fiber

	$R_1(I_{1595}/ I_{1105})$	$R_2(I_{1595}/ I_{2900})$
FNT	2.146	0.975
FTA	1.513	0.473
FTS	2.265	0.777

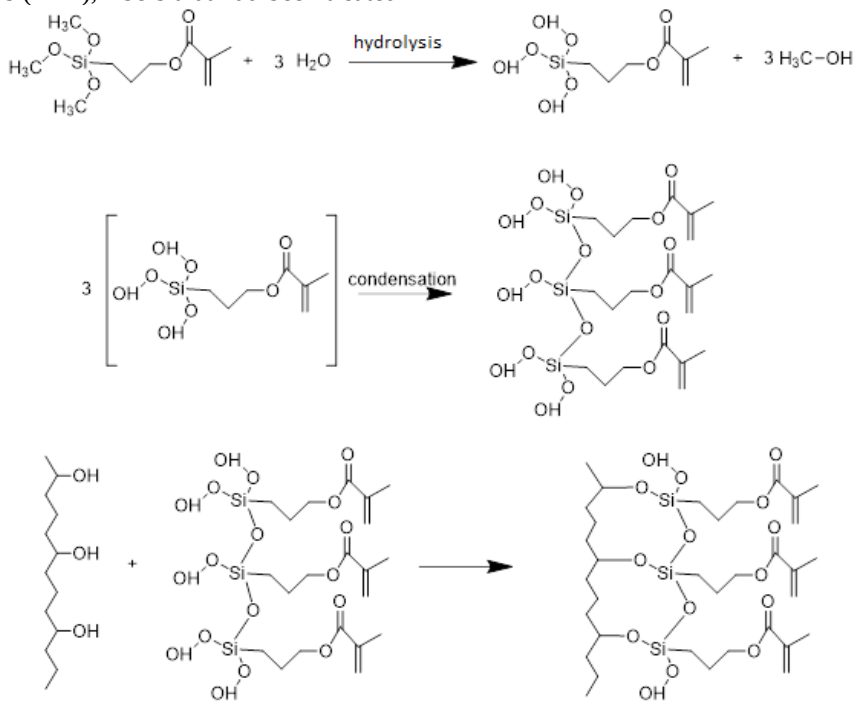

Figure 4. Reactional mechanism between date palm leaflet powder and silane (3-trimethoxysilyl) propyl methacrylate

3.2 Tensile test

3.2.1 Young's modulus

Figure 5 depicts the progression of Young's modulus of PVC composites with various filler treatments. Generally, Young's modulus rises with filler content, indicating that the addition of fillers to the PVC matrix (86.1 MPa) enhances the rigidity of the composites. This trend is attributed to the inherent rigidity of the dispersed filler, which is imparted to the final composites. The findings also reveal that surface treatments yield superior results compared to untreated particles. Specifically, PVC with 5% FTS exhibits the highest Young's modulus at 151 MPa, followed by PVC with 5% FTA at 118 MPa and PVC with 5% FNT at 92.3 MPa. Similarly, at a higher filler content of 25%, PVC with FTS outperforms PVC with FTA and PVC with FNT by 221.08 MPa, 208.13 MPa, and 169.33 MPa, respectively. Mercerization consistently yields better results, but silane treatment yields the best outcomes.

Quantitatively, the overall enhancement of Young's modulus is notable. In the case of 5% filler content, FTS composites exhibit enhancements of 75.37%, 37.04%, and 7.2% compared to the neat matrix, FNT, and FTA composites, respectively. Similarly, for 25% filler content, FTS composites show enhancements of 156.77%, 141.17%, and 96.66% compared to the neat matrix, FNT, and FTA composites. This increase is primarily attributed to improved fibre/matrix interfacial adhesion resulting from the excellent dispersion of the treated fibre in the PVC matrix.

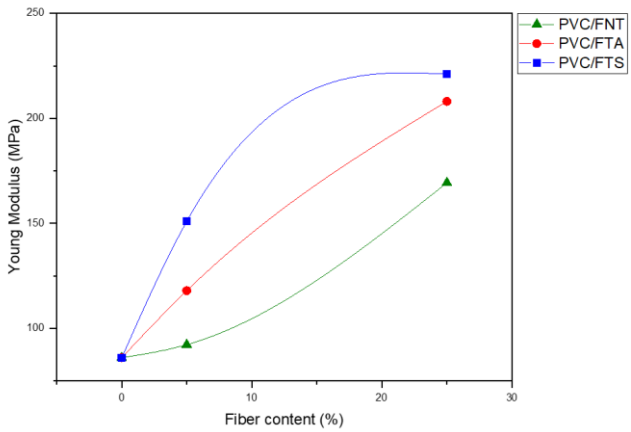

Figure 5. Young's modulus of neat PVC and composites

The treatment process leads to the formation of chemical bonds between the hydrophobic segment of the silane and the matrix surface, as well as between the hydrophilic segment and the fibre surface. This enhanced interfacial adhesion contributes significantly to the observed increase in Young's modulus of the composites.

3.2.2 Tensile strength

Figure 6 shows the evolution of the stress at fracture of the composites produced as a function of filler content. A noticeable decrease in the fracture stress for the composites is observed compared to the neat matrix (PVC) which have 13.4 MPa. However, better results are observed for PVC/FTA composites compared to PVC/FNT, at 5% filler content, PVC/FTA reached 10.1 MPa and PVC/FNT reached 9.66 MPa that means: 24.62% and 27.91% decrease compared to neat matrix. Similary, at 25% filler content, PVC/FTA outperforms PVC/FNT with 9.14 MPa against 8.58 MPa, which means 31.79% and 35.97% decrease, respectively. Because of the hydrophobic nature of the PVC matrix and the hydrophilic nature of the filler (FNT), the binding force between the fibres and the matrix is originally weak and the fibres tend to agglomerates inducing heterogeneities and non-uniform stress transfer within the matrix, leading to embrittlement and a reduction in the composite's strength.

PVC/FTS composites had the best results in 5% and 25% filler content, the results revealed 10.6 MPa and 9.21 MPa, respectively. This means 20.89% and 31.26% decrease compared to neat matrix. This is why the treatments improve the stress at the fracture of composites (PVC/FTA and PVC/FTS) compared to untreated fibres (PVC/FNT). Better adhesion between the treated fibres and the polymeric matrix is achieved.

It must be pointed out that the alkaline treatment removes impurities and parietal constituents such as lignins, pectins and waxy substances located on the fibres surface cell walls. This promotes the formation of fibrils leading to a rougher fibre surface to facilitate physico-chemical interactions at the filler-polymer interface. Figure 6 also shows that the samples treated with the silane coupling agent (PVC/FTS) have better tensile strength than mercerized ones (PVC/FTA). This can be explained by silane forming stronger interfacial bonding between the PVC and fibres. Thus, improved stress transfer between both phases is achieved compared to alkaline treatment.

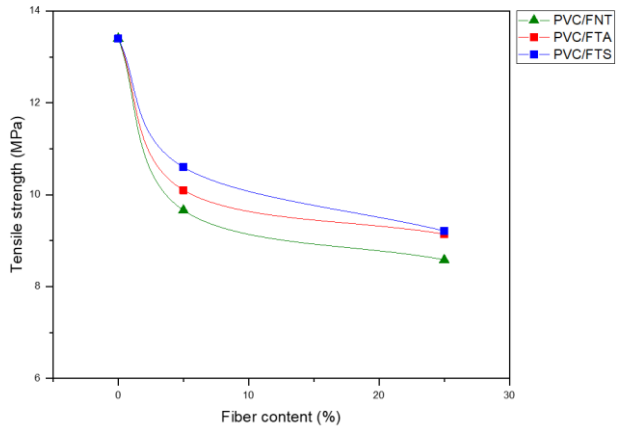

Figure 6. Tensile strength of neat PVC and composites

3.2.3 Elongation at break

The evolution of the elongation at fracture of the treated and untreated composites is represented in Figure 7. There is a significant decrease in the values for the composites made with untreated fibres to fall from 179.6% of the neat matrix to 100.3% and 31.2% for 5 and 25% filler content respectively. Quantitively, 44.15% and 82.62% decrease respectively. This decrease is explained by: i) the hydrophilic nature of untreated fibres that absorb more moisture and cause swelling in the PVC matrix, which causes the embrittlement of the material. ii) increasing volume of fibre creating defects in the system and reducing inter-chain interactions associated with fragile ductile variation in material behaviour.

Adding NaOH-treated fibres to significantly reduced the elongation at break as a function of the filler content. At 5% FTA in PVC, the result was 73.5%, which means 59.07% decrease and at 25% FTA in PVC the result was 34%, representing an 81.06% decrease. This is related to the

presence of lignin in the fibres, generating cracks and probable composite failure due to the time and concentration of soda used in the fibre treatment not having a major effect on lignin elimination. In addition, fibre agglomeration may have contributed to creating stress concentration zones requiring less energy for crack initiation and propagation.

Comparing the elongation at fracture of the different composites, it is clear that the silane coupling agent treatment produced a slight improvement with 105.5% and 70.46% for 5 and 25% filler content respectively, representing 30.33% and 51.74% improvement compared to 5 and 25% filler content FTA composites, respectively. Similarly compared to FNT composites, it can be noticed an improvement of 4.92% and 55.71%. However, comparing FTS to neat matrix it is shown a decrease of 41.25% and 60.76% respectively. This increase is mainly attributed to a better dispersion of the treated fibre, giving some flexibility to the material.

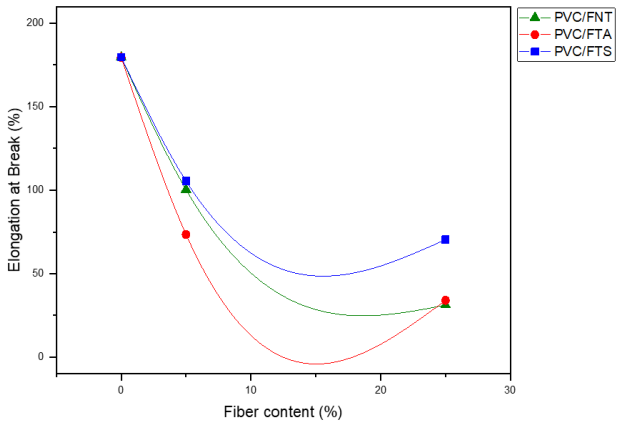

Figure 7. Elongation at break of neat PVC and composites

3.3 Bending test

3.3.1 Bending modulus

The bending modulus of the PVC composites is presented in Figure 8. It is evident that adding rigid fibres in the PVC matrix improved the composites bending modulus compared to the virgin PVC. Comparing the values shows that treated fibre with silane show the best properties. This is due improved interfacial adhesion compared to the other samples (FNT and FTA).

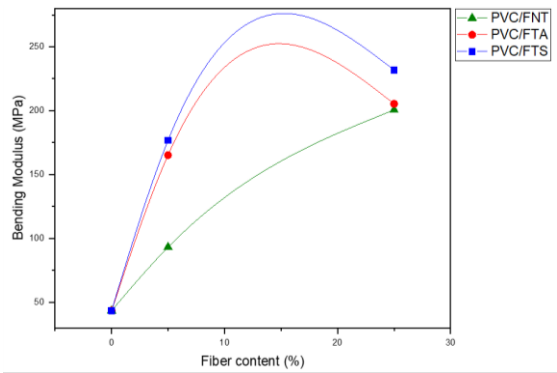

Figure 8. Bending modulus as a function of filler content and type of treatment

The variation of these properties can be explained based on changes in chemical interaction at the filler-matrix interface on the different treatments, as described in the section above.

3.3.2 Bending stress

The bending stress as a function filler concentration is illustrated in Figure 9. PVC reinforced with treated fibres have better bending strength than PVC composites based on untreated ones. Palm fibre treatments improve the filler/matrix interface, resulting in higher bending properties.

The lower bending resistance of untreated composites is due to hydroxyl and other polar groups' presence on the fibre surface leading to lower fibre-matrix interface quality. It should be noted that combining alkaline and silane modifications produces the best performance than each treatment taken alone. This is related to the alkaline treatment generating a network of H-bonds inside the cellulose structure which can be broken, but the cellulose hydroxyl groups are activated and thus improve the fibres hydrophilic character and their compatibility with silane agents. Table 3 and Table 4 summarize the mechanical parameters of all specimens evaluated in this work.

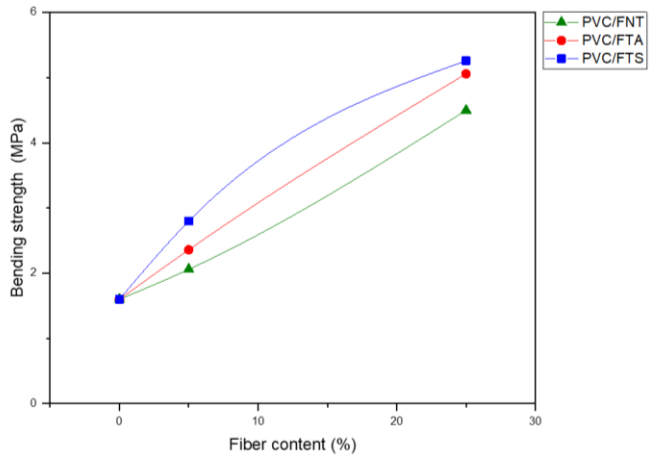

Figure 9. Bending strength as a function of filler content and type of treatment

Table 3. Tensile properties of treated and untreated palm fibers reinforced PVC composites

	Young's Modulus (MPa)	Tensile Strength (MPa)	Elongation at Break (%)
PVC	86.1	13.4	179.6
PVC/FNT$_5$	92.3	9.66	100.3
PVC/FNT$_{25}$	169.33	8.58	31.2
PVC/FTA$_5$	118	10.1	73.5
PVC/FTA$_{25}$	208.13	9.14	34
PVC/FTS$_5$	151	10.6	105.5
PVC/FTA$_{25}$	221.08	9.21	70.46

Table 4. Bending properties of treated and untreated palm fibers reinforced PVC composites

	Bending Modulus (MPa)	Bending Strength (MPa)
PVC	43.49	1.6
PVC/FNT$_5$	93.14	2.06
PVC/FNT$_{25}$	200.6	4.5
PVC/FTA$_5$	165.23	2.36
PVC/FTA$_{25}$	205.36	5.06
PVC/FTS$_5$	176.79	2.8
PVC/FTA$_{25}$	231.79	5.26

4. CONCLUSIONS

This study aimed at investigating the effect of applying a chemical treatment (mercerization and silane coupling agent) on date palm fibres to improve their properties when introduced into PVC. The samples were produced via calendering and compression molding with a range of filler content (0-25% wt.). The resulting composites were then characterized by their mechanical properties (tension and flexion).

A FTIR analysis firstly confirmed the surface modifications achieved on the surface of the date palm fibres via changes in peaks intensity, especially O-H, C-H and Si-O groups.

Then, the mechanical properties were analyzed from the stress-strain plots. It was observed that increasing the filler concentration led to higher bending and tensile moduli, but lower elongation at break and tensile strength. Nevertheless, improvement was observed when surface treatment was applied in the following order for all properties: untreated < mercerized < silane. These changes can be associated with improved fibre-matrix interfacial adhesion, better filler dispersion and improved compatibility leading to better stress transfer. Nevertheless, more work is needed to further characterize these samples and improve their possibilities in terms of other fibre contents and processing methods.

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