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Polypropylene Composites Reinforced by Marine Posidonia Fiber Waste: Effect of Silane and Alkali treatment

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Abstract: This paper investigates the effects of chemical treatment and the reinforcement rate on the mechanical properties of polypropylene composites reinforced with Posidonia waste fibers. Increasing the reinforcement rate from 20% to 30% improved Young's modulus and the flexural modulus of the composite. Silane treatment had a positive effect on all the mechanical properties of the composite; on the other hand, the alkali treatment improved the tensile strength but decreased the flexural property of the composite. The greatest Young's and flexural moduli were obtained in the case of 30% reinforcement treated with silane. The increases in these properties were 57.95% and 44.84% for the tensile and flexural moduli, respectively, compared to pure polypropylene. The mechanical properties of the composite obtained were higher than those of hemp and jute produced under the same conditions using the single-screw extrusion process. The results show that Posidonia waste fiber is an effective candidate to be utilized to produce composites for the automotive industry, such as rear shelves, boot linings, spare wheel compartments, and interior doors, and that it has economic and ecologic advantages in comparison with hemp and jute fibers.

Keywords: Posidonia fiber waste; Polypropylene composite; Silane treatment, Alkali treatment.

1 Introduction

Natural fiber composites have gained increasing attention due to their eco-friendliness (biodegradability and recyclability), specific strength and modulus, low costs, and lightweight nature [1, 2, 3]. Natural fibers have the potential to replace conventional composites in the construction, packaging, medicine and automotive industries [4, 5, 6]. However, it is known that the weak link between fibers and polymer matrix is due to the hydrophilic characteristics of natural fibers [7, 8]. For that, chemical or physical treatments can be applied to natural fibers to improve interfacial adhesion, to make their properties more uniform, and to reduce their hydrophilic state [9, 10].

Alkali, silane, benzoylation, acetylation, permanganate, peroxide and isocyanate treatments are chemical processes often used to alter the properties of natural fibers [11]. These processes can significantly affect the physical and mechanical properties of the fibers [12]. Sodium hydroxide is widely used for cleaning and bleaching the surface of natural fibers. It is utilized to eliminate pectin, lignin, and other materials covering the fiber surface. Plant fibers treatment with NaOH changes their surface topography and their crystallographic structure. Hence, impurities elimination on the surface of plant fibers has a positive effect on fiber–matrix adhesion, as it helps both bonding and mechanical locking effects [13, 14]. Silane compounds have many functional groups, a feature that allows them to be used in many ways in comparison with other chemical treatments. This adaptability makes silanes useful in composite materials. Silanes are commercially available on a large scale [15, 16, 17].

Posidonia is a marine plant with flowers; it loses its leaves in autumn, forming pellets several centimeters thick. According to Dural work [18], this non-exploited biomass represents a huge quantity of matter (between 5 and 50 M t per year in the Mediterranean region). Posidonia should be cleaned each summer to avoid ecological problems. Exploring this fiber's potential uses in technical products serves both economic and ecological interests.

Researchers have demonstrated that Posidonia fibers can be utilized as an adsorbent to remove dyes and phenol [18, 19],

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as a reinforcement to material building [20, 21], to produce cellulosic derivates [22], and for the manufacturing of nonwoven fabrics [23]. Bicomposites reinforced with Posidonia fibers have also been studied. Khiari [24] analyzed the effect of the Posidonia ratio on the mechanical and thermal properties of a composite film produced from Posidonia fiber and a bio-matrix using an extrusion process. He demonstrated that the addition of Posidonia fibers increased all of the mechanical properties of the composite except for the elongation at breaking point.

Garcia [25] utilized a hot molding process technique to produce a composite from a bio epoxy resin with 70% Posidonia fiber as a reinforcement. By applying an alkali and then a silane treatment to fiber and matrix, he proved that adhesion could be enhanced, and the mechanical properties of composites improved.

Mirpoor [26] extracted resin and nanocrystalline cellulose from Posidonia and used them as a reinforcement to manufacture a biofilm composite produced from hemp protein. The results indicated an enhancement of the bioplastic film's barrier and mechanical properties when the reinforcement was added. From an environmental point of view, bio composites have many benefits, but their industrial applications remain highly restricted [27]. On the other hand, there still exists a market for composites produced from synthetic resins, such as polypropylene and polyester, reinforced with natural fibers, especially in the automotive and construction sectors [28, 29, 30]. Due to their special properties and low costs, Posidonia fibers can be an effective candidate for this kind of composite [31, 32]. An essential condition for their use is to employ the optimal ratio and optimize fiber and matrix interaction. In this study, we investigate the effect of chemical treatments and reinforcement rate on the tensile and flexural properties of a polypropylene composite reinforced with Posidonia waste.

2 Materials and Methods

2.1. Fiber and Composite Preparation

2.1.1. Fiber Preparation

We collected the Posidonia balls from a Tunisian beach. We manually opened the substance and placed it on a horizontal opener to separate the fibers and dust. Then we used centrifugal force and aspiration to remove the good fibers upward and waste fell down. Afterward, we obtained fibers shown in Figure 1 using this mechanical action.

Fig. 1: Basic Posidonia Fibers (FPB).

In order to pick up the adhesion fiber/resin, the surface of the raw Posidonia fibers was modified using two chemical treatments: **silane and alkali**. Two different treated kinds of fiber are then obtained:

- Posidonia Fibers with alkali or Combined Treatment FPTC
- Posidonia Fibers with Silane Treatment FPTS

For the alkali treatment (or the combined treatment), raw Posidonia fibers were treated using a Datacolor AHIBA MSTRI (DATACOLOR Italia, Piazza della Republica 2, 24122 Bergamo Italy). First, we prepared a bath containing a liquor ratio of 1 by 40. Then, we immersed 5 g of untreated Posidonia fibers in it. Next, we added two components: H_2O_2 (hydrogen peroxide) and NaOH (sodium hydroxide) with concentrations respectively of 25 mL/L and 20 g/L. Finally, under the effect of stirring and pressure at 100 °C for 45 min, the entire intermingling was heated. Afterward, the obtained fibers were washed with cold water for 48 h at 25 °C before drying for 48 h at 25 °C.

Silane treatment was performed using amino-silane as a coupling agent. The treatment bath contained 1% by weight of the silane-coupling agent and 0.05% initiator and it was used to transform alkoxysilane silane groups into silanol groups. The pH was set to 3.5 using acetic acid; otherwise, polysiloxanes could form during the reaction. The total blend was stirred continually for 10 min, then we engrossed fibers in the solution for 1 h and finally desiccated them at 60 °C during 24 h. At a later step, the characteristics of the fibers were investigated using different laboratory analyses, and then the treated (FPTC and FPTS) and untreated fibers (FPB) were characterized. To carry out the analyses, we have conditioned fibers in a normal atmosphere (relative humidity: 65% ± 4%, temperature: 20 °C ± 2 °C).

2.1.2. Composite Preparation

The thermoplastic polymer used as a matrix for making composite structures is homopolymer polypropylene (PP). The polymers used are in the form of pellets. These pellets are approximately 3 to 4 mm in diameter. We use a RETSCH SK100 crusher to flatten the fibers used as reinforcement. We then screen using a 200 µm-micron sieve. In this way, we obtained fibers with a length of 200 to 2000 µm. These fibers will be unfeasible. Then we use 30% as the maximum percentage of reinforcement. Therefore, we prepared a mixture consisting of two parts by mass (20% and 30%) by weighing. Before that, we previously conditioned the fibers at 105 °C. Thereafter we used a single screw extruder fed with the above blend. Depending on the temperature of the screw, extrusion takes place in three temperature ranges. These temperatures were then 190°C, 200°C and 210°C, respectively. A thermo-compression was used to form our models. The used molding press was composed of two parallel dishes with adjustable temperature and distance. Then, the distance between these two plates is adjusted so that the mold can be inserted.

2.2. Mechanical Analysis of Composites

The tensile and flexural behavior of the samples was analyzed using a dynamometer Instron, according to ISO 527 and ISO 14125, respectively. The tensile speed was adjusted to 0.8 mm/min and the sensor used has a power of 30 kN for the tensile test and 1 kN for the bending test. The specimens of the polypropylene/Posidonia composites designated for tensile and bending tests had the following dimensions (width × thickness× length): (8 mm × 3 mm × 120 mm) and (10 mm × 3 mm × 120 mm), respectively. The distance between the clamps for traction and the distance between supports for bending were both fixed to 60 mm^3.

2.2.1. Scanning Electron Microscopy

The surface topography and shrinking surface of the composites were examined using a Scanning Electron Microscopy (SEM, HITACHI TM 3000 Schaumburg, Illinois 60173, U.S.A). The partial vacuum conditions were in order of 0.1 to 0.15 torr and the accelerating voltage was varying from 10 to 15 KV. The specimens were metellized before being analyzed with SEM microscope.

2.2.2. Fourier Transform Infrared Spectroscopy of the Posidonia Fibers

Using a Perkin-Elmer FT-IR Frontier spectrometer, we have determined the FTIR spectra of the used fibers (tread and crude fibers). This spectrometer was associated with an ATR accessory. The ATR method consists of pressing the fiber sample against a crystal (diamond). As a result, the interaction between infrared ray and the sample is interfacial. The absorbance was deliberate over a range of 4000 to 400 cm^{-1}.

3 Results and Discussion

3.1. Effect of Fiber Treatment on the Tensile and Flexural Properties of the Composites

Table 1 shows the results of the tensile and bending tests of the polypropylene matrix reinforced with the mixture of treated and untreated Posidonia fibers for a reinforcement rate of 30%.

Table 1: Mechanical properties of the PP/Posidonia composites.

		Tensile Properties			**Flexural Properties**		
Composite Designation	**Fiber Weight Ratio (%)**	**E (GPa)**	**Σ (MPa)**	**ε (%)**	**E (MPa)**	**Σ (MPa)**	**ε (%)**
PP	0	0.88 ± 0.03	20.34 ± 0.93	10.5 ± 0.27	358.5 ± 15.4	36.68 ± 2.05	0.09 ± 0.01
Comp PP/FPB	30	1.34 ± 0.07	17.7 ± 0.66	2.84 ± 0.34	493.1 ± 24.8	33.7 ± 3.08	0.07 ± 0.01
Comp PP/FPTC	30	1.16 ± 0.04	15.6 ± 1.52	3.03 ± 0.49	433.1 ± 21.8	33.81 ± 2.24	0.06 ± 0.01
Comp PP/FPTS	30	1.39 ± 0.07	19.2 ± 0.56	2.25 ± 0.14	519.3 ± 19.1	39.25 ± 2.04	0.06 ± 0.01

According to this table, the addition of Posidonia fibers to the polypropylene matrix modified the tensile and flexural properties of the composite material. Indeed, regardless of the nature of the fibrous reinforcement (raw or treated) added, the modulus of elasticity increased. The rate of increase of the respective tensile and bending parameters compared to pure polypropylene was approximately 52.27% and 37.54% for the raw Posidonia fibers, 31.81% and 20.8% for the fibers treated with a combined chemical treatment (FPTC), and 57.95% and 44.84% for fibers treated with silane (FPTS). This confirms the finding demonstrated by other researchers in the field that the incorporation of a reinforcement compatible with the matrix contributes to an improvement in the mechanical performance of the composite [3].

We also observed that composites reinforced with silane-treated fibers had the highest modulus of elasticity. The two bands at 1166 cm^{-1} and 940 cm^{-1} represent Si-O-C and Si-OH bonds, respectively (Figure 2). This showed that the cellulose hydroxyl groups were blocked with silane. Consequently, the surface of the fiber became more hydrophobic. Additionally, Wang et al. [33] observed that silane treatment of bamboo-fiber-reinforced polypropylene composites enhanced their mechanical properties.

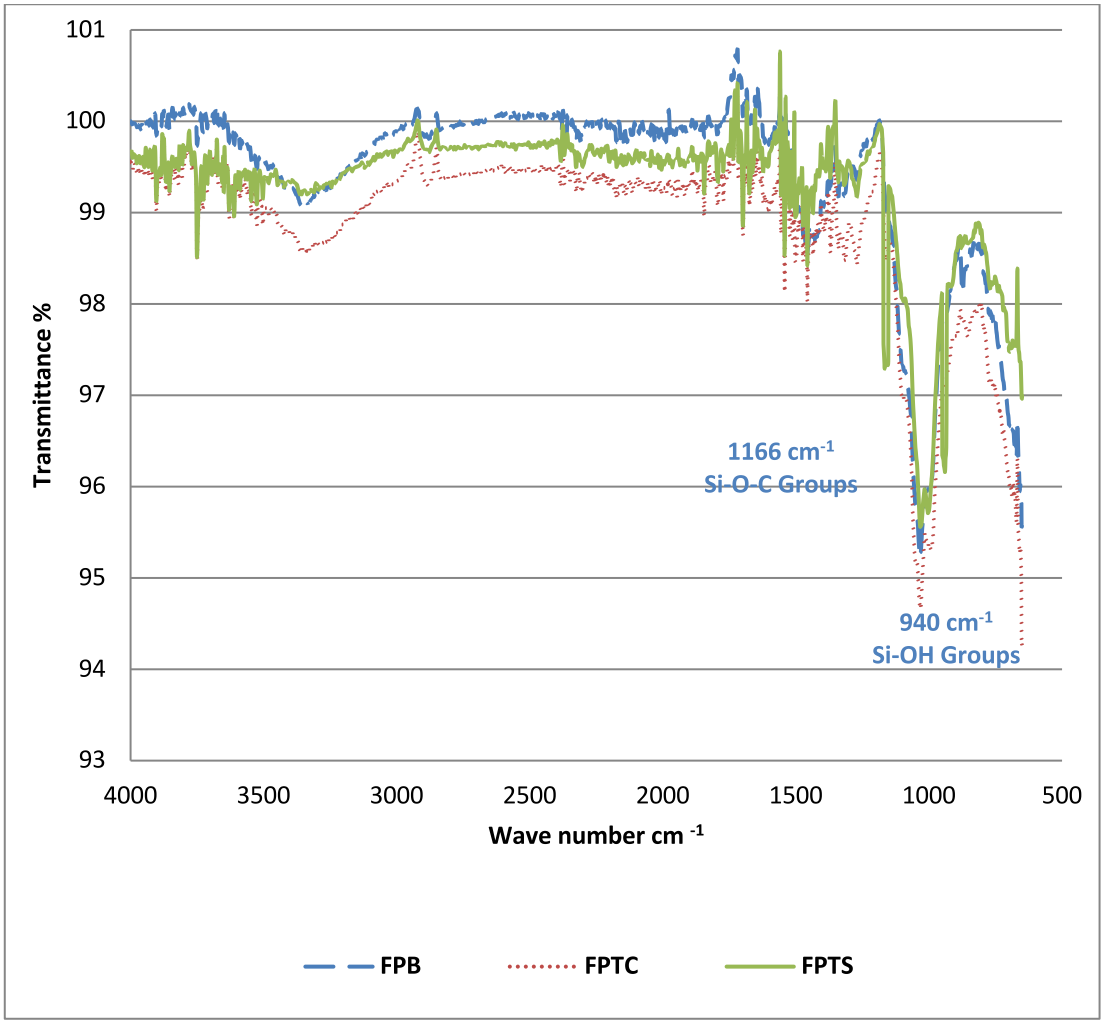


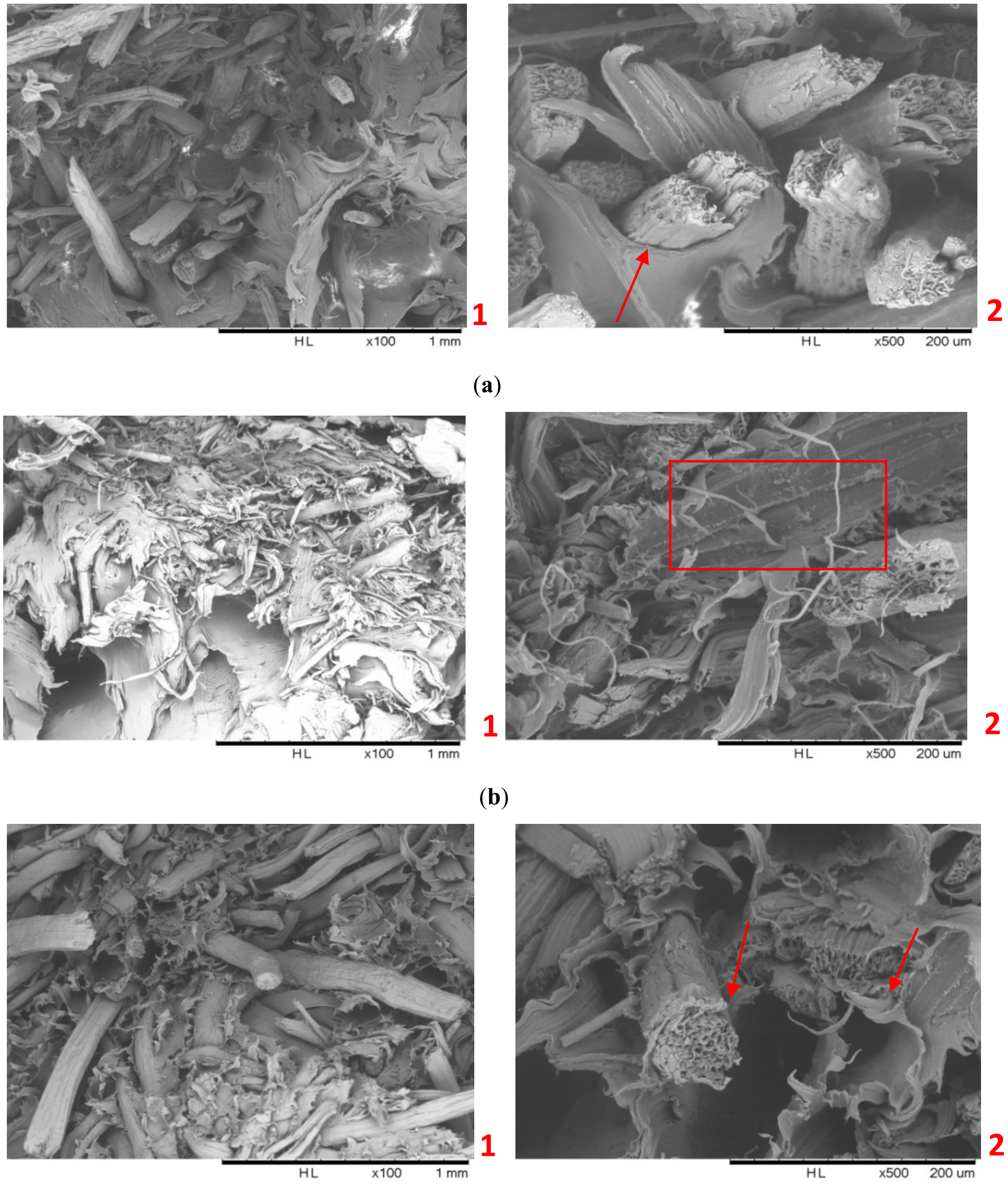
Fig. 2: FT-IR spectra of raw and treated Posidonia fibers.

3.2. Physical Structure

Figure 3 shows the SEM images taken at the fracture facies level of the tensile tests of the composite samples reinforced with treated and untreated Posidonia fibers.

These SEM photos clearly illustrate the modification of the mechanical properties of the composite materials reinforced by the treated and raw fibers. These figures reveal the presence of cracks between the fiber and the matrix. When the Posidonia fibers were treated with silane, this cracking was much less pronounced than that in the untreated fibers (Figure 3(a-2, c-2)). Hence, the fiber–matrix interface improved, which clearly confirms the improvement in the mechanical properties previously presented in Table 1. However, Figure 3(b-2) shows that the fibers (FPTC) were completely torn from the composite when stretched. This indicates the poor quality of the mechanical properties in this case compared to other fibers.

In addition, we observed that the addition of raw or treated reinforcement caused a decrease in the maximum stress at break and a decrease in the elongation at break under tension, which can be attributed to the reduction in the macromolecular chains on the specific surface of the material followed by the “stiffening” of the material. The one exception was found for tensile strength in bending, which was improved after the addition of fibers treated with silane (FPTS). This may be due to the improved adhesion between the fiber and the matrix after this treatment. Similar results were obtained by Chauhan et al. [34] when wood and palmyra fibers were treated with silane.



(**a**)

(**b**)

(**c**)

Fig. 3. Tensile fracture surfaces of composites; (**a**) comp. PP/FPB, (**b**) comp. PP/FPTC, (**c**) comp. PP/FPTS.

3.3. Effect of the Fiber Weight Ratio on the Tensile and Flexural Properties of the Composites

To better visualize the effect of the fiber content on the tensile and flexural mechanical properties of the thermoplastic composites reinforced with raw and treated Posidonia fibers, we plotted the histograms shown in Figure 4.

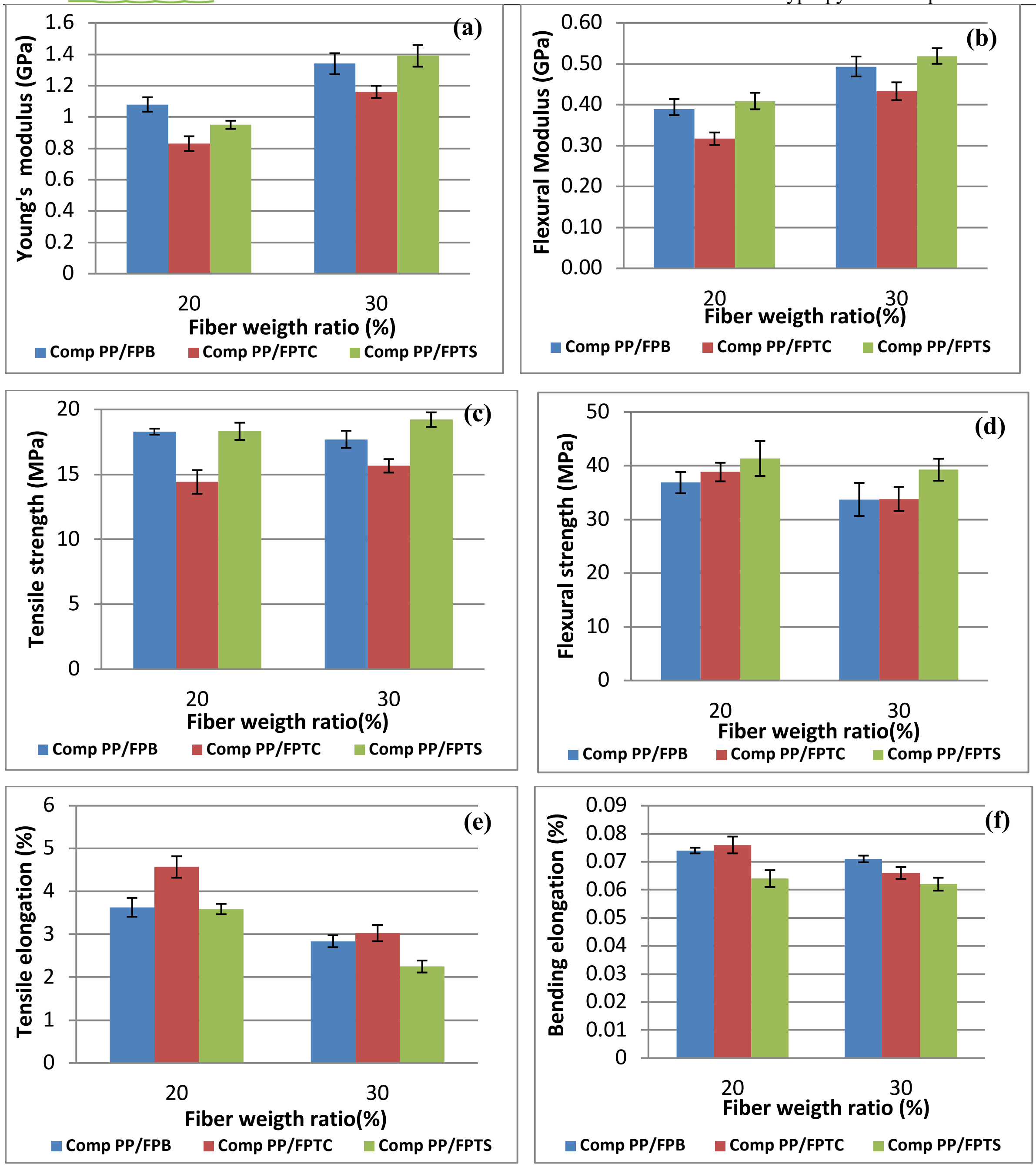


Fig. 4: Variations in the mechanical properties of PP/Posidonia composites as a function of the fiber content; (**a**) Young's modulus; (**b**) flexural modulus; (**c**) tensile strength; (**d**) flexural strength; (**e**) elongation tensile strength; (**f**) bending elongation.

The incorporation of raw reinforcements (20 and 30%) resulted in an increase in Young's modulus and the flexural modulus from 21% to 51% and from 8.76% to 37.54%, respectively, compared to the matrix alone. The more the fibrous reinforcement increased in the matrix, the more the contact surface between the fiber and the matrix increased, which led to greater cohesive energy between the two constituent elements while increasing the mechanical properties. Kaewkuk [35] also found that, with increasing sisal fiber content, the tensile strength and modulus of polypropylene composites increased.

For composites reinforced with treated fibers, increasing the reinforcement rate from 20% to 30% improved Young's modulus and the flexural modulus. The growth rate of the mechanical parameter was significant for a reinforcement rate of 30% of these fibers. There were increases of about 30% and 21% in the respective tensile and bending of the fibers subjected to a combined chemical treatment (FPTC), and of about 56% and 45% for those treated with silane (FPTS) compared to the pure matrix.

Furthermore, we observed that the addition of 20% raw and treated reinforcement improved the flexural strength of the composites compared to that of the matrix alone. Meanwhile, increasing this rate up to 30% for the raw fibers (FPB) and those treated using the combined chemical process (FPTC) decreased this property. The greatest flexural strength (41.34 MPa) was obtained when the fibers were treated with silane with a mass percentage of 20%. Concerning the elongation in bending, this characteristic decreased after the addition of the raw or treated fibrous reinforcement. This decrease was more pronounced in the case of tensile testing (a reduction rate of about 60% in tension against 25% in bending).

Moreover, these tests demonstrated that the addition of fibers decreased the deformation at breaking and the tensile strength of reinforced composites compared to the matrix alone. This decrease can be explained by the rigidity of the material following the reduction in the number of macromolecular chains in its specific surface.

Composites reinforced with hemp [36] and jute [37] fibers were generated under the same conditions as those in our case study (i.e., using a single-screw extrusion process). These composites had Young’s modulus values (1.013 GPa for hemp/polypropylene composites and 0.8 GPa for jute/polypropylene composites) that were lower than those of the composites reinforced with Posidonia fibers (1.16; 1.34; 1.39 GPa) studied in this work. These new thermoplastic composites (polypropylene/Posidonia) can therefore be used in the automotive field and in interior features such as rear shelves, boot linings, spare wheel compartments, interior doors, etc. [38].

4 Conclusions

This paper examined the effects of chemical treatment and the reinforcement rate on the mechanical properties of polypropylene composite reinforced with Posidonia waste fibers.

It was demonstrated that the increase in the reinforcement rate from 20% to 30% improved Young’s modulus and the flexural modulus of the composite. Silane treatment had a positive effect on all of the mechanical properties of the composite; on the other hand, the alkali treatment improved the tensile strength but decreased the flexural properties of the composite. The greatest Young’s and flexural moduli were obtained in the case of 30% reinforcement with silane treatment. Increases of 57.95% and 44.84% were observed for the tensile and flexural moduli, respectively, compared to pure polypropylene.

The mechanical properties of the obtained composite were higher than those of hemp and jute produced under the same conditions using the single-screw extrusion process. Our results prove that Posidonia waste fiber is an effective candidate to be utilized to produce composites for the automotive industry, for use in products such as rear shelves, boot linings, spare wheel compartments, and interior doors. Moreover, these composites have economic and ecologic advantages in comparison with hemp and jute fibers.

Conflicts of Interest Statement

The authors certify that they have NO affiliations with or involvement in any organization or entity with any financial interest (such as honoraria; educational grants; participation in speakers’ bureaus; membership, employment, consultancies, stock ownership, or other equity interest; and expert testimony or patent-licensing arrangements), or non-financial interest (such as personal or professional relationships, affiliations, knowledge or beliefs) in the subject matter or materials discussed in this manuscript.

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