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# Physicochemical, Morphological and Thermal Characterization of Composites Based on Olive Wood Flour

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**Abstract.** This study deals with the evaluation of the physicochemical, morphological and thermal properties of a biocomposite material based on polypropylene (PP) reinforced with vegetable fibers olive wood flour (OWF). Initially, the olive wood flour was chemically treated with a coupling agent (amino-silane) and then characterized to provide the modification influx. In a second step, various PP/OWF bio-composites with variable contents of OWF (0, 10, 20 and 30% by weight) were implemented by twin-screw extrusion and then shaped by injection molding. The effect of the OWF content on the one hand and the chemical modification of the latter by adding silane as coupling agent on the other hand, are the two parameters that are the subject of the present sensitivity study. Finally, the characterization results are compared and discussed. The significant influence of the treatment of OWF with the silane coupling agent is demonstrated, clearly demonstrating that the silane promotes the adhesion of the plant-based filler that is OWF with the PP polymer matrix.

**Keywords:** Physicochemical · Biocomposite · Coupling agent

## 1 Introduction

The development of a composite material makes it possible to obtain properties that are better than those of the constituents that compose it. Thus, it is imperative to know the composition and the properties of the constituents, as well as the stages of implementation to understand the interaction mechanisms between the constituents. This study aims to focus on this approach and allowed us to determine the physicochemical, thermal and morphological characteristics of the fibers, in this case olive wood flour (OWF), the matrix, polypropylene (PP), and elaborate composite (PP/OWF).

## **2 Materials and Method**

### **2.1 Materials**

The polypropylene (PP) used in this study is a standard homopolymer PP H9069 (Braskem, Brazil). FBO is recovered from wood waste generated during agricultural work. It is sieved and stored to protect it from moisture. Two types of FBO were used as reinforcing fillers, untreated (OWF) and treated (OWF) at 3% by weight with a coupling agent [1], amino-propyl-triethoxy-silane (OWFT).

### **2.2 Filler Characterization**

The biomass used in this study consisted of olive wood flour and sieved to obtain finer particles, from 84.6 to 254.9  $\mu\text{m}$  in size. The chemical composition of the dried fibers was determined according to [2]. It was determined by carrying out a series of extractions to isolate the various substances contained in the fibers such as: cellulose, hemicellulose and lignin. The length and aspect ratio of OWF filler was determined by Morfi analyses using JEOL JSM-5400 Morfi equipment

### **2.3 Elaboration of Bio-composite**

In order to eliminate all the moisture absorbed and to avoid the formation of agglomerates, the FBOs were pre-dried at 105 °C for 24 h before extrusion. The PP polymer and the FBO fibers were mixed in a co-rotating twin-screw extruder (DSM Xplore Netherlands) with 3 heating zones. The 10 mm diameter screw was set at a rotational speed of 100 rpm. The mixture is extruded through a cylindrical matrix of diameter 1 mm at a rate of 200 g/h. The rod is cooled, then cut into granules which are in turn dried for 24 h at 60 °C. After extrusion, the dried granules are injection molded using a molding machine (Demag Plastic Group Extra 50–200). The injection is produced at a temperature of 180 °C and a pressure of 1000 bar to produce the bio-composite samples required for the tests, in the form of plates and test pieces. Different charge rates of 0, 20 or 30% have been developed.

### **2.4 Scanning Electron Microscopy (SEM)**

Scanning electron microscopy (SEM) was performed with a JEOL JSM-5400 SEM microscope operating at 15 kV. The specimens were carried out under liquid nitrogen to avoid plastic deformation of the matrix and to obtain a well-defined fiber–matrix interface. Prior to analysis, all samples were coated with a 10 nm layer of gold to avoid sample charging under the electron beam.

### **2.5 Thermo-Gravimetric Analyzes (ATG)**

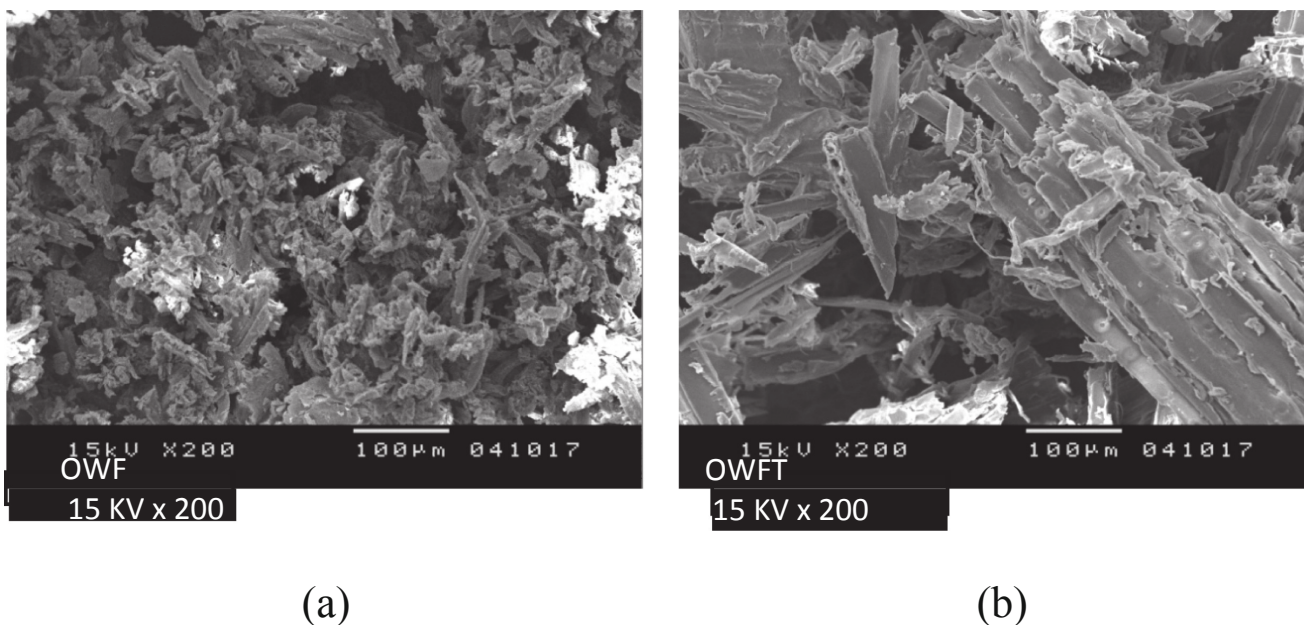
Thermogravimetric analyzes (ATG) were performed using Perkin Elmer Pyris-1 equipment (USA) under a nitrogen flow up to 800 °C and with a temperature rise rate

of 10 °C/min. The objective is to study the thermal stability of our samples (fibers alone, virgin polymer, and composite materials).

### 3 Results and Discussion

#### 3.1 Morphology and Chemical Composition of Olive Wood Flour

Olive wood flour is similar to a composite material reinforced by longitudinally bonded fibrils of cellulose, lignin and other non-cellulosic materials. Waxes and impurities regularly cover the whole [3, 4]. Figure 1 shows the micrographs obtained by the SEM show morphological transformations of the fibers following chemical treatment with silane. The surface of raw olive wood flour is smooth, while that of the treated FBO shows exposed fibrils. The reduction in the level of non-cellulosic material such as lignin, hemicellulose and wax, thanks to the treatment performed, has a positive effect on the roughness of the fiber surface, which increases visibly according to the micrograph [5].

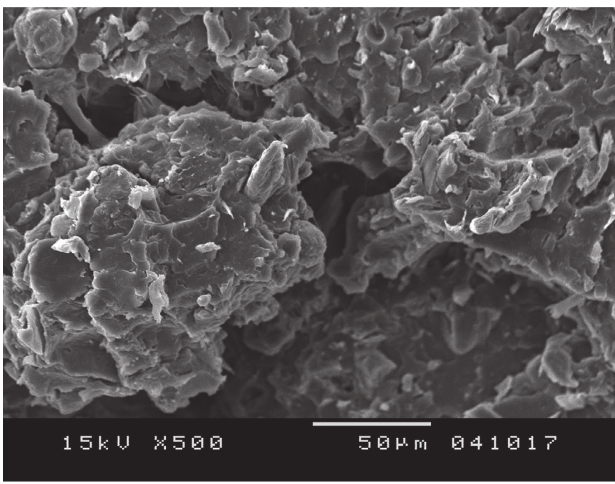


**Fig. 1.** SEM images of olive wood flour fibers: (a) untreated OWF, (b) OWF treated

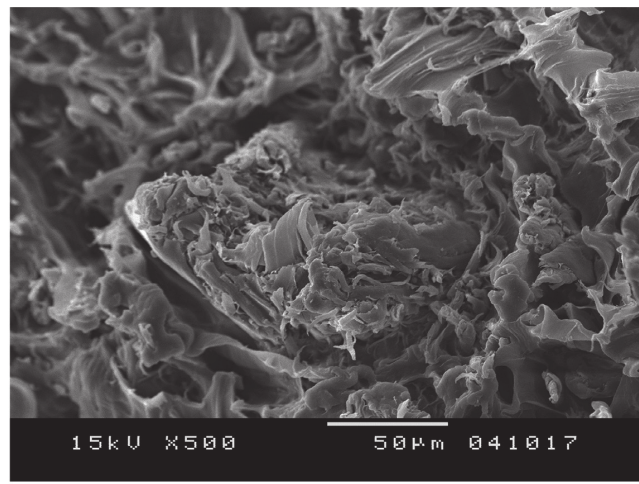
The chemical composition of the OWF. It confirmed the lignocellulosic character of the filler being composed of lignin, hemicelluloses and cellulose, with content about 21.9%, 29.2% and 45.9%, respectively.

#### 3.2 Morphological and Thermal Characterization of Elaborate Bio-composites

By switching from crude fiber-based biocomposite (Fig. 2 (a)) to processed fiber-based fiber (Fig. 2 (b)), the number of these cavities decreases. As a result, the improvement in adhesion between the fibers and the matrix can be distinguished by addition of the coupling agent (the treatment with the silane).



(a)



(b)

**Fig. 2.** SEM images of composite: (a) PP/OWF, (b) PP/OWFT

From the PP matrix. As shown in photo (a) of Fig. 2, the fiber surfaces are smooth and clean, showing poor inter-facial adhesion between the fibers and the matrix. On the other hand, the photo (b) of Fig. 2 shows that for the bio-composite loaded by the treated FBO the fibers are closely linked to the matrix with a surface characterized by a coating of a layer of the polymer matrix (PP). Indeed, it can be seen that the fracture occurred at the cross-section of the fibers themselves. This confirms that there is a transfer of the stresses from the matrix to the load, because of the adhesion to the contact between fibers and matrix. The fibers therefore participate in this case in the energy required for breaking and thus ensure good cohesion of the entire composite material.

These results are in agreement with previous studies which confirmed that the chemical treatment with silane of natural fibers used as reinforcements improves the cohesion between the fibers and the matrix of the corresponding bio-composite materials.

Biocomposites loaded with the raw and treated OWF were analyzed by thermogravimetry (ATG) in order to know the effect of chemical treatment of the load and the rate of reinforcement on the thermal stability of the material.

The results obtained by thermogravimetric analysis (ATG) are presented in Table 1 as a function of degradation temperature (Tmax).

**Table 1.** Degradation temperatures of raw and processed PP/OWF composites.

Composition	Degradation temperature of PP/OWF	Degradation temperature of PP/OWF
PP/	466.61	466.61
PP/OWF 10%	471.96	466.54
PP/OWF 20%	469.41	469.82
PP/OWF 30%	475.54	472.58

The addition of 10%, 20%, 30% of untreated fibers significantly increases the degradation temperatures respectively by 5, 3, and 9 °C relative to the matrix alone. This effect is comparable with the treated fiber content, for which the temperature



varies from 0, 3, and 6 °C with the addition of 10%, 20%, 30% of treated fibers, respectively. In our case, neither the addition of fiber content nor the chemical treatment significantly affected the thermal stability of the PP/OWF composite.

The literature [6, 7] shows that generally the concentration of chemical treatment is the factor that significantly influences the thermal stability of composites because of its ability to remove hemicellulose, lignin and impurities from the fiber. This allows an improvement in the thermal stability of the fibers. However, the modification made to the fibers in our study is not sufficient to affect the thermal stability of the composite in its entirety

## 4 Conclusion

In this study we characterized the raw olive wood flour and chemically treated with silane. Indeed, the development of new bio composites requires a prior knowledge of the different properties and the composition of fibers.

The SEM micrographs of both types of flours revealed an increase in the roughness of the treated versus untreated fibers due to the removal of non-cellulosic constituents. The chemical analysis reveals that after chemical treatment, the hemicellulose and lignin contents decrease, which causes the increase of the cellulose content in the treated fibers. Secondly, we have characterized morphologically and thermally elaborate bio-composites. SEM images showed good adhesion between the matrix and treated or untreated fibers. Thus, the cohesion at the fiber/matrix interface seems to improve with the chemical treatment.

Thermogravimetric analysis has shown that the thermal stability of bio-composites does not change much for both treated and untreated fibers.

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