

# Study of the Thermal, Rheological, Morphological and Mechanical Properties of Biocomposites Based on Rod-Of Typha/HDPE Made up of Typha Stem and HDPE

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## Abstract

The thermal, rheological and morphological properties of composite biomaterials made with mixture of high density polyethylene and typha rod powder (RD) were evaluated. The dynamic mechanical behavior of the samples was studied with 25%, 35% and 45% typha stem powder concentrations. The viscoelastic properties are mainly related to the nature of the polymer and the typha stem powder. Storage ( $G'$ ) and loss ( $G''$ ) moduli increased significantly, depending on the amount of powder in the molten mixture. After a viscosity increase was noticed in low frequency, it decreased in high frequencies, which demonstrates the pseudo-plasticity effect. Morphological and thermal characterization results have shown the dispersion state of the powder and its ability to modify the kinetics crystallization of biocomposites.

## Keywords

Biocomposite, Thermal, Rheological, Morphological, Mechanical Properties

## 1. Introduction

The development of polymeric biocomposites made up of natural fibers or wood particles as reinforcing components is attracting more and more attention from the industrial world because synthetic fibers are expensive, non-biodegradable

and their production consumes energy, with a negative impact on the environment [1]. The use of wood fibers and wood flour as a reinforcing agent for composite polymers has increased dramatically compared to the traditional synthetic fibers due to their advantages in terms of quality and production cost. Natural wood particles (fibers or granules) have been involved in the development of innovative, lightweight, solid materials and can be used in the engineering field. In addition, they are renewable, recyclable, biodegradable and very eco-friendly.

On the other hand, wood flour plays a major role in improving the elasticity modulus and the flexural strength of the composite [2]. However, the mechanical performance of WPC differs from kind of wood. For example, even though the flexural strengths of WPC and wood are close, the elastic properties of wood are higher. This statement is based on data comparison and experimental results [3].

Fiber content is an influential factor in the properties of WPC. Gisele *et al.* [4] studied the effect of wood content on the thermal behavior and molecular dynamics of composites made up of PVC and wood. They found that progressive addition of wood flour resulted in a small but gradual improvement in the decomposition temperature composites—while the glass transition temperature remained virtually unchanged—and a gradual decrease in relaxation time values ( $T_1$ ).  $H$  values have been observed with an increase in wood content, which indicates that composites have become less rigid.

Four formulations were made by Nourbakhsh and Ashori with 10%, 20%, 30% and 40% fiber mass concentrations (in terms of weight) to study the mechanical properties of poplar/PP composites. They observed that composites with 30% and 40% fibers allowed ideal reinforcement at 190°C to increase the tensile and flexural strength of the PP, and the modulus increased and the elongation and impact properties decreased as the fiber content increased. Other authors stated that there is an increase in torque and viscosity as wood fiber content increases at steady state.

A range of natural materials have been used for the manufacture of wood-polymer composites. Among those materials, wood fibers were found very interesting in the areas of basic research and applications due to their ease of handling and high availability [5] [6]. Many factors such as particle distribution, shape and size, chemical surface nature and impurity content have very large influence on the quality of the wood fiber. Among these factors, fiber size is the most determinant on the mechanical behavior of a composite made up of wood and polymer. However, the experimental results regarding fiber size role in mechanical behavior are contradictory. The experiments conducted by Maiti and Singh [7] on different wood particle sizes without coupling agent showed that despite poor adhesion between fiber content and the plastic, the sample produced by extrusion had an increasing elasticity modulus with a decreasing particle size. In contrast, Chen *et al.* [8] studied the influence of wood particle size

on wood/HDPE composites and found that composites made up of larger wood particles had greater strength. Furthermore, the addition of wood content to the thermoplastic polymer is combined with a considerable increase in viscosity in the molten state [9] [10].

Despite the rapid growth in the use of wood-plastic composite (WPC) in automotive and other industries [11] [12], there is limited information published in the field of WPC rheology in West Africa. In fact, people use a large number of plastic bags; the residues of these bags released in the nature constitute a serious pollution issue for both consumers and environmentalists. Rheology could allow the biocomposite development with a good interpretation of the interaction between the wood and the polymer matrix.

The mechanical and thermal properties and the dynamic behavior of the modified PP/WF composite were studied by Hristov and Vasileva [13]. Other authors studied the viscoelastic behavior; surface tearing and wall slip phenomena of high density polyethylene (HDPE)/WF composites [14] [15]. The rheological behavior of the HDPE/WF composite under steady shear and extension flux has been studied by Li and Wolcott [16]. They found that the extension viscosity is not significantly affected by the wood content. They also analyzed the composite slide and reported that the rate of this phenomenon is influenced by the wood content [17]. In another study, these authors examined wood content, particles size and maleated polyethylene effects on the rheological properties of the maple/HDPE composite [18]. The elastic component is given by the dynamic storage modulus, which indicates the inherent stiffness of the material while the mechanical damping or the internal friction ( $\tan\delta$ ) indicates the amount of energy dissipated in the form of heat during the deformation [19].

Our study focuses on the use of *Typha australis* fibers for the manufacture of composite material. *Typha australis* is an aquatic plant found on wetlands and it belongs to the Typhaceae family. This plant, which can be 3 m high [20], is particularly widespread on the Senegal River Valley. *Typha australis* is now considered a pest. Indeed, its proliferation on the Senegal River Valley affects the valley's ecosystem and reduces the socio-economic activities of local communities. *Typha* plants are widely used in wetland construction for ecological restoration [21] and wastewater treatment [22] [23]. In addition, *Typha* is also the raw material for heat preservation [24], weaving and paper [25] because of the length of its fiber, the plant's toughness, and its heat preservation performance.

The final objective of this study is to evaluate the rheological, thermal, mechanical, and morphological properties of original biocomposites made up of typha stem and high-density polyethylene.

## 2. Experimentation

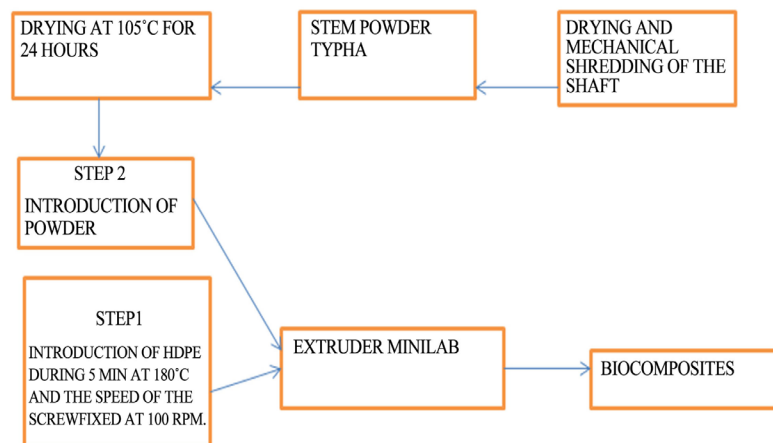
High density polyethylene (HDPE) has been used in the engineering field because of its good physical and mechanical properties.

It is one of the most versatile commercial polymers with its semi-crystalline

nature allowing treatment over a wide temperature range. The crystalline phase of the polymer provides resistance, while the amorphous phase provides flexibility [26]. HDPE is the most widely used polymer matrix for WPCs because of its relatively low processing temperature and property of being easily processable [27]. HDPE was supplied in the form of granules by Nova Chemicals. Its melt index was 7.0 g/10 min at 190°C and the density at room temperature was 0.962 g/cm<sup>3</sup>. Thus, prior to any mixing operation, the wood powder was oven dried at 105°C for 24 hours to remove moisture less than 1%.

No further processing took place on the polymer before use. The composite materials were obtained by mixing in a 7 cm<sup>3</sup> co-rotating twin screw microcompounder minilab mixer. For the extraction of harmful gases, the hood was open. In a first step, the polymer is introduced for 5 min at 180°C and the speed of the screw is set at 100 rpm. In a second step, typha stem powder is introduced. The system is mixed for 10 min at 180°C at 100 rpm (Figure 1). After this phase each sample is sandwiched for 1 min between two Teflon sheets and pressed at 180°C under a pressure of 200 bars to obtain films ranging from 100 to 200 microns thick for spectroscopic analysis, or of a few millimeters thickness for the study of different macroscopic properties. With the polymer matrix (HDPE), four formulations were produced with different typha stem powder contents at the Clermont-Ferrand Institute of Chemistry (ICCF) laboratory in France (Table 1). The temperature profile in the machine was kept below 180°C to minimize thermal degradation of the powder.

### Composition



**Figure 1.** Schematic diagram of the composites biomaterials.

**Table 1.** The percentage samples of wood and polymer.

RDHDPEC0	100	0
RDHDPEC1	75	25
RDHDPEC2	65	35
RDHDPEC3	55	45

### 3. Characterization

#### 3.1. Characterization of the Thermal Stability of the Composite

##### 3.1.1. Thermogravimetric Analysis (TGA)

Thermogravimetry is a technique that measures the mass variation of a sample, when it is subjected to temperature programming in a controlled atmosphere. This variation can be a loss of mass (vapor emission) or a mass gain (gas fixation, oxidation, etc.). The sample is weighed and the baseline established, then the sample is subjected to temperature rise. At different temperatures, chemical reactions can release gaseous species, or form oxides causing a mass variation of the sample. This mass change is recorded, according to the temperature. A large number of transitions (melting, crystallization, glass transition...) do not imply a mass variation. These transitions, which are not detected by thermogravimetric analysis, can be differential thermal and enthalpic analyses. Submitted to these high temperatures, wood and synthetic polymers are susceptible to have their mechanical properties deteriorated. It is necessary to optimize the temperature during the implementation of the WPC. This method of analysis is often used to determine the characteristics of materials such as oxidation kinetics, absorption, hydration, etc. [28]. For our tests, we used a Mettler Toledo thermogravimetric analyzer to measure the mass variations of the samples. Heating was carried out at temperatures ranging from 30°C and 600°C, setting during the analysis a thermal rate of 10°C/min. Nitrogen is used as carrier gas. This analysis makes it possible to know the thermal decomposition temperature of a polymer. The loss of mass is recorded according to a temperature programmed with a given gradient.

##### 3.1.2. Differential Scanning Calorimetry Analysis

Differential scanning calorimetry was used to analyze the thermal properties of composites. Samples with different wood particle concentrations were subjected to a temperature scanning ranging from 25°C to 210°C, at a heating rate of 10°C/min under a harmful inert gas (nitrogen). We performed two cycles (heating-cooling) under controlled atmosphere during the temperature programming. The fitness effects (processing) and the residual stresses in materials of thermal origin were eliminated during the first cycle. Then, the second cycle made it possible to measure the melting temperature  $T_m$ , the melting enthalpy  $\Delta H_p$ , the crystallinity degree of semi-crystalline polymers and the heat capacity variation  $C_p$  of the biocomposites.

#### 3.2. Rheological Characterization

The rheological characterization of the composites functions was carried out using dynamic low amplitude oscillatory shear tests. This method makes it possible to measure the dynamic properties of composites such as dynamic moduli  $G'$ ,  $G''$ , and the loss, complex and storage viscosity respectively  $\eta'$ ,  $\eta''$ , and  $\eta^*$ . The dynamic elastic moduli  $G'$  and  $\eta''$ , help measure the elastic energy stored in the

deformed material. This energy is reversible; it helps the material partially recover its shape before the deformation. While the dynamic moduli  $G''$  and  $\eta'$  represent the dynamic loss modulus, they correspond to the amount of energy dissipated by the materials due to the interaction between the molecular chains of the polymers in shear. The variation of these moduli, according to the frequency and at a temperature equal to 180°C for biocomposites with different wood contents, is studied.

### 3.3. Morphological Characterization

The state of dispersion of the wood inside the polymeric matrix was analyzed using optical microscopy on 100 - 200 mm thick samples. The fractured samples were coated with a thin gold layer to avoid electrostatic charging during examination. SEM was used to obtain microphotographs of the fracture surfaces of the wood composites. These fractures have been performed in liquid nitrogen to avoid any deformation. SEM has been performed using a Fei Quanta 400 microscope working at 20 kV [29].

## 4. Results and Discussions

### 4.1. Thermal Stability of the Composite

With a Mettler Toledo thermogravimetric analyzer, we obtain measures the mass variations of the samples (**Figure 2**: Heating ranging from 30°C to 600°C for biocomposites, a thermal rate of 10°C/min being set during the analysis). Nitrogen is used as carrier gas. Through this analysis, it is possible to know the thermal decomposition temperature of a polymer.

The thermal degradation of composites made up of wood fiber and pure HDPE in regard to the temperature is illustrated in **Figure 2(a)**. From the thermogravimetric curves, we notice in the first place a loss of mass for the different samples containing wood, which corresponds to the loss of water. By 225°C, starts a mass variation with a change in the shape of the curve. It corresponds to the degradation start temperature of wood. The curve of the first derivative is associated with the TGA curve.

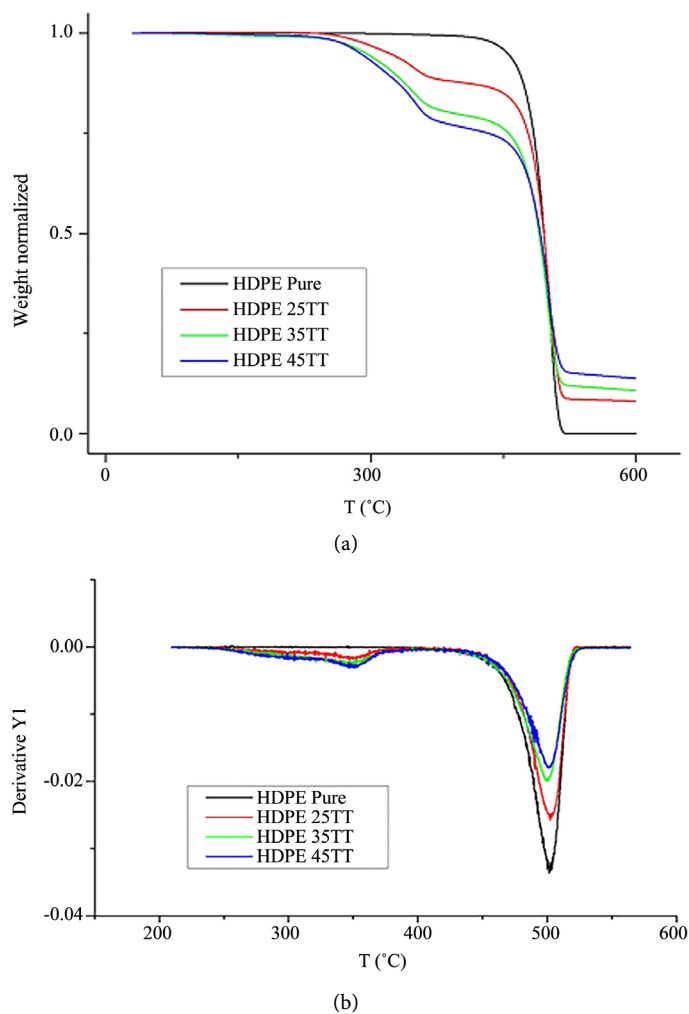
The pure HDPE shows negligible loss of mass at temperatures below 370°C. The significant decrease in mass loss starts from the temperature above 410°C. The TGA of typha stem fiber shows a two-step degradation process with an initial transition of about 100°C due to moisture evaporation.

It is known that cellulose in wood contains many strongly hydroxyl groups. The initial degradation temperature ( $T_d$ ) of typha plating was 240°C, and the maximum decomposition temperature was 381°C. Cellulosic substances normally have three degradation stages. The depolymerization of hemicelluloses is between 150°C and 350°C, the random cleavage of the glycosidic bonds of cellulose occurs between 280°C and 350°C, and the degradation of lignin occurs between 250°C and 500°C [30] [31].

In contrast, in other samples (**Figure 2(a)** and **Figure 2(b)**), depending on the

reinforcement rate inserted in the polymer matrix in both cases, a decrease in weight loss around 100°C due to the elimination of the moisture was observed. Because of the high flammability of cellulose, the addition of wood makes the composite less thermally stable [32]. The TGA measurements indicate that, when the polymer is heated, its thermal stability is generally evaluated from the loss of mass and its molecular weight decreases sharply due to the degradation of the molecular chains [33].

The degradation mechanism of wood/HDPE composites has not been sufficiently understood. However, the possible reasons for the increase in the thermal stability of these mixtures can be explained by the fact that the crystals in the amorphous phase of the polymer are initially protected by the surrounding polymer chains. Similarly, in the case of pure HDPE, the degradation began at around 450°C and the maximum decomposition rate appeared at 495°C. The degradation temperature range (about 450°C) of this polymer is displaced at lower temperatures.



**Figure 2.** (a) TGA thermograms of pure HDPE and its composites; (b) DTG curves of decomposition of pure HDPE and its composites.



**Figure 2(a)** shows the TGA curves of the pure polymer matrix and its composites.

The evolution of the mass is generally represented in relative values (in %) with respect to the initial mass of the sample. The derivative of the TGA curve thus obtained, the DTG curve, is often represented, according to the time or temperature for better clarity. It shows the speed of the evolution of the mass in (%)/min or in (%)/°C.

In DTG **Figure 2(b)**, the thermal degradation has two levels.

The temperature ranges of the first stages show dehydration up to 320°C approximately, a separation of the moisture and the combined water of the wood. The temperature at the beginning of the dehydration is taken as a measure of the thermal stability of the WPC.

These results are confirmed by those in the literature [34] [35] [36]. Therefore, it would be necessary to adjust the temperatures to around 200°C to prevent their degradation during manufacturing processes (extrusion). Mass loss according to time or temperature is an irreversible process due to thermal degradation. It can be concluded that the cellulose components and hemicellulose of wood are the main contributors to the decomposition between 250°C and 370°C, while lignin is mainly responsible for the carbonization of wood at a temperature above 400°C.

The loss of mass during its first stages are of 8%, 11% and 16.7% respectively for composites made up at 25%, 35% and 45% of typha powder. The remaining polymers decompose during the second stages. The sample loses a total of 92% of its mass up to 600°C. From the curve of this example, a thermal effect start temperature of 210°C can be observed. The mass loss information obtained by thermogravimetric analysis does not specify the types of decomposition products. This is only possible if the TGA device is coupled with a suitable interface. For example, equipment for gas analysis provided with an infrared spectrometer and gas-filled cells or a mass spectrometer.

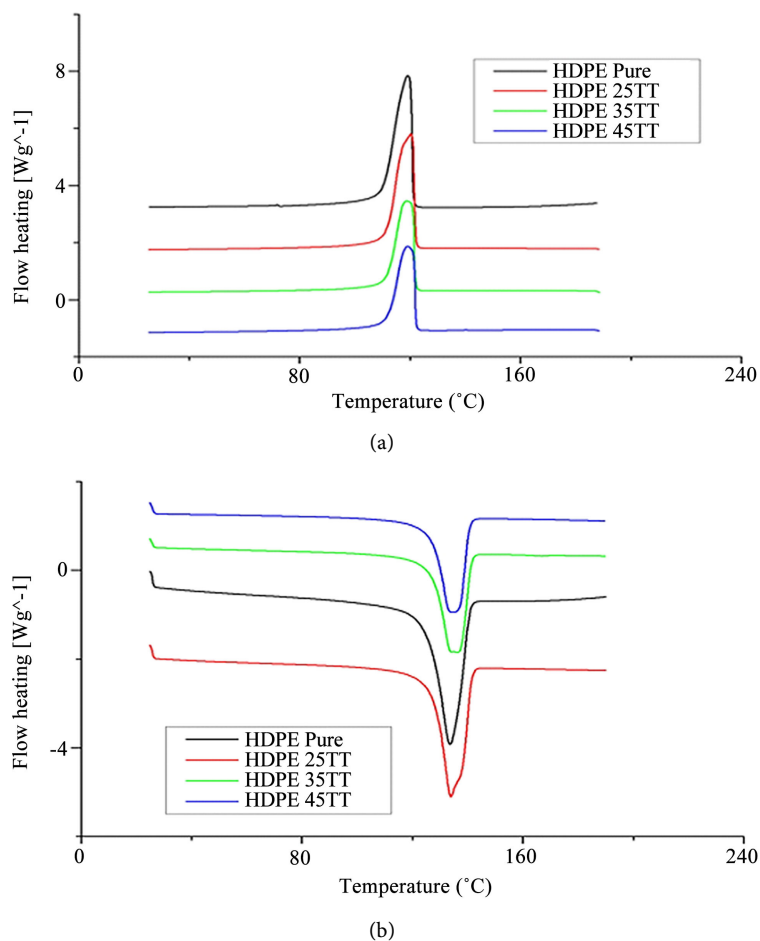
## 4.2. Differential Scanning Calorimetry

The DSC measurements were conducted on the various typha stem composite samples. The parameters resulting from these measurements are the melting temperature ( $T_m$ ), the crystallization temperature ( $T_c$ ), the crystallization enthalpy ( $\Delta H_c$ ), the melting enthalpy ( $\Delta H_m$ ), and the crystallinity percentage (%). The figure shows thermograms of composite samples containing different powder percentages. The results of these physical parameters quoted below were summarized in the following table.

**Figure 3** shows the DSC thermograms of the cooling curves (**Figure 3(a)**) and the melting curves (**Figure 3(b)**) of HDPE and its composites.

Like the thermograms in the figures, the thermograms of all the samples show endothermic peaks between 133°C and 135°C during heating and exothermic peaks between 118°C and 120°C during cooling.





**Figure 3.** (a) DSC cooling thermograms of neat HDPE and its composites containing different rates of typha rod, cooling; (b) DSC fusion thermograms neat HDPE and its composites containing different rates of typha rod, melting.

We notice that the addition of wood increases the melting temperature. Indeed, we notice an increase of the melting temperature with the wood percentage (**Table 2**).

This can be explained by the fact that the polymer needs heat to melt, but powder is not a good thermal conductor. Typha stem particles intercalated in the polymer matrix act as insulators and slow down the heat circulation process by conduction [14]. An increase in the degree of crystallinity is observed by adding the typha stem powder. The increase in the crystallinity level may be due to changes in the molecular weight that occurs during the degradation of the polymer, resulting in the breaking of chains and probably a secondary crystallization. In the composite, the increase in crystallinity is due to a decrease in molecular weight [19].

### 4.3. Rheological Behavior

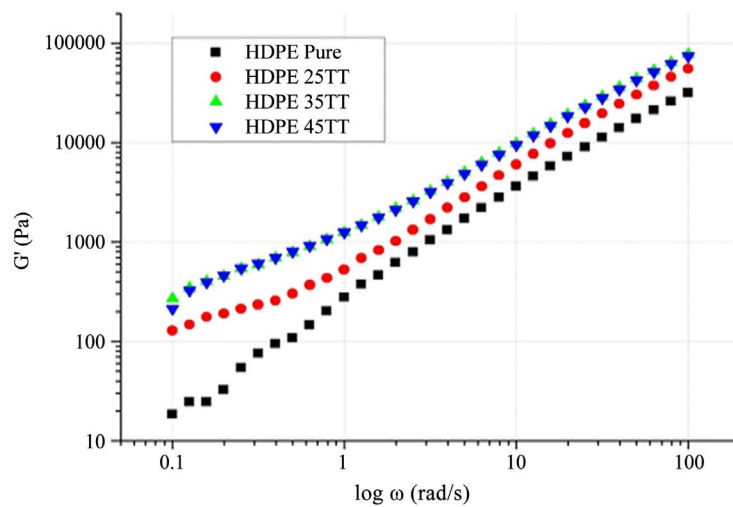
The rheological properties of all the samples in the molten state were determined by mechanical dynamic measurements. The conservation modulus  $G'$ , the loss

modulus  $G''$  and the dynamic viscosity  $\eta^*$  were measured according to the frequencies scanned between 0.1 to 100 rad/s.

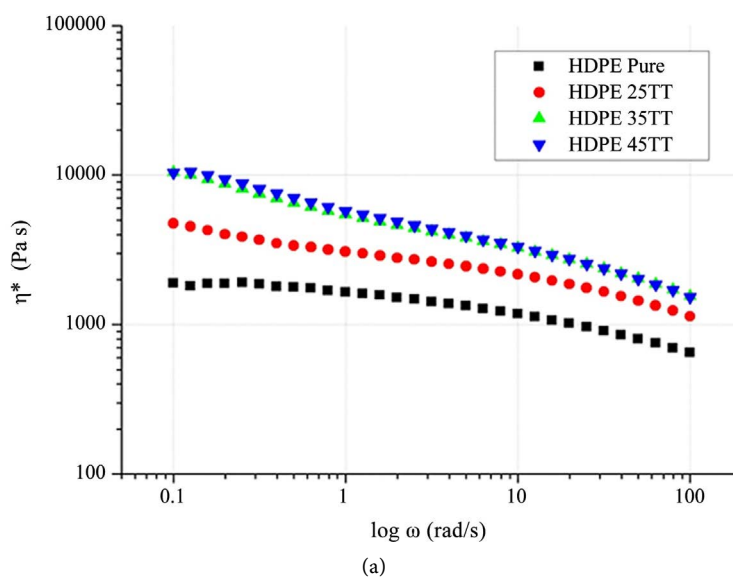
**Figure 4** and **Figure 5** show the variation of the dynamic moduli ( $G'$ ) and ( $G''$ ) and the viscosity according to the frequency with different percentages of typha.

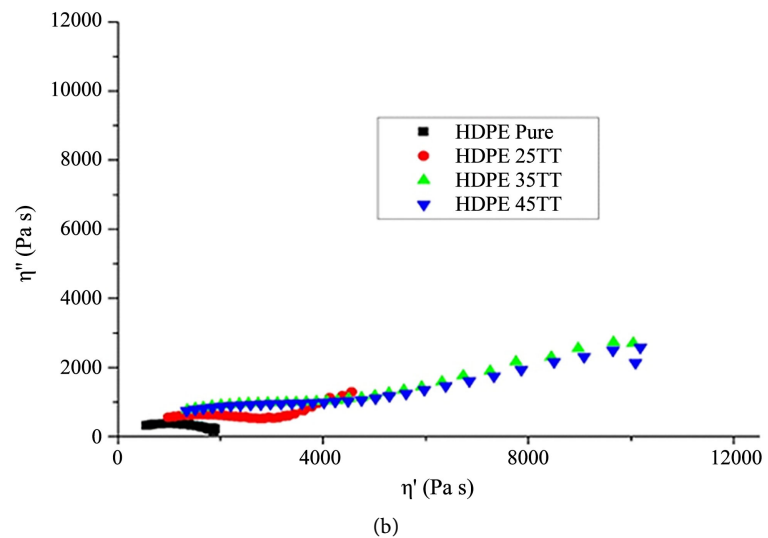
**Table 2.** Thermal properties of pure HDPE and its composites.

Sample	$\Delta H_f$ (J·g <sup>-1</sup> )	$\Delta H_c$ (J·g <sup>-1</sup> )	$T_f$ (°C)	$T_c$ (°C)	$X_c$ (%)
HDPE PUR	179.15	181.74	133.69	119.27	61.1
HDPE 25	156.73	163.94	133	120.45	71.3
HDPE 35	119.67	128	136.53	118.98	62.8
HDPE 45	107.07	119.75	135.73	119.18	66.5



**Figure 4.** The variation of the dynamic moduli ( $G'$ ) and ( $G''$ ) according to the frequency with different percentages of typha stem.





**Figure 5.** (a) Frequency variations of the complex viscosity of pure HDPE and its composites at  $T = 180^{\circ}\text{C}$ ; (b) Complex plane diagrams for the WPC (upper right curve for neat HDPE) at  $T = 180^{\circ}\text{C}$ .

The dynamic storage ( $G'$ ) and loss ( $G''$ ) moduli tend to increase with the typha stem powder content (Figure 4). This can be translated by a more elastic behavior for high contents. Quite obviously, the complex viscosity of pure polymer gradually increases with the increase of powder content from 25% to 45% (Figure 5). Indeed, in filled systems, wood particles disturb the flow of the pure polymer and hinder the mobility of chain segments in the direction of flow, its particles are often in the form of aggregates.

This can also be explained by an inhomogeneous dispersion and by the alignment of the cellulosic phase in the composites. These observations are confirmed by those provided in literature [31] [37]. In addition,  $G'$  and  $G''$  moduli tend to have the same values beyond 35%. Shear thinning behavior of the molten mass was observed, the viscosity of the composites is strongly influenced by the shear rate. The composites showed substantially the same viscosity above 35%. This phenomenon is probably due to the alignment of the fibers at high shear rates, which reduces the collisions between particles [38]. Indeed, the fluidity of the mixture is increased by an increase in the oscillation frequency, which decreases the complex viscosity. This reduction in viscosity shows the pseudo-plastic nature of materials in the molten state.

In the low frequency domain, the viscosity increases dramatically as the fiber content in the composite material increases. The addition of fillers has an influence on the rigidity, the density and the viscoelastic behavior of a polymer [37] [38] [39]. The highest value of complex viscosity was observed with the composite containing 45% of typha stem masses. In addition, the presence of agglomerates (due to high content) caused flow resistance and an increase in the viscosity of the composites. This behavior can be explained by a higher interaction rate between the polymer matrix and the content, which forces the mixture to have

greater shear stress and longer relaxation times for the composites to flow [40]. The rheological properties of high density polyethylene exhibit the same shear thinning behavior.

The elasticity moduli demonstrate an improvement in the dynamic behavior of composites.

#### 4.4. Scanning Electron Microscopy

The microstructure of the wood/HDPE composite shown in the figure is observed (after extrusion) using a scanning electron microscope. It is well known that the properties of wood/polymer composites strongly depend on the dispersion of the wood and the polymer matrix.

Visual observation reveals fairly smooth surface in composites (HDPE/Typha stem) probably due to a medium inter-facial adhesion. The wood particles are detected as white dots in **Figures 6(a)-(c)**.

When we compare **Figure 6(b)** and **Figure 6(c)**, we notice that the dispersion is better in **Figure 6(b)**, where there is less wood. These two results show that the dispersion decreases with a high content rate. For blends containing HDPE/RD (**Figure 6(a)**), we can see that the composite phase appears to be more uniform than the figures (**Figure 6(b)** and **Figure 6(c)**). The results suggest a smoother surface, which would promote interfacial adhesion between the two elements. From the images in **Figure 6**, the observations in **Figure 6(a)** are contrasted with the surface texturing of the samples from **Figure 6(b)** and **Figure 6(c)**.

The SEM micrographs presented (**Figure 6(b)** and **Figure 6(c)**) showed that the wood particles are more superficial on the surface of the samples.

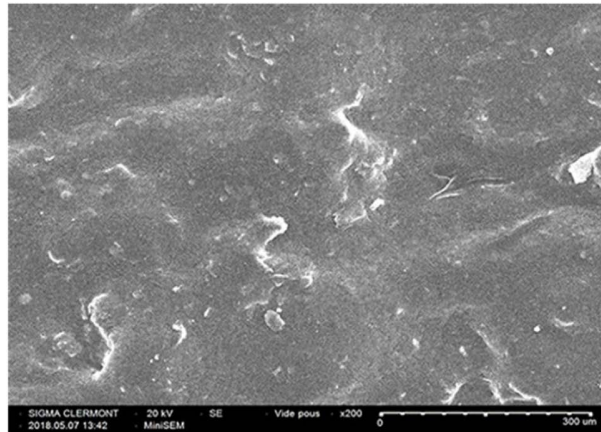
When the wood content increased up to 45%, the particles were not uniformly distributed in the HDPE matrix. The matrix did not sufficiently encapsulate the solid microparticles and microfibrils of wood. However, large aggregates were found and the aggregated size increased significantly in these surface microphotographs with a higher rate of wood.

#### 4.5. Mechanical Properties of Biocomposites HDPE/Typha Rod

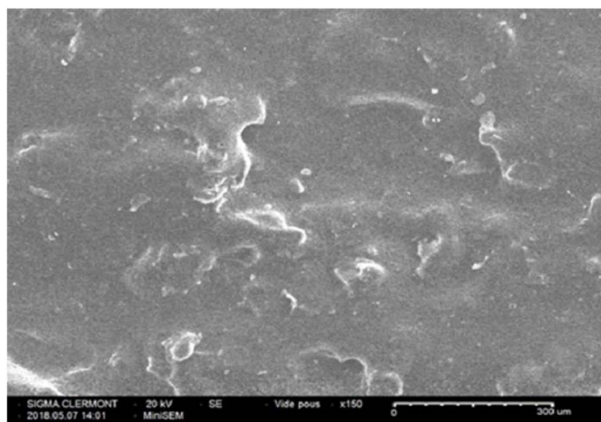
The following figures show the results of mechanical tensile tests of composites made up of high density polyethylene reinforced by typha stem powder with 25%, 35% and 45% of weight concentrations. The maximum stress and tensile elastic modulus of the HDPE tend to increase respectively with the reinforcement rates of 35% and 45%. However, there is a decrease in the maximum deformation at break with the powder concentration.

The slight fall in the maximum tensile stress observed when the wood particle content exceeded 35% (**Figure 7(b)**), is due to the formation of particles agglomeration at high concentrations, resulting in poor adhesion between the powder and the polymer matrix. Thus, one might think that there is an optimal concentration value for the manufacture of HDPE/typha stem composite. The weight

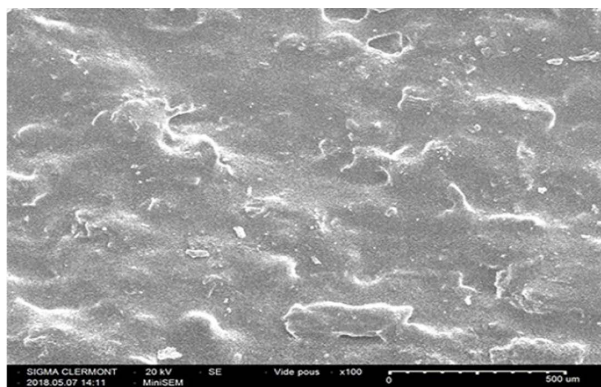
ratios of the powder increasing below this optimum value would cause a good content distribution in the polymer matrix; the direct consequences would be to obtain better tensile properties (**Figure 7(a)** and **Figure 7(b)**). However, an additional increase that is higher than the optimum mass value of powder would result in a decrease in tensile strain (**Figure 7(c)**), as described by most of the authors [41] [42].



(a)

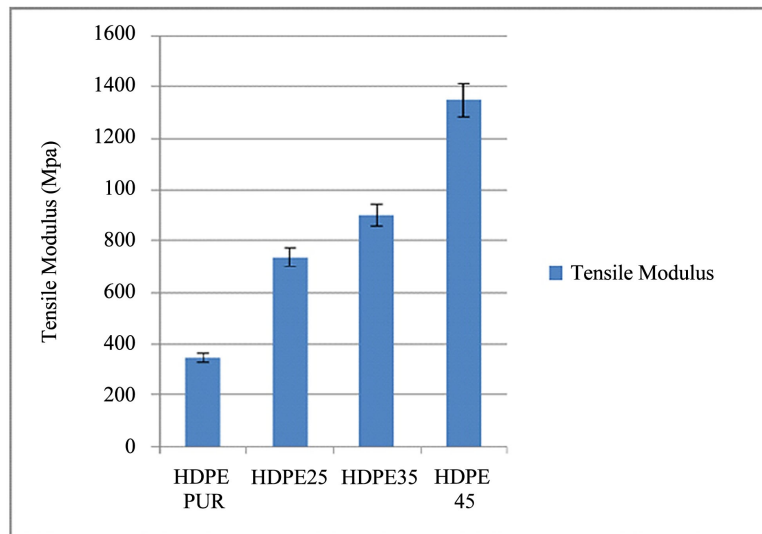


(b)

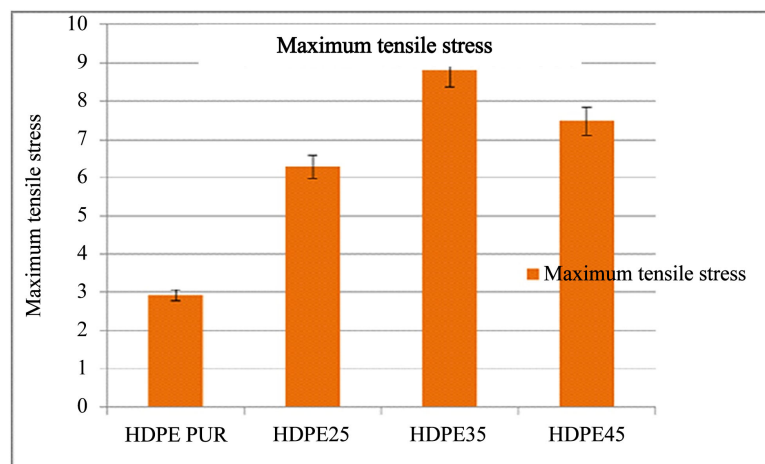


(c)

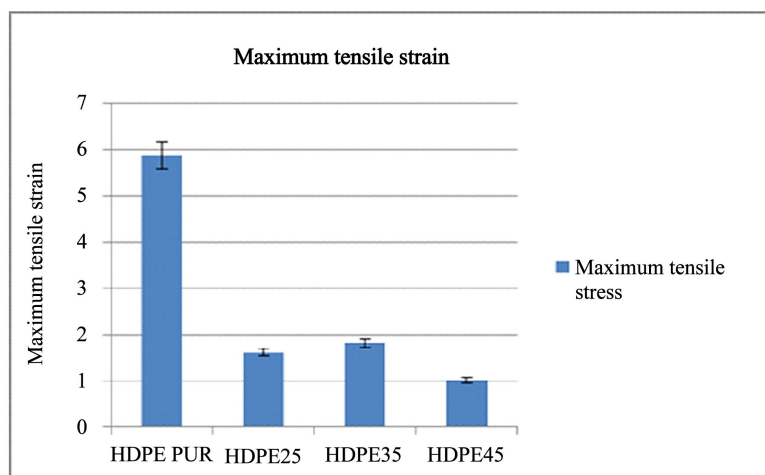
**Figure 6.** SEM images of HDPE composites: (a) HDPE/25 RD; (b) HDPE/35 RD; and (c) HDPE/45 RD.



(a)



(b)



(c)

**Figure 7.** (a) The Tensile Young modulus of pure HDPE and its composites; (b) The Maximum tensile stress of pure HDPE and its composites; (c) The Maximum tensile strain of pure HDPE and its composites.

## 5. Conclusions

The properties of mixtures made up of HDPE and typha stem flour have been studied. The dynamic behavior of the moduli in the molten state shows their increase for different powdered flour concentrations. In low frequency ranges, the Newtonian viscosity increased as the percentage of typha stem flour increased. However, the viscosity decreased with the increase of the frequency, which shows shear thinning behavior of the biocomposites.

Concerning the thermal properties of the composites, an increase in the crystallinity and a loss of more than 90% of mass were noted. The typha stem had served as a nucleating agent, and it caused a modification of the kinetic crystallization of the biocomposites.

Morphological analysis showed the state of the wood powder dispersion in the composite; it was observed that there was a better dispersion for the concentrations with 25% and 35% of powder. Tensile modulus and maximum deformation increased considerably with powder.

## Acknowledgements

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## Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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