

Rheological and electrical properties of EVA copolymer filled with bamboo charcoal

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Abstract The electrical and rheological properties of an ethylene vinyl acetate (EVA) copolymer filled with bamboo charcoal were investigated. The composites were prepared by melt process in an internal batch mixer. Size distribution analysis showed that $d_{(50)}$ and $d_{(90)}$ values of the bamboo charcoal particles are 12.7 and 40 μm , respectively, with a mean diameter of 22 μm . Scanning electron microscopy proved that the particles of bamboo charcoal present a rectangular shape. The electrical percolation threshold was observed at 0.18 volume fraction (35 wt%) of bamboo. Beyond the percolation threshold, a considerable increase in electrical properties was observed up to a limit value of 10^{-2} S/m. The rheological percolation was studied from different rheological models. As a result, the rheological percolation threshold was observed at 0.3 volume fraction (50 wt%) of bamboo charcoal contents. So, the electrical percolation occurs before the rheological percolation. This is principally due to the filler's characteristics such as the specific surface area, the aspect ratio, and the surface properties. Finally, the bamboo charcoal confers high electrical properties to the EVA composite without inducing strong changes in its viscoelastic properties.

Keywords Bamboo charcoal · EVA · Percolation · Viscoelasticity · Electrical conductivity · Biocomposites

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Introduction

Electrically conductive polymers present a great interest in many applications such as pressure-sensitive sensors, transducers, EMI-shielding materials, aircraft, telecommunication, and nuclear reactors. To improve the electrical conductivity of polymer materials, it is a common practice to load polymer matrices with conducting fillers like metallic fillers or more recently carbon fillers: carbon black, graphite, carbon fibers, and nanotubes. At a critical volume fraction of the conductive filler, an abrupt increase of the composite conductivity is observed, indicating the percolation threshold corresponding to the formation of a conductive filler network in the polymer. For example, Sohi et al. (2011) show that the percolation threshold for the electrical conductivity of ethylene vinyl acetate (EVA 2803) composites was 0.14, 0.07, and 0.03 per volume for conducting CB, SCF (carbon fiber), and multiwalled carbon nanotube, respectively. Actually, this study clearly showed that the percolation threshold principally depends, as expected, on the aspect ratio of the conductive particles.

In order to go further in the understanding of the composite microstructure, the rheological properties can also be investigated and compared with the electrical properties. Actually, due to the formed filler network above a critical concentration of fillers, the composite melt behaves solid-like rather than liquid-like. This liquid–solid transition corresponds to the rheological percolation threshold (Cassagnau 2008). This has been studied for various types of conductive fillers in different matrices (Du et al. 2004; Lee et al. 2007; Leboeuf et al. 2008; Bose et al. 2009; Martins et al. 2010; Poetschke et al. 2010; Charman et al. 2011; Sumfleth et al. 2011; Huang et al. 2012).

Generally, carbon fillers are produced by the incomplete combustion of heavy petroleum products. So, it will be

interesting to find an environmentally functional material that could have the same properties. Note that recently, bamboo and other natural fibers from renewable resources such as pineapple, banana, henequen, sisal, jute, wood, saw dust, coconut (coir), rice husk, and wheat straw have been extensively studied as reinforcing agents in different polymer matrices. Actually, the bamboo fiber is the most promising filler. It is one of the world's best known natural engineering materials and perhaps it is also one of the most underutilized natural resources available abundantly in Southeast Asian countries. Bamboo has been used traditionally in the fabrication of village houses as a structural material. In Hong Kong, 7,500 tons of bamboo scaffolding is generated from the construction industry annually (Kantarelis et al. 2010).

Finally, bamboo charcoal obtained by pyrolysis of bamboo gives good properties such as the prevention from electromagnetic waves, adsorbent, deodorization, good resolvability, antistatics, highly caloric fuel, and antibacterial (Mingjie 2004; Li et al. 2010; Huang et al. 2012). In 1878, a filament of bamboo charcoal has already been used by Thomas Edison for the first incandescent lamp.

Up to now, only thermal and mechanical properties of the bamboo-reinforced polymer composites have been studied as in the works of Chattopadhyay et al. (2010). However, its utilization has not been fully explored and the electrical conductive properties of bamboo charcoal-based composites are expected. Consequently, our work aims to study the electrical and rheological behavior of composites filled with bamboo charcoal. In order to compare our results with the works of Sohi et al. (2011), the matrix used in the present study is an ethylene vinyl acetate (EVA) copolymer.

Experimental part

Materials

The EVA used in this study was supplied by Arkema Evatane®. An EVA 2803 containing 28 wt% of vinyl acetate was used in the present study. The melt index of the grade is 3 g/10 min. The zero shear viscosity of the copolymer is 2.2×10^4 Pa s at 160 °C.

The bamboo charcoal (BC) was kindly supplied by Bamboo Fibers Technology, Lahontan, France. The mean diameter of the bamboo charcoal particles is 22 μm (Fig. 1

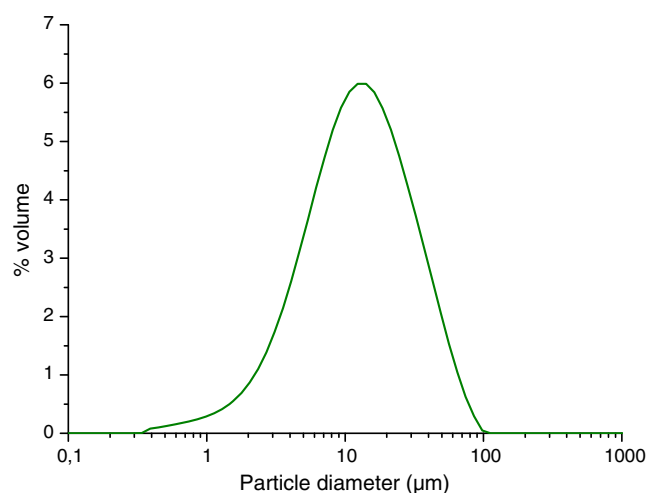


Fig. 1 Particle size distribution of bamboo charcoal particles

and Table 1) with a density of 1.9. The electrical conductivity of the compressed bulk sample of bamboo charcoal was measured between two electrodes with an electrometer Keithley 237 (high-voltage source measure). The electrical conductivity was measured to be 11 S/m. For comparison, the conductivity of a carbon black Ensaco 250 P, measured following the same conditions, was close to 35 S/m. Consequently, the electrical conductive properties of the bamboo charcoal particles are similar to those of conductive carbon black.

Composites preparation

The different composites were prepared by melt processing in an internal batch mixer (Rheomix 600) to obtain EVA composites filled with 20, 30, 35, 40, 50, 60, and 70 wt% of BC fillers. EVA granules were dried in a vacuum oven for 24 h at 50 °C prior to the composite preparation.

The mixing chamber has a capacity of 60 cm³, and the fill rate was fixed to 85 %. The melt dispersion of CB fillers in EVA was carried out at 120 °C for 10 min. The mixing speed of the rotors was set at 50 rpm.

The samples were then compression-molded into 0.6-mm-thick sheets in a Collin P200P press for electrical and rheological analysis. The composite compositions were precisely determined by a thermogravimetric analyzer with a

Table 1 Characteristic size of bamboo charcoal particles

$d_{(10)}$	$d_{(50)}$	$d_{(90)}$	Mean diameter	Specific area
μm	μm	μm	μm	m ² g ⁻¹
3.6	12.7	39.6	22	0.82

TA Instrument (TGA Q500). Samples were heated at 10 °C/min from room temperature to 700 °C in a nitrogen flow of 60 cm³/min.

Measurement of electrical properties

The ac conductivity of the different composites was performed using a TA Instruments DEA 2970 Dielectric Analyzer. The sample is placed between parallel-plate sensors. A maximum force of 250 N was applied to ensure an optimal contact. A sinusoidal voltage of 1 V was applied between parallel plate sensors. The frequencies tested ranged from 10 Hz to 100 kHz with six data points on each decade. The measurements were performed in ambient temperature conditions.

Capacitance and conductance were measured as a function of time, temperature, and frequency to obtain the dielectric constant, permittivity (ϵ'), dielectric loss (ϵ''), and ac conductivity (σ_{ac})

Measurement of rheological properties

The complex shear modulus (storage modulus G' and loss modulus G'' of different composites) was measured using an AR2000 Rheometer from TA Instruments, and 25-mm parallel plate fixtures were used for all the tests with a gap setting of about 0.6 mm. All measurements were performed at 160 °C. The linear viscoelasticity domain of the samples was determined from a stress sweep experiment at the frequency controlled at 6.23 rad s⁻¹. The experiments were performed in dynamic oscillation frequency sweeps

from 100 to 0.001 rad s⁻¹. The applied stress was 80 Pa in the range of 100–0.1 rad s⁻¹ and 150 Pa in the range of 0.1–0.001 rad s⁻¹.

Morphology analysis

A Mastersizer 2000 was used for measuring the bamboo charcoal particle size distribution which is shown in Fig. 1 (see Table 1 for the corresponding values). This distribution shows that $d_{(10)}$, $d_{(50)}$, and $d_{(90)}$ values are 3.6, 12.7, and 40 μm , respectively ($d_{(x)}$ is the diameter at which x (in percent) of the particles are smaller).

The mean diameter of bamboo charcoal particle is 22 μm . The specific surface area was also determined with a Mastersizer 2000 and the value is 0.82 m² g⁻¹. The uniformity coefficient of the sample is then 1.18. This coefficient, defined as the ratio between $d_{(60)}$ and $d_{(10)}$, measures the “width” of the distribution.

The filler particles were also studied by scanning electron microscopy (SEM), HITACHI S800 (HITACHI Corp.). Figure 2 displays the SEM micrographs of bamboo charcoal particles.

SEM images and the size distribution analysis show that bamboo charcoal particles present a rectangular shape with low specific surface area unlike carbon black fillers. The dispersion of these BC particles in the EVA matrix was also evidenced by a SEM observation. The samples were fractured in liquid nitrogen, and the fractured surfaces were gold–palladium-sputtered prior to observation. As a result, Fig. 3 shows the SEM micrographs of bamboo charcoal–EVA composites at different loadings.

Fig. 2 Scanning electron micrograph of bamboo charcoal particles

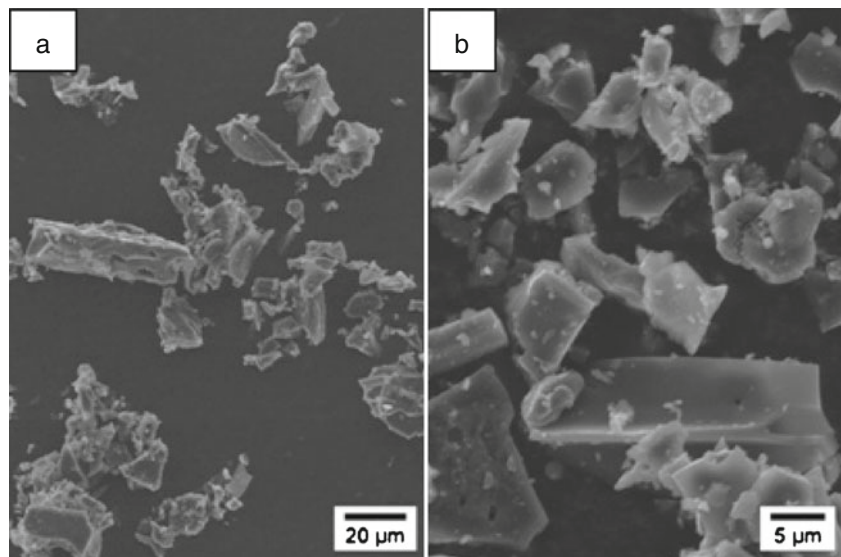
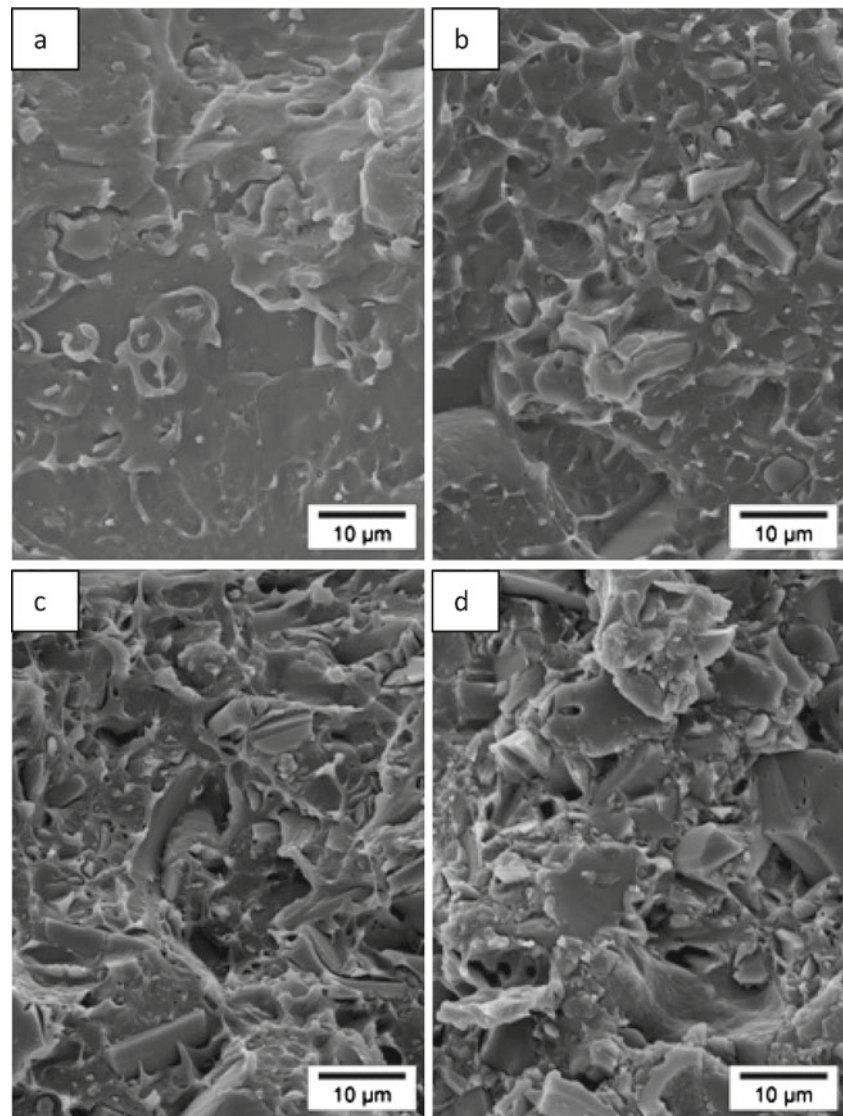


Fig. 3 SEM images of bamboo charcoal particles dispersed in EVA matrix at different loadings: **a** 11 vol% (20 wt%), **b** 25 vol% (40 wt%), **c** 33 vol% (50 wt%), **d** 54 vol% (70 wt%)



Results and discussion

Electrical properties

For different loading of bamboo charcoal, Fig. 4 shows the evolution of the ac conductivity as a function of frequency. At low filler concentrations, the electrical response of the composite is equivalent to the matrix response, which is an insulator because there is no possible contact between particles. At a critical volume fraction of filler around 0.2, the conductivity increases abruptly due to the formation of a conductive filler network (conductive path). The composite becomes electrically conductive. This critical volume fraction is defined as the electrical percolation threshold.

On the other hand, the electrical percolation threshold is observed when the ac conductivity is independent

of the frequency. This can be explained by the following equation (Jonscher 1977; Dyre and Schroder 2000; Barrau et al. 2003; Leyva et al. 2003; Dang et al. 2004; Sohi et al. 2011):

$$\sigma_{ac} = \sigma_{dc} + 2\pi f \varepsilon'' \quad (1)$$

where σ_{ac} is the ac conductivity, σ_{dc} is the dc conductivity, f is the frequency, and ε'' is the loss factor. σ_{dc} is the parameter that defines the ionic/electronic conductivity. It is nonfrequency dependent.

Consequently, when the composite is conductive, this component becomes predominant and σ_{ac} becomes nonfrequency dependent. Below 20 vol% of bamboo charcoal, the ac conductivity is dependent on the frequency showing that the conductivity is only due to the polarization. Beyond 20 vol%, the ac conductivity becomes frequency independent due to the conductive filler network. So, the

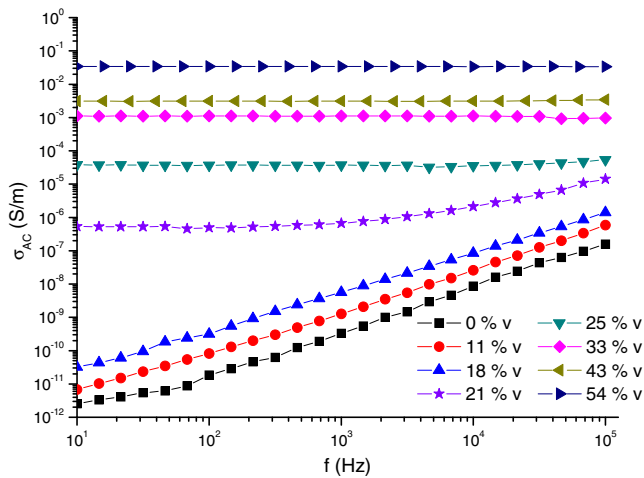


Fig. 4 ac conductivity versus frequency for different volume percent of bamboo charcoal

dc conductivity is dominant. The electrical percolation can be clearly evidenced from the variation of dc conductivity versus the filler’s concentration as shown in Fig. 5.

According to the literature (Stauffer 1979; 1985; Balberg 2002; Mierczynska et al. 2007; Bauhofer and Kovacs 2009; Bose et al. 2009; Dang et al. 2011; Levchenko et al. 2011), a power law can be derived above the percolation threshold:

$$\sigma(\Phi) \propto (\Phi - \Phi_c)^t \tag{2}$$

where Φ is the volume fraction of filler, Φ_c is the percolation threshold, and t is the critical exponent. According to Fig. 5, we derive the following values of Φ_c and t : $\Phi_c = 0.18$ (30 wt% of bamboo charcoal) and $t = 4.1$.

For spherical fillers, theoretically, $\Phi_c = 0.16$ and t is in the range of 1.6–2 (Dang et al. 2011). Kovacs et al. (2007) suggest that t increases with the percolation threshold. In

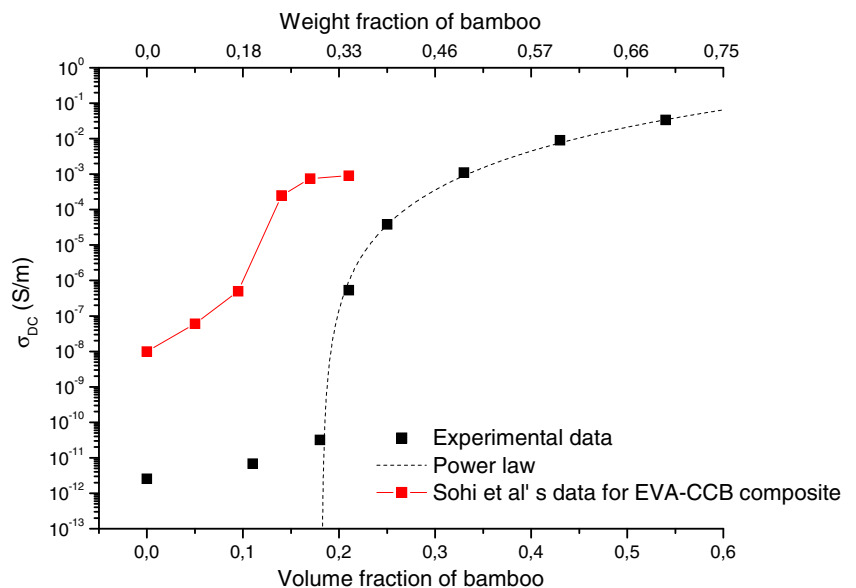
another study, Bauhofer and Kovacs (2009) suggest that the high value of t is due to the high tunneling barriers between fillers.

Balberg (1987) proved that this exponent t depends strongly on the geometrical properties of the filler. For example, Balberg (2002) found a value of $t = 6.4$ for polyethylene/carbon black composites with a low structure of carbon black (spherical shape with a large diameter giving a very low specific surface area). However, the high value of t could also be due to a heterogeneous filler distribution in the composite. On the other hand, Sohi et al. (2011) showed that the percolation threshold of a carbon black–EVA 2803 composite was observed at $\Phi_c = 0.14$. Compared with the present results, this slight difference is also probably due to the low specific area of bamboo charcoal ($0.82 \text{ m}^2 \text{ g}^{-1}$) compared with the one of CB used in their work ($\approx 240 \text{ m}^2 \text{ g}^{-1}$).

Beyond the percolation threshold, a considerable increase in electrical properties was observed with the concentration of bamboo charcoal till reaching the limit value of 10^{-2} S m^{-1} . Sohi et al. observed that beyond the percolation (from $\Phi = 0.17$), the conductivity of a CB–EVA 2803 composite was asymptotic at 10^{-3} S m^{-1} as shown in Fig. 5 for a comparative purpose. Consequently, when the conductive filler network is formed, the conductivity of the EVA composite is better with bamboo charcoal than with carbon black while the electrical conductivity of the CB ($\sim 500 \text{ S m}^{-1}$) used in a study by Sohi et al. is 50 times higher than the bamboo charcoal one ($\sim 11 \text{ S m}^{-1}$). Furthermore, as shown in Fig. 5, the variation of the electrical conductivity at the percolation threshold is straightforward for bamboo charcoal.

To sum up, the present work proves that bamboo charcoal can be a challenging and competitive filler for improving the

Fig. 5 Variation of the dc conductivity of the composite versus the volume fraction of bamboo charcoal. The dashed curve is the power law according to Eq. 2. For comparison with carbon black, the result of Sohi et al. (2011) have been plotted



electrical properties of polymer composites. Indeed, bamboo charcoal is as efficient as carbon black to be used as a conductive filler.

Rheological properties

The frequency dependence of the storage modulus $G'(\omega)$ and the loss modulus $G''(\omega)$ for different bamboo charcoal loadings is shown in Fig. 6a, b, respectively. As expected, the addition of bamboo charcoal considerably modifies the viscoelastic behavior of the molten EVA composite. However, at the lowest filler concentrations, ($\Phi = 11, 18,$ and 25 vol%), the composites have approximately the same response than the EVA matrix except in the low-frequency domain ($\omega < 0.01$ rad s^{-1}) where a second relaxation zone is clearly evidenced by the variation of the storage modulus. In this domain of relaxation, the loss modulus dominates the storage modulus and it can be then admitted that the percolation threshold is higher than the bamboo charcoal concentration of 25 vol%. The presence of this second

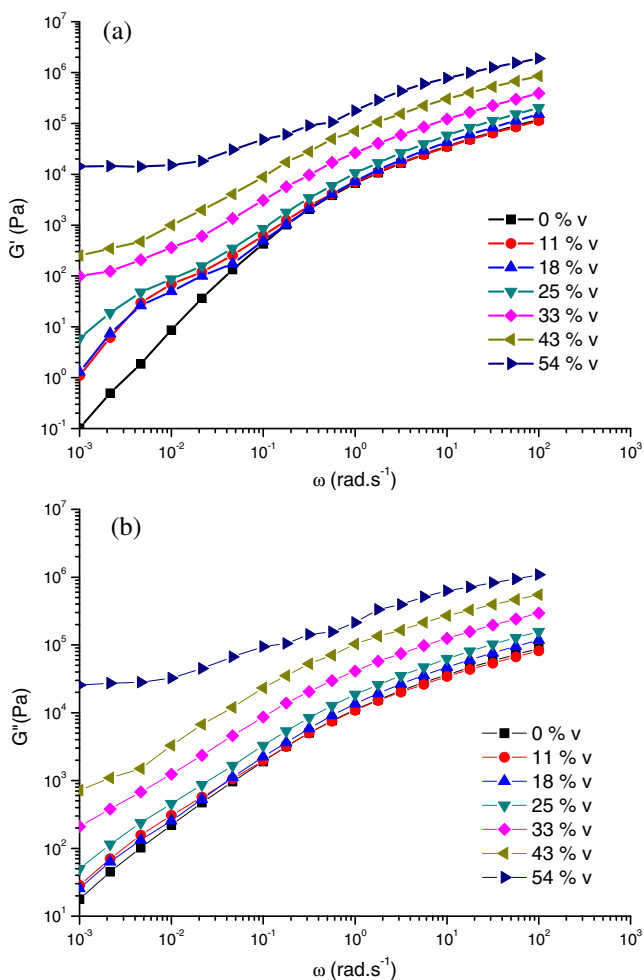


Fig. 6 Variation of storage G' (a) and loss G'' (b) modulus versus frequency for different bamboo charcoal loadings

relaxation domain ($\lambda \sim 200$ s) which appears in the low-frequency zone ($\omega\lambda = 1$) of the complex shear modulus can be explained by an adsorption of polymer chains on the bamboo charcoal surface.

Such phenomenon is well known in carbon black-filled rubbers and is called bound rubber (Choi 2002; 2004; Qu et al. 2011). According to the work of Vignaux-Nassiet et al. (1998), chain adsorption creates a monomolecular layer whose thickness is comparable to the bulk radius of gyration of the chains and whose relaxation time scales in the same way as branches of star polymers. More recently, Carrot et al. (2012) proved that this type of relaxation is also the trace of the existence of an effective interfacial tension between the agglomerates containing high amounts of filler and the polymer matrix.

By increasing the bamboo charcoal loading, filler–filler and filler–polymer networks are formed and the appearance of a transition from a liquid-like behavior to a solid-like behavior is expected (Krishnamoorti and Giannelis 1997; Du et al. 2004; Zhou et al. 2007; Cassagnau 2008; Leboeuf et al. 2008; Bose et al. 2009; Chapartegui et al. 2010; Song and Zheng 2011; Sumfleth et al. 2011) from the apparition of a secondary plateau of the storage modulus at low frequencies which is obviously higher than the loss modulus. This secondary plateau of the storage modulus G' is clearly observable for 50, 60, and 70 wt% (33, 43, and 54 vol%, respectively) filler contents. Thus, we can say that the sol–gel transition, which defines the rheological percolation threshold, appears between a 0.25 and 0.33 volume fraction (40 and 50 %wt, respectively) of bamboo charcoal in the EVA matrix. On the other hand, the change from liquid-like to solid-like behavior due to the formation of the filler–filler and filler–polymer network is also observable in Fig. 7 from the variation of the absolute complex viscosity

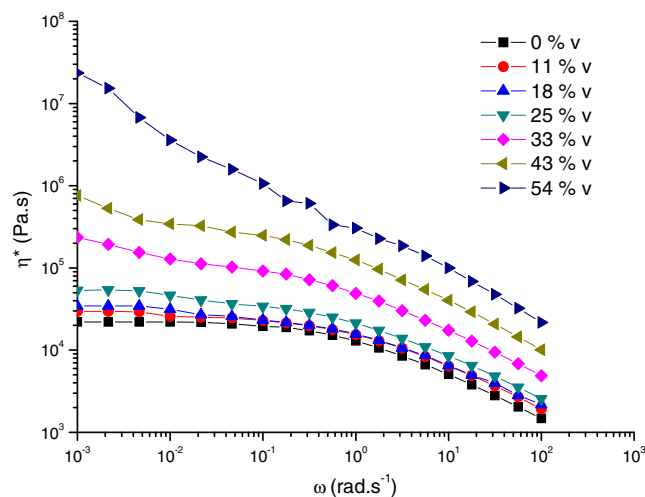


Fig. 7 Variation of the absolute complex viscosity $|\eta^*|$ versus frequency for different bamboo charcoal loadings

versus frequency. Actually, the solid-like behavior response is subsequently an apparent yield stress and a shear-thinning dependence on viscosity. According to Larson (1999), it was suggested that this yield behavior is due to filler–filler interactions.

From a quantitative point of view, Lertwimolnun and Vergnes (2006) used the Carreau–Yasuda model with yield stress to describe the frequency dependence of the absolute complex viscosity of nanocomposites:

$$|\eta^*(\omega)| = \frac{\sigma_0}{\omega} + \eta_0 \left[1 + (\lambda\omega)^a \right]^{\frac{n-1}{a}} \tag{3}$$

where σ_0 is the yield stress, η_0 is the zero shear viscosity, λ is the relaxation time, a is the Yasuda parameter, and n is the dimensionless power law index. Above the rheological percolation threshold, η_0 is not observable but is theoretically found with the Carreau–Yasuda’s model.

According to Carrot et al. (2010), the matrix mode behavior of the composite can be described by multiplying the viscosity of the virgin EVA matrix by a function describing the hydrodynamic interactions in the form of that proposed by Krieger and Dougherty (1959) and Krieger (1972). Assuming that the hydrodynamic interactions dominate at the high frequencies, the concentration dependence of the viscosity was then derived by applying the Krieger–Dougherty equation at $\omega = 100 \text{ rad s}^{-1}$:

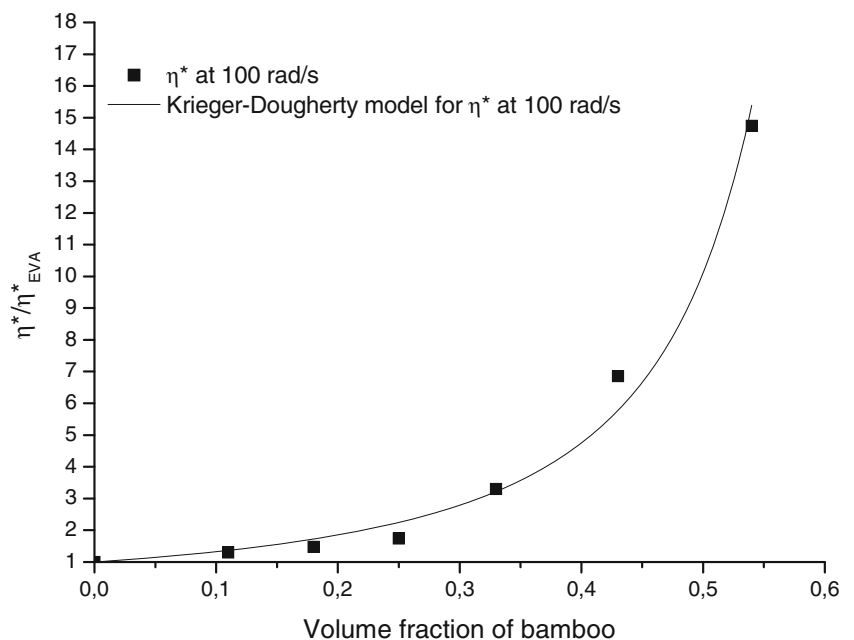
$$\eta_c = \eta_m \left[1 - \frac{\Phi}{\Phi_m} \right]^{-[\eta]\Phi_m} \tag{4}$$

where η_m is the viscosity of the matrix, Φ_m is the maximum packing fraction of the particle at which the viscous flow can occur, and $[\eta]$ is the intrinsic viscosity. We finally found $\Phi_m = 0.69$ and $[\eta] = 2.6$. These values mean that

the arrangement of bamboo charcoal particles is random and that their particle size is fairly homogeneous ($\Phi_m = 0.64$ for a random close packing and $\Phi_m = 0.74$ for a closest packing) (German 1989; Pishvaei et al. 2006). Actually, for ideal spherical particles $[\eta] = 2.5$ according to the Einstein law. The variation of the relative viscosity of the composite versus the volume fraction of particles is shown in Fig. 8.

To model the variation of the absolute complex viscosity according to Eq. 3, the dependence of the zero viscosity on the filler concentration was derived from the Krieger–Dougherty equation (Eq. 4). Actually, Eq. 3 expresses the complex behavior of the filler concentration dependence of the viscosity versus frequency. The modeling was divided in two parts depending on the concentrations below or beyond the percolation threshold. Figure 9a shows that below the percolation threshold, the hydrodynamic interactions are the dominant ones as a simple Carreau–Yasuda model ($\sigma_0 = 0$) fits well the variation of the absolute complex viscosity. However, as previously discussed, a second mechanism of relaxation is observed due to chain adsorption on the bamboo charcoal surface. This second mechanism of relaxation cannot be depicted by the present model, a second relation domain should be added in the Carreau–Yasuda equation. Beyond the percolation threshold, Fig. 9b shows that Eq. 3 fits perfectly the yield stress and shear thinning behavior of the EVA composites. The values of the yield stress are reported in Table 2. As expected, the yield stress increases with increasing bamboo charcoal concentration. Actually, this model means that the hydrodynamic interactions dominate in the high-frequency range whereas the particle–particle interactions are dominant in lower frequency due to the formation of the particle network.

Fig. 8 Variation of the relative viscosity η_c/η_m as a function of the particle volume fraction at 100 rad/s. The solid line shows the fit according to Krieger–Dougherty’s model (Eq. 4)



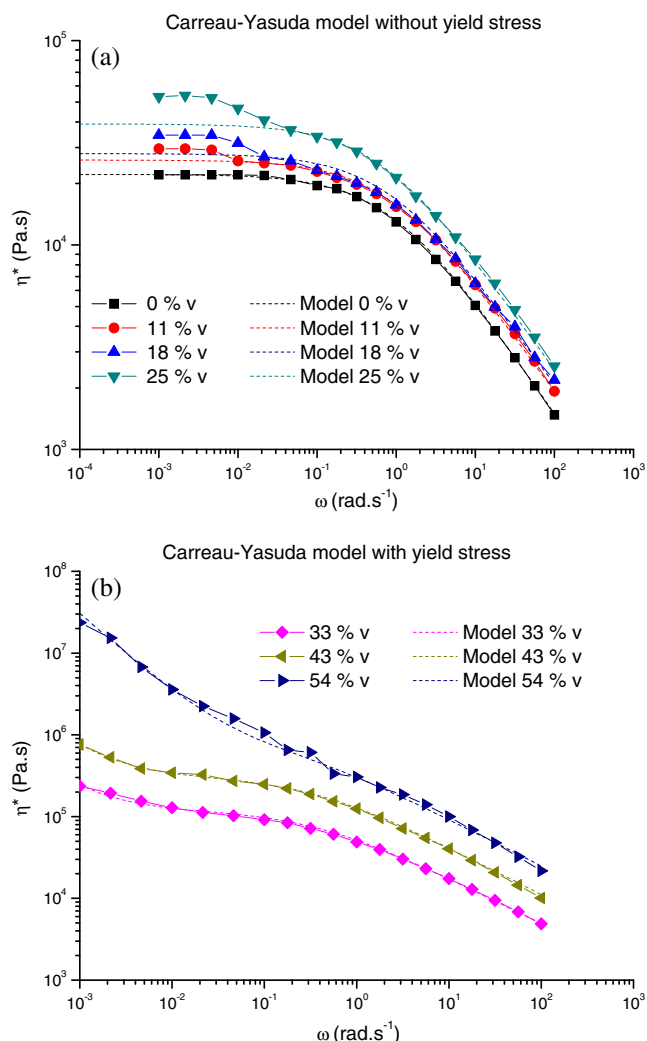


Fig. 9 Modeling of the variation of the absolute complex viscosity using the Carreau–Yasuda’s model (*dashed line*) at different bamboo charcoal concentrations. **a** Concentrations lower than the percolation threshold, $\sigma_0 = 0$. **b** Concentration higher than the percolation threshold, the model parameters are reported in Table 2

Finally, it can be concluded that the formation of the three-dimensional filler network, defined as rheological percolation, appears at nearly $\phi_R \approx 0.3$ volume fraction of bamboo charcoal. This rheological percolation threshold must be compared to the electrical percolation threshold $\Phi_c \approx 0.18$ as determined in the first part of the present

work. This strong difference could be explained by the filler–filler distance required to induce electron transfer in the first case and stress transfer in the latter (Huang et al. 2012). An electron may tunnel quantum mechanically between conductive fillers if the distance between each is less than 10 nm (Sherman et al. 1983; Zou et al. 2002). But this distance is too high for interaggregate polymer layers transfer the stress from a load to another. This is principally due to the filler’s characteristics such as the specific surface area, the aspect ratio, and other surface properties. For carbon nanotube composites, the rheological percolation threshold is smaller than the electrical percolation threshold due to filler’s shape and filler’s large surface areas (Lee et al. 2007; Martins et al. 2010; Poetschke et al. 2010; Sumfleth et al. 2011). In the case of carbon black composites, the rheological percolation threshold is higher than the electrical percolation threshold due to the filler’s spherical shape and its low surface areas. In fact, a filler–polymer network is more difficult to form than conductive network (Poetschke et al. 2010; Sumfleth et al. 2011; Huang et al. 2012). However, the bamboo charcoal particles do not have an aggregate structure like CB aggregates. With the bamboo charcoal particles having a large size (mean diameter of 22 μm and specific surface area of 0.82 $\text{m}^2 \text{g}^{-1}$) and a sample of 600- μm thickness, only some particles need to make contact between each other or need to be spaced less than 10 nm to create a conductive pathway across the sample. Actually, in the case of microscale fillers, only a few conductive pathways are necessary for the sample to become conductor whereas much more contact is necessary to modify the viscoelastic behavior from a liquid to a yield stress fluid.

Conclusion

The electrical and rheological properties of an EVA copolymer filled with bamboo charcoal particles were investigated. SEM and size distribution analysis showed that bamboo charcoal particles present a rectangular shape with a low specific surface area (0.82 $\text{m}^2 \text{g}^{-1}$).

The electrical percolation threshold was observed at $\Phi_c \approx 0.18$ volume fraction of bamboo charcoal composite contents. Furthermore, the maximum of the electrical conductivity at higher concentration was observed around

Table 2 Parameters of the Carreau–Yasuda’s model including yield stress (Eq. 3)

Vol% of bamboo	0	11	18	25	33	43	54
σ_0 (Pa)	–	–	–	–	130	460	30,000
η_0 (Pa s)	2.2×10^4	2.6×10^4	2.8×10^4	3.9×10^4	1.2×10^5	3.1×10^5	6.5×10^5
λ (s)	1.1	1.12	1.2	1.5	2.9	3.0	3.1
a	0.8	0.8	0.8	0.8	0.83	0.83	0.83
n	0.44	0.45	0.46	0.45	0.44	0.43	0.43

10^{-2} S/m. This level of electrical conductivity proves that bamboo charcoal is as efficient as carbon black and carbon nanotubes for electrical applications of polymer composites.

The rheological percolation threshold is more difficult to define from a relevant criteria point of view. The yield stress behavior of the absolute complex viscosity was used as the rheological criteria to determine the percolation threshold. Furthermore, the rheological behavior was successfully modeled from a modified Carreau–Yasuda equation. The rheological percolation was found at $\Phi_R \approx 0.3$ volume fraction of bamboo charcoal composite contents.

Finally, this work shows that electrical percolation occurs before rheological percolation. This difference can be explained from the microscopic scale of the bamboo charcoal particles. As a result, bamboo charcoal confers high electrical properties to the polymer matrix without inducing strong changes in its viscoelastic properties.

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