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MECHANICAL PROPERTIES OF BAMBOO POWDER REINFORCED ETHYLENE PROPYLENE DIENE MONOMER (EPDM) COMPOSITES: EFFECT OF FILLER LOADING AND PARTICLE SIZE

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ABSTRACT

Due to the light weight, high specific strength and non hazardous nature of bamboo fiber, it is preferred over synthetic fibers in composite materials for a wide range of applications such as automotive industry and including household sectors. As was noticed, little attention has been given to the effect of bamboo powder on the mechanical properties of rubber composites. Hence, an attempt has been made in this paper to the study the effect of loading and particle size of bamboo powder on the mechanical properties of EPDM composites. Thermo-gravimetric analysis (TGA) was carried out to study the thermal stability of composites. Results indicated that the thermal stability of EPDM was further improved with increasing in bamboo loading and decreasing in particle size. The stress- strain curves of the composites were studied and fitted according to Ogden's model. Mechanical parameters for the studied composites were improved with increasing bamboo loading. Besides, properties such as rupture stress, and internal friction were found to be maximum for composites containing certain content of bamboo powder, depending upon its particle size. Moreover, composites containing the smallest particle size of powder, at all levels of bamboo loading, showed mechanical properties superior to all other composites. From the dynamic mechanical measurements, the dynamic modulus, internal friction, and thermal diffusivity were calculated. The observed variations were explained in view of the role played by both the loading level and the particle size of bamboo powder. These findings were supported by scanning electron microscopy (SEM) micrographs.

Keywords

(Bamboo powder; EPDM-rubber, Composite; Thermo-gravimetric analysis; Stress- strain; Dynamic mechanical properties)

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INTRODUCTION

The use of natural fiber for the reinforcement of the polymer composites has received increasing attention both in academic sector and in industry. Numerous studies have been carried out on the utilisation of natural fillers such as flax, hemp, jute straw, wood, rice husk, wheat, cane (sugar and bamboo), kenaf, oil palm empty fruit bunch, sisal, coir, banana fiber, and cellulose fibres as reinforcement materials [1-10].

Bamboo is one of the agricultural crops which can be exploited for the design and development of polymer composites [11]. Many previous studies have shown that the addition of bamboo fiber improves morphology and mechanical properties of polymer composites. Short bamboo fibers reinforced epoxy composites and their chemical resistance and tensile properties with fiber length have been studied [12]. Researchers studied the effect of fiber length on the mechanical properties of polymer composites by using starch resin and short bamboo fibers [13]. Flexural properties of bio-based polymer composites made from bamboo and biodegradable resin were evaluated in comparison with kenaf composites [14]. The effects of bonding agent on the curing characteristics, morphology and mechanical properties of bamboo fiber reinforced natural rubber composites have been studied [15]. Morphological and mechanical properties of bamboo flour filled high density polyethylene (HDPE) based composites were investigated in respect of crystalline nature of maleated elastomer modifier[16]. Morphological, and thermal properties of Polypropylene/polylactic acid/bamboo fiber composites were investigated and compared with those of neat polymers [17]. Thwe et al. [18] have investigated the effect of environmental aging on the mechanical properties of bamboo-glass fiber reinforced polymer matrix hybrid composite. The mechanical properties (tensile strength, elastic modulus, flexural strength and flexural modulus) of untreated and alkali-treated bamboo matting-reinforced epoxy composites were investigated [19]. The effects of alkalization and fiber loading on the mechanical properties and morphology of bamboo fiber composites have been studied [20]. The study aimed to evaluate the influence of caustic concentration on the mechanical properties of bamboo-strip-reinforced resol composites with a constant 50% loading of the reinforcement. The maximum improvement in the properties was possibly achieved with 20% acoustically treated reinforcements. A correlation was found to exist between the mechanical properties and the morphology that developed. Shibata[21] has investigated the relationship between processing pressure, temperature, holding time, and the flexural properties of bagasse and bamboo plastic composites. The optimum fiber volume fractions, which indicated the maximum flexural values, were found to be in the range of 75% to 80% for both bagasse and bamboo plastic composites.

To our knowledge, the effect of particle size of bamboo powder on the dynamic mechanical properties of rubber composites has not been undertaken deeply. In this study, bamboo powder was used as a filler to produce biocomposites based on ethylene propylene diene monomer (EPDM) rubber. In this work, the effect of both the loading level and the particle size of bamboo powder on the static and dynamic mechanical, thermal, and microstructural properties were investigated.

EXPERIMENTAL WORK

Materials and Samples Preparation

Ethylene propylene diene monomer (EPDM, Grade 4045), used as a host material in this study and filled with different ratios of bamboo powder, was purchased from Jilin Chemical Industry (China). The bamboo, used as reinforcing filler, was collected from Egypt. Bamboo powder of different particle sizes was obtained using a laboratory scale extraction technique. Cylindrical bamboo columns without nodes were cut into small pieces and soaked in water for 24 h. After soaking the columns were manually sliced into small chips with a knife. Bamboo powder was obtained by grinding bamboo chips with a high speed blender for approximately half an hour. Ground bamboo powder with four different size ranges: (1) $\leq 400\mu m$, (2) $\leq 250\mu m$, (3) $\leq 150\mu m$ and (4) $\leq 90\mu m$ were obtained using sieves of different aperture sizes. The extracted powder was dried in an oven at 100 ^{o}C for 72 h in order to remove moisture. The composites with those powder sizes are designated as A_x, B_x, C_x, and D_x, respectively. Where x stand for the concentrations of bamboo in phr (part per hundred parts of rubber by weight); x = 0, 10, 15, 20, 25, 30 and 40 phr. Chemical compositions of bamboo powder used in this study are given in Table 1.

Table 1. Chemical compositions of bamboo.

Chemical composition	(%)
Holo-cellulose	67.6
Lignin	21.3
Hot water solubles	6.1
Alcohol- toluene solubles	3.5
Ash	1.3

Other chemicals such as sulphur used as vulcanizing agent, zinc oxide as vulcanizing activator, stearic acid used to improve the dispersion of ZnO in elastomer and tetramethylthiuram disulfide (TMTD) as ultra-accelerator and vulcanizing

agent were all purchased from Transport and Engineering (Rubber Manufacturing) Company, Alexandria, Egypt. Flectol H (poly-1,2-dihydro-2,2,4- trimethylquinoline) used as an antioxidant and stabilizer was supplied by Monsanto (M) Company. Bonding agents used in this study were phenol formaldehyde [Borden Chemical (M) Ltd] and hexamethylenetetramine [Fluka Chemical (M) Ltd]. All samples were prepared according to the recipe presented in Table 2.

Table 2. Recipe for bamboo powder reinforced ethylene propylene diene monomer (EPDM) composites(*).

Ingredients	Ax	Bx	Cx	Dx
EPDM	100	100	100	100
Stearic acid	2	2	2	2
Zinc oxide	5	5	5	5
Processing oil	20	20	20	20
Flectol H	1	1	1	1
Phenol formaldehyde (PF)	10	10	10	10
Hexamethylene tetramine (Hexa)	5	5	5	5
TMTD	2	2	2	2
Sulphur	3	3	3	3
Bamboo powder	Varied (0 - 40)	Varied (0 - 40)	Varied (0 - 40)	Varied (0 - 40)

(*) All values are expressed as (phr).

The rubber composites were prepared on a two rolls mill of 300 mm length, 170 mm diameter with speed of slow roll (18 rev per min.) and gear ratio (1.4). After mixing, the rubber compositions were left for about 24 hours and then molded in an electrically heated hydraulic press at 160 ± 2 ^{o}C under a pressure of 4 MPa and the time of vulcanization was 20 min.

Testing

Thermo-gravimetric analysis (TGA) was carried out using Shimadzu TGA-50H thermogravimeter analyzer and the sample was heated from 25 °C to 600 °C at a rate of 10 °C /min in an inert nitrogen atmosphere.

Stress-strain measurements were performed on dumbbell shaped samples of constant width (3 mm) and thickness of about (1 mm). Stress–strain measurements were conducted according to ASTM D 412 with a tensile testing machine (AMETEK, USA). A digital force gauge (Hunter Spring ACCU Force II, 0.01 N resolution, USA) connected to a microprocessor was used to measure extension force. A home-made motor attachment was used to control the displacement rate through a gearbox. Calibration of this part was previously carried out by comparing the obtained results with those of similar samples tested on other standard equipments. The displacement rate was preset using a variable DC power supply, and was measured using a micro-switch attached to the apparatus wheel. The accuracy of displacement measurement was about ± 0.1 mm. The displacement rate throughout the experiment was fixed at 1 mm/sec. Three pieces of the same sample were used and the results were averaged for each data point. From the tensile data, the following parameters were calculated as follows follows [22]

$$\lambda = L / L_{\circ} \quad , \quad \sigma = F / A_{\circ} \quad , \quad \varepsilon_T = \ln \lambda \ , and \ \ \sigma_T = \sigma\lambda \qquad (1)$$

where λ is the extension ratio; L, and L_o are the lengths of the deformed and nondeformed samples, respectively; σ is the nominal stress; F is the force; A_o is the area of the unstrained sample; ε_T is the true strain; and σ_T is the true stress.

The dynamic elastic modulus (Y_d), internal friction (Q^{-1}) (taken as the ratio of energy dissipated as heat to the maximum energy stored in the material during one cycle) and thermal diffusivity (D_{th}) measurements were carried out by the application of the dynamic resonance technique described elsewhere follows [22]. The mechanical parameters were obtained as follows follows:

$$Q^{-1} = \Delta f \ / \sqrt{3} f_{\circ} \quad , \quad D_{th} = 2d^2 f_{\circ} / \pi \ , \quad Y_d = (2\pi L^2 f_{\circ} / K\, Z^2)^2 \rho \qquad (2)$$

where $f_{\circ}$ is the resonance frequency, Δf the full width at half-maximum, d is the thickness of the sample, K is the radius of gyration of the cross-section about the axis perpendicular to the plane of vibration, the constant Z depends on the mode of vibration and equals 1.8751 for the fundamental mode, ρ is the density of the sample, and L is the length of the vibrating part of the sample.

ISSN 2347-3487

Scanning electron microscopy (SEM) was used to investigate the surface morphology of the bamboo fiber/EPDM rubber composites. All samples were coated with a thin gold layer prior to SEM examination, and then examined under an electron microscope (model JEM-850, JEOL), operated at 15 kV. The SEM data were obtained using Leica Qwin 500 Image Analyzer Computer System (England).

RESULTS AND DISCUSSIONS

Thermal stability of bamboo fiber/EPDM vulcanizates

Thermal decomposition of the D_x composite specimens (bamboo particle size $\leq 90\mu m$, with EPDM rubber) are shown in Fig. (1a). There is no obvious variation at the onset temperature of thermal degradation for different specimens. With increasing bamboo content, a shift of rapid degradation region towards higher temperature can be seen at temperature range (>400 C).

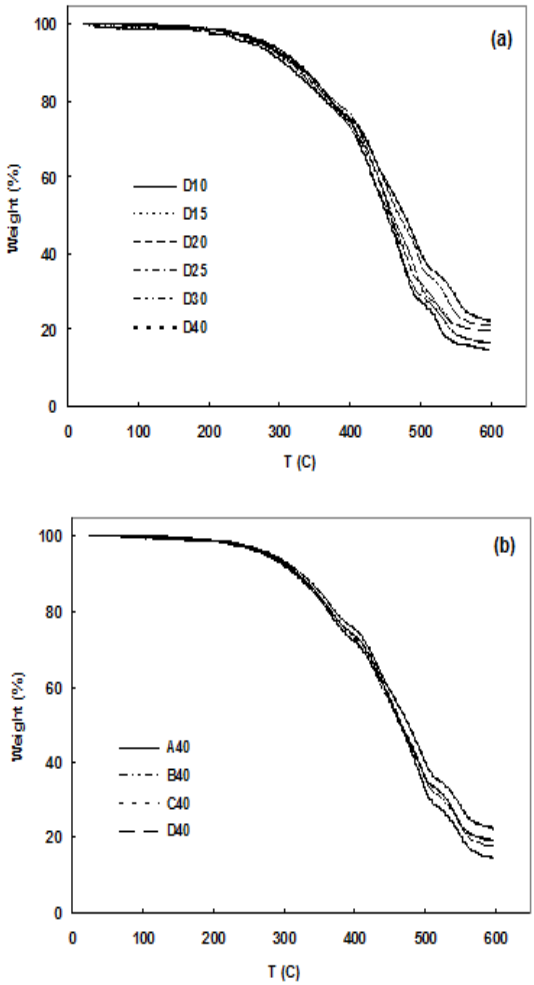

Fig 1: Thermo-gravimetric curves (TGA) of (a) composites D_x with varying bamboo powder content and (b) composites A_{40}, B_{40}, C_{40} and D_{40}.

The percentage weight loss data of the studied composites at 500, and 600 ^{o}C are given in Table 3, which clears that weight loss of the studied composites, at the same temperature decreases with increase of bamboo content. The reason is that bamboo particles can impose restriction on the mobilization of molecular polymer chains in the heating environment. Besides, the particle size of bamboo powder was found to affect the thermal stability of the composites.Fig. (1b) shows the TGA curves of A_{40}, B_{40}, C_{40} and D_{40} composites (different particle size, 40 phr of bamboo with EPDM rubber). It is clear that, decreasing the particle size of bamboo powder shifts the rapid degradation region towards higher temperature in the temperature range (>400 ^{o}C). Also, the weight loss of the studied composites, at the same temperature, decrease with the decrease of bamboo particle size: i.e., from 66.9% for A_{40} composites to 64.2% , 63.4% and 59.5% for B_{40}, C_{40} and D_{40} composites at 500^{o}C, respectively. This could be attributed to the fact that, as the particle size of bamboo powder decreases its surface area increases, thereby increasing the physical and chemical crosslinking points between bamboo particles and rubber matrix. Therefore, the thermal stability of EPDM was further improved with increasing bamboo loading and decreasing in particle size.

ISSN 2347-3487

Table 3. Percentage weight loss data of the studied composites at 500 and 600 ^{o}C

Sample	Weight loss (%) at 500 ^{o}C	Weight loss (%) at 600 ^{o}C
D_{10}	72.5	85.1
D_{15}	71.3	84.0
D_{20}	68.4	83.7
D_{25}	67.7	80.7
D_{30}	62.8	79.3
D_{40}	59.5	77.7
A_{40}	66.9	85.6
B_{40}	64.2	82.5
C_{40}	63.4	80.9

Mechanical Properties

Static mechanical properties (stress - strain behavior)

Mechanical properties in filled rubber vulcanizates can be explained in view of the filler content and/or particle size. Fig. (2), depicts the stress - strain curves for bamboo powder /EPDM composites. It is clear that, the stiffness as governed by the slope of the initial linear part increases with increasing the concentration of bamboo which acts as reinforcing filler. Accordingly, the elongation at break decreases.

The energy absorbed per unit volume (W) in deforming the rubber composites to a strain ε is simply the area under the stress - strain curve and can be written as [23]

$$W = \int \sigma(\varepsilon) d\varepsilon \qquad (3)$$

where $\sigma(\varepsilon)$ is the stress as a function of the strain. Obviously, the higher the stress – strain curve, the higher is the energy absorption capacity.

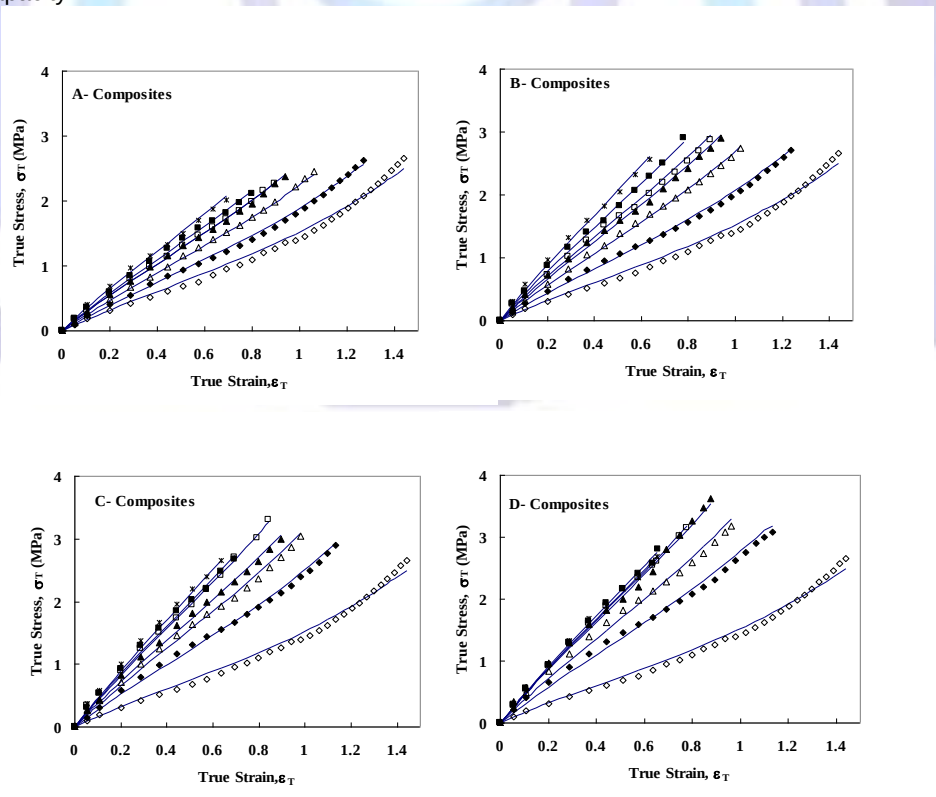

Fig 2: True stress vs. true strain of bamboo powder/EPDM rubber composites with different content of bamboo [◇ 0 phr, ♦10 phr, △15 phr, ▲20 phr, □25 phr, ■30 phr, and ✳ 40 phr].

ISSN 2347-3487

The mechanical stress-strain parameters; the absorbed energy W(MJ/m^3 at 64% strain), Young's modulus Y, rupture stress σ_R and rupture strain ε_R of the samples as deduced from the stress-strain curves of Fig. (2), were found to be appreciably affected by varying bamboo content and particle size.

Fig. (3), shows the bamboo content dependence of these parameters. From Fig. (3) it is evident that the values of W and Y increased with increasing in bamboo loading and decreasing in particle size [Fig. (3a,b)]. The increase in these parameters was because with the increase in bamboo loading, the extent of bonding between the bamboo particles and the matrix increased, and the vulcanizates became stiff. However, peaks in the σ_R – bamboo content graph were observed, and a shift in the peak position towards higher bamboo content was observed with increasing bamboo particle size [Fig. (3c)].

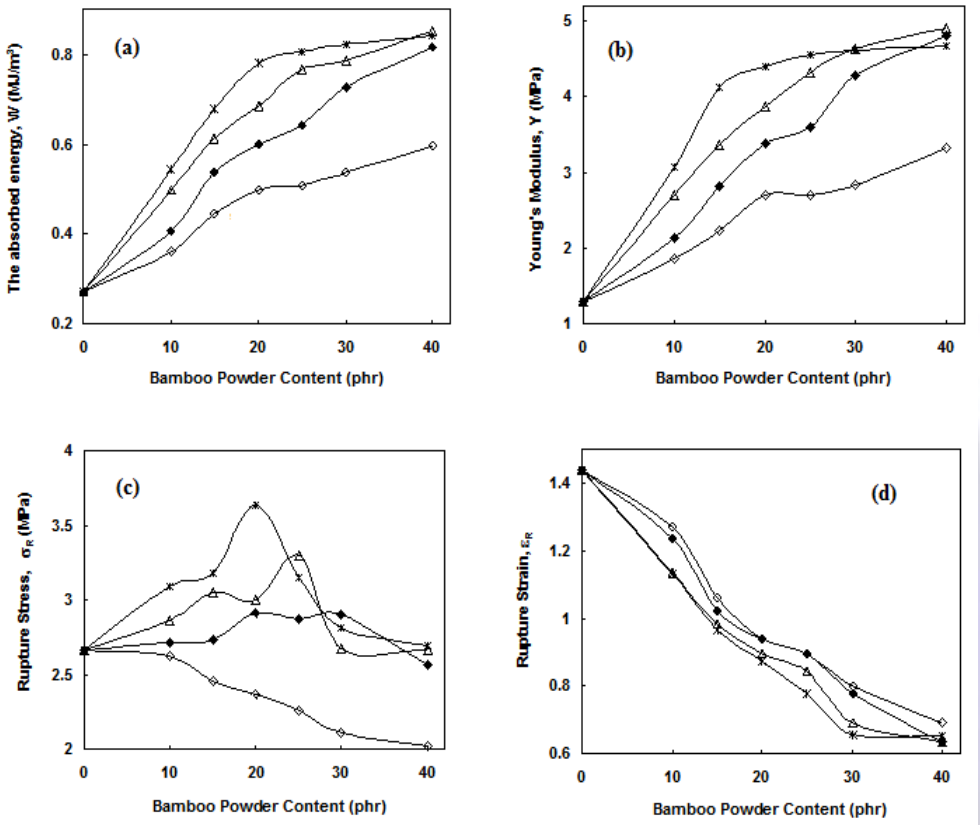

Fig 3:Variation of (a) the absorbed energy, (b) Young's modulus, (c) rupture stress and (d) rupture strain with bamboo powder loading for bamboo/EPDM rubber [◇ A_x , ◆ B_x , △ C_x , and ✳ D_x] composites.

These peaks were observed for D_{20} composite (particle size $\leq 90\mu m$), C_{25} composite (particle size $\leq 150\mu m$) and B_{30} composite (particle size $\leq 250\mu m$). As the vulcanizates became more and more stiff with increases in bamboo loading, the values of σ_R gradually decreased. Because of the increase in bamboo concentration, stress transmission from matrix to fiber was very difficult. Therefore, the continuity of the matrix phase was disturbed by the increase of bamboo content, which was corroborated by the SEM micrograph. Moreover, these parameters for the studied composites, at all levels of bamboo loading, were increased with decrease of the bamboo particle size. As the particle size of bamboo decreases its surface area increases. This consequently leads to stronger adhesion at the bamboo/matrix interface. So the stress transfer becomes more efficient and consequently enhances these parameters. On the other hand rupture strain ε_R shows a reduction with increasing bamboo loading (Fig. (3d)). Increasing bamboo loading in the rubber matrix caused composites to become stiffer and harder. This will reduce composites resilience and toughness and lead to lower resistance to break. Flink et al. [24], Akhtar et a1. [25] and H. Ismail et al. [26] also reported similar observations. Besides, the values of ε_R decreased with decrease of the bamboo particle size at similar loading. This is due to better strength and stiffness achieved from strong adhesion between bamboo particles and rubber matrix. Consequently, the chain mobility decreased, resulting in lower rupture strain ε_R.

The stress - strain curves were fitted by the Ogden model [27], Eq. (4), and plotted in Fig. (2).

$$\sigma_T = \frac{2\mu}{\alpha}\left((1+\varepsilon)^{\alpha-1} - (1+\varepsilon)^{-(\frac{\alpha}{2}+1)} \right) \qquad (4)$$

where σ_T is the true stress, ε is the nominal strain in the loading direction, μ represents the shear modulus and α is a material parameter depending on the host polymer. These two parameters can be determined by fitting experimental stress - strain curve. It was found that the fitting parameter α is 2.04. Fig. 4 represents the variation of shear modulus (derived from fitting of the stress– strain curves by Ogden model, Eq. (1) with bamboo powder loading for bamboo/EPDM rubber composites. Fig. (4), shows that the shear modulus, μ follow a similar trend to the parameters, W and Y. The decreasing of the bamboo particle size is also observed to enhance the values of shear modulus of the composites at similar loading.

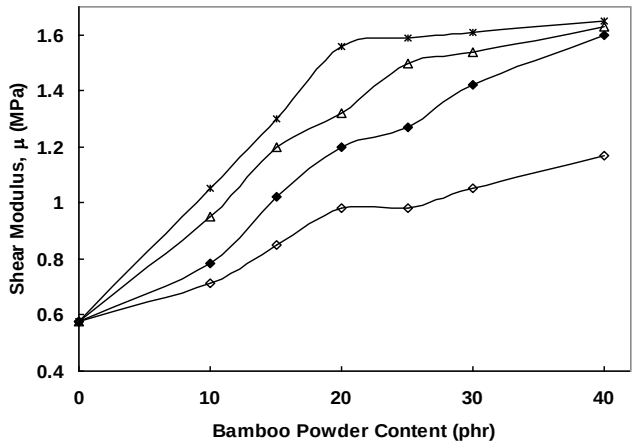

Fig. (4):Variation of shear modulus (derived from fitting of the stress– strain curves by Ogden model, Eq. (4) with bamboo powder loading for bamboo/EPDM rubber [◇ A_x , ◆ B_x , △ C_x , and ✳ D_x] composites.

Dynamic mechanical properties

Dynamic mechanical testing, in general, gives more information about the tested polymer than static testing because of to its high sensitivity to the chemical and physical structures of a polymer. The resonance curves of the composites B and D with different bamboo contents as representative examples are given in Fig. (5).

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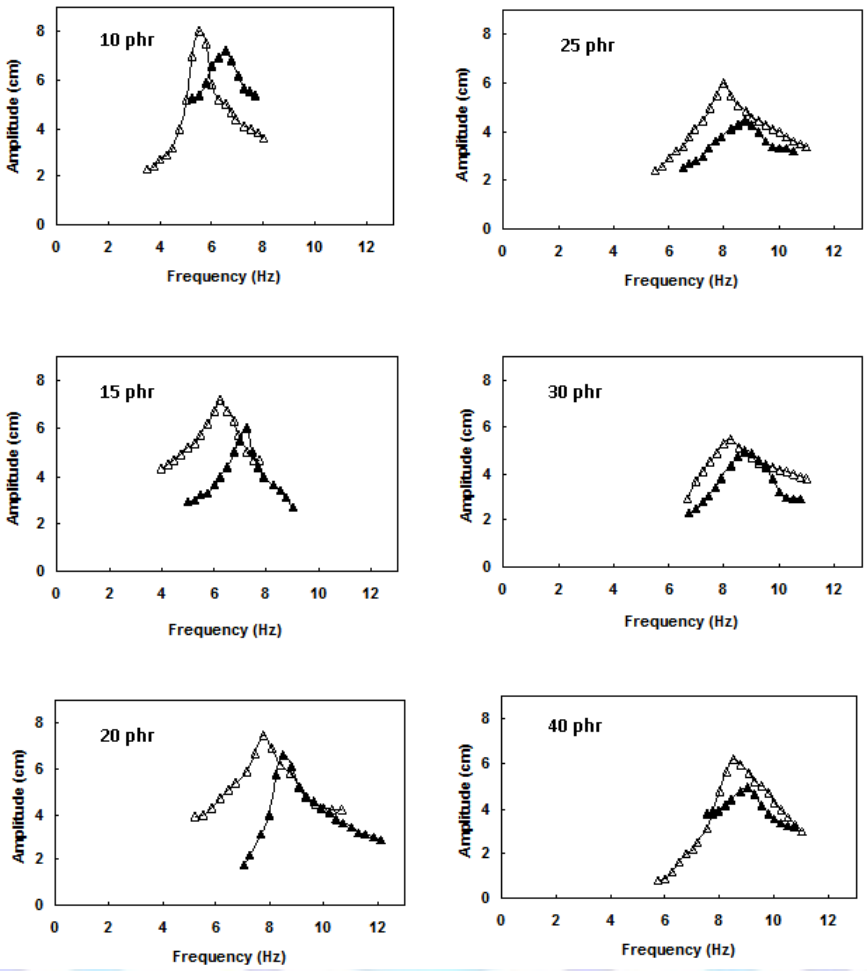

Fig 5: Resonance curves of bamboo /EPDM rubber [(△) B_x composites and (▲) D_x composites].

In general, thermal diffusivity (D_{th}), dynamic elastic modulus (Y_d) and internal friction (Q^{-1}), are the important indicators in characterizing the dynamic mechanical properties of rubber composites. From Fig. (5), the values of Y_d, D_{th}, and Q^{-1} are given as function of bamboo content in Fig. (6). Results show that the addition of bamboo powder to the EPDM matrix decreases the thermal diffusivity (D_{th}) of the composite. Thermal conductivity of bamboo =0.04 W/mK[28] is much smaller than that of EPDM rubber = 0.17 W/mK[29]. Hence, the addition of bamboo powder into EPDM rubber would decrease its thermal conductivity, and the content and particle size of filler have effects on the thermal conductivity. On increasing the content of bamboo powder, the interfacial thermal barrier at the interface between the fillers and the polymeric matrix increases. This will in turns increase the scattering of phonons thereby, reduces the thermal transport in the overall composite. So, the thermal diffusivity decreases with increasing bamboo powder content Fig. (6a). Hence; these composites may be suitable for use as building components to reduce heat transfer in air conditioned buildings and consequently decrease energy consumption.

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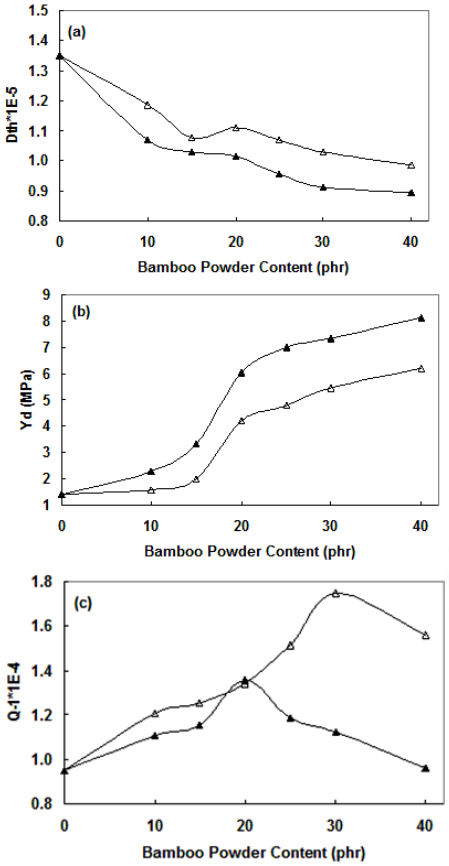

Fig 6: Dependence of (a) D_{th}, (b) Y_d and (c) Q^{-1} on the bamboo content for [(△) B_x composites and (▲) D_x composites].

Besides, Y_d for all of the tested samples showed a steady increase with an increase of bamboo content. The dispersion of bamboo particles led to the creation of physical crosslinks that brought the polymer chain segments to a more stable state and increased the strength of the sample, as is clear in Fig. (6b). However, Q^{-1} increased with increasing bamboo content, showing maxima values at certain content of bamboo, depends upon the particle size [D_{20} composite and B_{30} composite](Fig. (6c)). The less the Q^{-1} is, the less the energy dissipated as heat during one cycle is. Basically, dynamic heat build-up mainly results from the frictions of various networks such as rubber-rubber, filler-rubber, and filler-filler friction[30]. The initial increase in Q^{-1} and Y_d was because the low crosslink density created with low bamboo content, which consequently increased the number of isolated stabilized chain segments and hence the friction generated at the interfacial crosslink-free areas adjacent to the stabilized crosslinked segments [31]. Therefore, a maximum for Q^{-1} was reached at [20 phr for D composite and 30 phr for B composite]. Further increases in bamboo content led to increased crosslink density. The stabilized segments, which became less in number and greater in size reduced the polymeric chain movements, and therefore, Q^{-1} decreased with increasing bamboo content.

Moreover, the values of Q^{-1} and D_{th}, at all levels of fiber loading, of D_x composites were lower than those of B_x composites. The lower values of Q^{-1} and D_{th} for D composites could be attributed to the smaller particle size of bamboo powder in case of D_x composites. As the particle size of bamboo decreases its surface area increases which can enhance the filler-rubber interfacial interaction and weakens the relative slippage and friction of filler-rubber. As well as, the formation of steadier filler network (in case of D_x composites which are loaded with the smaller particle size of filler) decreases the filler-filler friction greatly.

Morphological Studies

Morphology of the composites was examined SEM. Because the highest thermal stability as well as mechanical parameters were obtained for D_x composites, the effect of bamboo loading on the microstrucure properties was studied for this particular formulation. Fig. (7) shows the SEM micrographs of EPDM and bamboo/EPDM composites D_x with different bamboo content. The results indicated that no porous structure appeared on the surface of the pure EPDM composite; only a smooth surface texture was found, as shown in Fig. (7a). The microstructure of the pure EPDM composite affected by filler addition of 10 phr bamboo (D_{10} composite) as observed from Fig. (7b). Dispersion of the bamboo particles was relatively homogeneous in most areas of the matrix. Further addition of bamboo to 20 phr (D_{20} composite), the structure of the surfaces appears to be more compact, with few aggregates of bamboo particles inside the rubber matrix (see Fig.

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(7c)). This observation supports the results of the mechanical tests where the D_{20} composite displayed higher rupture stress σ_R (Fig.3c) and internal friction Q^{-1}, (Fig. (6c)), than the other D_x composites. On increasing the bamboo content to more than 20 phr (D_{25}, D_{30} and D_{40} composites), it is observed that the dispersion of bamboo particles in the polymer matrix are the main factors for the reduction of σ_R and Q^{-1} with an increase of filler content in the composites. Besides, the particle size of bamboo was found to affect the microstructure of the composites. Fig. (8), shows the SEM micrographs of bamboo /EPDM composites with different particle sizes of bamboo powder at bamboo content of 20 phr (A_{20}, C_{20} and D_{20} composites). In comparison, the morphologies of rubber composites are different depending on the particle size of bamboo. Large clusters of the raw bamboo particles can be clearly seen in the composite micrograph, in case of A_{20} and C_{20} composites, (see Figs. (8a,b)). While, in the case of D_{20} composite, the filler and matrix was not clearly differentiable, with few aggregates of bamboo inside the rubber matrix, i.e. the dispersion of the bamboo particles was relatively good (Fig. (8c)). This good dispersion of the bamboo particles was reflected in the mechanical stress-strain parameters; W, Y, σ_R and ε_R (Fig. (3)).

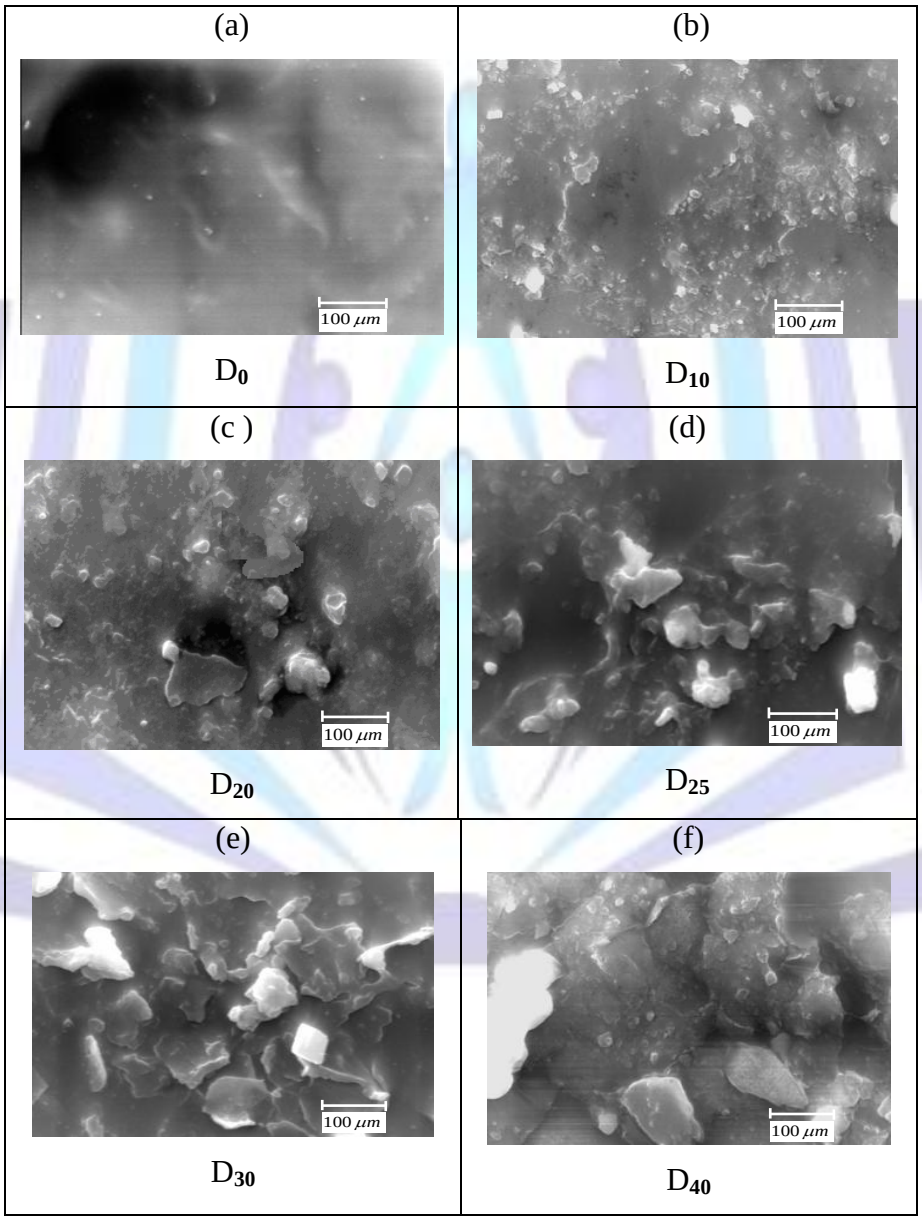

Fig 7: SEM micrographs of EPDM and bamboo/EPDM composites D_x (200 X)

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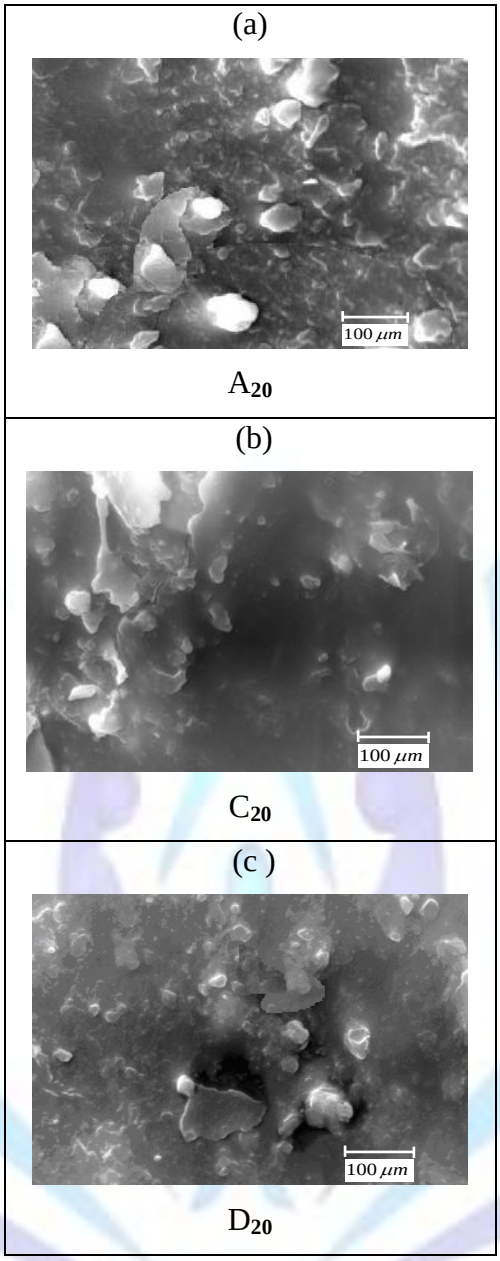

Fig 8: SEM micrographs of bamboo/EPDM composites (200 X) with different

CONCLUSION

The mechanical, thermal, and microstructure properties of bamboo powder reinforced ethylene propylene diene monomer (EPDM) have been investigated as a function of bamboo loading and particle size. Based on the results obtained from this study, the following can be concluded:

1. The thermal stability of EPDM composites was enhanced with increasing the bamboo loading and /or decreasing the particle size as endorsed by TGA.

2. Composites containing the smallest particle size of bamboo powder, at all levels of bamboo loading, showed mechanical properties superior to all other composites as confirmed by SEM micrographs.

3. The absorbed energy W, and Young's modulus Y, for the investigated composites increase with increasing the bamboo content. On the other hand rupture strain ε_R showed a reduction with increasing bamboo loading.

4. Peaks in the σ_R – bamboo content graph were observed, and a shift in the peak position towards higher bamboo content was observed with increasing bamboo particle size.

5. The variation of shear modulus, μ (derived from fitting of the stress– strain curves by Ogden model) with bamboo loading followed a similar trend to that of W and Y.

6. The D_{th}, Y_d and Q^{-1} values obtained from the dynamic mechanical testing revealed the effect of bamboo content and its particle size on the observed variations of the data obtained for the different investigated composites.

7. The proper selection of the particle size and content of bamboo powder is necessary for optimum performance of the rubber composite end product. The optimum bamboo powder contents in EPDM rubber composites, which indicated the maximum rupture stress σ_R values, were found to be 20 phr for (particle size $\leq 90\mu m$), 25 phr for (particle size $\leq 150\mu m$), and 30 phr for (particle size $\leq 250\mu m$).

8. Finally, the results of this study indicate that the (bamboo powder/EPDM) composite is an insulating material. Therefore, this composite may be suitable for use as building components to reduce heat transfer in air conditioned buildings.

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