Mechanical Characterization of Graphite and Graphene / Vinyl-Ester Nanocomposite Using Three Point Bending Test

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This paper analyses the influence of carbon filler content over the mechanical properties of vinyl ester nanocomposite. The carbon fillers used in this study were graphite and graphene nano sized particles in the following weight percentage: 0.10, 0.15 and 0.20. The mechanical properties such as modulus of elasticity, flexural stress and flexural strain were determined using three point bending tests.A significant enhancement of overall mechanical properties were achieved for some of the nanocomposite materials studied.

Keywords: mechanical properties, vinyl-ester nanocomposite, graphite, graphene, three point bending test

Trend in recent years has been for researchers to use nano-scaled particles to reinforce polymers in order to obtain materials with enhanced properties[1-10]. Different types of nano particles were used as filler in polymer matrix to obtain better properties. Several studies reported significant changes in properties such as mechanical, optical, electrical, thermal by using clay, graphite, graphene and carbon nanotubes as filler in thermosetting polymer matrix [11-21].

Improving the overall properties of resulted materials by addition of filler to a polymer matrix is requesting using nanoparticle due to its high contact surface but paying attention to the degree of dispersion of the filler in matrix system. It is well known the tendency of nanoparticle to agglomerate and to form clusters which is a challenge for the researcher to avoid this occurrence [22]. Since its discovery, graphene became one of the most promising materials. Was reported in literature that by the addition of small amount of graphene nano particle to polymer matrix can be obtained significant enhancement of the overall properties [23-26].

Vinyl ester resin is used for many applications due to its strength and durability, very low viscosity and the ability to cure at ambient temperature [27]. Vinyl ester is more suitable for application that required better chemical resistance and physical properties than polyesters.

Experimental part

The vinyl ester resin type Polimal VE-11 M was used as matrix phase. The graphite and graphene used as filler into the vinyl ester matrix were purchased from Cheap Tubes Inc. Company. The diameter of carbon nanoparticles was approximately 2-4 μm with more than 97 wt.% purity.

The mechanical mixing method used to obtain graphite and graphene / vinyl-ester nanocomposite materials consist in the following: progressive addition of carbon nanoparticle in polymer matrix followed by magnetic stirring at room temperature for one hour at 600 rot/min to obtain a good homogenization; starting the polymerization using methyl-ethyl-ketone peroxide as initiator; to avoid the agglomeration, the mixture was exposed to ultrasonic waves for 5 min; after degassing the mixture, the materials was poured into mold which consist of ten parallelepiped spaces that provide ten specimens at each pouring. For every batch were used three mold with ten parallelepiped spaces.

In order to achieve a complete curing of resulted composite material, all the specimen were introduced in the oven and kept 8 h at 80 degree Celsius.

Parallelepiped specimens with 8 mm width, 4 mm height and 40 mm length were obtained by pouring in special dimensioned mold. A total of minimum 25 samples for each materials has been obtained. Five samples of each material were tested for all testing speeds. The mechanical properties characterization has been done using the three point bending test according to ASTM D790, that provides values for the modulus of elasticity, flexural stress and flexural strain. The Instron machine type 8800 was used for testing the materials. The tests were performed with a constant strain rate of 1, 5 10 25 and 50 mm/min.

Results and discussions

According to the standard requirements, all test were performed on at least five specimens. The results for modulus of elasticity, flexural stress and flexural strain determined using 3-point bending tests are synthesized in the figures 1 to 6. In the tables 1 to 3 are presented the

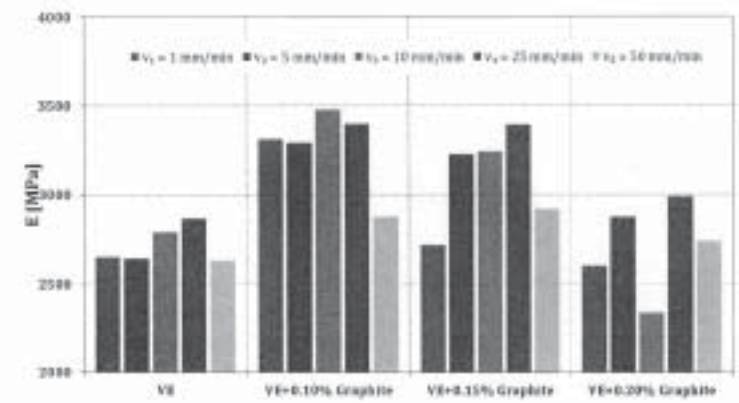

Fig. 1. Modulus of Elasticity for graphite/vinyl ester nanocomposite with following weight percentage filler: 0.10, 0.15 and 0.20 tested with a constant strain rate of 1, 5, 10, 25 and 50 mm/min

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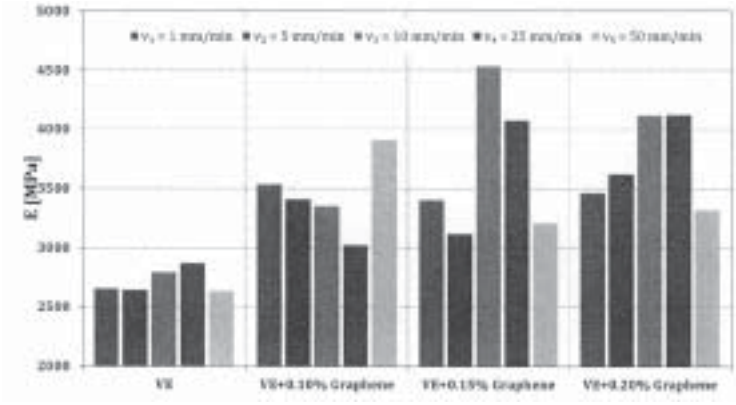

Fig. 2. Modulus of Elasticity for graphene/vinyl ester nanocomposite with following weight percentage filler: 0.10, 0.15 and 0.20 tested with a constant strain rate of 1, 5, 10, 25 and 50 mm/min

Modulus of elasticity	VE+graphite, graphene vs. neat VE				
	1 mm/min	5 mm/min	10 mm/min	25 mm/min	50 mm/min
VE+0.10wt.% graphite	24.94 %	24.60 %	24.68 %	18.52 %	9.19 %
VE+0.15wt.% graphite	2.56 %	22.14 %	16.19 %	18.34 %	10.9 %
VE+0.20wt.% graphite	-1.96 %	8.81 %	-16.15 %	4.28 %	4.10 %
VE+0.10wt.% graphene	33.03 %	29.06 %	20.13 %	5.52 %	48.33 %
VE+0.15wt.% graphene	28.35 %	18.01 %	62.41 %	41.98 %	21.91 %
VE+0.20wt.% graphene	30.61 %	37.05 %	47.54 %	43.72 %	26.09 %

Table 1
THE COMPARISON OF MODULUS OF ELASTICITY OF VINYL ESTER / GRAPHITE AND GRAPHENE NANOCOMPOSITE WITH NEAT VE

increasing /decreasing of the obtained values for modulus of elasticity, flexural stress and flexural strain of nanocomposites tested compared with the neat vinyl ester polymer.

The highest values of modulus of elasticity of the VE+graphite nanocomposite were obtained for 0.10 wt.% at 10 and 25 mm/min with 3480 MPa and 3399.4 MPa (fig.1). Compared to the modulus of elasticity of neat vinyl ester the maximum increasing is obtained by 0.10 wt.% graphite at 1 mm/min speed test of about 25%. The lowest values are obtained by 0.20 wt.% graphite nanocomposite at 50 mm/min (table 1).

All the other values of modulus of elasticity shows an increasing compared to neat vinyl ester with the exception of 0.20 wt.% nanocomposite which decreased with about 1.96%% at 1 mm/min and with 16.15% at 10 mm/min speed test.

In the case of graphene based vinyl ester nanocomposite, the highest values of modulus of elasticity were obtained for 0.15 wt.% graphene at 10 mm/min test speed with 4533 MPa followed by 0.20 wt.% concentration of graphene at 10 and 25 mm/min with 4118 MPa and 4112 MPa respectively (fig. 2). The lowest values of modulus of elasticity were obtained for 0.10 wt.% graphene at 25 mm/min test speed with 3026 MPa. Overall, the nanocomposite materials of vinyl ester with graphene presents higher values of modulus of elasticity compared with neat vinyl ester for all studied concentrations and test speed (table 1)

In figure 3 can be observed that the highest values of flexural stress of the VE+graphite nanocomposite were obtained for 0.10 wt.% at 1 mm/min with 64 MPa and 0.15 wt.% at 25 mm/min with 60 MPa. Compared to the flexural

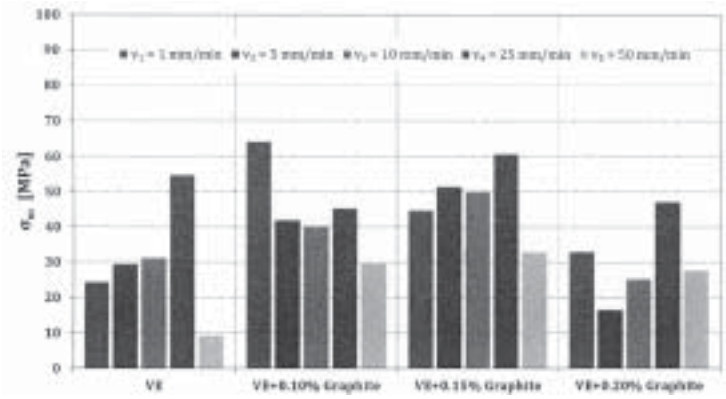

Fig. 3. Flexural stress for graphite /vinyl ester nanocomposite with following weight percentage filler: 0.10, 0.15 and 0.20 tested with a constant strain rate of 1, 5, 10, 25 and 50 mm/min

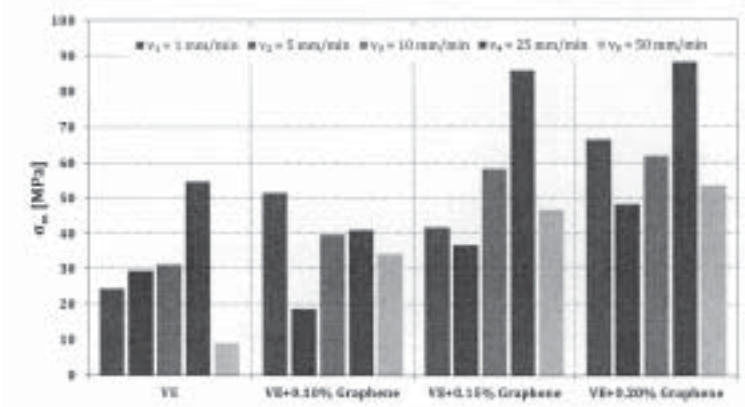

Fig. 4. Flexural stress for graphene /vinyl ester nanocomposite with following weight percentage filler: 0.10, 0.15 and 0.20 tested with a constant strain rate of 1, 5, 10, 25 and 50 mm/min

Flexural stress	VE+graphite, graphene vs. neat VE				
	1 mm/min	5 mm/min	10 mm/min	25 mm/min	50 mm/min
VE+0.10wt.% graphite	163.78 %	42.35 %	34.10 %	-17.29 %	222.7 %
VE+0.15wt.% graphite	83.3 %	74.77 %	60 %	10.9 %	257.2 %
VE+0.20wt.% graphite	35.27 %	-43.5 %	-18.79 %	-13.7 %	201.74 %
VE+0.10wt.% graphene	111.2 %	-36.09 %	26.8 %	-25.3 %	272.8 %
VE+0.15wt.% graphene	71.32 %	24.64 %	86.29 %	56.85 %	407.6 %
VE+0.20wt.% graphene	172.35 %	63.49 %	98.43 %	72.44 %	482.2 %

Table 2
THE COMPARISON OF FLEXURAL STRESS OF VINYL ESTER / GRAPHITE AND GRAPHENE NANOCOMPOSITE WITH NEAT VE

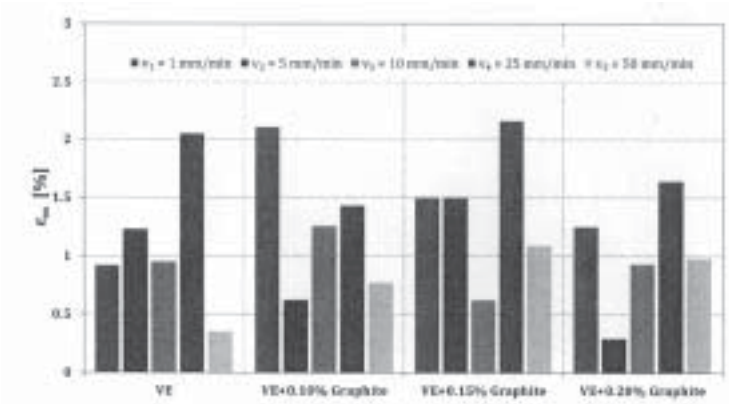

Fig. 5. Flexural strain for graphite/vinyl ester nanocomposite with following weight percentage filler: 0.10, 0.15 and 0.20 tested with a constant strain rate of 1, 5, 10, 25 and 50 mm/min

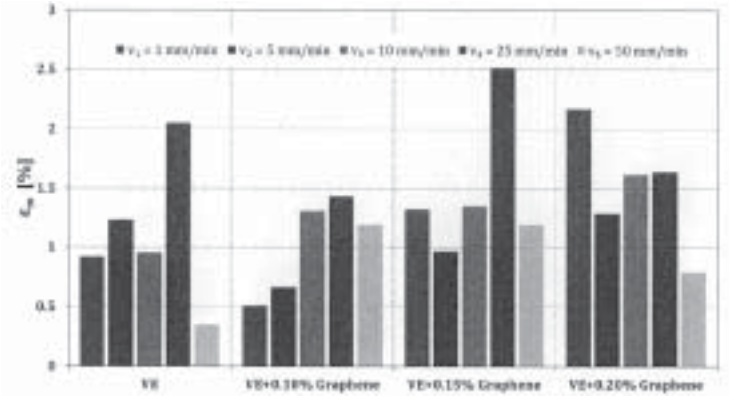

Fig. 6. Flexural stress for graphene/vinyl ester nanocomposite with following weight percentage filler: 0.10, 0.15 and 0.20 tested with a constant strain rate of 1, 5, 10, 25 and 50 mm/min

stress of neat vinyl ester the maximum increasing is obtained at 50 mm/min speed test for all graphite concentration with more than 200% increasing. The lowest values are obtained for 0.20 wt.% graphite nanocomposite at 5 mm/min (table 2). An increasing of flexural stress values for all VE /graphite nanocomposite are obtained for 1 and 50 mm/min test speeds. Better results are also obtained for 0.15 wt.% graphite for all test speeds studied, compared with neat vinyl ester. The 0.20 wt.% graphite content show a decreasing for testing speed of 5, 10 and 25 mm.min most probably due to aglommeration of the graphite particles.

Figure 4 present the result for vinyl ester/graphene composites regarding flexural stress. Except for 0.10 wt.% graphene content that present a decreased of flexural stress at 5 and 25 mm/min all the other results for VE / graphene composite are increased compared to neat polymer. The maximum increased values is about 482 % for 0.20 wt.% content at 50 mm/min speed.

The highest values of flexural strain of the VE+graphite nanocomposite were obtained for 0.10 wt.% at 1 mm/min and 0.15 wt.% at 25 mm/min and the lowest values for 0.20 wt.% at 50mm/min (fig. 3). Vinyl ester/ graphene nanocomposite show a maximum of flexural strain for

Flexural strain	VE+graphite, graphene vs. neat VE				
	1 mm/min	5 mm/min	10 mm/min	25 mm/min	50 mm/min
VE+0.10wt.% graphite	129.04 %	-49.6 %	32.63 %	-30.24 %	120 %
VE+0.15wt.% graphite	63.04 %	21.95 %	-34.7 %	5.36 %	211.4 %
VE+0.20wt.% graphite	35.86 %	-76.4 %	-2.1 %	-20 %	180 %
VE+0.10wt.% graphene	-44.6 %	-46.3 %	37.89 %	-30.24 %	240 %
VE+0.15wt.% graphene	43.47 %	-21.13 %	42.1 %	30.73 %	245 %
VE+0.20wt.% graphene	135.8 %	4.87 %	70.52 %	-20 %	125.7 %

Table 3
THE COMPARISON OF FLEXURAL STRAIN OF VINYL ESTER / GRAPHITE AND GRAPHENE NANOCOMPOSITE WITH NEAT VE

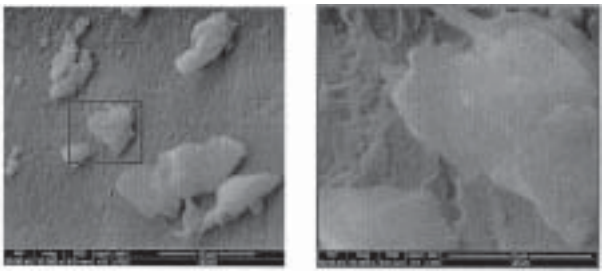

Fig. 7. SEM images of vinyl ester / graphite 0.10 wt. % nanocomposite

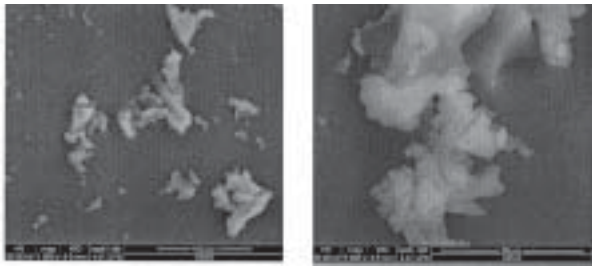

Fig. 8. SEM images of vinyl ester / graphene 0.20 wt. % nanocomposite

0.15 wt.% at 25 mm/min and a minium for 0.10 wt.% at 1 mm/min (fig. 4) (table 1).

SEM images of the specimens of vinyl ester / 0.10 wt. % graphite nanocomposite and vinyl ester / 0.20 wt. % graphene nanocomposite are presented in figure 7 and 8 showing the morphological aspects. It is observed the presence of graphite and graphene respectively throughout the entire mass of the polymer matrix. It also noticeable the cluster formation of carbon nanoparticles.

Conclusions

Nanocomposite materials formed by addition of graphite and graphene nanoparticle to the vinyl ester thermosetting polymer matrix has been fabricated and characterized for their mechanical properties. The filler content and testing speed influence on mechanical properties have been studied by three point bending test. It has been found that the modulus of elasticity of vinyl ester / graphene and graphite nanocomposite increased compared to neat vinyl ester in all testing condition except for 0.20 wt. % graphite content at 1 and 10 mm/min test speed. The maximum values has been achieved for 0.10 et.% graphite at 1 mm/min and 0.15 wt. % graphene at 10 mm/min. Improvement of flexural stress was obtained for all nanocomposite tested at 1 and 50 mm/min speed. A good behaviour was obtained for 0.15 wt.% graphite for all speed test. By the experimental results can be concluded that by addition of small content of carbon nanoparticle such as graphite and graphene to vinyl ester matrix can be obtained an improvement of the overall mechanical properties.

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