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Improving of rheological and mechanical properties of natural and EPDM rubbers *via* multi-walled carbon nanotubes (MWCNTs) reinforcement

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In this study, natural rubber and EPDM rubber were reinforced with multi-walled carbon nanotubes (MWCNTs) by three different methods. These methods are homogenous mixing, spraying and hand laying methods. The rheological and mechanical properties of two different rubbers produced by the three different methods were examined. As a result, six samples of nano composite material with different rheological properties were produced. The maximum torque (MH), the minimum torque (ML), the start time (tc10), the time of vulcanization (tc90) and the time of early scorch were calculated from the rheological properties. The mechanical behavior of MWCNTs reinforced rubber samples was determined by tensometer test in two different ways in tensile and compression directions. As a result of this experiment, it was observed that the difference in tensile and compressive load is that the rubber has a hysteretic structure and doesn't lose elasticity. Torsional stiffness (Kt) torque - angle (N rad^{-1}) values were found. Although torsion joint of EPDM rubber differs in three methods, this difference hasn't been observed in natural rubber. The hardness values of MWCNTs reinforced mixtures were tested in shore form and it was stated that these values could not be related to torsional stiffness. The natural frequency test was performed with a hammer and natural frequencies of the system (without rubber) and variable natural frequencies in the system when EPDM and natural rubber were observed. It was determined that the vibration damping of nano-particle reinforced EPDM rubber is different from that of natural rubber.

Keywords: EPDM rubber, Multi-walled carbon nanotubes, Natural rubber.

INTRODUCTION

Rubber is a remarkable engineering material found in every aspect of our daily lives. It is preferred because of its viscoelastic structure, being an alternative to metals, and providing ease of use. Its usage areas increase each day in many sectors such as work environments, home applications, games, sports, automotive industry, aircraft industry, railway transportation, and construction.

Elastomers, which have a structure between a linear polymer and a spatial polymer, consist of long molecular chains that are twisted and entangled in the form of pellets. These materials are called elastomers because they show a highly reversible behavior (elastic deformation) as a result of the opening of the lumps and bond rotation under the effect of force. In industry, elastomers are divided into two groups as “thermoplastic” and “thermoset” in terms of behavior under the effect of temperature [1]. Thermoplastic elastomers are linear polymers that soften when the temperature is increased, harden when cooled, and tend to soften again when reheated. Thermoset elastomers are materials that tend to soften when heated. All of them are called plastic because of this reason. The difference between elastomers and thermoset plastics is due to their mechanical properties. For example, if an elastomer material is subjected to a certain tension in both directions, the material begins to elongate, and when the stretching process is released, it tends to take its former shape. This situation is called elasticity. Thermosets, on the other hand, maintain their elasticity up to a certain limit (yield limit). After this limit, the material breaks and undergoes deformation. This condition is permanent and irreversible. However, elastomers have less elastic modulus and tensile strength than thermosets. Because of this situation, although elastomers start to elongate immediately, thermoplastics cause elongation under higher load [2].

The average molecular weight of natural rubber is 68.12 g mol^{-1} and varies in a wide range. Due to its structure, it crystallizes when stress is applied or at low temperatures. The crystallization has some positive effects on natural rubber, resulting in its characteristics. It has very good tensile and tear resistance, elasticity, and fatigue properties. During the vulcanization process, the sulfur molecule, which is a ring with eight atoms, is opened.

Cross-linking occurs by linking the sulfur molecules to the polyisoprene chains [3].

Ethylene-propylene diene monomer is formed as a result of the chemical combination of ethylene and

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propylene monomers. By combining two monomers, an amorphous, semi-crystalline, and crystalline elastomer is obtained. EPM, which has a saturated structure, is produced as a result of the copolymerization of ethylene and polypropylene. EPDM is obtained by adding diene to the chemical structure obtained from EPM. Today, it has become an engineering material that can be widely used in the industrial field.

Nanotechnology is the most important and rapidly developing technology of the 21st century, it is in a key position for many researchers and has managed to attract attention in this regard. The rapid increase in global competition causes this science to have strategic importance among the countries. With the developing technology in the production techniques of rubber and nanomaterials, different approaches are being made every day. Because of this situation, many researchers have tried to bring these two different structures together with new methods. With the emergence of new composite materials, the view of manufacturing methods has reached a very important point. The physical and chemical effects on the material are investigated.

In a study supported by the National Natural Science Foundation of China and the Postdoctoral Science Foundation of China, nanoparticles were added to the rubber to reduce vibration and noise in tubes operating under water, and tribological and mechanical properties were investigated. As a result of the study, it was observed that the friction coefficient of nano-particle reinforced NBR rubber decreased at critical speeds, and besides, the damping ability of its mechanical properties improved [4].

Ramzan *et al.*, in their study, obtained a composite material by reinforcing elastomers with nanomaterials. The damping capabilities of natural rubber by throwing nanoparticles into it were investigated and as a result, design parameters were determined in different variations. They proposed nanocomposites for their strong damping capabilities [5]. In another study, Ramzan and Kumar prepared a test setup and demonstrated the damping ability with this test method [6]. Ziraki e*t al.* added silica nanoparticles and polypropylene fibers to the silicone rubber matrix. They examined the viscoelastic behavior and mechanical properties of the samples they took from this nanoparticle reinforced rubber. As a result, they proved that silica particles increase the effect on the elastic modulus on rubber and reach higher hardness [7]. Huang and Tsai characterized the vibration damping response of composite layers containing silica nanoparticles and rubber particles. They compared the experimental results they obtained with their study with the finite element method. These results showed that the reduction of flexural stiffness of fiber composites, especially for (90/10) layers, decreased as the damping properties of the layers were improved. Vibration damping responses of composite layers obtained using micrometric analysis were shown to be compatible with experimental data together with modal analysis [8].

Muhammad *et al.* have designed a new nanocomposite vibration damper. They used NR rubber and alumina zirconium as test specimens. Modal analysis and natural frequency results were compared with analytical and finite element methods. Then they revealed the first five modes. They proved that the vibration amplitude was reduced by 25% [9].

Azizli *et al.* have prepared a series of elastomeric nanocomposites based on NR/EPDM rubber blends. It was determined that with the addition of graphene oxide to the samples, the mechanical properties increased with fatigue resistance, hardness, tensile strength, and elongation at break. At the end of all analyses, they found a good coherence between the experimental and theoretical results [10]. Ruksakulpiwat *et al.* used natural rubber NR and EPDM rubber with various contents as impact modifiers for composites. The composites were prepared using injection molding. The rheological, morphological, and mechanical properties of the composites were investigated. They observed a significant increase in impact resistance and elongation when NR or EPDM was added to the composites. They also explained the effect of NR and EPDM rubbers on the mechanical properties of PP composites. They stated that composites with EPDM rubber showed slightly higher tensile strength and impact resistance than composites with NR [11]. Enew *et al.* investigated the mechanical properties of the effect of aramid fiber in Kevlar (KP), carbon mono fiber (CMF), and nano-carbon black (NCB) pulp form as filler for Trilene liquid polymers on EPDM rubber. They found that EPDM rubber improved its mechanical properties such as thermal insulation, tensile strength, elongation, and hardness [12]. In this work, MWCNTs reinforced natural and EPDM rubbers were developed to investigate their rheological and mechanical properties by using three different methods. The aim of this study is to determine the most suitable method and MWCNTs reinforced rubber which can be added to the literature as a remarkable engineering materials.

Table 1. Ratios of homogeneously mixed NR and EPDM mixture raw materials.

Raw Materials	%	phr		Raw Materials	%	phr
NR Rubber	100	40.88		EPDM Rubber	100	29.03
MWCT Nano material	50	20.44		MWCT Nano material	50	14.51
N330 (Carbon black)	76	31.07		N550 (Carbon black)	120	34.83
Oil	4	1.64		Oil	45	13.06
Plasticizer	2	0.82		Plasticizer	4	1.16
Stearic acid $(CH_3(CH_2)16COOH)$	3	1.23		Calcium carbonate $(CaCO_3)$	19	5.52
Zinc oxide (ZnO)	6	2.45		Zinc oxide (ZnO)	5	1.45
Sulfur (S)	1.7	0.70		Sulfur (S)	0.1	0.03
Antiozonants	0.9	0.37		Antiozonants	0.4	0.12
Antioxidants	0.2	0.08		Antioxidants	0.1	0.03
Accelerators	0.8	0.33		Accelerators	0.9	0.26
Total	244.6	100.00		Total	344.5	100.00

MATERIALS AND TEST METHODS

While preparing the test samples, MWCT reinforcement to NR and EPDM rubbers was handled by three methods. These methods are homogeneous mixing, hand-laying, and spraying methods, respectively. The ratios of homogeneously mixed NR and EPDM mixture raw materials are given in Table 1.

MWCNTs were applied to natural rubber and EPDM rubber by spray gun with the help of 3 – 4 bar air pressure. In this method, MWCNTs are permeated to rubber by making a special solution out of them. 5 mg of MWCNTs and 20 ccs of a special solution were used in this mixture. MWCNTs were spread on the sample rubbers with the help of a roller. As in the spray method, the solution and the nanomaterial were made at the same scale. The MWCNTs reinforced natural (NR) and EPDM rubbers produced by three different methods are presented in Figure 1.

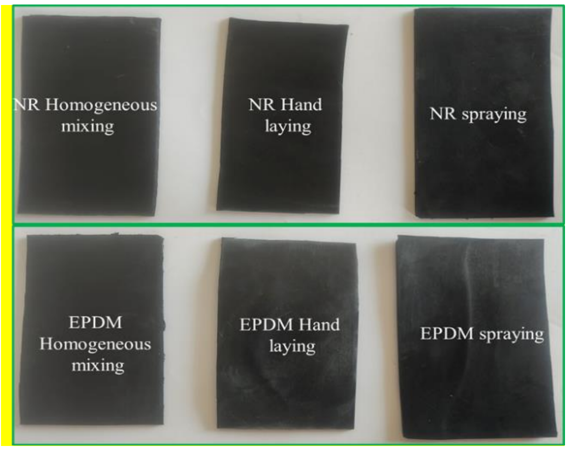


Fig. 1. MWCNTs reinforced natural (NR) and EPDM rubbers produced with homogeneous mixing, hand laying and spraying methods.

Rubber is formed by the combination of many raw materials such as carbon black, oil, accelerators, resins, fillers. Before the curing of this mixture, it was tested in rheometer test devices to see the results such as curing time and scorch time. Experimental samples (GOTECH Moving Die Rheometer M2000 A) were tested with boundary conditions of 150 °C for 15 minutes. Both tensile and compressive behavior of metals in the elastic region show the same properties. This can be expressed by Hooke's laws. Features such as Poisson's ratio and modulus of elasticity can easily be found. However, the same statements are not true for elastic materials such as rubber. It is aimed to make the measurements easier by subjecting the rubber sample to deformation by a force that will provide homogeneous distribution. Thus, it was found to be a simpler method since the forces applied to the sample would cause the same deformation and elongation at all points [13].

There are two types of tensile test methods: uniaxial and multi-axis. Due to the different behavior of rubber in the tensile and compression regions, it is insufficient to determine only the mechanical properties. Due to the accumulation of knowledge on this subject, standards such as ASTM D412, DIN 53 504, and ISO 37 have emerged [14-16]. Mechanical properties of rubber-type materials change with the rate of deformation applied to the material. For the test samples, rubbers were designed according to ASTM D412 – C sample and subjected to uniaxial tensile and compression test (LLOYD LR50KPlus) according to different preloading speeds.

Elastomer materials such as rubber have similar characteristic properties as a metal spring and a viscous liquid. This viscoelastic behavior is defined as deformation and energy-absorbing. Crank pulleys, which are used as automotive parts, are an important test of torsional stiffness, and their resistance to torsion is a torsional behavior. The rubber structure on the crank pulleys is determined by the torsional stiffness (kt). Torsional stiffness is an important parameter because it affects the natural frequency of the crank pulley. The test specimens were subjected to tests in the DEVOTRANS Torque Life Test device at room temperature, providing angles of 1 º – 30 º.

Many researchers have developed test methods for natural frequency and vibration measurements. In the modern automotive industry, natural frequency measurements are made according to modal test methods. For crank pulleys, it is available as torsional (torsion) frequency. A modal test on the rotating component takes place by actuation by mounting accelerometers in a constant tangential fashion to both inertial masses in the same direction. Therefore, frequency functions (FRF) are formed here. These FRFs form a theory in finding physical properties [17]. Known as the current theory, it is known as the hysteretic damping of the rubber, dissipating the input energy and accelerating the system without resonating [18]. Natural frequency measurements are generally measured with the hammer method because it is cheap and easy. The vibration frequency distribution of the force is found in the test performed with the hammer in the modal test setup. With these distributions, natural frequency, damping, and modal shapes can be determined [19, 20]. Experimental samples were measured between two sheet metal plates, using a bedding element to dampen the vibrations coming from the ground, and data collector DEWESoft DAQ with the help of a hammer.

RESULTS

Figure 2 (a, b, c, d) shows the SEM image of the EPDM sample, MWCNTs blended EPDM sample, NR sample and MWCNTs blended NR sample, respectively. The SEM image of the EPDM sample obtained by the homogeneous mixing method displayed a rough surface morphology. After adding of 0.5 wt% MWCNTs to EPDM, the composite materials showed that MWCNTs were placed as nanotube groups in the composite material. The mechanical test results indicated the enhancement after MWCNT addition, as mentioned above. According to the SEM results, the dispersion of CNT groups was not completely homogeneous and if it could be provided the enhancement of mechanical properties also could be increased. By functionalization of CNTs or using a different method the homogeneity could be increased and will be the subject of future studies. SEM image of the NR and MWCNTs blended NR sample obtained by homogeneous mixing method displayed a rough surface morphology like EPDM samples.

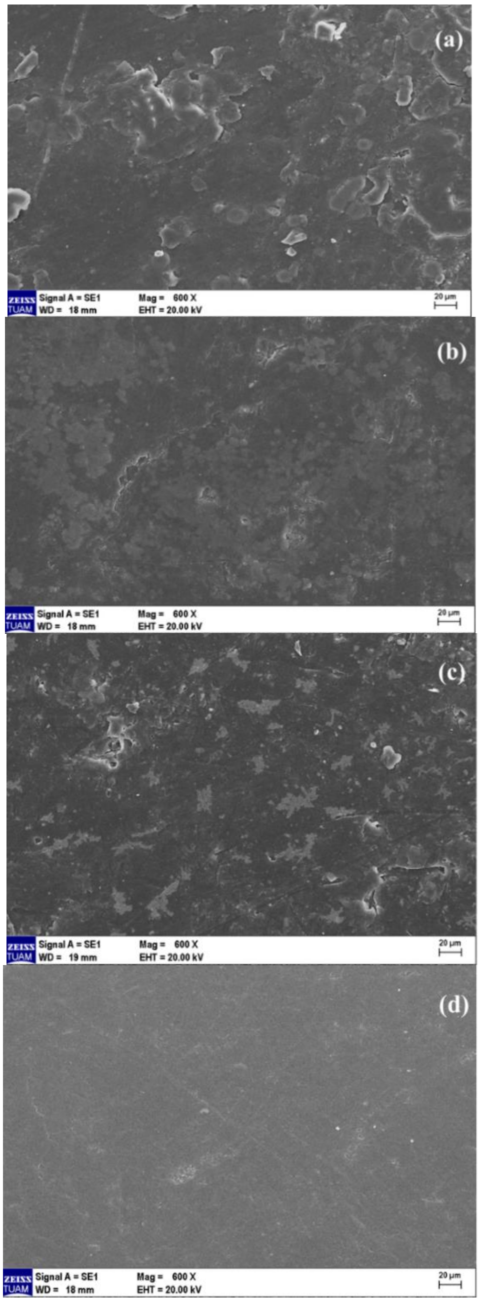
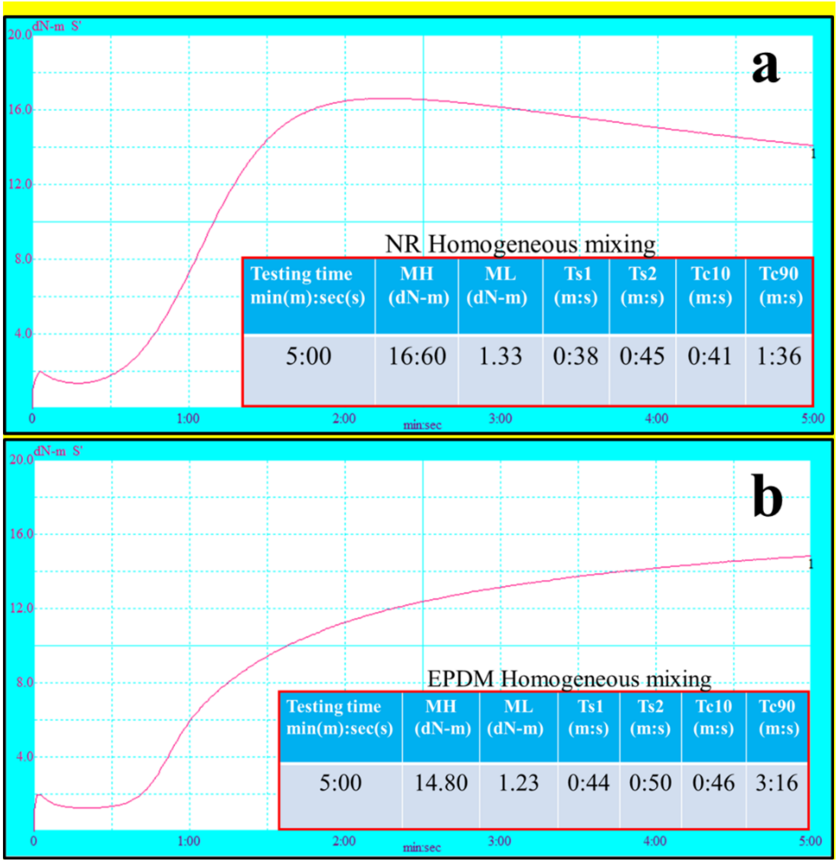


Fig. 2. SEM image of EPDM (a), MWCNTs blended EPDM sample (b), NR sample (c), MWCNTs blended NR sample (d).

Rheometer test results of MWCNTs reinforced natural rubber and EPDM rubber produced *via* homogeneous mixing method are given in Figure 3. The MWCNTs reinforced natural and EPDM rubbers produced *via* other two methods are given in Table 2 for hand-laying and spraying methods, respectively.



NR Homogeneous mixing

Testing time min(m):sec(s)	MH (dN-m)	ML (dN-m)	Ts1 (m:s)	Ts2 (m:s)	Tc10 (m:s)	Tc90 (m:s)
5:00	16:60	1.33	0:38	0:45	0:41	1:36

EPDM Homogeneous mixing

Testing time min(m):sec(s)	MH (dN-m)	ML (dN-m)	Ts1 (m:s)	Ts2 (m:s)	Tc10 (m:s)	Tc90 (m:s)
5:00	14.80	1.23	0:44	0:50	0:46	3:16

Fig. 3. Rheometer test results of MWCTs reinforcement (a) natural rubber (b) EPDM rubber produced *via* homogeneous mixing method.

Table 2 Rheometer test results of MWCNTs reinforced natural and EPDM rubbers produced *via* hand-laying and spraying methods.

Materials/Method	Testing time min(m):sec(s)	MH (dN-m)	ML (dN-m)	Ts1 (m:s)	Ts2 (m:s)	Tc10 (m:s)	Tc90 (m:s)
NR Hand laying method	5:00	15.06	0.32	0:20	0:28	0:23	1:23
EPDM/Hand laying method	5:00	16.30	1.26	0:53	1:02	0:57	3:29
NR Spraying	5:00	17.22	1.74	0:40	0:48	0:43	1:45
EPDM Spraying	5:00	14.87	1.17	1:00	1:09	1:02	3:16

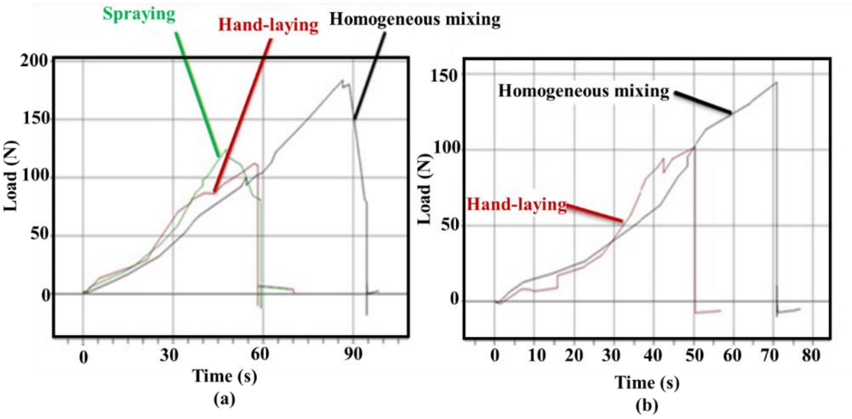


Fig. 4. MWCT reinforced (a) natural rubber (b) EPDM rubber rupture test results

According to the tensile test of MWCT reinforced composite specimens, the preload stress and velocities are equally given in Figure 4. In Table 3, tensile and compression results according to different sample types with preload stress velocities (50 – 100 – 200 mm/min) are shown. In addition, the hardness values of nanocomposite materials were measured in shore – A hardness measuring device (HUATEC).

Torsional stiffness graphs for two different rubbers, angle – torque graphs between 1º – 30º (0.0174 – 0.523 radian) with three different methods are given in Figures 5 and 6.

Fig. 5. MWCTs reinforced NR rubber angle-torque graph

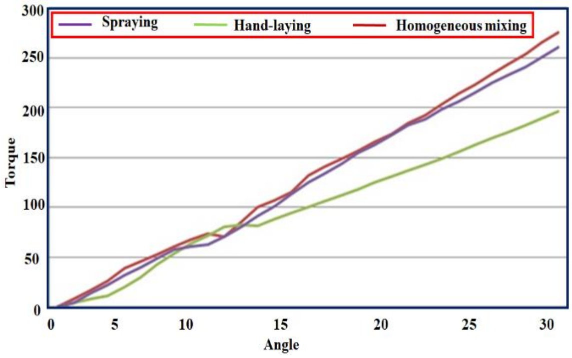


Fig. 6. EPDM rubber angle-torque graph with MWCTs reinforcement

Natural frequency measurements were made and the test results of MWCT reinforced NR and EPDM rubbers are given in Figures 7, 8 and 9. The chart data of Figure 7 is given in the inset of Figure 7. The chart data of Figure 8 and 9 are given in Table 4 and Table 5, respectively.

As the preload stress rate increases, the difference between the tensile and compressive loads of the sample mixtures increases. For MWCNTs-reinforced NR, the highest load was with the spraying method, while the least load was formed in the homogeneous mixing method. In EPDM rubber, while the highest load is in the homogeneous mixing method, the least load is seen in the hand laying method. Due to this situation, three different methods that are subjected to the highest load are given in Figure 10 below.

DISCUSSION

The rheological and mechanical properties of natural rubber and EPDM rubber with MWCTs reinforcement were experimentally investigated. In the tests, it was observed that the nano-material reinforcement had different effects on the material and different nano-composite materials emerged due to the different methodological processes.

The data on the rheological properties of MWCTs reinforced natural rubber and EPDM rubber are shown in Figure 3, where they differ. From the rheometer curve, it was observed that the nanocomposites produced by the homogeneous mixing and spraying method for natural rubber were the same. In the hand lay-up method, which has different firing start times (tc10) and maximum torque (MH), it was observed that the rubber cooked later than other methods. The curing time (tc90) time was taken as the same value for all methods of natural rubber. It is seen that MH and ML of EPDM rubber vary. When the rheometer curves were examined, it was observed that the cooking additive used the same raw material in both rubbers and the firing condition was stable.

When the mechanical properties were examined, the effect of nanomaterial reinforcement for the NR mixture (Figure 4) on the tensile strength and maximum elongation amount, the highest tensile strength and elongation amount were obtained by the homogeneous mixing method. while the tensile direction is subjected to more load in general, the compression direction is subjected to less load due to the hysteretic nature of the rubber (Table 3). It was determined that the method that affects the hardness value the most among the nanocomposite samples is the hand lay-up method. Torsional stiffness of MWCTs reinforced NR and EPDM rubber was tested and is given in Figures 5 and 6. For NR and EPDM rubber, the same torsion is observed in all methods up to 7º. The torque values after this degree are ordered as spraying, hand-laying, and homogeneous mixing for NR rubber and for EPDM rubber, homogeneous mixing, spraying, and hand-laying are listed from the largest to the smallest. For EPDM rubber, homogeneous mixing, spraying, and hand-laying are listed from the largest to the smallest.

It was observed that the MWCTs reinforcement has little effect on the torsional stiffness in natural rubber, but differences occur in EPDM rubber.

Natural frequency test results are given in Figs. 7, 8 and 9.

Table 3. Tensile and compression results of reinforced NR and EPDM rubber. Tensile – compression test results of nano composite materials

Test Conditions				NR Rubber			EPDM Rubber		
Sample Type/ Method	Method	Preload Stress (N)	Preload Stress Rate (mm/min)	Hardness (Shore A)	Max. Load (N)	Max. Elongation (mm)	Hardness (Shore A)	Max. Load (N)	Max. Elongation (mm)
Spraying	Tensile	100	50	63	79.876	100.76	67	71.075	100.04
Spraying	Compression	100	50	63	72.727	84.548	67	59.061	69.965
Spraying	Tensile	100	100	63	88.775	101.49	67	60.094	96.007
Spraying	Compression	100	100	63	82.189	82.829	67	58.647	100.8
Spraying	Tensile	100	200	63	102.75	118.06	67	88.792	144.79
Spraying	Compression	100	200	63	35.619	105.48	67	20.704	63.297
Hand Lay	Tensile	100	50	68	72.081	94.694	69	64.066	78.063
Hand Lay	Compression	100	50	68	61.478	91.86	69	-	-
Hand Lay	Tensile	100	100	68	667.553	96.016	69	37.566	46.182
Hand Lay	Compression	100	100	68	53.806	79.5	69	-	-
Hand Lay	Tensile	100	200	68	73.09	107.26	69	39.351	108.42
Hand Lay	Compression	100	200	68	39.351	103.42	69	-	-
Homogeneous Mixing	Tensile	100	50	61	52.07	103.03	63	73.212	99.342
Homogeneous Mixing	Compression	100	50	61	38.333	102.58	63	63.553	71.39
Homogeneous Mixing	Tensile	100	100	61	50.107	99.405	63	78.959	121.561
Homogeneous Mixing	Compression	100	100	61	44.039	105.84	63	60.002	115.294
Homogeneous Mixing	Tensile	100	200	61	53.144	96.428	63	106.72	160.25
Homogeneous Mixing	Compression	100	200	61	43.975	56.849	63	21.021	92.238

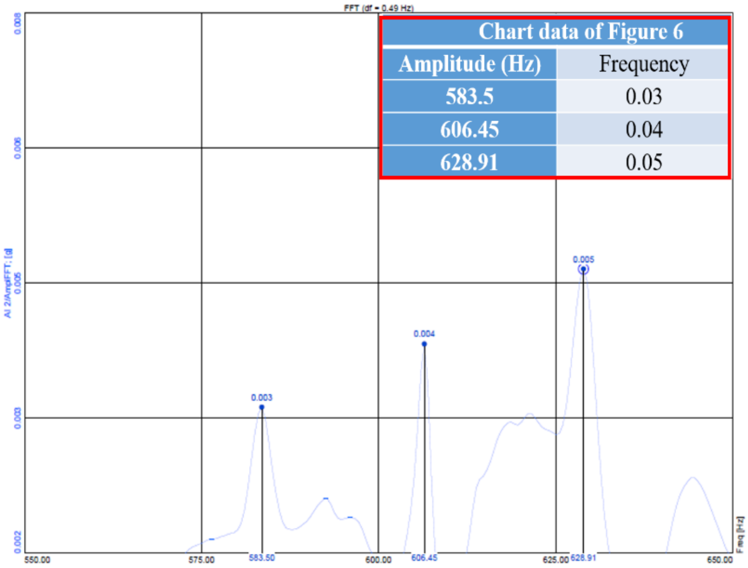


Chart data of Figure 6	
Amplitude (Hz)	Frequency
583.5	0.03
606.45	0.04
628.91	0.05

Fig. 7. Natural frequency of the system (without rubber)

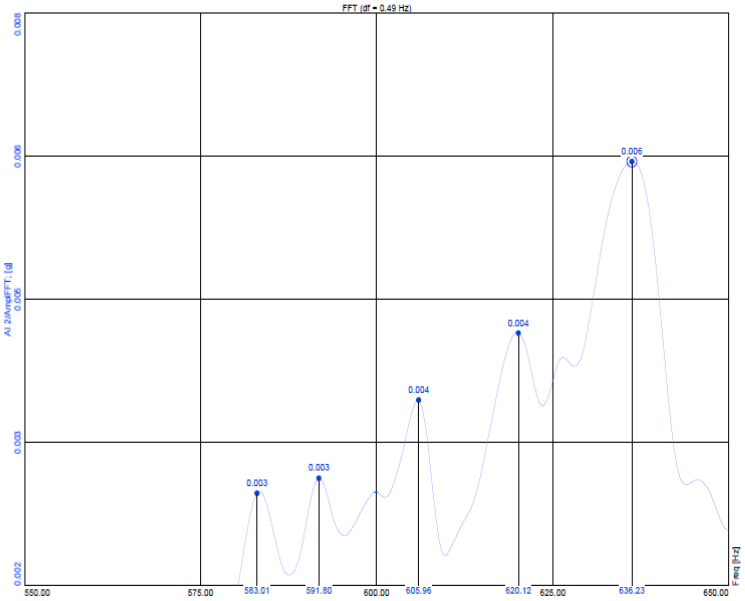


Fig. 8. Natural frequency graphs of MWCNTs reinforced NR rubber produced with homogeneous method

Table 4. Chart data of natural frequency graphs of MWCNTs reinforced NR rubber produced by three methods

MWCNTs -reinforced NR by homogeneous mixing method		MWCNTs -reinforced NR by hand lay method		MWCNTs -reinforced NR by spraying method	
Amplitude (Hz)	Frequency	Amplitude (Hz)	Frequency	Amplitude (Hz)	Frequency
583.01	0.003	606.45	0.005	586.91	0.004
591.80	0.003	613.77	0.007	606.93	0.008
605.96	0.004	630.86	0.007	621.58	0.006
620.12	0.004			635.25	0.005
636.23	0.006				

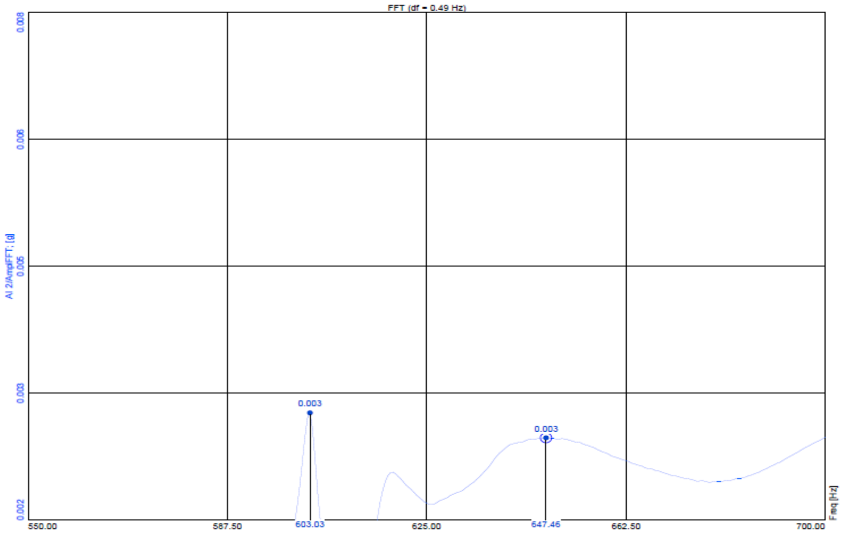


Fig. 9. Natural frequency graphs of MWCT reinforced EPDM rubber produced by homogeneous mixing method

Table 5. Chart data of natural frequency graphs of MWCNTs reinforced EPDM rubber produced by three methods

MWCNTs -reinforced EPDM by homogeneous mixing method		MWCNTs -reinforced EPDM by hand lay method		MWCNTs -reinforced EPDM by spraying method	
Amplitude (Hz)	Frequency	Amplitude (Hz)	Frequency	Amplitude (Hz)	Frequency
603.03	0.003	604.98	0.004	616.21	0.005
647.46	0.003				

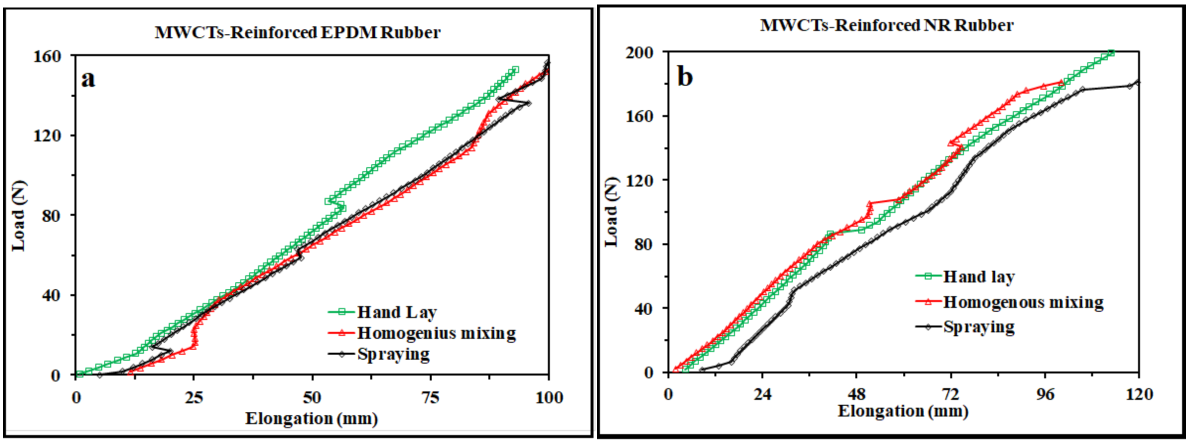


Fig. 10. Tensile loads – elongation plot of MWCTs reinforced (a) EPDM and (b) NR rubbers

The natural frequency values of the system should be in all test setups and these frequencies should be distinguished. It is seen that the natural frequency of the crankshaft without a crank pulley is the closest to the natural frequency measurement values of the pulleys, with a homogeneously mixed MWCNTs reinforced EPDM rubber pulley. When the crankshaft enters the resonance state, it will get by 1.09% closer to the crank pulley and will attract torsional vibrations. A different natural frequency interpretation could not be made for nano-reinforced NR rubber since it has the same natural frequencies as the system. However, since MWCT reinforced EPDM rubber has different natural frequencies, it has destroyed the natural frequencies of the system. As can be seen in Figure 9, the vibration damping ability of EPDM rubber changes with nano reinforcement.

As the preload stress rate increases, the difference between the tensile and compressive loads of the sample mixtures increases. Due to this situation, three different methods that are subjected to the highest load are given in Figure 10. For MWCNTs -reinforced NR rubber, the highest load was with the spraying method, while the least load was formed in the homogeneous mixing method. In

EPDM rubber, while the highest load is in the homogeneous mixing method, the least load is seen in the hand laying method.

CONCLUSION

The mechanical behavior of the MWCNTs reinforced sample rubbers was carried out in two different ways in tensile and compression directions with the tensometer test, and it was observed that the difference in tensile and compression load was that the rubber had a hysteretic structure and did not lose its elasticity. Although there was a difference in torsional stiffness in EPDM rubber within the three methods, such a difference did not occur in natural rubber.

In the natural frequency test, natural frequencies of the system (without rubber) were observed and variable natural frequencies in the system when EPDM and natural rubber were used. It was observed that vibration damping of EPDM rubber with nano reinforcement is different from that of natural rubber.

Declarations: *The authors declare that they have no conflicts of interest.*

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