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Effect of Tungsten concentration on the thermal properties of Ethylene Propylene Diene Monomer (EPDM) composite

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Keywords: Polymer materials, Ethylene Propylene Diene Monomer (EPDM), rubber, Nanocomposite, Tungsten powder (W) nanoparticles.

Abstract:

This study investigates the tungsten concentration effect of Ethylene Propylene Diene Monomer [EPDM] composite on thermal properties, which is synthesized as anti-degradation nanocomposite. The composite was fabricated via ball milling. Structural, morphological and thermal properties of the fabricated nanocomposites were examined by different characterization techniques such as X-ray diffraction (XRD), Scanning Electron Microscopy (SEM) and thermal analysis (TGA) & (DMTA).

It was found that the weight losses due to heat degradation decreased gradually due to the increase in concentration of Tungsten nanoparticles, which confirms the improved thermal stability of EPDM matrix. The onset temperature and the maximum decomposition temperature increase with the increase of tungsten ratio to reaches the maximum at 70% tungsten loading. Conferring to this, the thermal properties and flame resistance of the composite was enhanced with the increase in tungsten concentration due to the high melting temperature of tungsten enforcement and their high latent heat coefficient. The overall thermal properties were enhanced with the increase in tungsten percentage in the composites.

1 Introduction:

Many works were made to enhance EPDM properties including their strength, stiffness and flame resistance to extend their range of applications especially in fields related to healthcare and military applications while maintaining their unique physical and mechanical properties like good dielectric properties, good electricity and heat insulation [1]. Ethylene Propylene Diene Monomer (EPDM) rubber has an excellent resistance to oxidation, heat, ozone and weather ageing due to its stable and saturated polymer backbone structure and for these reasons they are widely used in applications like wire and cable coatings [2]. The growing demand for EPDM in electrical applications is also due to its excellent resistance to degradation and ease of accepting large amounts of fillers [3]. Generally, advancements in polymer composites using dissimilar fillers, adhesive bonding and low-cost materials allow designers and producers to achievement well the benefits of these materials [4,5,6,7]. While disadvantages include complex rheological behavior and difficult fabrication techniques [8,9,10,11,12,13,14]. The experimental work in

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this research was designed conferring to the prerequisite of the military applications (armored vehicles designing needs) to examined their resistance to heat of the produced alloy in addition to their microstructure of the composite.

Radiation hazard has inevitably become a noticeable issue in the world range. In this regard, the preparation of flexible and light materials that efficiently protects radiation workers and the environment become a research effort [15]. E. Kusiak prepared the EPDM composites filled with selected fillers as radiation shielding substance [16]. Jaewoo K. offered a kind of polymer composites which were prepared by dispersing Tungsten nano-additives into ethylene-propylene-based polymer combination using melt mixing and the attenuation of gammas for the composites was enhanced up to 75 % for Ba-133 (0.3 MeV) [17, 18]. Due to its necessary physico-chemical properties and its capability to accept additives, EPDM is widely used in many applications. This can reflect the extent of its industrial importance and potential of use. However, because of its low tensile strength, dense smoke generation as well as some toxic gases release during combustion its practical application has been strictly limited. To overcome these problems and in order to achieve the required strength, and thermal stability, several studies have been conducted to reinforce it with different types and shapes of materials to suit usage in both military and medical applications. In this work, the thermal properties of EPDM/W Nanocomposites with different weight ratios (0, 30, 45, 60, and 70 wt.%) were studied for shielding application.

2 EXPERIMENTAL:

2-1 Materials and Methods

Ethylene Propylene Diene Monomer (EPDM J-4045) containing 5-ethylidene-2-norbornene (ENB) as Diene, which is manufactured by Jilin Petrochem., SINOPEC. The EPDM consists of 52.0 wt. % ethylene, 40.3 wt. % propylene, and 7.7 wt. % ENB. Tungsten powder with purity 99.9%, Particle size 0.5-2 μm, density 19.3 g/cm3, and melting temperature. 3410˚ C was supplied by Sigma - Aldrich, the powder was continuously ball milled for particle size reduction until it reaches Nano scale. Milling was carried out using a SPEX 8000M MIXER/MILL. The ball milling process was performed using balls and tank made from chromium steel with a ball to powder mass ratio of 10:1.

Figure 1. The Two instruments used in the clay modification (a) Magnetic stirrer and (b) High speed mixer.

2-2 Sample Preparation (Preparation of EPDM Nanocomposites)

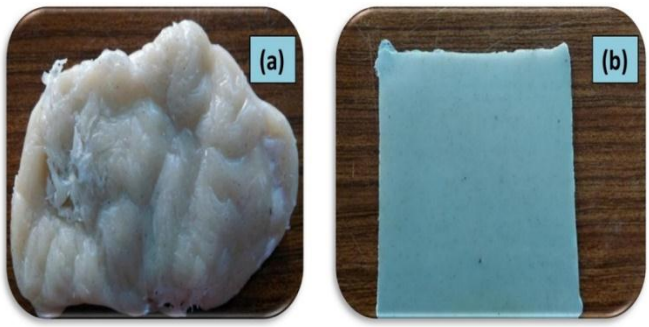

Figure 2. The shape of the prepared composites. (a) After melt mixing and (b) After compression.

The EPDM nanocomposites were prepared by masticating the EPDM on a two-roll mill for 10 min with different weight percentages (0, 30, 45, 60, and 70wt. %) of tungsten (W) nanoparticles with respect to EPDM content. According to ASTM D-1928, the fabricated nanocomposites were molded into sheets with dimensions of 150 x 150 x 2 mm3 at temperature of 150 ℃ and pressure of 15 MPa for 3 minutes. At the end, the samples were cooled for further testing.

2-3 Characterization

2-3-1 Powdered X-ray Diffractometer

The crystallite phases of rectangular sheet samples of EPDM containing of 0, 30, 45, 60, and 70 wt. % tungsten Nano powder with dimensions of 10x10x1 mm3 were identified by Powdered X-ray diffraction (XRD) using a step size of 0.04◦ and at a scanning rate of 1.2◦/min with continuous Scan mode on a ARL™ X'TRA Powder diffract meter, Thermo Fisher Scientific Inc. Available at Science and technology of excellence (STCE) with crystallographic data software Winxrd program attached with ICDD laboratory information using metal ceramic tube Copper Target (with Cu-K-alpha wave length=1.5405981 Å) radiation operating at accelerating voltage and applied current were 44 kV and 45 mA, respectively, Ni Filter and scintillation detector (NaI (TI) scintillation crystal). The diffraction data was recorded for 2Θ values between 10◦ and 70◦.

2-3-2 Microstructure examination of the produces composite using Scanning Electron Microscopy (SEM)

The microstructures of the Tungsten powder and the sheet samples of EPDM contents of 0, 30, 45, 60, and 70% Tungsten powder nanoparticles were also analyzed using Quanta FEG 250 scanning electron microscope (FEI Comp., USA. Samples were mounted onto SEM stubs. Applied SEM conditions were a 10.1 mm working distance, with in-lens detector with an excitation voltage of 20kV.

2-3-3 Thermal Analysis of the produced material to characterize its thermal properties

Thermo-gravimetric analysis (TGA) was carried out using a (TGA Q500, TA instruments) that used to evaluate the thermal stabilities of the sheets, thermo balance sensitivity 0.1µg. Samples were heated in nitrogen, flowing rate at 50 ml min−1 from 25 to 500℃ at a heating rate of 10℃ min−1. Sample weights ranging from 5 mg to 10 mg were used.

Dynamic mechanical thermal analysis (DMTA) was carried out using a (TA Instruments DMA Q800). The sample dimensions were (20 mm x 7 mm x 0.5 mm) and analyzed in tension mode. The tests were carried out from -20 to 100℃ at frequency (1 Hz) with a standard heating rate of 5℃ min−1.

3 Results and discussion:

3-1Powdered X-ray Diffractometer

X-ray data shown in Fig.3 indicate that the main peaks of tungsten powder are identified at angles 40.41◦, 73.32◦ and 58.35◦ as in the standard card no: 00-01-1204 that indicate the tungsten powder did not react with the polymers but physically dispersed. The diffraction patterns displayed in Fig.1. shows that adding of the tungsten nanoparticles with different weight percentages from 0% (pure polymer EPDM) up to 70% change the degree of crystallinity, which measured by area under the peaks to the whole area [19,20,21,22,23,24].

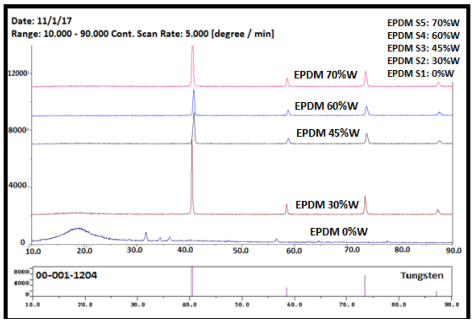

Fig. 3. XRD patterns of EPDM /

3-2 Scanning Electron Microscopy (SEM)

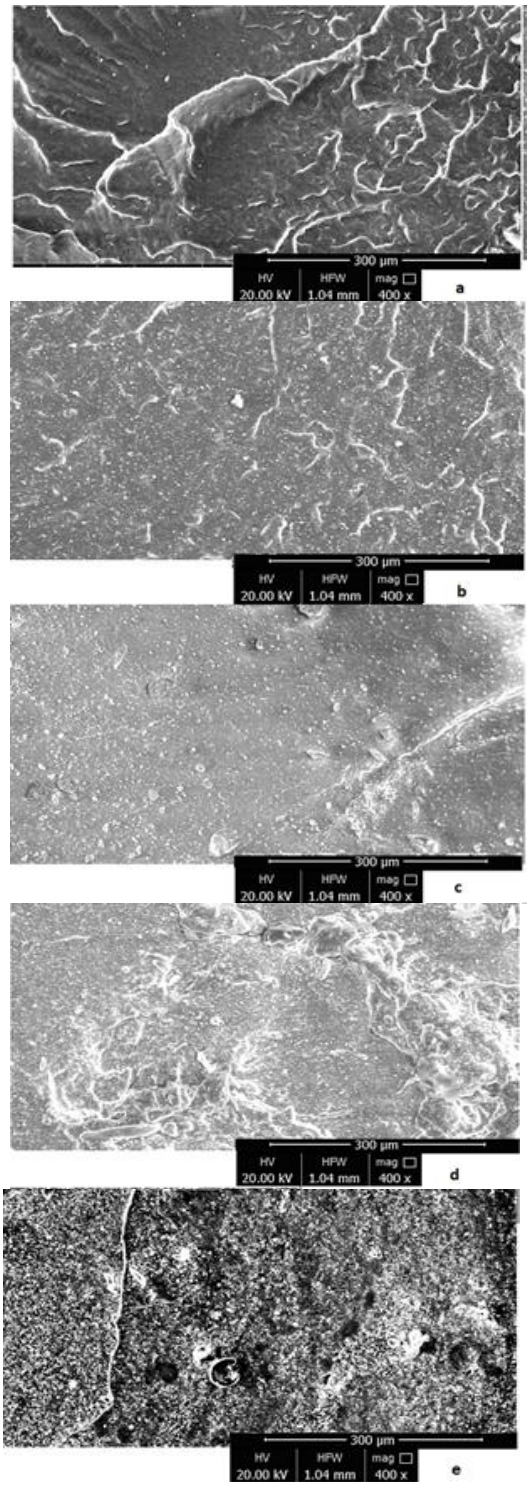

Fig. 4. SEM images of EPDM (a) 0wt% W, (b) 30wt% W, (c) 45wt% W, (d) 60wt% Wand (e) 70wt% W.

Fig.4 (a, b, c, d and e) show the scanning electron microscope (SEM) imaginings of the microstructures of EPDM 0 wt% W, 30wt% W, 45wt% W, 60wt% W, and EPDM-70% W Nanocomposites, respectively using liquid nitrogen fractured samples. The images show the Tungsten nano-additives are well dispersed and adhered in the EPDM matrix and the Tungsten particles spreads consistently within the polymer matrix

3-3 Thermal Analysis

Thermal stability of EPDM and its Nanocomposites

Table (1): Data derived from TGA curves of EPDM composites

sample	Mass fraction of total weight loss (%) at 600°C	Onset temp, °C	Max. decomp. Temp. °C	Residue % at 600°C
S1 (EPDM-0%)	97.2	220	462	2.8
S2 (EPDM-30%)	68.68	224	464	31.34
S3 (EPDM-45%)	56.79	226	463	43.21
S4 (EPDM-60%)	43.6	229	469	56.4
S5 (EPDM-70%)	35.26	232	471	64.74

TGA curves of EPDM with different tungsten doses are seen in Fig.5. The thermal stability data is shown in Table (1) indicated that the pure EPDM sample (S1) showed degradation at two stages. The first from about 200 ^{o} C to 380 ^{o} C and the second one from 390 ^{o} C to 500 ^{o} C. The thermal degradation of EPDM is attributed to the haphazard chain scission and hydrogen abstractions from the tertiary carbon involved in intermolecular transference [25]. In EPDM composites, the

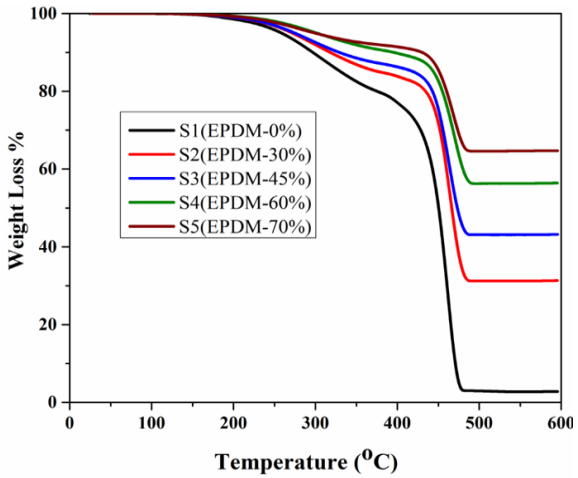

Fig 5. TGA curves of EPDM/W nanocomposites

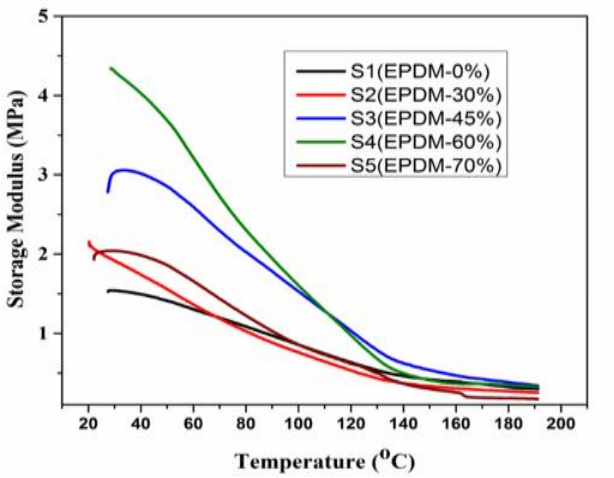

Fig. (6): Storage modulus of EPDM and its nanocomposites

filler-polymer networks minimize the permeability of volatile components in the polymer matrix and to suppress thermal degradation of the elastomers, which may be caused by barrier effects or a reduction of the pyrolysis rate due to the decrease of the polymer mobility. In fact, it has been shown that polymer chains restrained in the mesoporous structures show greater thermal stabilities. The weight loss percentage decreased gradually, this confirms that the introduction of tungsten could improve the thermal stability of EPDM matrix. This is might be due to the core/shell structured nano-W particles with solid adhesive encapsulation caused by atomic interstitial interdiffusion of W and the methylene group of PE of EPDM on the surface without any chemical reaction among the rubber chain and nanoparticles using simple dry blending. Moreover, mass fraction of the residual (or residue) percentage, the onset temperature and the maximum decomposition temperature increase with the increase of tungsten ratio to reaches the maximum at 70% tungsten loading.

Fig.6 shows the Dynamic Mechanical Thermal Analysis DMTA traces (storage modulus versus temp.) of EPDM nanocomposites. For EPDM and its Nanocomposites, EPDM sample (S1) showed lower storage modulus than other samples. By incorporation of tungsten nano into the matrix of EPDM sample (S2), a small increase in the value of storage modulus was observed in the rubbery region. More increase in the filling content leads to more growth in the storage moduli up to sample (S4). Because of the strong filler-rubber interaction, nanofillers increase storage moduli greatly, and the dynamic heat build-up. In fundamental, dynamic heat, build-up mainly results from the frictions of various networks such as rubber-rubber, filler-rubber, and filler-filler friction. In theory, steadier filler network will decrease the friction between filler particles effectively, and stronger interfacial interactions between filler particles and rubber chains will decrease the relative slippage and the filler-rubber friction. On the basis of these analyzes, the higher storage moduli of modified nano tungsten filled composites should be attributed to enhancing the interactions between filler particles and rubber chains through physical bonding, improving the dispersion of filler and forming the steadier filler network. More increase of tungsten filler leads to failure in the storage modulus of sample (S5). This is may be attributed to the aggregation and agglomeration of nanofiller in the matrix of EPDM.

4 Conclusions

EPDM composites samples with different percentage of Tungsten Powder nanoparticles were prepared. The structural, morphological, and thermal properties of the prepared nanocomposites were examined. It was found that the produce composite has homogenous dispersion of the reinforcement particles within the composite matrix, the thermal properties and flame resistance of the composite was enhanced with the increase in tungsten concentration due to the high melting temperature of tungsten enforcement and their high latent heat coefficient. the onset temperature and the maximum decomposition temperature increase with the increase of tungsten ratio to reaches the maximum at 70% tungsten loading. Overall thermal properties were enhanced with the increase in tungsten percentage in the composites.

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