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Thermal Properties of Nano Tungsten - Ethylene Vinyl Acetate (EVA) Composites

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Abstract. Ethylene vinyl acetate (EVA) with different percentage of nano tungsten powder (0wt%, 30/wt %, 45/wt %, 60/wt%, and 70/wt %) composites (EVA/W nano-composites) were prepared by melt blending in a twin-screw brabender. The structural, morphological, thermal and mechanical properties of the prepared nano-composites were investigated using powder X-ray diffraction (PXRD), Scanning Electron Microscopy (SEM), thermogravimetric analysis (TGA), and dynamic mechanical thermal analysis (DMTA). It was found that the EVA composites show a higher thermal stability at high levels of tungsten loading.

Keywords. EVA, Nano tungsten powder, Higher thermal stability.

1 Introduction:

Polymers in general have low stiffness and strength, poor thermal properties, good dielectric properties and they are good insulators of electricity and heat. In addition, they are non-magnetic. Many attempts have been made over the years to change some of these properties for use in more applications. One of these attempts is reinforcing polymers with metal nanoparticles to enhance the polymer properties in different applications. The overall composites reinforced with nano-fillers was found to vary widely depending on type and content of reinforcing filler, dispersion, filler size and shape, and type of coupling agent. For polymeric based composites type and percentage of reinforced nano-composites are selected to impart some degree of metallic behavior to the polymer matrix. [1]

Ethylene vinyl acetate (EVA) is the copolymer of ethylene and vinyl acetate segments characterized by different acetate contents, and typically formed via free radical polymerization [2, 3]. Due to its desirable physico-chemical properties and its ability to accept additives, EVA is widely used in many applications: it can be used in electronic devices, electrical engineering, wire and cables, buildings, and transportation (aircraft, cars) [4]. This reflects the extent of its industrial importance and potential use. However, because of its low tensile strength, dense smoke generation as well as release of toxic gases during combustion its practical application has been strictly limited [5–7]. To overcome these problems and to improve its strength, and thermal stability, several studies have been conducted [8–11].

In this study, EVA with different percentage of Nano Tungsten powder -composites (0%, 30%, 45%, 60%, and 70%) was prepared, to improve its dynamic mechanical and thermal properties.

2 Experimental

2.1. Materials and Methods



EVA copolymer with a content of 18% vinyl acetate by weight, a melt flow index of 1.83 g/10 min and density of 0.98 g/cm³ was supplied by Tane Elf Atofina, China. Tungsten powder with purity 99.9%, particle size 0.5-2 μm, density 19.3 g/cm³, and melting point 3410°C was continuously ball milled for particle size reduction until reaching 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. Scherrer equation, was used to calculate size of sub-micrometer crystallites in a solid to the broadening of a peak in a diffraction pattern using X-ray diffraction and crystallography, and the results showed that the tungsten particles after milling was in the Nano scale range.

2.2. Sample Preparation (Preparation of EVA Nano-composites)

EVA/W nano-composites, with different amounts of W nano particles (0, 30, 45, 60, and 70 wt. %) with respect to EVA content, were extruded. The EVA/W nano-composites were prepared via melt blending. Melt blending was performed in a twin-screw extruder (10 mm twin-screw extruder, Rondol, England) at 160 °C and screw speed of 60 rpm for 10 min. The extrudates were cooled down through passing in a water bath and then pelletized (10mm Diameter * 2 ± 0.05 mm thickness).

2.3. The phase structure

2.3.1. Powder X-ray Diffractometer.

The crystallite phases of sheet samples with dimensions 10×10×1 mm³ of the prepared EVA composites were identified by Powder X-ray diffraction (PXRD) using a step size of 0.04° and at a scanning rate down to 1.2 °/min. with continuous scan mode using ARL™ X'TRA powder diffractometer. Thermo-Fisher Scientific Inc. The diffraction data was recorded for 2θ values between 10° and 70°.

2.3.2. Scanning Electron Microscopy (SEM)

The microstructure of the prepared EVA composite samples was also analyzed using Quanta FEG 250 scanning electron microscope (FEI Company, USA). Samples were mounted onto SEM stubs. Applied SEM conditions were: a 10.1 mm working distance.

2.3.3. Thermal Analysis

TGA instrument model Q500 (TA instruments), characterized by thermo balance with sensitivity 0.1 μg, was used to evaluate the thermal stability of the EVA composite sheets. Samples were heated in nitrogen, flow rate at 50 ml min⁻¹, from 25 to 500 °C at a heating rate of 10 °C min⁻¹. Sample weights ranging from 5 mg to 10 mg were used. The storage modulus of EVA and their nano-composites were conducted by DMA Q800 (TA instruments) from RT to 190 °C with heating rate 10 °C min⁻¹ using dual cantilever mode.

3 Results and discussion

3.1. Powder X-ray Diffractometer

Our data in figure 1 shows that the main peaks of tungsten powder are identified at angles 40.41°, 73.32° and 58.35° as per the standard card no. 00-001-1204 which indicates that tungsten powder did not react with the polymer but physically dispersed.

The diffraction patterns displayed in figure 2, show that adding tungsten nano powder with different weight ratio from 0% (pure polymer EVA) up to 70% changed the degree of crystallinity, which measured by area under the peaks to the total area, of the prepared samples.

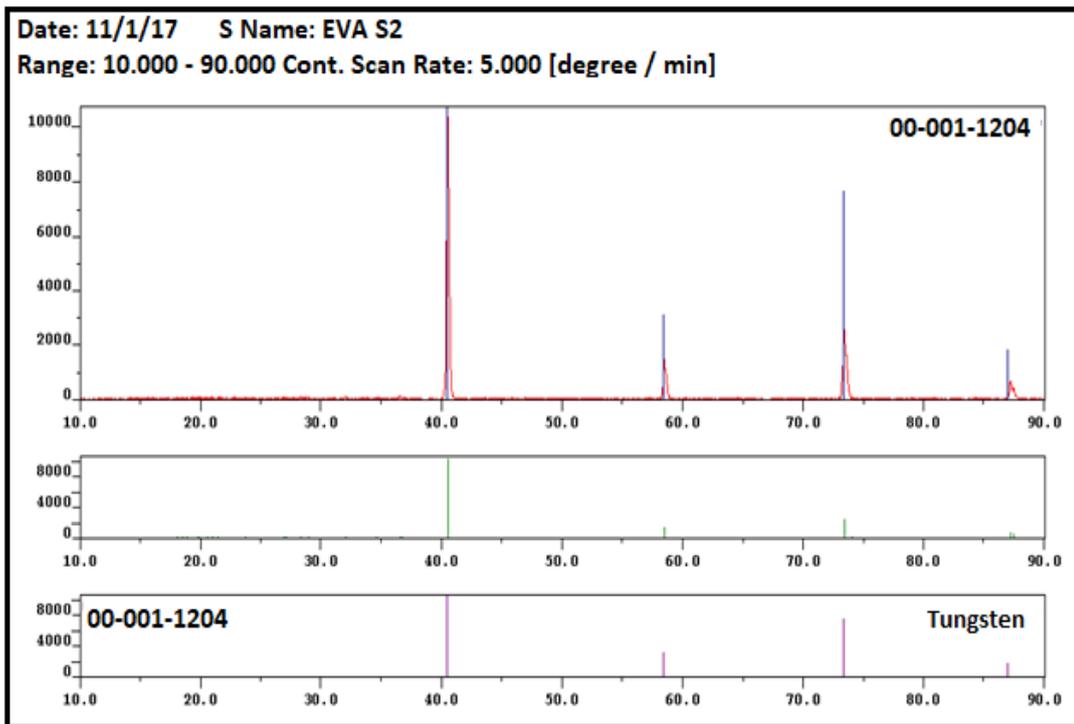


Figure 1. EVA-30%W sample with respect to W XRD card

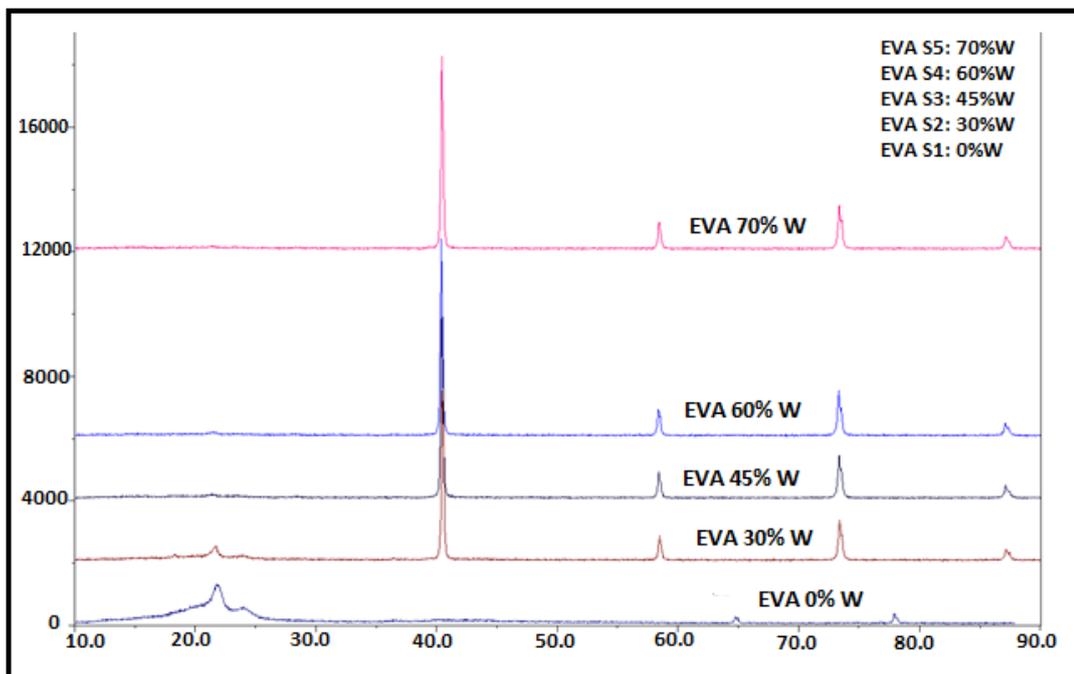


Figure 2. The PXRD profile of EVA samples with 0 wt% W,30 wt% W,45 wt% W,60 wt% W,70 wt% W

3-2 Scanning Electron Microscopy (SEM)

Figure 3 a, b, c, d and e, show the scanning electron microscope (SEM) images of the microstructure of EVA-0%W, EVA-30%W, EVA-45%W, EVA-60%W, and EVA-70%W nanocomposites, respectively using liquid nitrogen fractured samples.

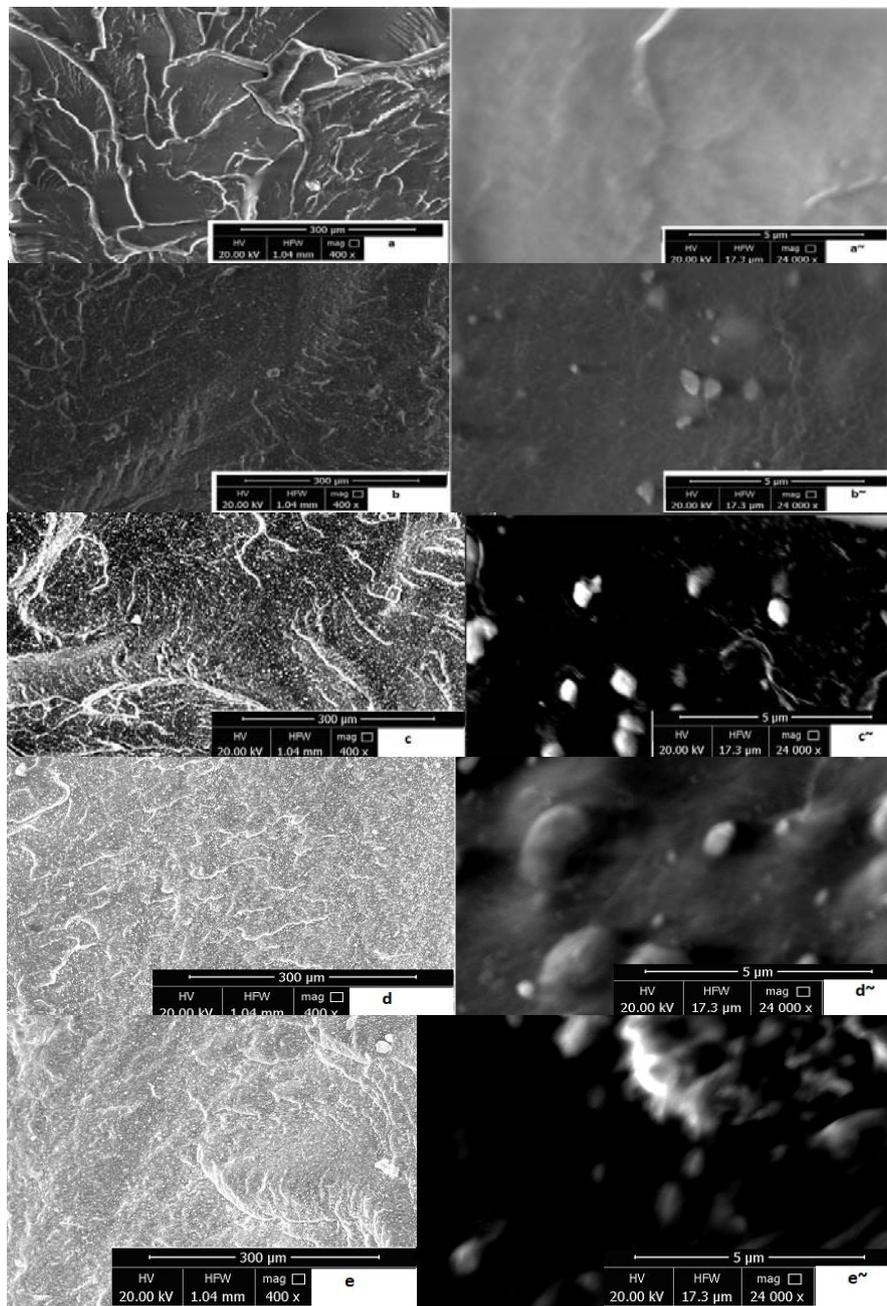
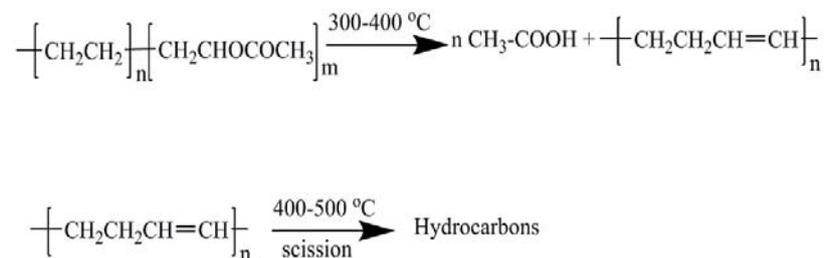


Figure 3. SEM images of EVA composites: (a) 0 wt% W, (b) 30 wt% W, (c) 45 wt% W, (d) 60 wt% W and (e) 70 wt% W.

The images show that the nano-tungsten particles are well dispersed and adhered in/with the EVA matrix and the tungsten powder spreads homogeneously within the polymer matrix. The examination also shows that the matrix/ nanoparticles adhesion and dispersion are very efficient since the fractured cracks tend to develop through the matrix itself, not at the interface between the particles and polymer matrix, which confirm the strong mechanical bond (Particle mechanical interlock with in polymer matrix) of the reinforced composite.

3.3. Thermo-gravimetric analysis (TGA)

The mass loss profile of EVA in figure 4 shows a two stage of degradation pathway with the first step attributed to the elimination of acetate side groups (de-acetylation) leaving behind an unsaturated polymer backbone or polyene [2], while the second step is due to the allylic chain scission of the polyene to reach full thermal degradation at high temperatures [12], Scheme 1.



Scheme 1. Degradation of EVA

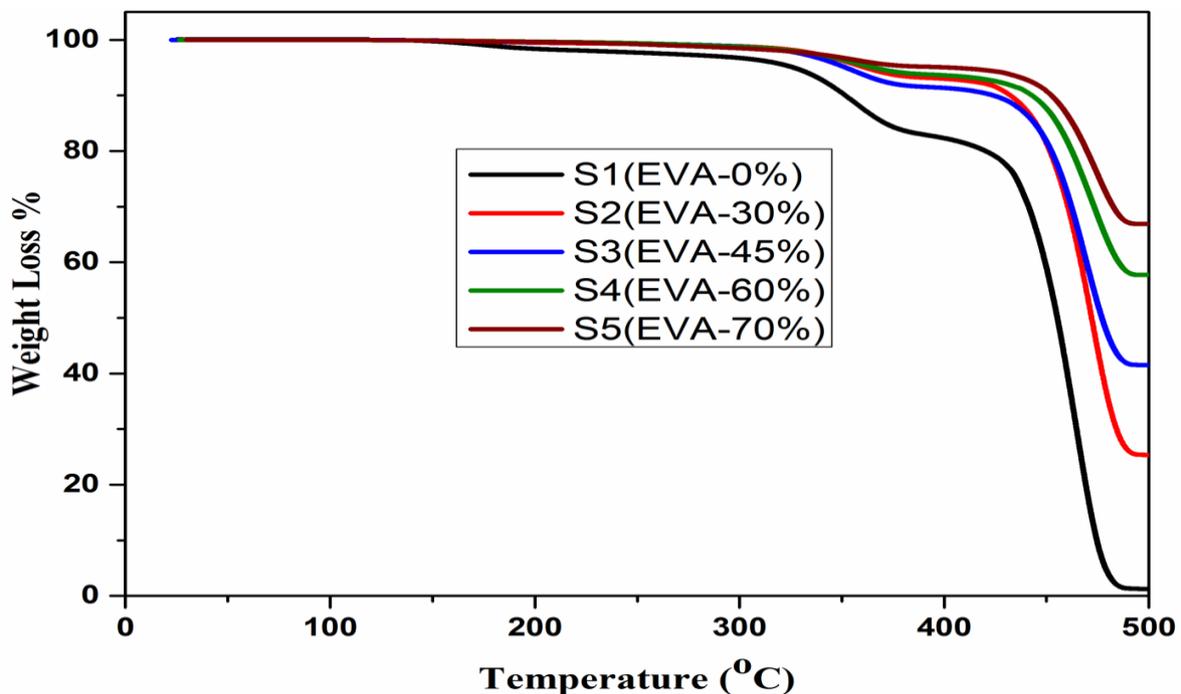


Figure 4. TG curves of EVA and its nano-composites

Introducing tungsten into the matrix of EVA has enhanced its thermal stability as shown in the previous figure. The TGA profiles of pure EVA and its composites are similar however, the curves shift to a higher degradation temperatures relative to pure EVA with the increase of reinforcing filler contents. The total weight loss of pure EVA, which was 98.97%, decreased by introducing of tungsten as shown in table 1, where the residue at 500 °C reaches its maximum value (66.97%) in sample S5 (70%W).

Table 1. Data derived from TGA curves of EVA composites

sample	Mass fraction of total weight loss (%) at 500°C	Residue % at 500°C
EVA samples		
S1 (EVA-0%W)	98.79	1.24
S2 (EVA-30%W)	74.63	25.37
S3 (EVA-45%W)	58.48	41.53
S4 (EVA-60%W)	42.23	57.77
S5 (EVA-70%W)	33.11	66.89

3.4. Dynamic Mechanical Thermal Analysis (DMTA)

DMTA is a technique in which the storage modulus (elastic response) of the sample, under oscillating load, is monitored against temperature, time or frequency of oscillation. During the test, a fraction of energy is absorbed (E'' , viscous component), whereas the other fraction is returned elastically (E' , elastic component).

Figure 5 shows the DMA traces (storage modulus versus temperature) of EVA and EVA nano-composites. Pure EVA sample (S1) showed lower storage modulus than other samples. By incorporation of tungsten nano into the matrix of EVA sample (S2), a remarkable increase in the value of storage modulus was observed in the rubbery region.

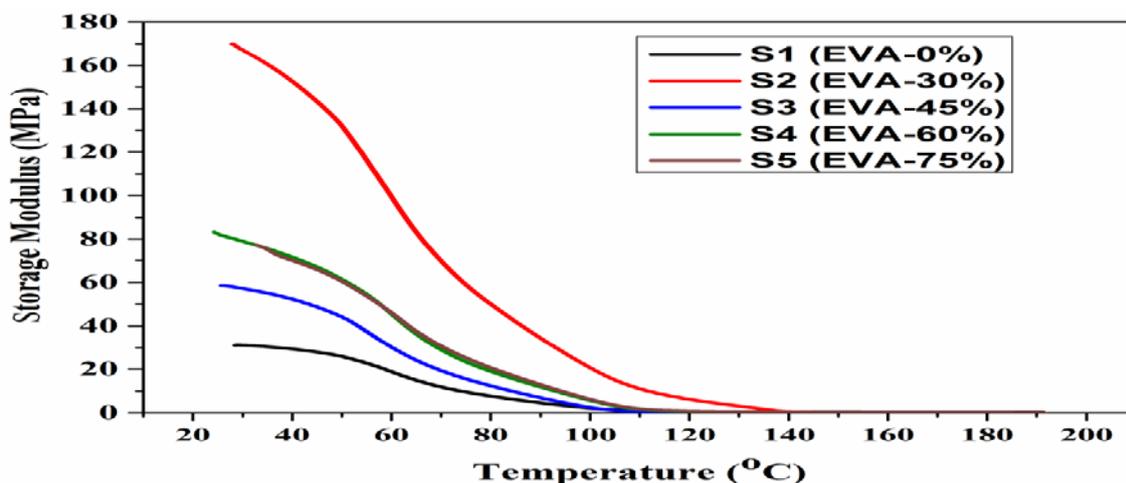


Figure 5. Storage modulus of EVA and its nano-composites

More increase in the filling content leads to decrease in the storage but still higher values than virgin polymer, this means that 30% nano-filler is sufficient to give the best optimum condition in the case of EVA. The high storage modulus of EVA nano-composite samples could be explained by the reinforcing role of the tungsten nanoparticles and strong established link. More increase of tungsten filler leads to failure in the storage moduli. This is may be attributed to the aggregation and agglomeration of nano-filler in the matrix of EVA.

4 Conclusions

EVA composite samples with different percentage of Tungsten nano Powder (0 wt%, 30/wt%, 45/wt%, 60/wt%, and 70/wt%) were prepared by melt blending in a twin-screw brabender mixer. The structural, morphological, thermal and mechanical properties of the prepared nanocomposites were investigated. It was found that the prepared composite have homogeneous dispersion of the reinforcement particles within the composite matrix. The thermal and dynamic properties of the composite were enhanced with the increase of tungsten concentration due to the high melting point of tungsten.

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