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Preparation and Characterization of Nano Composite Materials Based on Polyisoprene Added to Nano Ternary Silica

Fayq Hasan Jabbar[1], Fadhil Muhi Mohammed[2*]

[1]Department of Materials Engineering, College of Engineering, Mustansiriyah University, Baghdad, Iraq
[2] Department of Civil Engineering, College of Engineering, Al-karkh University of Science, Baghdad, Iraq

[*]Email: fadhil.mohammed@kus.edu.iq

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Abstract

Polyisoprene (PI), whether naturally or synthetically produced, is used in rubber compounds for many applications, such as tires, belts, and shoes. The effect of Nano ternary silica (NTS) addition on the thermal and mechanical characteristics of PI was investigated in this research. According to the results, the addition of NTS enhances the hardness and mechanical strength of PI, improves thermal properties, reduces thermal deformations, and enhances the ability of the resulting composite materials to withstand high-temperature conditions and resist environmental deformations. The weight percentages for the added NTS in the prepared composite materials followed five models (2 %, 4 %, 6 %, 8 %, and 10 wt. %). The optimal percentage of polyisoprene nanocomposite was determined using X-ray diffraction (XRD), thermogravimetric analysis (TGA), Transmission Electron Microscopy (TEM), and atomic force microscopy (AFM). It was found that at 4 wt.% NTS with a d-spacing between layers of 4.1 nm, the best thermal stability and mechanical properties are obtained. These results suggested that the stability and properties of PI depend on the amount and particle size of the NTS distribution mixture.

Keywords: Fumed Silica, Nano Material, Polymeric Material, Thermal Characterization, Thermogravimetric.

1. Introduction

Polyisoprene is available from plant sources as natural rubber latex and from petroleum derivatives as synthetic polyisoprene. Synthetic polyisoprene exhibits high elasticity and stability over a wide temperature range, and it offers a high degree of flexibility, with properties similar to those of natural rubber. These characteristics make it suitable for various industrial applications, including the manufacture of tires, belts, footwear, and hoses. Polyisoprene is a latent and lightweight material known for its high ductility, low thickness, low stiffness, and resistance to surface abrasion. Additionally, polyisoprene has been widely applied in the electrical cable industry due to its excellent electrical insulation properties. Polyisoprene is a diene polymer derived from a monomer and containing two unsaturated carbon-carbon double bonds [1]. It is known for its ease of manufacturing and processing, which has led to widespread applications in structural engineering and other fields [2],[3]. Typically, X-ray Diffraction of polyisoprene shows an amorphous pattern rather than distinct peaks, indicating the absence of long-range intermolecular order [4].

Likewise, the composite material comprises the base matter (Matrix Material) and the reinforcing matter (Reinforcement). These matters are characterized by low density, high durability, low weight, low production cost, and resistance to corrosion or rust. Due to the high mechanical and thermal properties of reinforced polymers, which have led various industries to adopt them, the best composite materials are those based on polymers [5]-[7]. Depending on the intended purpose, the base material's reinforcements come in a variety of shapes and types. They either take the shape of scales, fibers, particles, or powder [8]-[10].

Currently, studies on the nanoscale have made significant contributions to the development of new types of nanocomposites by using nanoparticles with diameters below 100 nm. Consequently, they are employed to strengthen and support nanomaterial (Nano supports). Also, in view of nanoscale utilization, nanoparticles have the potential to enhance the mechanical and thermal properties of the base material and increase its resistance compared to conventional micro particles [11]-[13]. The process of polymeric materials supported by particles, such as natural SiO_2 nanoparticles, is

important, as it has encouraged scientists and researchers to conduct numerous studies [14], [15].

Previous studies [16], [17] examined the mechanical properties of silica (SiO_2)- reinforced polyester resin and polyvinyl resin at different weight ratios. The modulus of mechanical experiments showed that the bending and shear strengths of polyester and polyvinyl increased with the addition of SiO_2. The results also showed that the base material has a lower elastic modulus than the composite material. In addition, it was found that the hardness of these polymers increased with increasing nano-silica concentration. Also, similar results have been reported by Alnamel and Mudhaffer [18] with the addition of 3% and 5% SiO_2 to the polymethyl methacrylate denture (PMMD). In comparison, a significant reduction was observed at 7% SiO_2 in impact- and transverse-strength specimen tests. In addition, a significant increase in surface hardness was observed at 3, 5, and 7% SiO_2 additions. The preparation of NTS gel was carried out by Koch and Holthausen [19], who illustrated the transformation of primary nanosilica into secondary and, subsequently, ternary porous nanosilica gel. A distance of around 2.4 nm separates the tiny conglomerates of this gel. Using a built-in blender (H. Poly-drive), the planned quantity of PI was prepared according to the procedures described by Numan et al. [20]. In the current work, a new nanocomposite (PI/NTS) was fabricated. The effect of the relative weight of NTS addition to PI on the thermal and mechanical properties, in addition to the structure and surface morphology of the produced nano-PI, was studied.

2. Materials and Methods

2.1 Materials

This experiment is conducted using the Standard Malaysian Rubber (controlled-viscosity, CV60) Score PI, kindly provided by the Malaysian rubber industry, as well as nano-ternary silica (NTS). NTS, also known as fumed silica with the chemical formula of SiO_2, is originally available as zero nano-silica.

2.2 Methods

2.2.1 Preparation of PI / NTS

The PI was first relaxed for 1 minute, then combined with the required amount of NTS over the next 2 minutes. After that, a pressure regulator was used to heat the electrically heated part to 130 °C for 10 minutes under a stress of 150 kg cm^{-2}. After the molding process, the compounds were immediately cooled for five minutes at room temperature. Table 1 includes the PI and NTS dosages utilized in this study.

2.2.2. Characterization

The prepared material was characterized using XRD, TGA, TEM, and AFM. Copper target (Cu Kα) emission from the SHIMADZU XRD 6000 interferometer (λ = 0.15406 nm) was used to conduct the XRD investigation. The intrinsic structure was examined at a scan rate of 1° per minute in the range of 2° to 10°. Additionally, a thermogravimetric analyzer made by PERKIN ELMER, the TGA 7 model, was employed to investigate the samples' thermal stability.

Table 1. The quantities of polyisoprene and nano-ternary silica in the nano-composite.

Model Character	%wt. nano ternary silica	Mass of PI, gm	Mass of NTS, gm
PI/NTS0	0	50.00	0.00
PI/NTS1	2	49.00	1.00
PI/NTS2	4	48.00	2.00
PI/NTS3	6	47.00	3.00
PI/NTS4	8	46.00	4.00
PI/NTS5	10	45.00	5.00

The samples were heated to temperatures between 35 and 1000 °C at a rate of 10 °C min-1 while under a nitrogen atmosphere at a flow rate of 20 mL. min^{-1}. Energy-filtered transmission electron microscopic (EFTEM) was used to examine the dispersion of PI. TEM images were captured using a LEO 912 AB EFTEM with an acceleration voltage of 120 keV. The samples were prepared using a Reichert-Jung Micrometeorite Quick E, and 100 nm-thick sections were cut using a diamond knife at -120 °C. Additionally, an atomic force microscope (AFM), a high-resolution scanning probe microscope, consists of a cantilever arm with a sharp tip used to scan the sample surface. The cantilever arm is typically made of silicon or silicon nitride with a radius of a few nanometers.

3. Results and Discussion

3.1 XRD

X-ray diffraction is a powerful tool for characterizing crystalline polymers. XRD determined the d-spacing of the NTS gel. According to Bragg's law, $n\lambda = 2d \sin h$ where n is a positive integer determined by the order of diffraction, d is the separation between two NTS layers in succession, and λ represents the wavelength of the X-rays that are incident at an angle h. XRD examination is utilized to measure the scattering signal at $2h = 3.91°$ using raw NTS. This value indicates a d-spacing of 2.40 nm for the NTS. It was also used to measure the NTS in the PI matrix. Table 2 provides a summary of the intercalated NTS in PI/NTS nanocomposites as determined by the XRD examination.

Table 2. XRD analysis of PI/NTS Nano-composites.

Nanocomposite	Percentage of NTS				
PI/NTS	2%	4%	6%	8%	10%
d-spacing (nm)	2.93	4.11	3.43	3.07	2.50

The comparison of d-spacing between layers for composite materials (NTS with IP) calculated in this work with those determined by Jabbar and Latif [2] is shown in Table 3. It can be observed from this table that natural rubber (NR) blends with fatty amides (FAs) synthesized from vegetable oil were used for the modification of montmorillonite (MMT), NR/MMT-FAs have a lower d-spacing compared with modified bentonite blends with fatty amides synthesized with corn oil (FAco) added to polymethyl butadiene (PMB), PMB/FAco-BTT. The lower d-spacing between intercalated layers of the modified polymer results in better thermal stability and mechanical properties.

Table 3. Comparison of d-spacing layers for different nanocomposite materials.

Nanocomposite	2*h* degree	d-spacing (nm)	Ref.
PI/NTS	3.91	4.11	This work
PMB/FA_{co}-BTT	4.71	3.28	[3]
NR/MMT-FAs	2.75	3.14	[2]

3.2 TGA

Thermo-gravimetric analyses between 200 °C and 1000 °C was used to demonstrate the weight loss of raw NTS correspondence to the nanocomposite of grafted PI at 2%, 6% and 4% weight percent of NTS as shown in Fig.1. This weight loss was determined using tangent method on curves presented in Fig.1.The breakdown of the nanocomposites began faster than it was expected, i.e., at 345, 364, and 380 °C for PI containing 2%NTS, 6%NTS and 4%NTS, respectively. It was observed that with the 4 wt. % NTS added to PI, the thermal stability was enhanced compared to the resulting nanocomposite when adding 2 wt. % or 6 wt. % of NTS. This is due to the high homogeneity between PI and NTS at 4% weight percent of NTS, as shown in Figs. 2 and 3 for TEM and AFM images, respectively.

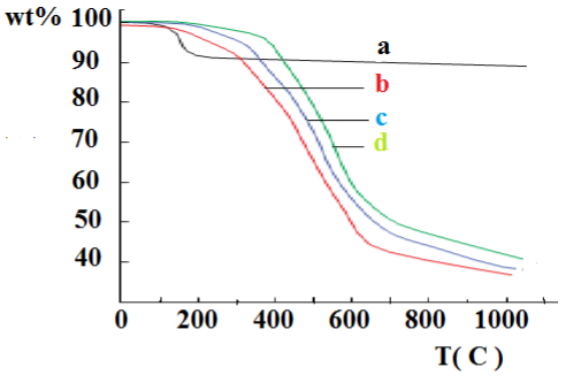

Figure 1. Thermo-gravimetric analyses of (a) raw NTS, (b) PI/2 wt.% NTS, (c) PI/6 wt.%, and (d) PI/4 wt.% NTS.

3.3 TEM

Pure PI composites were reinforced with NTS. The TEM image of the PI/4 wt. % NTS nanoform, which displays good features and compound occurrences, was presented in Fig. 2. The PI/4 wt.% NTS demonstrates a more uniform dispersion of silica without NTS aggregates. Additionally, the PI/NTS XRD pattern clearly demonstrates that the d-spacing decreases with a substantial rise in NTS contents.

Figure 2. TEM image of a PI/4wt.%NTS nanocomposite.

3.4 AFM

The model's exterior surface is depicted in Fig. 3 using atomic force microscopy, which also shows the size, shape, and homogeneity of the manufactured nanoparticles. The spherical shape exhibits effective sorting, with a homogeneous size distribution and no agglomeration. The figure also shows that the NTS nanoparticles exhibit different sizes and spherical shapes, yet they remain in the nano range. The photo was captured at 100 nm. Additionally, the AFM test supports TEM and XRD when naturally obtaining a PI nanocomposite.

Figure 3. AFM image of a PI/4wt.% NTS nanocomposite.

4. Conclusions

Fabricating composite polymers with reinforcing agents is a good approach to improve thermal and mechanical properties. In this research, nano-ternary silica (NTS) was added to polyisoprene to create nano-PI. TGA, TEM, and AFM were used for diagnosis and to obtain the best readings and images, achieving better morphology and nanostructure.

The results from the diagnostic devices used indicated that adding 4% by weight of NTS to the polymer used (PI) resulted in the best crystalline structure for the resulting nanopolymer in terms of crystalline arrangement. By distributing the nanocrystals evenly within the polymer, the best mechanical properties in strength and durability of the polymer were achieved, along with improvements in its thermal properties. Therefore, it was concluded that adding 4 wt.% NTS to the PI led to improvements in the thermal and other mechanical properties, with a d-spacing of 4.11 nm.

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Author Contributions

Fayq Hsan Jabbar proposed the research problem and verified the analytical methods.
Fadhil Muhi Mohammed developed the theory, performed the computations, and revised the English language grammar.

Conflict of interest

The authors declare that there are no conflicts of interest regarding the publication of this manuscript.

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