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Mechanical and physical properties of nanosilica/nitrile butadiene rubber composites cured by gamma irradiation

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ABSTRACT

Nitrile Butadiene Rubber (NBR) nanocomposites are prepared with different contents ranging from 5 to 20 phr (part per hundred parts of rubber) of Sinai sand nanoparticles and fumed silica micro particles (SiO2) and mixed with other additives to improve its properties. The nanocomposites were irradiated with gamma irradiation from 25 to 150 kGy. The effect of the silica content and irradiation dose on the morphology, the mechanical and the physical properties of nanocomposites was investigated. The scanning electron microscope (SEM) showed that there is a fine dispersion of nanoparticles into NBR matrix, also some aggregates were observed and their size depends on the SiO_2 content. The results indicated that there was an improvement in tensile strength by increasing irradiation doses up to 50 kGy and by increasing silica loading up to 15 phr. Enhancement of volume fraction by increasing irradiation doses and also by increasing silica loading was observed. The thermal gravimetric analysis (TGA) indicated that the thermal stability of NBR increases by increasing silica contents and the increase in irradiated samples is higher than that in un-irradiated. Finally, it is found that the presence of silica in the nanocomposites enhances the electrical insulating properties of the NBR.

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1. Introduction

Nowadays, mechanical, physical, and electrical properties of polymer nanocomposites are improved and developed during industrial and academic research laboratories. Their concern to develop such composites is mainly because of the fact that nanoparticles present a high surface-to-volume ratio, which may induct individual properties to these nanocomposites as compared to macroscale composites [1]. Industrial fillers such as carbon black, calcium carbonate, mica and talc [2–6] are used for many of the works performed on such systems. The effect of naturally occurring fillers such as clay [7–9] river mine and beach sand [10] on some properties of filling polymeric systems is also investigated. Among the naturally occurring fillers, sand is relatively cheap and is found more abundantly than the aforementioned industries [11].

Among the numerous polymer composites, silica/polymer nanocomposites are the most commonly reported in the literature and are also employed in a variety of applications, such as electronics, automotive and aerospace industries as well as used in many industrial products due to their good mechanical characteristics [12–14].

Nitrile butadiene rubber is a polar rubber, has good resistance to a wide variety of oils and solvents and hence it is widely used in products like oil seals, pipe protectors, automotive and aeronautical industries [15]. NBR in the unfilled form has very low tensile strength and is not electrically conductive. Silica and other fillers are used for reinforcement. It is found that nitriles exhibited the highest interaction with silica probably through the hydrogen bond interaction between the -CN group and silanol groups. Incorporating silica to NBR enhances its electrical insulating properties. Huang et al. consider that inorganic particles may reduce the carrier mobility in the composite materials, thereby increasing their electrical insulating properties [16,17].

Irradiation of polymers causes formation of free radicals and ions that can react to produce cross linked polymers. The products exhibit new properties such as better resistance to heat and chemical action and improvement in the mechanical strength. Vulcanization of rubber by irradiation has also been accomplished and offers some advantages over the conventional methods, such as the complete control of crosslinking density, the formation of cross

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links in solid state, no need for catalysts or other additives, no heat treatment and the ability of saturated polymers to crosslink which, because of the absence of unsaturated groups are largely resistant to conventional chemical treatment. Moreover, it is cleaner and more efficient alternative to heat and certain costly chemicals. Generally, radiation sensitizers are used, which give high yields of radicals under the influence of radiation, promote the crosslinking at a much lower dose and consequently improve the properties of the base polymer.

The purpose of this work is to investigate the effect of the silica type sand and fumed as well as the effect of gamma irradiation on different properties of NBR.

2. Experimental

2.1. Materials

Nitrile Butadiene Rubber (NBR) has acrylonitrile content 40%, density 0.97 g/cm^3, Mooney viscosity ML1 + 4 (100 °C) = 50. Its commercial name is KRYNAC40.50 (Bayer Rubber Company Division France). Antioxidant, chemical name is 1, 2-Hydro.2, 2, 4-Trimethyl quinoline (TMQ), its red pellets' molecular weight: 173.258, molecular formula: $C_{12}H_{15}N$, density: 1.05 g/cm^3, Supplier: Intatrade chemical (GmbH), Germany. Di-Octyl-phthalate (DOP) used as plasticizer, it is colorless, oily, liquid, its molecular weight: 390.6, molecular formula: $C_{24}H_{38}O_4$, density 0.80 g/cm^3, boiling point: 384 °C and supplier: Henan Tianfu Chemical Co., Ltd., China. Hexamethylenetetramine (HMTA) used as coupling agent for reaction, molecular formula: $C_6H_{12}N_4$, molecular weight: 140, it is white and has a density of 1.3 g/cm^3, its sublimation and decomposition reach 265 °C, supplier: Heliopolis for chemical industries, Cairo, Egypt. Polyethylene glycol (PEG) M_n = 600 g/mol, OH functionality = 2.0, 6-Hexanediol diacrylate (HDDA) as crosslinking agent (Fluka, Germany). Fumed Silica (Pyrogenic Silica) used as received, HDK® N20, synthetic: hydrophilic amorphous silica, produced via flame hydrolysis, white colloidal powder of high purity, SiO_2-content > 99.8%, density of SiO_2 = 2200 g/l, silanol group density = 2 SiOH/nm^2, Surface area 170–230 m^2/g, particle size >40 μm, supplier: Wacker Chemie AG, Wacker Silicons, Hanns-Seidel-Platz, and München, Germany. Silica sand originated naturally from Sinai, Egypt.

2.2. Preparation of silica sand nanoparticle

Silica sand is washed with deionized water to remove any clay particles followed by drying in an oven at 100 °C for 8 h. The dried silica sand is sieved to about < 325 μm. The silica sand is grounded to nanoparticles for 16 h using dry ball milling, (PlanitaryBM 400 type) retsch company, Germany. The chemical composition of silica sand nanoparticles is analyzed by Energy Dispersive X-ray (EDX) technique. The nanoparticles' results are checked microscopically through TEM.

2.3. Preparation of the NBR/silica nanocomposites

Nitrile butadiene rubber (NBR) and the ingredients are mixed on two-roll mill, the formulations are shown in Table 1. Rubber is first masticated for 5 min. The other ingredients are mixed well to ensure homogeneous distribution, and then added to rubber. Mixing is continued at the rubber mill for another 20 min. Finally, rubbers containing the ingredients are taken out of the mill and stored at room temperature for 24 h before molding. The rubber mixes are pre-heated for 7 min at 160 °C and a pressure of 10 MPa, then full pressed for 12 min using hot press. In order to ensure predetermined sheet size, the hot pressed sheet is cold pressed afterward in another press and cooled with water.

2.4. Gamma irradiation of the nanocomposites

The molded samples, in the form of thin sheets, have been irradiated by a Cobalt 60 gamma cell source (made in Canada), installed at the National Centre for Radiation Research and Technology, Egyptian Atomic Energy Authority, Cairo, Egypt. The samples are cured by using gamma rays at 25 to 150 kGy.

2.5. Transmission electron microscopy (TEM)

Transmission electron microscopy (TEM) is adopted to characterize the nano-particles modification. TEM is performed by TEM-1230 with an accelerating voltage of 100 kV (JEOL Co., Japan).

2.6. Scanning electron microscope (SEM)

Scanning electron microscope SEM studies of NBR composites are carried out by JEOLJSM–5400 high resolution (Japan), at 20 MA and 15 kV. Samples' film is cut and is sputter–coated with gold using a microscope sputter coater, viewed through the microscope and also EDX technique is used for analysing elements of SiO_2 through the microscope.

2.7. Measurements for nanocomposites

2.7.1. Swelling properties

All samples of known weight (W_O) are immersed in thimbles containing toluene. The swelling measurements are carried out at room temperature. The samples were then removed, blotted quickly to remove the attached solvent on the sample surface and weighed. The swelling equilibrium is reached after 24 h. Samples are dried in oven at 50 °C to constant weight for 48 hr.

The volume fraction of the swollen rubber (V_F) is calculated according to the following equation:

$$V_f = \frac{volume\,of\,rubber\left(\frac{W_0}{\rho_r}\right)}{volume\,of\,rubber\left(\frac{W_0}{\rho_r}\right) + volume\,of\,solvent\left(\frac{W1-W_0}{\rho_s}\right)} \quad (1)$$

where Wo is original weight, W_1 = weight after swelling + solvent, ρr = density of rubber, and ρs is the density of solvent.

2.7.2. Mechanical properties

Tensile strength and elongation at break are tested at room temperature. Every data point is the average of 5 tests. (Mecmesin tester, Mecmesin Limited, UK.), equipped with software is used, employing a crosshead speed of 500 mm/min.

2.7.3. Crosslinking density measurements

It has been shown that the true stress (σ) in simple extension can be considered as a sum of two contributions [18]:

$$\sigma = \sigma_o(\lambda) + Ge(\lambda^2 - \lambda^{-1}) \quad (2)$$

where σ is the true stress (nominal stress multiplied by λ), λ is the extension ratio, σ_o depends on the chemical nature of the rubber, but the Crosslink density, Ge is a parameter that depends on the degree of crosslinking. Plotting σ vs. λ^2-λ^{-1} gives Eq. (2), the slope of the curve is Ge.

According to Zang et al. [19], M_c (molecular weight between crosslinks) can be estimated from the value of Ge as follows:

$$Mc = A_\phi \rho RT/Ge \quad (3)$$

The prefactor A_ϕ is assumed to be equal to 1; T is absolute temperature, ρ is the density of the specimens, R (8,314,472 cm^{-3} Pa K^{-1} Mol^{-1}) is the gas constant.

2.7.4. Thermal properties

Thermo gravimetric analysis (TGA) is determined by using Shmadzu-50 (TGA-50H) (Japan) at a heating rate of 10 °C/min in

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