

**Ministry of Higher Education
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***Preparation and characterization of nano silica
using precipitation and sol-gel method and its
application in rubber reinforcement***

A Thesis

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Abstract

Silica nano particles were prepared from Iraqi rice husk (al-Najaf province) by two methods, precipitation and sol-gel. Rice husk was treated with organic acid 1N CH₃COOH and inorganic acid (0.1, 1, 3) N HCl, 1N HCl is the best one to reduce the metallic ingredients and to obtain highly purified silica powder; followed by thermal combustion under controlled conditions at 700 °C to give 23.40% ash of which 99.17% was silica. In precipitation method, rice husk ash (RHA) was purified by alkaline extraction method using 2.0, 2.5 and 3.0 N sodium hydroxide, the optimum particle size of nanosilica was extracted by 2.5 N. In sol-gel method, the nano silica was extracted from RHA using 2, 3 and 4 N sodium hydroxide solution to produce a sodium silicate solution and then precipitated nano silica by adding H₂SO₄ at pH = 4 in the mixture of sodium silicate solution, cetyltrimethylammonium bromide (CTAB), water and butanol, the optimum particle size of nanosilica was extracted by 3 N sodium hydroxide. The prepared nanosilica by the previous two methods was characterized by particle size analyzer (PSA), X-ray diffraction (XRD), Scanning electron microscope (SEM), and Fourier transform infrared spectroscopy (FTIR). The particle size obtained of the prepared nanosilica by the above two methods were found to be 25 and 45 nm, respectively. X-ray results revealed a single strong broad peak of 2 θ at about 22.4 and 22.5 for the previous two methods respectively. It is clearly seen that the particle size and specific surface area of nanosilica prepared by precipitation method are better than

sol-gel method. Then, nanocomposites were prepared from incorporation of different amounts of the prepared nanosilica into the rubber matrix. Two types of rubber material were used in this work: Natural rubber (NR) and Nitrile rubber (NBR). The nanocomposite was carried out in a controlled manner in two-roll mixing mill, then the composite was vulcanized at 155° C and a pressure of 320 psi. The effect of nanosilica with loading level (0.5, 1, 2.5, 3, 5 phr) on some mechanical properties of NR and NBR (tensile strength, elongation at break, hardness, compression set and abrasion resistance) was studied.

The effect of nanosilica prepared by precipitation method, when increased the amount of nanosilica up to 2.5 phr, the tensile strength for the two types of rubber NR and NBR increased from (5.97 to 13.7) MPa and (6.2 to 16.5) Mpa, respectively , then tensile strength decreased. Also, the elongation at break increased when the amount of nanosilica increased up to 2.5 phr, then the elongation at break decreased. The hardness increased from (52 to 84.5) and (56.3 to 88.1), respectively when the amount of nanosilica was increased. The compression set increased from (12% to 55%) and (15% to 59%), respectively when the amount of nanosilica was increased. The abrasion resistance increased when the amount of nanosilica was increased up to 2.5 phr with different loads, then the abrasion resistance decreased.

The effect of amount of nanosilica prepared by sol-gel method on the mechanical properties for the two rubbers NR and NBR was studied, the tensile strength increased when the amount of nanosilica was increased up to 2.5 phr from (4.86 to 9.1) MPa and (5.24 to 10.5) Mpa, respectively , then tensile strength decreased. The elongation at break increased when the amount of nanosilica was increased up to 2.5 phr, then elongation at break decreased. The hardness increased from (43 to 70) and (48.84 to 73.3),

respectively when the amount of nanosilica was increased. The compression set increased from (5% to 50%) and (11% to 52%), respectively when the amount of nanosilica was increased. Generally, it is clearly seen that the mechanical properties of NBR nanocomposite are much better than NR nanocomposite, and the effect of nanosilica on the mechanical properties is the better than commercial silica and carbon black.