بسم الله الرحمن الرحيم

MONA MAGHRABY

شبكة المعلومات الجامعية

التوثيق الالكتروني والميكروفيلم

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شبكة المعلومات الجامعية

التوثيق الإلكتروني والميكروفيلم

جامعة عين شمس

التوثيق الإلكتروني والميكروفيلم

قسم

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Faculty of Science
Chemistry Department

"Synthesis and Characterization of Some Copolymers of *p*-phenylenediamine using eco-friendly nanomaterial for improving mechanical and electrical properties"

A Thesis Submitted for Degree of Ph.D. in Chemistry

By

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Abstract

Eggshell is a bio-friendly substance containing by weight of 95 % $CaCO_3$ and 5 % the oxides (Mn, Si, Al, Fe, Cr, Cu and Ca) and organic substances. The oxidation was used to prepare the poly(*p*-phenylenediamine) in the presence of potassium persulphate as an oxidant. Poly (*p*-phenylenediamine) and their nanocomposites with nano eggshell (ESN), carbon nanotube (CNT), and nano eggshell /carbon nanotube were prepared and compared with ground eggshell (ESG). All prepared samples were confirmed via FTIR in addition to raman spectra and ^{1}HNMR. The morphology has been studied by transmission electron microscope. The thermal stability of the synthesized polymer nanocomposites was evaluated via thermogravimetric analyzer, the results proved that the addition of ESN and ESG to CNT gives better thermal stability than each compound. The addition of 0.04gm (2%) ESN+CNT improved the thermal stability of polymer than 0.02gm (1%) ESN+CNT. The electrical conductivity properties were studied, the results showed efficiency in the improvement of electrical properties $\sigma = 10^{-5}$ $ohm^{-1}.cm^{-1}$, which form a better composite with higher conductivity, so it can be used as a semiconductor. The hardness value of P*p*PDA and its nanocomposites at 0.02 gm (1%) and 0.04 gm (2%) of (ESN+CNT) with different values of pressure was studied. It appears a moderate value of

hardness at 1Kg and 5Kg and it was observed that as the pressure increases, the hardness value increases.

Copolymer of (*p*-phenylenediamine) and ethylene glycol was synthesized by chemical oxidative polymerization with potassium persulfate as an oxidant at different ratio 1:1, 1:2 and 2:1. (*p*-phenylenediamine/ethylene glycol) copolymer was treated with eggshell ground and nano, CNT, (ESG+CNT) and (ESN+CNT) with different concentration 0.02gm (1%) and 0.04 gm (2%). This prepared samples were characterized using FT-IR, Raman spectra, H^1NMR and XRD. The morphology has been studied by TEM. It is observed that the presence of (ESN+CNT) enhanced the stability of the copolymers due to calcium carbonate starts decomposing into calcium oxide (CaO) and carbon dioxide (CO_2) at 650–700^oC as well as CNT usually start to oxidize at higher temperature between 600 and 700 °C. Accordingly, The most stable (P*p*PDA/EG) with ratio (2:1) at 0.04 gm (2%) of (ESN+CNT). The conductivity of (P*p*PDA/EG) 1:2 was 3.46x 10^{-6} which the highest value than that of (P*p*PDA/EG) 1:1 = 2.99x10^{-7} and (P*p*PDA/EG) 2:1 =3.56x10^{-8}. The copolymer (P*p*PDA/EG) at ratio 1:2 in the presence of CNT+ESN has the most conductivity respect to CNT and ESN at different temperature. With different concentration of the filler, the conductivity with 0.04 gm (2%) CNT+ESN= 1.58x 10^{-4} was higher than 0.02gm (1%) = 4.5 x 10^{-5}, this mean that the conductivity was increase with increasing the concentration. The hardness value increases in the weight

0.04 gm (2%) (CNT+ESN) more than weight 0.02 gm (1%) for all ratios of the copolymers. The hardness value in presence of (CNT+ESN) at load 1Kg was high and out of the scale.

Copolymer of (*p*-phenylenediamine) and glycerol was synthesized by the same previous procedure with the same ratio of the monomers. The nanocomposite of this copolymer was prepared with the same filler and their concentration. This prepared samples were characterized with the same method. The behavior of the thermal stability of prepared copolymers nanocomposites with different ratio and different concentration of the filler were recorded. It is observed that the presence of 0.04 gm (2%) (ESN+CNT) enhanced the stability of the copolymers with ratio (2:1). The conductivity of (P*p*PDA/Gl) 1:2 was 8.46×10^{-6} which the highest value than that of (P*p*PDA/EG) 1:1 = 4.0×10^{-6} and (P*p*PDA/Gl) 2:1 =2.9×10^{-6}. Depending on the result, the best enhancement was for 0.04 gm (2%) of (ESN+CNT) for the ratio 1:2 for copolymer. The highest values of the hardness of the Poly(*p*PDA/Gl) copolymer at ratio 2:1 with 0.04 gm (2%) (ESN+CNT).