

Physicomechanical Properties of Nano Polystyrene Nanocomposites

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PREPARATION of nano metal oxides (Fe₂O₃, ZnO and TiO₂) and nano sphere polystyrene (PS) has been done in micellar solution. Nanocomposite films have been prepared by doping nano polystyrene with different ratios of nano metal oxide ranged from 0.25 up to 3 wt%. These nanocomposites were characterized using transmission electron microscopy (TEM), X-ray diffraction (XRD) and Fourier transform infrared spectroscopy (FTIR). Thermo gravimetric analysis (TGA) and mechanical properties have also been investigated. By comparison with the values obtained for the PS, it was observed that the thermal stability is higher for the nanocomposites and increases with increasing the nano metal oxides concentration in these samples. The results indicate that the tensile strength, elongation at break and Young's modulus of all the nanocomposites enhanced with the addition of metal oxides nanoparticles compared with the pure PS.

Keywords: Polystyrene, Nanocomposites, Nano metal oxides, Thermal stability, Mechanical properties.

Composite structures comprising metal nanoparticles (NPs) and polymer sub-micro spheres have attracted much attention due to their applications in such areas as catalysis, optics, biological sensing [1-6]. The usage of nano-crystals (NCs) as polymer fillers relates to the strong current interest in development and application of new materials [7,8]. These, so-called, polymer nanocomposites take advantage of large specific surface area and size-dependent properties of NCs as well as desired properties of host polymers such as possibility to be designed in various shapes, long-term stability and reprocess ability [9]. The inorganic nanoparticles have been used to create nanocomposites since the early 1990s, and improvements the properties of polymer matrix in strength, modulus, and fracture strength have been seen for a variety of nanocomposite systems. The nanoparticles used provide improved interfacial surface area and smaller inter particle distances, which are improved the mechanical properties [10-12]. The polystyrene (PS) is a hydrophobic polymer frequently used as a matrix for a

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variety of fillers such as organoclays [13,14], metals [15,16], oxides [17,19], metal chalcogenides [20-22] and graphite [23]. Due to their high surface to bulk ratio, nanoparticles can significantly change thermal as well as mechanical properties of the polymer matrix even when the content of the inorganic phase is quite low [9]. The influence of ferric oxide nanoparticles on the mechanical and thermal properties of polymer nanocomposites has been widely studied [9,17,18,24-29]. In addition, ZnO nanoparticles have been demonstrated to improve mechanical, optical, thermal properties and crystallization extent in polymers [30]. The ZnO/polymer nanocomposite materials, due to the good dispersion of ZnO nanoparticles in the polymer, is interesting in applying in coatings, plastics, sealants and fibers [31]. TiO₂ is an ideal inorganic reinforcing component in manufacturing nanocomposites materials with promising properties and applications [32]. The influences of TiO₂ nanoparticles on the properties of some TiO₂/polymer nanocomposites, such as antibacterial activity, the mechanical properties, UV absorber and photo catalysis degradation were reported [33-39]. The combination of sodium dodecyl sulfate (SDS) surfactant and a polymer and their synergetic effects to stabilize the colloidal systems have been studied [40-42]. (SDS) also, has been used as a good dispersing agent for halloysite (HNT) in polystyrene/halloysite nanocomposites [43].

In this work, sodium dodecyl sulfate (SDS) have been used as capping agent for preparing nano metal oxides (Fe₂O₃, ZnO and TiO₂) and nano sphere polystyrene (PS). Also, the nanocomposites were prepared by solution casting method. The prepared nanocomposites were characterized. The influence of nano-metal oxides on the mechanical and thermal properties of the metal oxides/PS nanocomposites was discussed.

Experimental

Materials and chemicals

Sodium dodecyl sulfate (analytic reagent (AR), 99.5%), potassium persulfate (AR, 99.5%), styrene (chemically pure (CP), 99.0%) were purchased from Sinopharm Chemical Reagent Co., Ltd. Fe(NO₃)₃, Zn(NO₃)₂, TiCl₄, acetone and xylene. The chemicals were used as received, except removal of inhibitor from the styrene with 10 wt% sodium hydroxide solution in a separating funnel twice to remove the anti polymerizer. Deionized water with a resistivity exceeding 18.0 MV cm from a JL-RO 100 Millipore-Q Plus water purifier was used throughout.

Preparation of nanosphere Polystyrene

PS nanosphere was prepared using emulsion polymerization [1]. Sodium dodecyl sulfate (SDS) 0.15 g and potassium persulfate 0.1g were dissolved in 70 ml aqueous ethanol in a 250 ml two-neck flask, and then 4.5 ml of styrene was added to the flask under nitrogen atmosphere. The resulting mixture was rapidly

stirred for 8 h at 70 °C to ensure polymerization. The suspension was removed from nitrogen atmosphere, and the product obtained by de-emulsification, the product was washed, and dried. The yield of prepared PS was about 70 -75% with Mwt about 65,000.

Preparation of nano metal oxides

In the typical procedure, 100 mL of 0.05 M solution of surfactant (sodium dodecyl sulfate (SDS) was made in distilled water and divided into two parts. These two parts were used to prepare 0.5 M metal precursors [$\text{Fe}(\text{NO}_3)_3$, $\text{Zn}(\text{NO}_3)_2$ and TiCl_4] and 2 M NaOH separately. The solution of NaOH was added using a dropping funnel under stirring. The stirring was continued for 2 h after the completion of addition of NaOH. The solution was left to age for 12 hrs. The solution was centrifuged to obtain the precipitate. The precipitate was washed with water and acetone to remove the unreacted materials and organic impurities. The precursor was dried in an electric oven at 110°C. The dried product was then crushed and calcined at 200°C for 3 hrs [44].

Preparation of metal oxides /PS nanocomposites

The preparation of metal oxides (Fe_2O_3 , ZnO or TiO_2)/PS nanocomposites is based on the dispersion of nanometal oxides in to the organic solvent by ultrasonic radiation for 10 min. The nano metal oxides colloid was then combined in appropriate ratio with PS dissolved in xylene, the solution also dispersed ultrasonically for 20 min. and finally after evaporation of the solvent, a transparent metal oxides /PS nanocomposite film was obtained.

Characterization and measurements

Transmission electron microscopy (TEM) micrographs were taken using a JEOL JX 1230 TEM with micro-analyzer electron probe. X-ray diffraction (XRD) measurements were performed on a Philip's X-ray diffractometer PW1390 with Ni-filtered Cu $K\alpha$ radiation at generator voltage of 40 kV and wavelength of 0.154 nm at room temperature. The diffraction angle, 2θ , was scanned at a rate of 2°min^{-1} . FTIR spectra of samples were obtained using a Jascow FTIR 430 series infrared spectrophotometer equipped with KBr discs. Thermogravimetric analysis TGA was carried out using a detector type Shimadzu TGA-50H at a heating rate of 10 deg min^{-1} from 50 to 600°C under nitrogen atmosphere and the sample weight was about 8–10 mg. The physico-mechanical properties (viz., the tensile strength, elongation at break and Young's modulus) of the prepared nanocomposites were determined according to standard methods using an electronic Zwick tensile testing machine, model 1425.

Results and Discussion

Characterization of the nanocomposites

Particle texture

The morphology of Nano PS, Fe₂O₃, ZnO and TiO₂ nanoparticles (Fig.1.a-d) and Nano metal oxide/PS composites was examined by TEM (Fig. 2a-c). (Fig. 1a) shows the TEM image of prepared PS spheres in (SDS). The individual particles have smooth and bare surfaces. The PS spheres show good mono-dispersity with diameters of 60–80 nm. While, the images of the prepared metal oxide samples indicated that the nanoparticles were highly crystalline, and could be well indexed to the hexagonal structure of pure metal oxides (ZnO and TiO₂) and appearance of some small like rod (Fe₂O₃) uniform single particles. Solans *et al.* [45] had reported that in the binary system (water/surfactant), self-assembled structures of different types can be formed, ranging, for example, from spherical and cylindrical micelles to lamellar phases, which may coexist with predominantly aqueous phases. So, the micelles route can be employed for obtaining the shape and size controlled of the metal oxide NPs. The metal precursors will interact with the (SDS) surfactant molecules in the solution and some chemical reactions and capping effects will happen. The metal precursors are hydrolysis to their initial product (metal hydroxide) and finally form metal oxide nanoparticles after calcination. When SDS solution is bigger than its critical micelle concentration (CMC), the capping effect of surfactant becomes dominant as the SDS molecules will form a protection bilayer or surface micelles on the particle surface. As shown in Fig.1b, the Fe₂O₃ nanoparticles aggregate and elongated into big rod-like particles, with average particle sizes of (20-85nm), while the obtained ZnO and TiO₂ nanoparticles are hexagonal, with average particle sizes of (11-28nm), (12-30 nm), respectively (Fig.1c& d).

Moreover, the TEM image of metal oxide/PS nanocomposites (Fig.2), the dark background represented polymer matrix and black dots displayed nanometal oxide nanoparticles distributed in the PS as the filler. Evidently, different capability for charge transport of the filler and the polymer matrix has resulted in the contrast between the nanoparticles and the polymer matrix.

The EDX analysis is shown in Fig.3. which shows the presence of Fe, Zn and Ti beside the presence of O₂ these results are been confirmed by XRD.

XRD diffraction

X-ray diffraction spectra of pure nano PS and metal oxides/PS nanocomposites are shown in Fig.4.

The XRD pattern of pure PS is specified by a broad diffraction peak at $2\theta = 20^\circ$ indicated that the PS nanoparticles were amorphous materials.

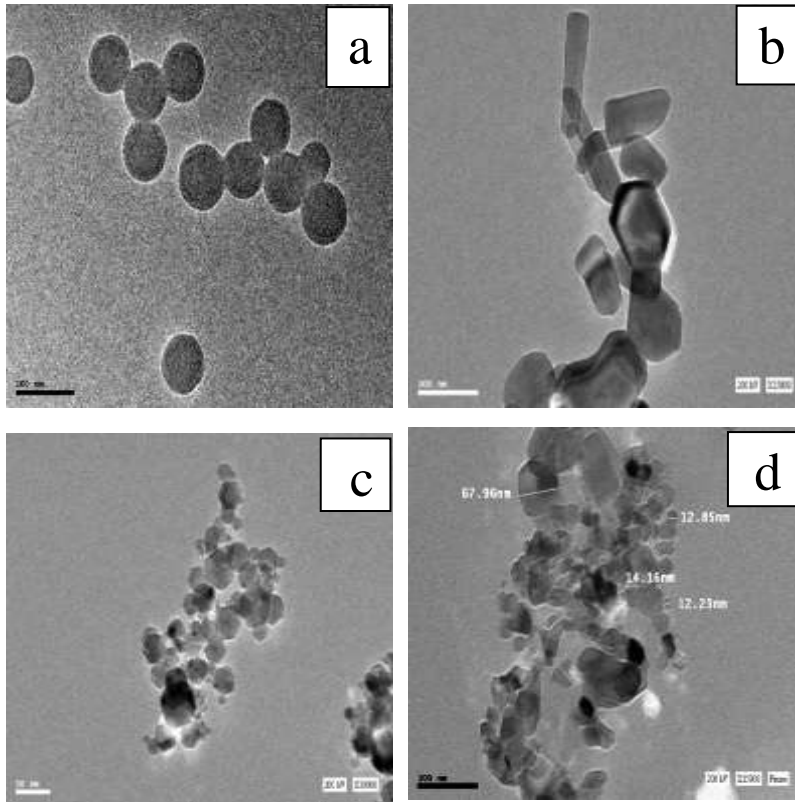


Fig.1a. TEM images of as-prepared PS spheres (b-d) Fe_2O_3 , ZnO and TiO_2 nanometal oxides.

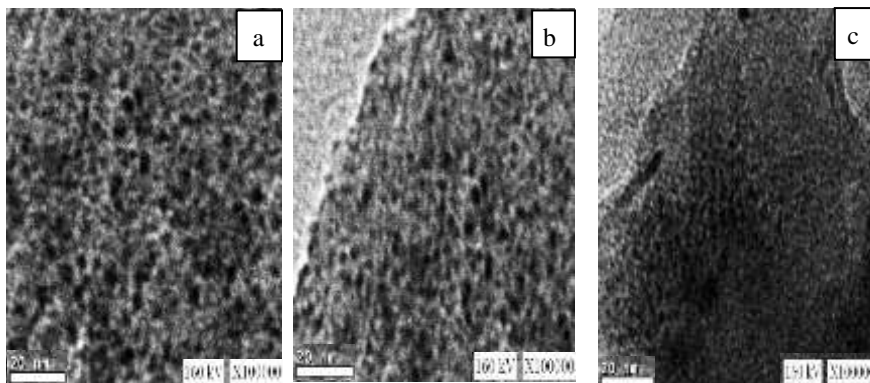


Fig.2. TEM of nanocomposites (a) $\text{Fe}_2\text{O}_3/\text{PS}$, (b) ZnO/PS and (c) TiO_2/PS .

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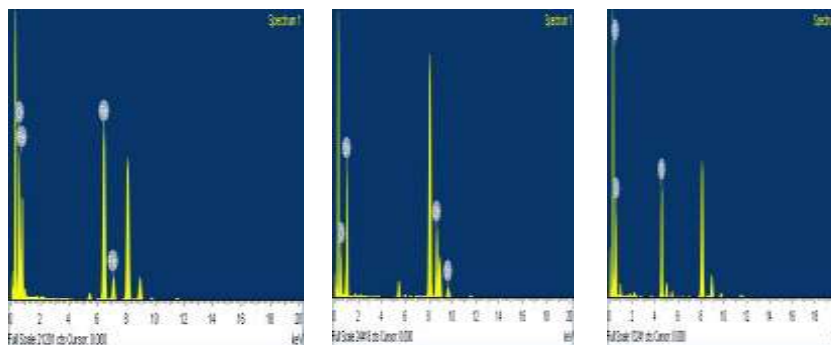


Fig.3. EDX pattern of PS/Fe₂O₃, ZnO/PS, andTiO₂/PS nanocomposites

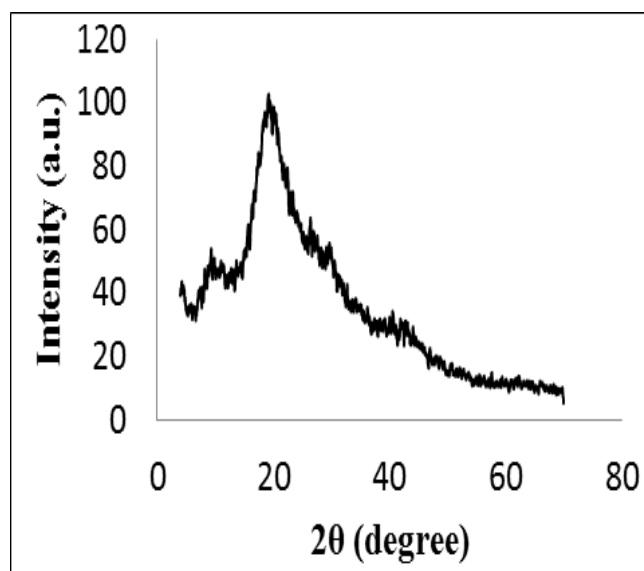


Fig.4. XRD pattern of PS sphere nanoparticles.

The X-ray diffraction spectra of Fe₂O₃/PS nanocomposites is shown in Fig.5 a. Existence of characteristic peaks of iron oxide indicates presence of this inorganic material in hybrid PS nanocomposite. The locations of expected Bragg reflections and peaks at the 2θ angles of 30.18° (d=2.962 Å); 35.68° (d=2.522 Å); 43.48° (d=2.083 Å); 53.98° (d=1.606 Å); 59.88° (d=1.546 Å); and 62.48° (d= 1.487 Å) corresponding to α-Fe₂O₃ crystalline structure [9].

Meanwhile, Fig.5 b shows sharp diffraction peaks of ZnO can be observed at scattering angles (2θ) value of 31.83°, 34.49°, 36.26°, 47.59°, 56.56°, 62.86°, *Egypt. J. Chem.* **60**, No. 2 (2017)

68.12°, and 69.14° correspond to d spacing 2.81 Å, 2.60 Å, 2.47Å, 1.91 Å, 1.62 Å, 1.47 Å, 1.37Å and 1.35Å, respectively [46]. In the case of TiO₂/PS nanocomposite appearance of the peaks at $2\theta = 25.4^\circ, 37.9^\circ, 48.0^\circ, 53.9^\circ$ and 55.2° , corresponding to the anatase crystalline form of TiO₂ as shown in Fig.5 c [32,39]. As well as, it is observed that the peak intensities of the metal oxide phases increased with increasing metal oxides contents.

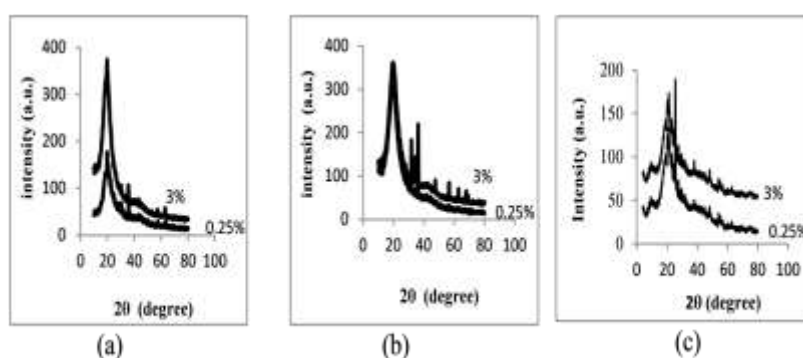


Fig.5. XRD pattern of (a) PS/Fe₂O₃ (b)PS/ZnO(c) PS/TiO₂ nano-composite particles at two different wt %

FTIR analysis

The FTIR spectra of the pure PS nanoparticles and Fe₂O₃ and ZnO/PS Nanocomposite are shown in Fig.6, the prominent PS absorption peaks at 3024, 2920, 1720, 1460, and 1056 cm⁻¹ indicating aliphatic C–H stretching, aromatic C–H stretching, aliphatic CH₂ and different conformation sensitive vibrational modes of PS, respectively. There was no significant change in the peak positions apart from the peak intensity, indicating that the PS nanoparticles in pure and nanocomposite form show the same structural features [47].

The properties of the nanocomposites

Thermal Gravimetric Analysis TGA

The thermal stability of polymeric blend is usually studied by thermogravimetric analysis (TGA). The weight loss due to the formation of volatile products after degradation at high temperature is monitored as a function of temperature. When heating occurs under an inert gas flow, a non-oxidative degradation occurs, while the use of air or oxygen allows oxidative degradation of the samples. Generally, the incorporation of nano metals into the polymer matrix was found to enhance thermal stability [48].

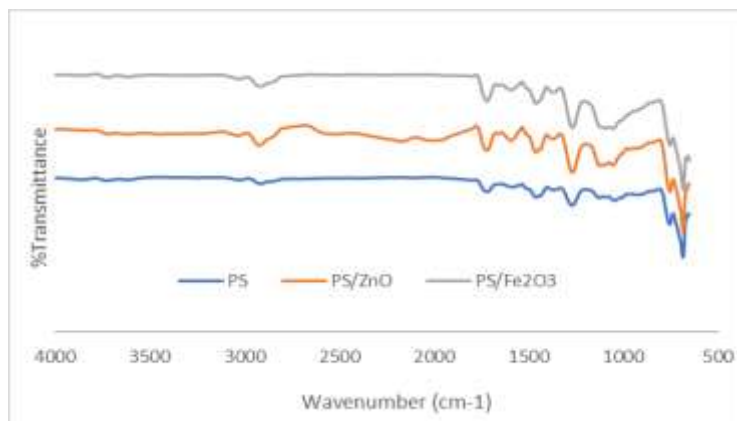


Fig.6. FTIR spectra of the pure PS nanoparticles and PS/Fe₂O₃ and PS/ ZnO nanocomposites

TGA thermograms of the PS and PS/metal oxides nanocomposites are shown in Fig.7. It is clearly seen from the curves that the starting decomposition temperatures of the nanocomposites were nearly equivalent to that of pure PS sphere. The decomposition temperatures of all samples at 10%, 50% loss of the weight and maximum temperature were showed in Table 1.

We could see from Table 1, the decomposition temperatures of Fe₂O₃/PS, ZnO/PS and TiO₂/PS at 10 wt.% loss increased by 6, 2 and 4°C respectively compared with unreinforced PS sphere. As well as, the maximum decomposition temperatures increased by 2, 5 and 10 °C respectively, this indicated that the thermal stability of the nanocomposites was enhanced. The reason may be due to formation of chemical bonds between nano-filler and polymer chains [2].The char residue is found to be higher for the nanocomposites than that of the neat PS, which confirms that the thermal stability of the PS nanocomposites was enhanced due to the presence of the nano-particles.

TABLE 1. Thermal Parameters of the polymer nanocomposites.

Sample	T _{10%} °C	T _{50%} °C	T _{max} °C	Char Residue Wt % at 600°C
PS	246	384	405	0.87
PS/Fe ₂ O ₃	252	384	407	3.31
PS/ZnO	248	375	410	5.81
PS/TiO ₂	250	401	415	6.41

Mechanical properties

It is well known that the addition of nanosized materials into the polymer alters its mechanical properties [32]. The influence of nanoparticles (Fe₂O₃, ZnO

and TiO_2) on the mechanical properties of PS was investigated by tensile strength, the elongation at break (Eb) and Young's modules of the film. The corresponding values of the tensile strength, elongation at break and the modulus of the nanocomposites were determined and plotted against the content of the nanoparticles weight %. These plots are shown in Fig. 8, 9 and 10 respectively.

For the tensile strength, some observations can be seen from Fig. 8. First, there is an increase in the tensile strength for the nanocomposites samples than that of the Pure PS. Second, there is an enhancement of the tensile strength for TiO_2 and Fe_2O_3 nanocomposites till 0.75 wt.% nanoparticles content, where the tensile improved by 46% and 100% for them respectively at 0.75wt%. Then the tensile strength decrease with increasing the content in the range of 1-3 wt%. Third, the tensile strength increases till 2 wt.% for nano-ZnO and improved by 204% at 2%, then the tensile decline only at 3 wt%. It is logically expected that the agglomeration of nanoparticles at relatively high nanoparticles contents in polymer matrix should be responsible for the above tensile loss. Fourth, ZnO/PS nanocomposites give the higher values of the tensile and the Fe_2O_3 nanocomposites give the least.

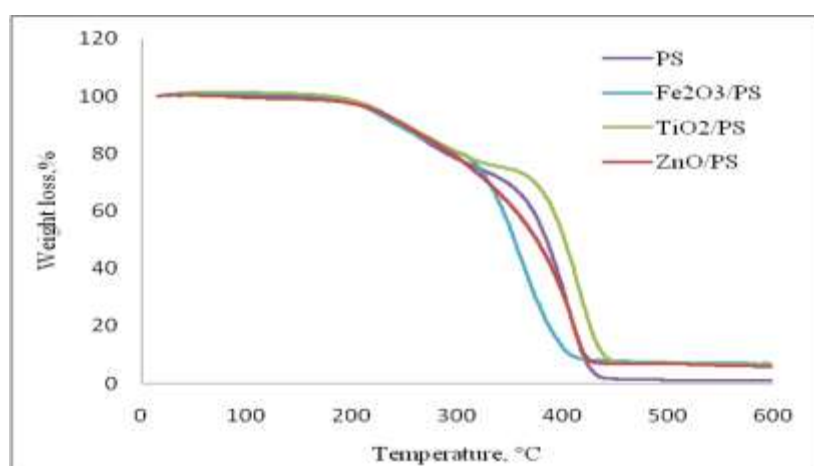


Fig.7. TGA curves of PS and PS/ nanocomposites particles

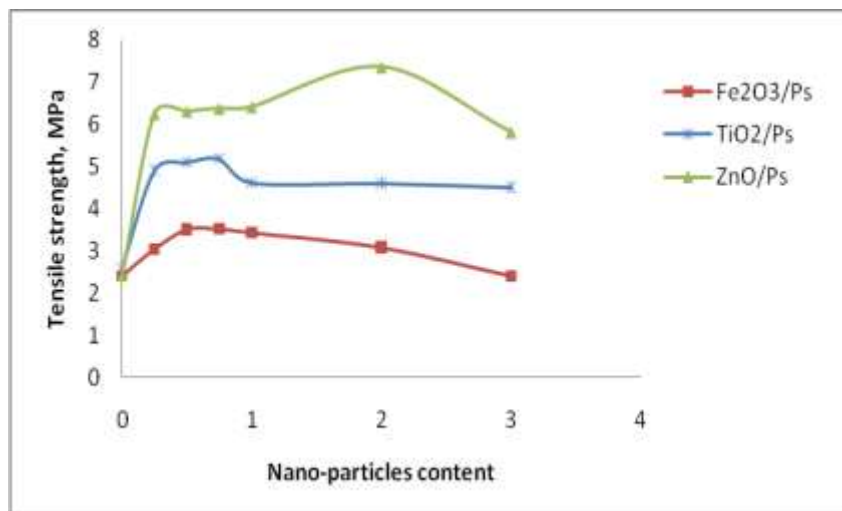


Fig.8. The effect of nano-particles content on the tensile strength of PS/nanocomposite

The increase in the strength of the composites up to 0.75% nano-Fe₂O₃, TiO₂, and up to 2% nano- ZnO may be due to the well-dispersed nanoparticles and the improved interfacial adhesion up to these concentrations. The homogenous dispersion of nanoparticles in high concentrations is difficult. Likely, nano-sized particles have high surface energy and thus, they are easy to aggregate, which results in decreasing the strength of the composites, at high concentrations of nano-particles [49].

The effect of nano-particles addition on the values of elongation at break of nanocomposites is shown in Fig. 9. It is shown from the figure that the elongation enhanced with the addition of the nano- (Fe₂O₃ and TiO₂) up to 0.75%, then decay up to 3% which are in good agreement with tensile strength experiment. However, for ZnO nanocomposites the elongation decreases than the pure PS. This reduction is attributed to stiffening of the matrix by the nano-particles which due to restrictions on the mobility of polymer chains during stretching by the tethering nano particles [50]. Chae, D.W *et al.* [51] found that the introduction of ZnO nanoparticles into polystyrene (PS) decreased both the tensile strength and elongation to break. This implies that the interfacial adhesion is not strong enough to stand up to large mechanical forces. The effect of nano-particles addition on tensile modulus is seen in Fig. 10. Tensile modulus of pure PS is 0.007 MPa. It is observed that the modulus of nanocomposites increases tangentially with increasing nano-Fe₂O₃ content up to 0.75%, where the modulus is enhanced by 15 % only at 0.75% content. However on addition of nano-TiO₂ and nano-ZnO, the modulus increases dramatically up to 2% content. The highest improvement of the modulus for nano-TiO₂ and nano-ZnO

respectively was 97.6 and 355% at 0.75 wt%. The reason for the increase in mechanical properties is due to the fact that the small diameter of the particles themselves will contribute a very high-interfacial surface area and increase the mechanical properties of the polymer.

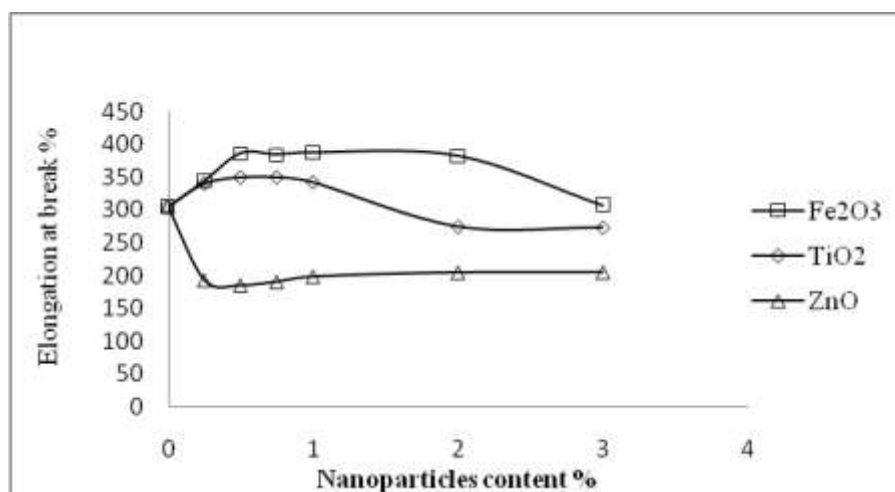


Fig.9. Effect of nano-particles content on the elongation at break of PS/nanocomposite

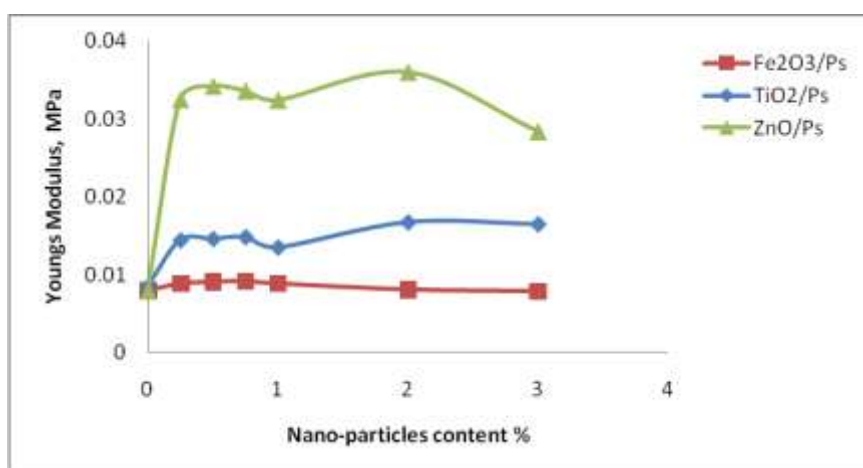


Fig.10. Effect of nano-particles content on the Young's modulus of PS/nanocomposite

Conclusion

Nano polystyrene sphere had been prepared through emulsion polymerization method using (SDS), also preparation of nano metal oxides depending on using (SDS) as capping agent, then nano polystyrene/nanocomposites prepared by sonication through solution casting method methodology at different concentrations of nano metals. The PS/nano metals composites were characterized by TEM, XRD and EDX. TEM images showed that PS have good mono dispersity with diameters of 60–80 nm., different nano metals also have diameters in nano scale, as well as nano polystyrene/nano metals oxide composites showed a good dispersion on polystyrene film. Results from X-ray diffraction of nano metal oxides confirmed the presence of nano metal oxides in the samples. TGA results showed that adding metal oxides NPs in polystyrene film would increase thermal stability of product. Evaluating the effect of amount of metal oxide NPs on mechanical properties of polystyrene showed that the nanofiller increased the stability and elasticity module of polystyrene, and the results confirms that zinc oxide is the best filler which improved mechanical test due to the fact that the small diameter of these particles themselves and contribute a very high-interfacial surface area.

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الخواص الفيزيوكيميائية لمتراكبات البوليمر النانوية

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تم تحضير مواد نانوية اكسيد المعادن هي اكسيد الزنك واكسيد التيتانيوم واكسيد الحديد كذالك تم تحضير بوليمر البوليستيرين النانوي ذات الشكل الكروي في وجود محلول ميسلي. تم تحضير افلام من البوليمر و متراكباته مع اكسيد المعادن النانوية المحتوية على نسب مختلفة من اكسيد المعادن وهي تتراوح ما بين ٠.٢٥ الى ٣%. ولقد تم توصيف تلك المتراكبات النانوية باستخدام الميكروسكوب الإلكتروني و حيود الأشعة وقياسات الاشعة تحت الحمراء بالمقارنة الى النانو بولي ستيرين المحضر. خلال دراسة الثبات الحراري والخواص الميكانيكية وجد ان الثبات الحراري اعلى للمتراببات النانوية ويزداد مع زيادة تركيز أكاسيد المعادن النانوية الموجودة في هذه العينات.

وتشير النتائج إلى أن قوة الشد، استطالة عند الكسر ومعامل يونج للمتراببات تحسنت بإضافة أكاسيد المعادن النانوية بالمقارنة مع البوليستيرين النقي.