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Synthesis and Characterization Unsaturated Polyester Resin Nanocomposites Reinforced by Fe_2O_3+ Ni Nanoparticles: Influence on Mechanical and Magnetic Properties

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P A P E R I N F O

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A B S T R A C T

This investigation aims to study the effect of Fe_2O_3, Ni nanoparticles as a reinforcement material on the mechanical properties of unsaturated polyester (UPR) as a matrix to produce a nanocomposite material using a casting route. Various examinations and tests were conducted to define the characteristics of the manufactured nanocomposite, such as Field Emission Scanning Electron Microscopy (FESEM), Energy Dispersive Spectrometry (EDS), and Fourier Transform Infrared Spectrometer (FTIR) analysis. The mechanical tests, including tensile, bending and hardness were performed on samples at the room temperature according to ASTM standards, while the magnetic characteristics were defined by vibrating sample magnetometer (VSM). Fe_2O_3 nanoparticles were incorporation into unsaturated polyester resin by different weight percentages that vary from 0 wt% to 20 wt% and a constant concentration 3 wt% of Ni nanoparticles. The images of FESEM and EDS evinced the homogeneity of F_2O_3, Ni nanoparticles into the pure unsaturated polyester resin (UPR). While, the improvement in Young's modulus, tensile strength, bending strength, and hardness was compared with those for the UPR. The improvement was 10.02% in young's modulus, 44.08% in tensile strength, 13.55% in bending strength, and strength in hardness. Also, the magnetic properties, including saturation magnetization (Ms), residual magnetization (Mr) and coercivity force (Hc) enhanced with an increase in the concentration of nanoparticles. The preferred percentage to improve the mechanical properties was found at 15 wt% of Fe_2O_3 and then decreased above this concentration, whereas the enhancement in hardness was achieved at 20 wt% of Fe_2O_3.

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NOMENCLATURE

UPR	unsaturated polyester resin	W t%	Weight percent
FESEM	Field Emission Scanning Electron Microscopy	FTIR	Fourier Transform Infrared Spectrometer
EDS	Energy Dispersive Spectrometry	Hc	Coercivity field
Mr	residual magnetization	VSM	Vibrating sample magnetometer

1. INTRODUCTION

In recent years, polymer nanocomposites (PNCs) have become an interest for many researchers because of their ability to manipulate the thermal, electrical, and thermomechanical properties [1]. Thermosetting as polymer matrix are characterized as a low molecular weight solids and low viscosity liquids which they need reinforcement materials as a cross-linking agent to be cured and formulated. Also, thermosetting polymer can be incorporated with nanoparticles of fibers reinforcements to improve the mechanical, thermal, electrical and magnetics properties [2].

The polyester resin was used as a matrix owing to its network structure, resistance to moisture and toxin [3]. Polymer nanocomposites (PNCs) consisting of unsaturated polyester (UPRs) have been widely used for the automotive and electrical fields, structural,

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biomedical and tribological applications [4]. PNCs are an important type of a hybrid material comprising a reinforcement material (inorganic) in nanoscale incorporated into (organic) polymer matrix [5]. Different nanoparticles have been used as fillers in nanocomposites, including ZnO, Fe_2O_3, SiO_2, Al_2O_3, TiO_2, and $CaCO_3$. Owing to their excellent stability, inertness, biocompatibility, non-toxicity, low cost, iron oxide (Fe_2O_3) nanoparticles are very popular among them [6].

It is worth noting that Fe_2O_3 is stable in different ambient conditions, low cost, non-toxicity and high resistance to the corrosion. Polymer/Fe_2O_3 nanocomposites are particularly interested owing to the combination of properties between polymer matrix (organic) and Fe_2O_3 nanoparticles (inorganic) [7, 8]. A highly cross-linking thermoset polymer, such as the unsaturated polyester resins (UPRs), is used as the matrix in nanocomposites. The reinforcement unsaturated polyester resins are basically used in marine and transportation industries. UPRs offer many advantages comparing with other thermosetting resins, like thermal and mechanical properties, easy to cure at room temperature, can be molded at low pressure and temperature, and low cost. Although UPRs are very brittle owing to their covalently bonding network, they are low inhibitors to the initiated cracks and their propagation. However, many researches have been able to improve many properties with addition of organic and inorganic materials [9]. The magnetic nanoparticles have been extremely used as a reinforcement material into many polymers owing to their characteristics and applications, the magnetic iron oxide (Fe_3O_4) is an important oxide nanoparticle having special properties in manufacturing nanocomposites. Its unique properties, such as a high ratio of the spin polymerization, ferromagnetic ordering, high magnetic moment and high conductivity are important to choose this material as reinforcements to prepare nanocomposites [10]. However, the disadvantages of polyester like low toughness limits its industrial applications for engineering components subjected to the impact energy [11]. There are many investigations published in this field, Rahman et al. [12] investigated the effect of different gamma radiation doses about 0-15 KGy on the mechanical properties of (UPR) unsaturated polyester resin reinforced by Fe_2O_3 nanoparticles. Fe_2O_3 nanoparticles were prepared by sol-gel route, while the nanocomposite UPR/Fe_2O_3 nanoparticles were manufactured by solution casting route. The results of this work showed that increasing the dose of gamma radiation to 5 KGy will increase the Young's modulus, tensile strength and decrease the elongation. In another study by Rahman et al. [13] depicted the effect of $NiFe_2O_4$, TiO_2 and Fe_2O_3 nanoparticles on the electrical, optical and mechanical properties of the unsaturated polyester resin (UPR). Results of this investigation revealed the improvement in the DC electrical conductivity and low resistivity for $NiFe_2O_4$ + Fe_2O_3 + TiO_2, and the enhancement in the optical properties for Fe_2O_3 was about 30.38%. Finally, the improvement in the mechanical properties developed was about 6.56% in Young's modulus, about 21.62% in tensile strength, and the highest microhardness obtained for $NiFe_2O_4$. While Seyhan et al. [14] investigated the effect of CNTs carbon nanotubes on the mechanical, thermal and electrical properties of unsaturated polyester resin (UPRs) with and without NH_2 amine functional groups. The results of this study showed that the polyester resin incorporated by carbon nanotubes with amine has better mechanical properties comparing with the carbon nanotubes. Also, the images of the TEM examination revealed a homogeneous dispersion of carbon nanotubes into the unsaturated polyester resin. The main objective of this paper is to improve the mechanical and magnetic properties of (polyester/Fe_2O_3 + Ni) nanocomposites.

2. 1. Materials Used In this work, the materials used are unsaturated polyester resin (UPR) supplied by Petrochemicals Pvt. Ltd., India with accelerator methyl-ethyl-ketone-peroxide (MEKP, Turkey). The reinforcement materials are Fe_2O_3 nanoparticles (with an average diameter of 15-20 nm and the purity ratio at 97.5%, specific surface area (SSA) 40-45 m^2/g) supplied by USA research nanomaterial, while the Ni nanoparticles (at size 40 nm, specific surface area (SSA) at 40-60 m^2/g and the purity ratio at 99.9%) provided by (Hongwu International Group Ltd, China) are further reinforcing material. Tables (1-3) list the properties of the all used materials in this work.

2. 2. Nanocomposite Fabrication The UPR/Fe_2O_3 nanocomposites were fabricated by the casting route. At first, the nanoparticles of Fe_2O_3 and Ni were added to the unsaturated polyester with different weight percentages and stirred for 10 minutes to ensure

TABLE 1. Properties of unsaturated polyester used in this work

Average Particle size (APS)	Purity	Specific Surface Area (SSA)	Density	Color	Morphology
30 nm	99.5%	40-60 m^2/g	5.24 g/cm^3	Red brown	Spherical

TABLE 2. Properties Fe_2O_3 nanomaterial used in this work

Density (gm/cm^3)	Tensile strength (MPa)	Elongation (EL%)	Thermal conductivity W/m.°C	Viscosity at 25°C
1.2	65-100	< 2.8	0.17	200-300 cps

TABLE 3. Properties Ni nanomaterial used in this work

Average Particle size (APS)	Purity	Specific Surface Area (SSA)	Density	Color	Morphology
40 nm	99.9%	12-16 m^2/g	8.9 g/cm^3	Black	Spherical

the better mixing of Fe_2O_3 and Ni nanoparticles with the unsaturated polyester resins. Then, a hardener about 5 wt% of methyl ethyl ketone peroxide (MEKP) was added to the mixture. Afterward, the mixture was stirred by hand at a slow rate in an ultrasonication bath for 10 min to obtain a homogeneous dispersion of Fe_2O_3, Ni nanoparticles into a polymer. The mixture was poured into templets of silicon molds with standard dimensions according to ASTM specifications for each mechanical test's specimen to be tested and cured for 72h at room temperature [15-18]. The as-received sample of unsaturated polyester resin (UPR) with hardener (MEKP) was prepared by mixing process as mentioned above.

2. 3. Microstructural Examinations

2. 3. 1. FESEM and EDS Examinations Field emission scanning electron microscopy (FESEM) type (MIRA3 TESCAN) operating at 10 kV was used to examine the surface morphology of the manufactured polymer nanocomposites. While, the EDS examination was done using SEM analysis for the same sample.

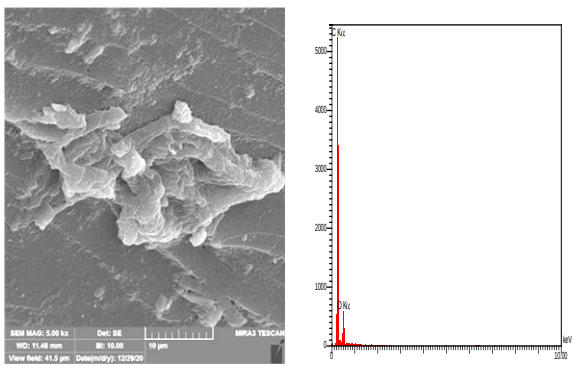
2. 3. 2. FTIR Examination The chemical structure of nanocomposites and the bonding between the oxide nanoparticles and the unsaturated polyester resin were examined by Fourier-transform infrared spectrometer) (FTIR) type (Shimadzu IRAffinity-1, Japan) in the 400–4000 cm^{-1} range. FTIR was used to show the interaction between the nanoparticles and the polymer matrix.

2. 4. Mechanical Properties The mechanical tests, including tensile, bending, and hardness tests were performed for the samples prepared in the present work. The tensile test was conducted using a computerized universal testing machine (Laryee Company) with full capacity (50 KN). Bending test was carried out using a bending apparatus type Microcomputer Controlled Electronic Universal Machine, and the shore (D) hardness device type Bareiss to measure the hardness of the samples. All specimens were tested according to ASTM standards, comprising (ASTM D 638) for the tensile test specimens, (ASTM D 790) for the bending test specimens (ASTM D 790), and (ASTM 2240) for hardness test specimens (ASTM 2240).

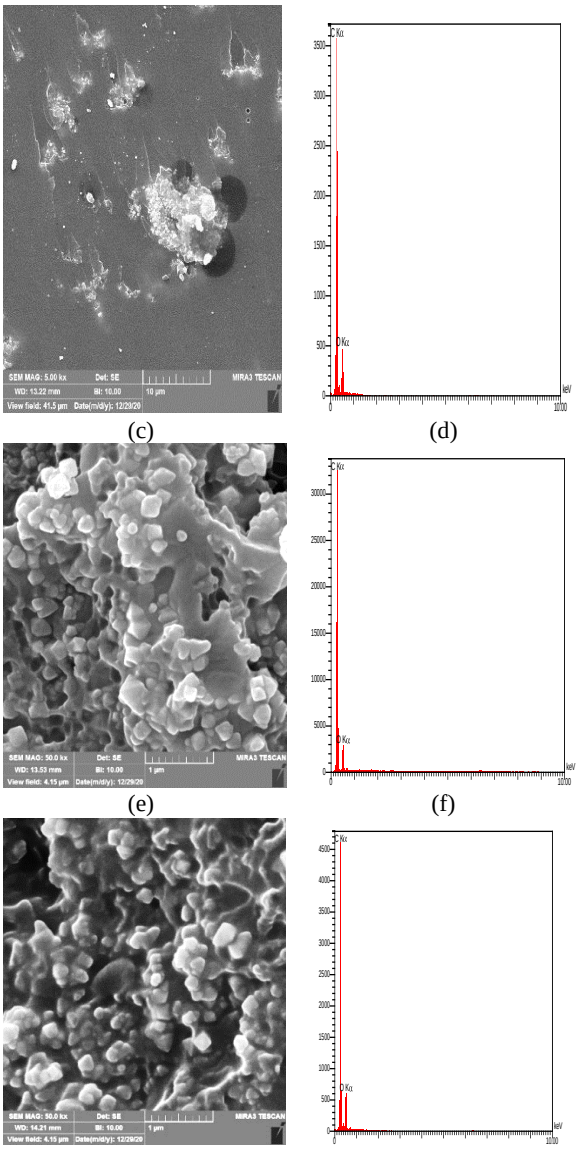
2. 5. Magnetic Properties At ambient temperature, the magnetic characteristics of (UPR/Fe_2O_3+Ni) nanocomposite were examined using a vibrating sample magnetometer (VSM) measuring device. The curves of the magnetic hysteresis loops were used to calculate the residual magnetization (Mr), saturation magnetization (Ms), and coercivity field (Hc) values for each specimen. The saturation magnetization manifests the specimen's response to the external magnetic field, whereas the coercivity field represents the force necessary when the specimen is subjected to an external magnetic field that is the polar opposite of the initial external magnetic field. The coercivity field (Hc) is utilized to minimize the magnetization of the specimen and then the external magnetic field; the magnetization returns to the zero, while the residual magnetization is not reduced to zero [19].

3. RESULTS AND DISCUSSION

3. 1. Microstructural Analysis (FESEM and EDS) FESEM and EDS analysis of the manufactured nanocomposites manifested a good dispersion and homogeneous distribution of Fe_2O_3 and Ni nanoparticles into a polymer matrix. This is due to mechanical and chemical techniques which in turn improve the dispersion of Fe_2O_3 and Ni nanoparticles into polymer matrix [20], and the main factor increases the interaction between the nanoparticles and polymer matrix depending on the chacteristics of nanoparticles, such as the size, shape and surface area. However, the dispersion mechanism depends on the characteristics of polymer matrix like the molecules of polymer and how they are reacted together to improve the dispersion of nanoparticles in it. In the present work, the ultrasonication stirring also helps the dispersion of nanoparticles in the polymer due to creating a homogenous dispersion [21]. Moreover, in all samples, a limited agglomeration is present. This is attributed to a heterogeneous dispersion and causes stress concentration and interfacial failure; such phenomena were reported by Kim et al. [22]. Figure 1 shows the images of FESEM and EDS.



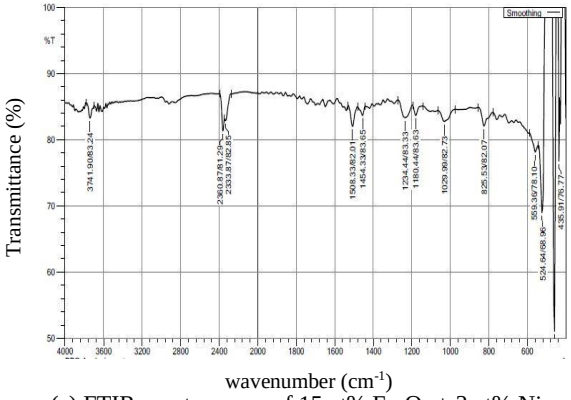
(a) (b)



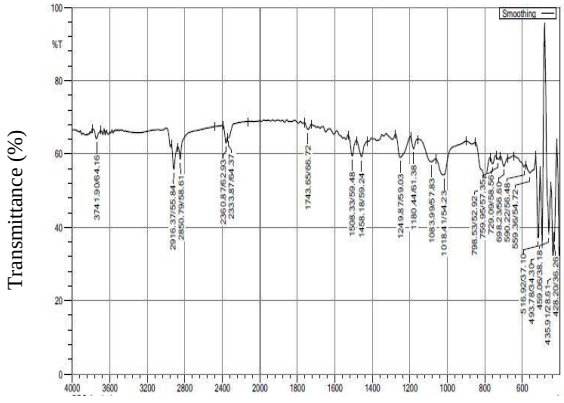
(c) (d) (e) (f) (g) (h)

Figure 1. FESEM image and EDS for polymer nanocomposites with different weights (a, b) FESEM and EDS for 5wt% Fe_2O_3 + 3wt% Ni, (c, d) FESEM and EDS for 10wt% Fe_2O_3 + 3wt% Ni, (e, f) FESEM and EDS for 15wt% Fe_2O_3 + 3wt% Ni, and (g, h) FESEM and EDS for 20wt% Fe_2O_3 + 3wt% Ni

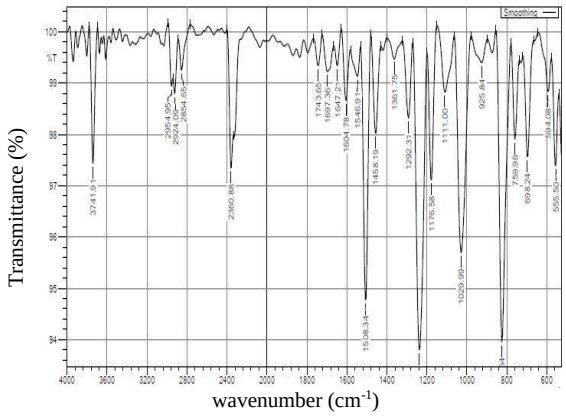
3. 2. FTIR Analysis of the Nanocomposite The FTIR range of the polyester with different weight percentages of Fe_2O_3 nanoparticles and the constant weight percentage of Ni is shown in Figure 2. The stretching vibration modes of the Fe–O functional groups bonds in Fe_2O_3 are similar to the bands of absorption peaks at wavelengths around (450–480 cm^{-1}) and (500–600 cm^{-1}). The vibrations of the following chemical bonds/groups produce IR bands in the range of (700 – 4000 cm^{-1}), which are observed in the range of (700 – 4000 cm^{-1}). The stretching of asymmetrical aromatic C-O with vibrations is at (1029.99–1035 cm^{-1}) and (1234.44–1292.31 cm^{-1}). The asymmetrical aliphatic C-O is stretching at (1111-1180.44 cm^{-1}), while the symmetrical aliphatic C-O is stretching at (1111-1180.44 cm^{-1}). The vibration is at (1450–1850 cm^{-1}) during the C-C stretching. C-H stretching bands were observed at around (2850.79 - 2920.23 cm^{-1}). The aliphatic and aromatic C–H bond stretching is at (2330.01-2366.87 cm^{-1}). The peaks at around (3738.04 -3741.90 cm^{-1}) are assigned to the stretching vibration of the OH group.



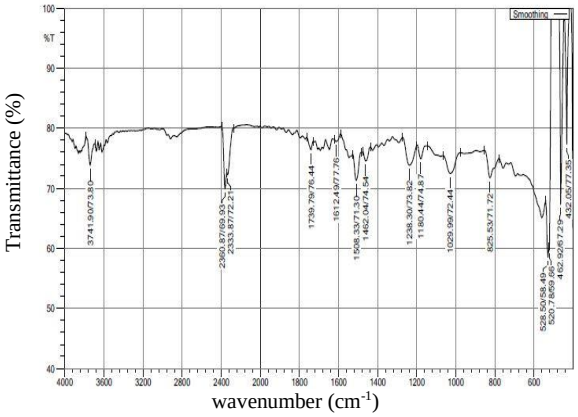
(a) FTIR spectroscopy of 15wt% Fe_2O_3 + 3wt% Ni



(b) FTIR spectroscopy of 10wt% Fe_2O_3 + 3wt% Ni



(c) FTIR spectroscopy of 15wt% Fe_2O_3 + 3wt% Ni



(d) FTIR spectroscopy of 15wt% Fe_2O_3 + 3wt% Ni

Figure 2. FTIR spectroscopy of (UPR /Fe_2O_3 + Ni) nanocomposites with different weights (a, b) FTIR for 5wt% Fe_2O_3 + 3wt% Ni and 10wt% Fe_2O_3 + 3wt% Ni, and (c, d) for 15wt% Fe2O3 + 3wt% Ni and 20 wt% Fe_2O_3 + 3wt% Ni

3. 3. Mechanical Tests Results In general, the aim of incorporating ceramic nanoparticles Fe_2O_3, Ni into unsaturated polyester resin is to enhance the mechanical properties, including Young's modulus, tensile strength, bend strength and hardness through reinforcement mechanisms defined by nanocomposites theories. Table 4 shows the mechanical properties of the all manufactured samples of (UPR /Fe_2O_3+ Ni) nanocomposites.

3. 2. 1. Results of Tensile Test The tensile test is one of the most important mechanical tests, and this test was done to assess the improvement in the tensile properties of nanocomposites with different percentages of reinforcement materials. The role of surface area for Fe_2O_3 and Ni nanoparticles is extremely affected on the Young's modulus and tensile strength for the manufactured nanocomposites. As shown in Table 1, the Young's modulus and tensile strength of pure polyester are about 810 MPa and 18.6 MPa, respectively. While,

TABLE 4. Mechanical properties of (UPR/Fe_2O_3+ Ni) nanocomposite

Material (Polyester+ Fe_2O_3)	Young's Module (MPa)	Tensile Strength (MPa)	Bending Strength (MPa)	Hardness Shore D
Pure Polyester (UPR)	810	18.6	59	68
Polyester + 5 wt% Fe_2O_3 +3wt% Ni	865.3	22.2	61	72
Polyester + 10 wt% Fe_2O_3 +3wt% Ni	871.3	25.3	65.3	75
Polyester + 15 wt% Fe_2O_3 +3wt% Ni	891.2	26.8	67	82
Polyester + 20 wt% Fe_2O_3 +3wt% Ni	775.6	19.7	62.9	85

an increase in the weight percentage of Fe_2O_3 by 5, 10 and 15 wt% as well as a constant percentage of Ni (3wt, %) leads to increase the Young's modulus about 865.3, 871.3 and 891.2 MPa, respectively. Moreover, the tensile strength increases about 22.2, 25.3 and 26.8 MPa, respectively. This is owing to the increasing of cross linking of unsaturated polyester molecules and then the enhancement of the interfacial bonding between the hybrid nanoparticles (Fe_2O_3, Ni) and the unsaturated polyester matrix. When the concentration of nanoparticles is increased to 20 wt%, the Young's modulus and tensile strength decreased to 775.6 MPa, 19.7 MPa, respectively. This is attributed to the aggregation of the hybrid nanoparticles (Fe_2O_3, Ni) into the unsaturated polyester matrix [21]. Additionally, it can be seen that the Young's modulus of the synthesized UPRNCs firstly increased till 15wt% of Fe_2O_3 nanoparticles and then decreased at 20wt% of Fe_2O_3 as displayed in Figure 3(a and b).

3. 2. 2. Results of Bending Test The Fe_2O_3 nanoparticles have the same effect on the polymer in the bending test. Also, the bending strength increases with an increase in weight percentage of Fe_2O_3 nanoparticles till

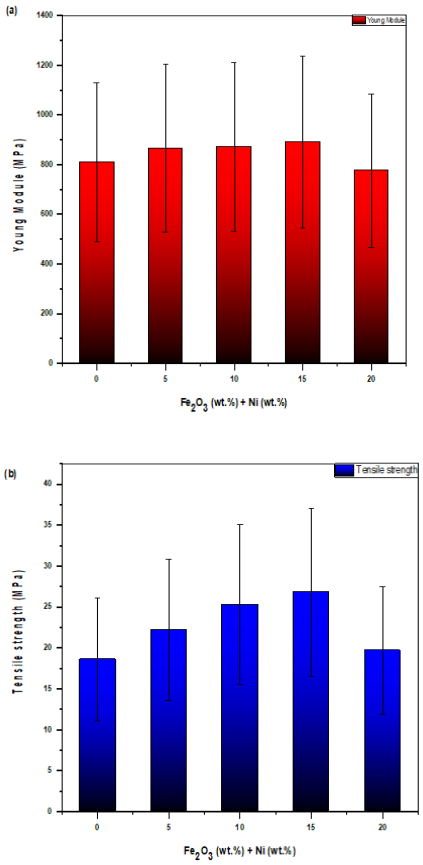


Figure 3. (a) Young's modulus vs wt% of Fe_2O_3 + Ni and (b) tensile strength vs wt% of Fe_2O_3 + Ni

15wt% and decreases above this ratio. This is owing to the same reasons mentioned in tensile test. Figure 4 evices the bending strength of (UPR/Fe_2O_3+Ni) nanocomposites.

3. 2. 3. Results of Hardness Test The hardness test was done using hardness shore D. Figure 5 reveals the results of hardness for nanocomposite samples. Four readings were recorded for each sample, and then an average of these readings was calculated to define the accuracy value of hardness. The hardness (Shore D) value also increased with an increase in the weight percentage of Fe_2O_3, and the maximum value for the unsaturated polyester resin was obtained with 20wt% of Fe_2O_3 nanoparticles. This is attributed to the stiffness and rigidity for the (UPR/Fe_2O_3 + Ni) nanocomposites and the strong bonding between unsaturated polyester molecules and Fe_2O_3 + Ni nanoparticles.

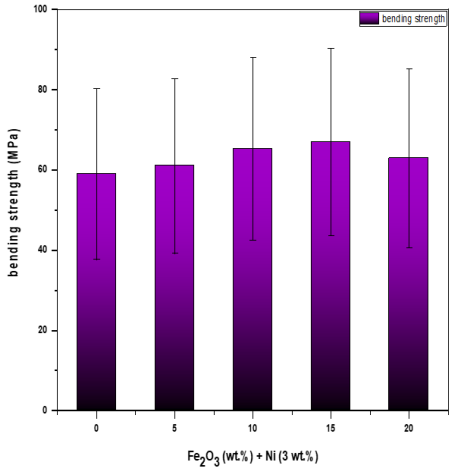


Figure 4. Bending strength *vs* wt% of Fe_2O_3 + Ni

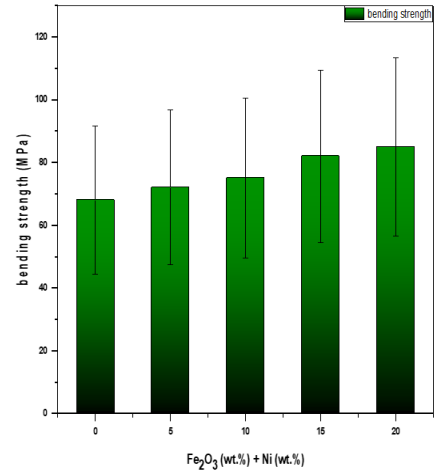


Figure 5. Hardness *vs* wt% of Fe_2O_3

3. 3. Magnetic Characterization Figure 6 elucidates the magnetic hysteresis loops (M-H) at the room temperature for the pure unsaturated polyester resins and (UPR/Fe_2O_3+Ni) nanocomposites. The residual magnetization, saturation magnetization and coercivity field values were extracted from the curves of hysteresis loop to describe the magnetic behavior of each sample. The results demonstrated that increasing the amount of Fe_2O_3, Ni nanoparticles into the unsaturated polyester resins improved the residual magnetization, saturation magnetization and coercivity field values. The maximum values of (Ms), (Mr) and (Hc) were obtained at (20wt% Fe_2O_3 + 3wt%. Ni) as 1.23 emu/g, 12.19 emu/g and 99.7 Oe, respectively. Magnetic characteristics of the manufactured nanocomposites are listed in Table 5.

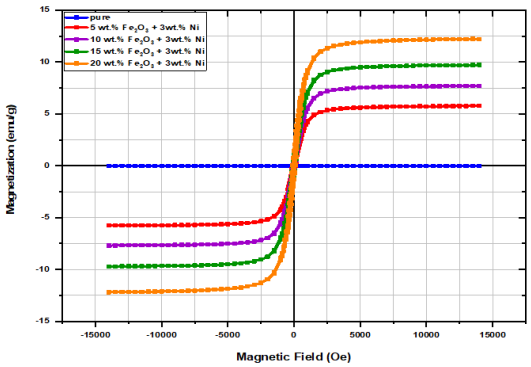


Figure 6. Magnetic hysteresis loops for (UPR/Fe_2O_3+Ni) nanocomposite

TABLE 5. Magnetic properties of (UPR/Fe_2O_3+Ni).

Specimens No.	Fe_2O_3 wt%	Ni wt%	Saturation Magnetization (M_s) (emu/g)	Residual Magnetization (M_r) (emu/g)	Coercivity Field (H_c) (Oe)
1	0	0	0	0	0
2	5	3	5.77	0.40	94.18
3	10	3	7.72	0.79	95.5
4	15	3	9.66	1.02	97.05
5	20	3	12.19	1.23	99.7

4. CONCLUSIONS

In this work, the nanocomposites UPR/Fe_2O_3+Ni were successfully manufactured by casting technique using different weight percentages (5, 10, 15 and 20wt%) of Fe_2O_3 nanoparticles and a constant weight (3wt%) of Ni nanoparticles dispersed in the unsaturated polyester resins matrix. FESEM, EDS and FTIR examinations were conducted for all samples. The mechanical and magnetic properties of the synthesized nanocomposites

were defined. The best mechanical properties were achieved at 15wt% Fe_2O_3 + 3wt% Ni (UPR/Fe_2O_3 + Ni) nanocomposites having (891.2 MPa) Young modulus, (26.8 MPa) tensile strength, (67 MPa) bending strength and (82 shore D) hardness and then decreased for the samples containing Fe_2O_3 more than 15wt%. This is perhaps attributed to the agglomeration of Fe_2O_3, Ni nanoparticles during the long pouring period and causes heterogeneous dispersion of Fe_2O_3, Ni nanoparticles into the unsaturated polyester resins. While, the maximum improvement in the hardness (85 shore D) was obtained at 20wt% of (UPR/Fe_2O_3+Ni) nanocomposites. Magnetic experiments manifested that the improvement of magnetic characteristics, including residual magnetization (Mr), saturation magnetization (Ms), and coercivity field (Hc) was obtained at 20wt% Fe_2O_3 + 3wt% Ni. The values of Mr, Ms and Hc increased from the zero in a pure polyester resins to (1.23 emu/g), (12.19 emu/g) and (99.7 Oe) for 20wt% (UPR/Fe_2O_3 + Ni) nanocomposites.

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Persian Abstract

چکیده

این تحقیق با هدف بررسی تأثیر نانوذرات Fe_2O_3، Ni به عنوان یک ماده تقویت کننده بر خواص مکانیکی پلی استر غیراشباع (UPR) به عنوان یک ماتریس برای تولید یک ماده نانوکامپوزیت با استفاده از یک مسیر ریخته گری انجام شده است. آزمایشات مختلفی برای تعیین ویژگیهای نانوکامپوزیت تولید شده انجام شد ، مانند میکروسکوپ الکترونی روبشی انتشار میدان (FESEM)، طیف سنجی پراکندگی انرژی (EDS)و طیف سنج مادون قرمز تبدیل فوریه .(FTIR)آزمایشات مکانیکی شامل کشش ، خمش و سختی بر روی نمونه ها در دمای اتاق مطابق با استانداردهای ASTM انجام شد ، در حالی که ویژگی های مغناطیسی توسط مغناطیس سنج نمونه ارتعاشی (VSM)تعیین شد. نانوذرات Fe_2O_3 با درصدهای وزنی مختلف که از ۰ تا٪ وزنی تا ۲۰ درصد وزنی و غلظت ثابت ۳ درصد وزنی از نانوذرات نیکل در رزین پلی استر غیر اشباع گنجانیده شد. تصاویر FESEM و EDS یکنواختی نانوذرات Fe_2O_3، Ni را به رزین پلی استر غیر اشباع خالص (UPR)نشان داد. در حالی که ، بهبود مدول یانگ ، مقاومت کششی ، مقاومت خمشی و سختی با موارد UPR مقایسه شد. بهبود در مدول جوان ۱۰.۰۲، ، در مقاومت کششی ۴۴.۰۸، ، در مقاومت خمشی ۱۳.۵۵ and و در سختی مقاومت بود. همچنین ، خواص مغناطیسی ، از جمله مغناطیس شدن اشباع مغناطیس شدن باقی مانده و نیروی اجباری افزایش غلظت نانوذرات افزایش می یابد. درصد مطلوب برای بهبود خواص مکانیکی در٪ ۱۵ درصد وزنی Fe_2O_3 یافت شد و سپس از این غلظت بیشتر شد ، در حالی که افزایش سختی در٪ ۲۰٪ وزنی Fe_2O_3 به دست آمده است.