



## Investigating The Effect of Magnetite ( $Fe_3O_4$ ) Nanoparticles on Mechanical Properties of Epoxy Resin

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### KEY WORDS

Magnetite nanoparticle,  $Fe_3O_4$ , polymer nanocomposites, mechanical properties, microstructure.

### ABSTRACT

*In this paper, study the effects of magnetite nanomaterial  $Fe_3O_4$  on the mechanical properties of epoxy. Dispersion of  $Fe_3O_4$  nanoparticles in the epoxy resin was performed by ultrasonication. The samples of the nanocomposites were prepared using the casting method. The nanocomposites contain epoxy resins as a matrix material incorporated by different weight percentages of magnetite  $Fe_3O_4$  that varies from 0wt.% to 15wt.% as a reinforcing material. The epoxy with the additive reinforcement materials  $Fe_3O_4$  was slowly mixed in a sonication bath for 15 minutes, then the mixture poured into silicon molds. Field Emission Scanning Electron Microscopy FESEM and X-ray diffraction spectra XRD were used to characterize the morphological and structural properties of preparing samples and the distribution of  $Fe_3O_4$  nanoparticles to the epoxy resin. Mechanical testing consists of tensile, hardness shore, and three-point flexural tests were performed on the samples at room temperature according to ASTM standards. The results showed that reinforcement by 15wt.% of  $Fe_3O_4$  nanoparticles maximizes these mechanical properties of nanocomposites compared with pure epoxy except for the young modulus's preferred weight at 9 wt.%, this is due to aggregation of the additives nanomaterials in epoxy resin above 9 wt.%.*

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## 1. INTRODUCTION

Polymer nanocomposites (PNCs) have become one of the important research issues among study scientists in the field of composites. PNCs have constituted a great position between the technically important materials because of their wide scope of applications such as microwave absorption, bioplastics, sensors, dielectrics, biomedical field, food packaging and coatings [1]. Polymer nanocomposites (PNCs) are an interesting large concern of national research labs and manufacturing owing to their outstanding mechanical properties such as increasing strength, large modulus of elasticity, barrier characteristic and flame retardant [2]. The advancement in nanocomposite discipline depends extensively on the manufacture of nanomaterials as nanoparticles and nanofibers with different shapes and sizes [3]. Polymer nanocomposites are an important type of hybrid material where the inorganic materials in nanoscale distributed in a polymer matrix. It is commonly recognized to obtain a correlation between the desired property, cost, processability and performance. The lower concentrations of nano reinforcement materials enable greater retention of the inherent possibility of the pure polymer [4,5]. Between various polymers, epoxy is the most normally used thermosetting polymer matrix in PMCs, has gained formidable concern from both industry and academia [6]. The properties of epoxy can be improved by incorporating different types of inorganic materials in nanoscales, such as nanoparticles, carbon nanomaterials, fibers, sheets or tubes, nano clay, metallic oxides and organic nanoparticles [7,8]. The interesting characteristics of epoxy include low shrinking after cure, good adhesion to different substrates, great stiffness, low creep, chemical and thermal resistance, low cost and high significance of hardness and strength. Epoxy resins exhibit some advantages in mechanical properties [9-13]. These excellent properties make epoxy resin a selection resin for many high-performing engineering applications [14]. Many types of research have dealt with various nanomaterials reinforcing epoxy resin with different weight ratios carried out experiments to enhance mechanical properties [15]. In this work nanocomposite system composed an epoxy resin as matrix incorporated by magnetite nanomaterial ( $\text{Fe}_3\text{O}_4$ ) with different weight percentages (0wt. %, 3wt. %, 6wt. %, 9wt. %, 12wt. %, 15wt. %) as reinforcing materials, have been produced and examined. The effect of nanomaterials ( $\text{Fe}_3\text{O}_4$ ) on the mechanical properties of the epoxy resin was tested by using mechanical tests including (tensile, bending and hardness). The microstructure of the epoxy/ $\text{Fe}_3\text{O}_4$  nanocomposite was investigated using Field Emission Scanning Electron Microscope (FESEM) and X-ray diffraction (XRD). The results of tests show that improvement in mechanical properties. Were the mechanical properties of epoxy resin increasing with increases adding weight percentages of magnetite ( $\text{Fe}_3\text{O}_4$ ) nanomaterials. The results of (FESEM and XRD) investigation show that the homogeneous dispersion of magnetite ( $\text{Fe}_3\text{O}_4$ ) into epoxy resin.

The objective of this research is to study the effect of  $\text{Fe}_3\text{O}_4$  nanoparticles on the mechanical properties of epoxy resin nanocomposites manufactured by the casting method.

## 2. EXPERIMENTAL WORK

### I. Materials Used

The materials used in this work to preparing nanocomposite sample are epoxy resin type (Sikadur 52 A) and the hardener (Sikadur 52 B) that were produced by Sika company placed in Turkey, the mixing of the epoxy resin with the hardener in a 2:1 ratio the added material is magnetite nanoparticles ( $\text{Fe}_3\text{O}_4$ ) supplied by (US Research Nanomaterials, Inc. company). The purity ratio of ( $\text{Fe}_3\text{O}_4$ ) is 99.5% with a particle size of about 30 nm and a specific surface area (SSA) 40-60 m<sup>2</sup>/g used in the manufacture (epoxy/ $\text{Fe}_3\text{O}_4$ ) nanocomposites.

### II. Preparing of nanocomposites samples

The nanocomposites samples were manufactured using the casting method. The magnetite ( $\text{Fe}_3\text{O}_4$ ) nanomaterial was added with different percentages (0, 3, 6, 9, 12 and 15wt. %) to the epoxy resin and mixed for 10-15 minutes. The mixture was stirred in a sonication bath at 50 °C to ensure homogeneous dispersion of the nanoparticles, because of using the ultrasonic system, it has been a high energy level used to cause dispersion of nanoparticles in epoxy with a lot of bubbles and collapse processes [16-18]. The hardener was added and mixed well for 5 minutes, mixing of the epoxy resin with the hardener in a 2:1 w/w ratio according to the supplier datasheet. Subsequently, the mixture was poured into templates of silicon molds with standard dimensions according to ASTM standard for each form of

mechanical test to be tested and cured for seven days at room temperature. The specimens of tensile test manufactured according to (ASTM D 638-I) at dimension (165 x 25x 5) mm, bending test (ASTM D 790) with dimension (130x12.5x5) mm and Hardness according to (ASTM 2240) with dimension (30x15x5) mm. The resulted sample was put in an oven at 100 °C [19]. The above procedure applied for all additive weight percentages of (Fe<sub>3</sub>O<sub>4</sub>) nanomaterial. The total weight of epoxy resin with hardener is 30 gm. Table I shows the weight percentages of epoxy resin and nanomaterials using to manufacture the samples.

**TABLE I: The weight percentages of epoxy resin and nanomaterial in making specimens.**

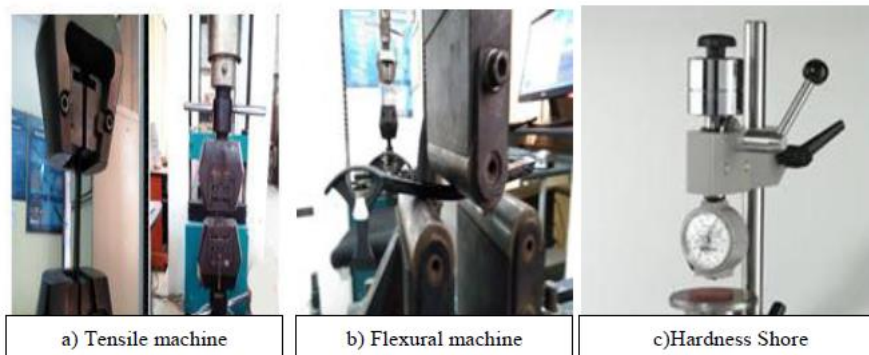
Sample	Weight Percentages of Materials	
	Epoxy resin + hardener (wt.%)	Magnetite Fe <sub>3</sub> O <sub>4</sub> (wt.%)
S <sub>1</sub>	100 (i.e., pure epoxy)	0
S <sub>2</sub>	97	3
S <sub>3</sub>	94	6
S <sub>4</sub>	91	9
S <sub>5</sub>	88	12
S <sub>6</sub>	85	15

### III. Characterizations of nanocomposites

The microstructure of the fractured surfaces of epoxy/Fe<sub>3</sub>O<sub>4</sub> nanocomposites was examined using a field-emission scanning electron microscope (FESEM). The sample surfaces were sputtering coating with platinum and then examined by FESEM type (MIRA3 TESCAN). FESEM examination aimed at finding out the effect of nanoparticles on the microstructure of the nanocomposites and the quality of the samples through homogeneous dispersion of nanoparticles in epoxy resin. Another examination carried out was the X-ray diffraction (XRD) type (XRD-6000) SHIMADZU. The XRD analysis was used to characterize the samples of pure epoxy and epoxy/Fe<sub>3</sub>O<sub>4</sub> nanocomposites. The objective of this examination was the indicative phase formation before and after adding Fe<sub>3</sub>O<sub>4</sub> nanomaterial.

### IV. Mechanical Tests

The mechanical tests were performed for all samples produced in this work, including (tensile test, flexural test and hardness). The tensile test has been carried out for the nanocomposite samples by using a computerized universal testing machine (Laryee Company) with full capacity (50 KN). The flexural test was done at a three-point bending test process using device type (Microcomputer Controlled Electronic Universal Machine). The hardness test was done by the shore (D) scale (Durometer) device type (Bareiss) to measure the hardness of the samples. All specimens were manufactured according to the ASTM standards (American society for testing and materials). The samples of tensile test manufactured according to (ASTM D 638-I) at dimension (165x19x5) mm with the 3.18 mm width of the narrow part. The sample of flexural test manufactured according to (ASTM D 790) with dimension (100x10x5) mm and the hardness samples with dimension (10x10x5) mm. Figure 1 shows the machine tests using in this work and Figure 2 shows the manufactured samples.



**Figure 1: The machine tests using in this work**

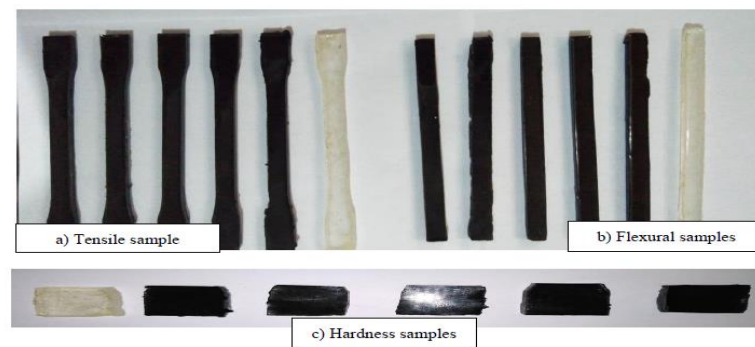


Figure 2: Mechanical test samples

### 3. RESULTS AND DISCUSSION

#### I. Morphology properties

The morphology behavior of the manufactured sample for both pure epoxy and nanocomposites with different weight percentage loadings from  $\text{Fe}_3\text{O}_4$  nanomaterial was examined by Field Emission Scanning Electron Microscope (FESEM). FESEM images of pure epoxy show a smooth fracture surface while (FESEM) images of (epoxy/ $\text{Fe}_3\text{O}_4$ ) nanocomposites with a different additive of  $\text{Fe}_3\text{O}_4$  nanomaterials from 3wt.% up to 15 wt.% revealing that a homogeneous dispersion of  $\text{Fe}_3\text{O}_4$  nanomaterials in epoxy resin with a limited agglomeration of nanomaterial when the additive at high weight percentage. Figure 3 shows (FESEM images). FESEM image analyses are consistent with [20,21].

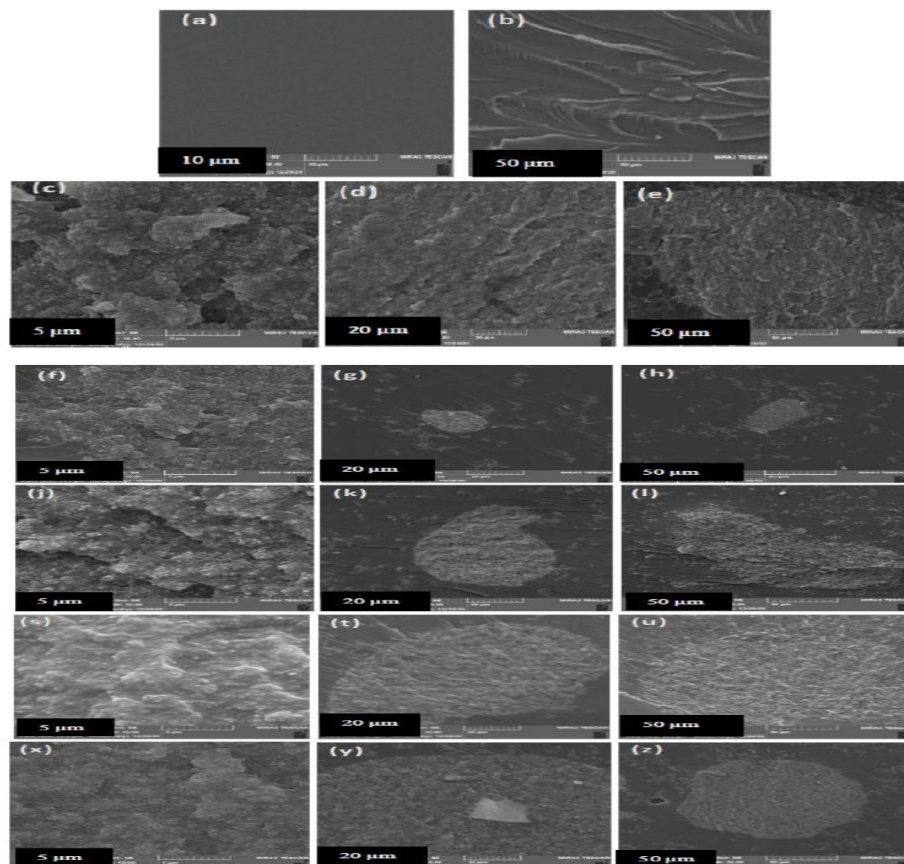
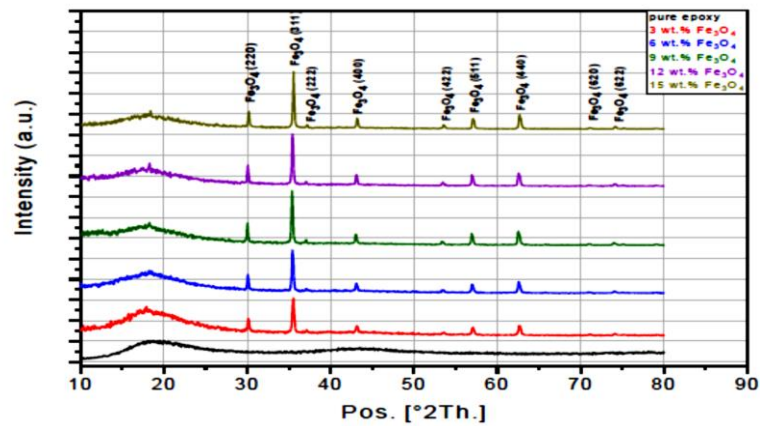


Figure 3: FESEM images of the examined samples for epoxy resin with different weights of  $\text{Fe}_3\text{O}_4$  (a, b) pure epoxy, (c, d, e) 3wt.%  $\text{Fe}_3\text{O}_4$ , (f, g, h) 6wt.%  $\text{Fe}_3\text{O}_4$ , (j, k, L) 9wt.%  $\text{Fe}_3\text{O}_4$ , (s, t, u) 12wt.%  $\text{Fe}_3\text{O}_4$  and (x, y, z) 15wt.%  $\text{Fe}_3\text{O}_4$

**II. X-ray Diffraction (XRD) analysis**

X-ray diffraction analysis was carried out by X-Ray Diffractometer lab model (XRD-6000) SHIMADZU Europe with CuK $\alpha$  radiation at a wavelength ( $\lambda$ ) =1.5405 Å for all the samples of (epoxy/Fe<sub>3</sub>O<sub>4</sub>) nanocomposites. These XRD analyses were performed at room temperature from (10° phase position) to (80° phase position) of (2 Theta°). It can be seen from the XRD patterns as shown in Figure 4 for all samples that the main peak of magnetite (Fe<sub>3</sub>O<sub>4</sub>) nanoparticles occurred at 2 $\theta$  ranging about 30.077°, 35.427°, 37.058°, 43.055°, 53.412°, 56.937°, 62.522°, 70.928°, and 74.964° with (hkl) about (220), (311), (222), (400), (422), (511), (440), (620) and (622) respectively. Also, there is a strong peak at 20° for epoxy matrix, the planes of epoxy/Fe<sub>3</sub>O<sub>4</sub> nanocomposites were defined according to reference code 01-079- 419 for Fe<sub>3</sub>O<sub>4</sub> peak. Where this reference code is for iron oxide (Fe<sub>3</sub>O<sub>4</sub>) that was used and whose peaks appeared on the X-ray diffraction test. The results of XRD analysis exhibited that a homogeneous dispersion of Fe<sub>3</sub>O<sub>4</sub> nanomaterial into epoxy resin for all samples. These results agree with [6, 22].



**Figure 4: X-Ray Diffraction analysis**

**III. . Mechanical results**

The results of mechanical testing shown in Table II recorded that the mechanical properties improved by increasing the added ratio of (Fe<sub>3</sub>O<sub>4</sub>) nanomaterials in epoxy resin. The percentage increase for Young's modulus, tensile strength, flexural strength, and Hardness was calculated according to equations as shown below:

$$\text{Percentage Increase} = \frac{\text{Final Value} - \text{Starting Value}}{|\text{Starting Value}|} \times 100 \% \tag{1}$$

were final value after adding nanomaterial and starting value for pure epoxy.

**TABLE II: Test results of deep beam specimens.**

Sample no.	Epoxy (wt.%)	Fe <sub>3</sub> O <sub>4</sub> (wt. %)	Tensile strength (MPa)	Young modulus (MPa)	Flexural modulus (GPa)	Flexural strength (MPa)	Hardness shore (D)
S <sub>1</sub>	100	0	15	1220	1	60	66
S <sub>2</sub>	97	3	15.5	1333.3	1.5	72	69
S <sub>3</sub>	94	6	16	1381.7	3	78	70
S <sub>4</sub>	91	9	20	1555.5	3.2	84	72
S <sub>5</sub>	88	12	20.6	1403.5	3.5	138	74
S <sub>6</sub>	85	15	22	1293.3	4.5	142	77

#### IV. Tensile test

A tensile test is one of the main tests being used to calculate the improvement in the mechanical properties of the nanocomposites taking into account various percentages and types of additives. This experiment is very significant to understand the behaviors of mechanical properties for nanocomposite material under loading. The test procedure requires placement of the test sample in the testing machine and slowly stretching it until it fractures. The ultimate tensile strength of epoxy/Fe<sub>3</sub>O<sub>4</sub> nanocomposite starts improving with increasing the percentages of additives. The value of tensile strength increases from (15 MPa) for pure epoxy to (22 MPa) at 15wt. % of Fe<sub>3</sub>O<sub>4</sub>. The young's modulus of epoxy/Fe<sub>3</sub>O<sub>4</sub> nanocomposite increases with increasing nanomaterials dispersion up to 9 wt.%, but reduced above this percentage, this is due to aggregation of the additives in epoxy resin above 9wt%. The measured young's modulus for the pure epoxy was (1220 MPa) increasing to (1555.5 MPa) at (9wt.%). The tensile strength and young's modulus increased by 46.66% and 27.5%, respectively compared with pure epoxy as shown in equations below. A.A. Javidparvar et al. [9] and A. Sanida et al. [23, 24] have also reported the increase in tensile strength and young modulus for (epoxy/ Fe<sub>3</sub>O<sub>4</sub>) nanocomposite. Figure 5 a and b show the tensile strength and Young's modulus with weight percentages of Fe<sub>3</sub>O<sub>4</sub>.

$$\text{Increasing in tensile strength} = \frac{22-15}{|15|} \times 100\% = 46\% \quad (2)$$

$$\text{Increasing in Young's modulus} = \frac{1555.5-1220}{|1220|} \times 100\% = 27.5\% \quad (3)$$

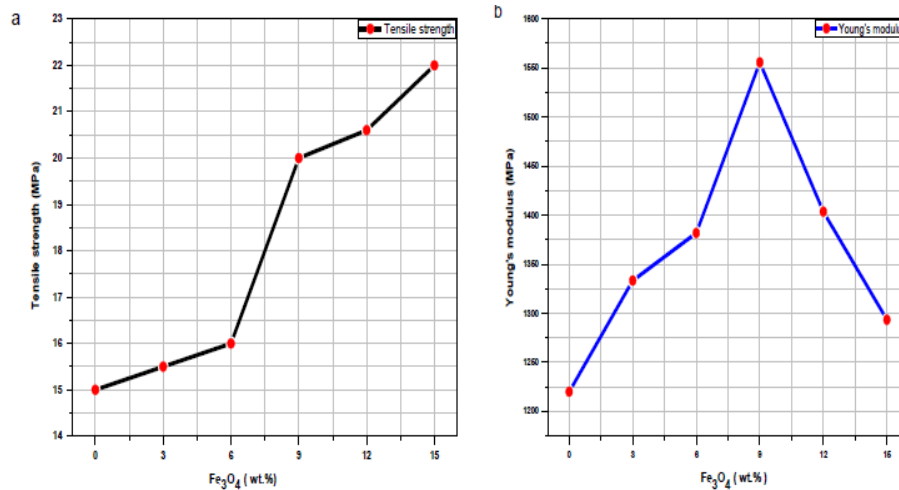


Figure 5: (a) Tensile strength and (b) Young modulus with different weight percentages of epoxy/Fe<sub>3</sub>O<sub>4</sub> nanocomposites

#### V. Flexural test

The flexural test measures the force needed to bend a sample were carried out according to three-point loading requirement at room temperature. The reinforcing effect of Fe<sub>3</sub>O<sub>4</sub> nanomaterials on flexural properties of an epoxy resin was reported. Results show that the flexural modulus and strength increases with the increasing weight of nanomaterials. The maximum improvement of flexural modulus and flexural strength are (4.5 GPa) and (142 MPa) respectively as compared to the pure epoxy for 15wt% of (epoxy/Fe<sub>3</sub>O<sub>4</sub>) nanocomposites as shown in Figure 6 a and b, and caused the significant improvement of flexural properties to good interaction of epoxy chains and the nanomaterial. Flexural strength increased 136.66% as shown in the equation below. Wanda D. Jones et al. [25] have also indicated the improvement in flexural strength and flexural modulus for epoxy/Fe<sub>3</sub>O<sub>4</sub> nanocomposite.

$$\text{Increasing in flexural strength} = \frac{142 - 60}{|60|} \times 100\% = 136.66\% \quad (4)$$

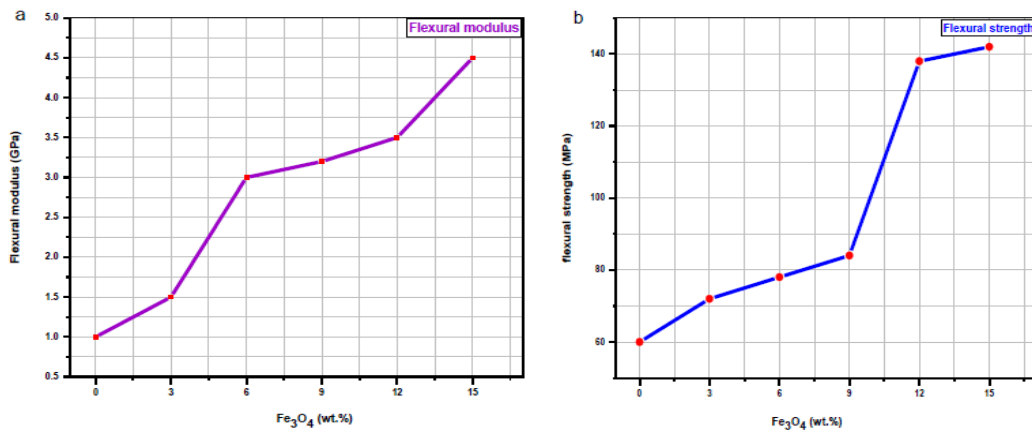


Figure 6: (a) Flexural modulus and (b) Flexural strength with different weight percentages of (epoxy/Fe<sub>3</sub>O<sub>4</sub>) nanocomposites

### VI. Results of Hardness Shore D

Hardness is a measure of the resistance of the surface of the material to scratching or damaging or fracture or dent in any method as a result of force or pressure. In the present study, the hardness shore (D) was used to measure the hardness of the polymer nanocomposite material [26,27]. The results show that with increased (Fe<sub>3</sub>O<sub>4</sub>) nanomaterial loading in the epoxy matrix, the hardness Shore D value also increased and noted the highest value in hardness of (epoxy/Fe<sub>3</sub>O<sub>4</sub>) nanocomposites with filler loading of 15wt.% from Fe<sub>3</sub>O<sub>4</sub> nanomaterial as shown in Figure 7. Therefore, the samples with higher content of nanomaterials demonstrate higher resistance to the indenting force, consequently exhibit increased Shore D values. The hardness increased by 16.66% as shown in the equation below compared with pure epoxy. This is owing to the inherent hardness of the additive nanomaterial compared with pure epoxy sometimes includes color. In another explanation, adding the nanomaterial to the epoxy leads to strong bonding occurrence between the nanoparticle surface and the epoxy chains at the first interfacial nanolayer, and this, in turn, leads to an increase in hardness compared to the hardness of pure epoxy without adding nanomaterials.

$$\text{Increasing in hardness} = \frac{77 - 66}{|66|} \times 100\% = 16.66\% \quad (5)$$

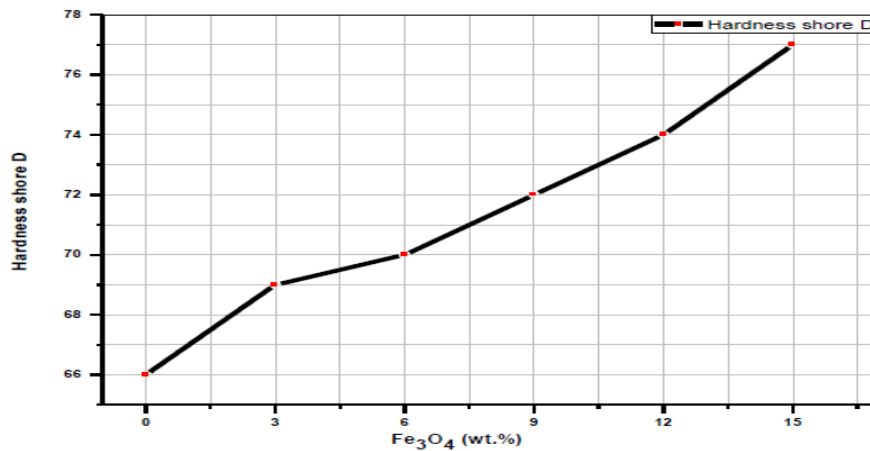


Figure 7: Shows the relationship between hardness and Fe<sub>3</sub>O<sub>4</sub> (wt.%).

#### 4. CONCLUSIONS

In this work, the magnetite nanoparticles ( $\text{Fe}_3\text{O}_4$ ) with different weights were incorporated into an epoxy resin and their effect on the mechanical properties of the manufactured nanocomposites was investigated. The casting method was used to manufacture (epoxy/ $\text{Fe}_3\text{O}_4$ ) nanocomposites and the ultrasonication process was done for dispersion of ( $\text{Fe}_3\text{O}_4$ ) nanomaterial in an epoxy matrix. The nanocomposite system was successfully manufactured and characterized Microstructurally by using FESEM and XRD examinations. The mechanical properties were studied by tensile, flexural, and hardness tests at room temperature according to ASTM standard. The FESEM and XRD results show that a homogeneous dispersion of ( $\text{Fe}_3\text{O}_4$ ) nanomaterial into epoxy resin. From the experimental results of mechanical tests, it seems that improved the mechanical properties by increasing the wt.% of ( $\text{Fe}_3\text{O}_4$ ) additives nanomaterial. It is found that the value of tensile strength, flexural strength, and hardness, increased by 46.66%, 136.66%, and 16.66%, respectively, while the flexural modulus is increased by 3.5 times for 15wt. % of ( $\text{Fe}_3\text{O}_4$ ) compared with the pure epoxy, Where the value of the flexural modulus of pure epoxy is (1 GPa) and when 15% of ( $\text{Fe}_3\text{O}_4$ ) iron oxide is added, the value of the flexural modulus increases to (4.5 GPa), which is 3.5 times more than pure epoxy. Young's modulus increased by 27.5% at 9 wt.% and reduces above this ratio, this is because it increases the settlement and aggregation of the nanoparticles in an epoxy matrix.

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