Effect of Micro Powder on Mechanical and Physical Properties of Glass Fiber Reinforced Epoxy Composite

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ABSTRACT

In the present study, composites were prepared by Hand lay-up molding. The composites constituents were epoxy resin as the matrix, 3% volume fractions of Glass Fibers (G.F) as re enforcement and 2%, 4%, 6% volume fraction of micro powder (Aluminum Oxide Al_2O_3, Silicon Oxide SiO_2 and Titanium Oxide TiO_2) as filler. Studied the, hardness test, flexural strength, density, water absorption measurements and tests were conducted to reveal their values for each type of composite material. The results showed that the non – reinforced epoxy have lower properties than nano composites material. Measured density results had show an incremental increase with volume fraction increase and water absorption, hardness, and flexural strength had show an incremental increase with volume fraction increase and with smaller particle size.

Keywords: Density, Water absorption, Hardness, Flexural Strength, Glass fibers, Composites.

INTRODUCTION

Composite materials are those containing two or more than two bonded materials whose mechanical performance and properties are designed to be superior to those of the constituent materials acting independently [1]. One of the phases is usually discontinues, stiffer and stronger and is called reinforcement, whereas the less stiff and weaker phase is continuous and is called matrix [1]. Figure (1) shows a typical reinforcement geometric for composites [2]. A composite material is originally considered to be a combination of two materials but now this class of material is regarded as any combination which has particular physical and mechanical properties [3]. Many of common materials are metals, alloys, doped ceramics and polymers mixed with additives. Favorable properties of composite materials are high stiffness and high strength, low density, high temperature stability, high electrical and thermal conductivity, adjustable coefficient of thermal expansion, corrosion resistance, and improved wear resistance [4].
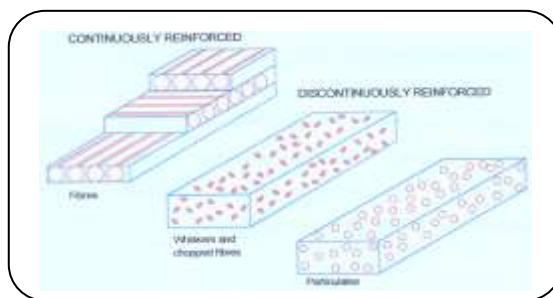
**Figure (1): Typical reinforcement geometric for composites [2]**

Patil.D and Vijaykumar.N have studied the development and mechanical characterization of new polymer composites consisting of glass fiber reinforcement, epoxy resin and filler materials such as (TiO_2) and (ZnS). The newly developed composites are characterized for their mechanical properties. Experiments like tensile test, three point bending and impact test are conducted to find the significant influence of filler material on mechanical characteristics of (GFRP) composites. The test results have shown that higher the filler material volume percentage greater the strength for both (TiO_2) and (ZnS) filled glass epoxy composites, (ZnS) filled composite show more sustaining values than (TiO_2) [5].

Ramish k.et.al have study the effect of epoxy modifiers (Al_2O_3, SiO_2, TiO_2) on mechanical performance of epoxy/glass fiber hybrid composite. The results shown that mechanical properties like flexural strength, flexural modulus and LLSS are more in case of SiO_2 modified epoxy composite compare to other micro modifier. This is may be because of smaller particle size of silica compare to other. Alumina modified epoxy composite increase the hardness and impact energy compare to other modifiers [6].

Amar Patnaik et.al have studied the fiber reinforced polymer composites often have to function in severe erosive environment in which they encounter solid particle erosion. In hybrid composites consisting of reinforcing fibers and particulate filled polymer matrices, the filler material plays a major role in determining the magnitude and mechanism of damage due to erosion. Study of the influence of three different particulate fillers namely fly ash, alumina (Al_2O_3) and silicon carbide (SiC) on the erosion characteristics of glass polyester composites. For this purpose, an air jet type erosion test configuration and the design of experiments approach utilizing Taguchi's orthogonal arrays are used. The wear rates are found to be in good agreement with the theoretical values obtained from an existing prediction model. This study reveals that addition of hard particulate fillers like flyash, (Al_2O_3) and (SiC) improves the erosion resistance of glass polyester composites significantly [7].

Experimental Work

The basic materials used in the preparation of research samples consisting of glass fibers (Woven E- Glass Fiber) from the Tenax company, England, and epoxy resin Euxit (50) base as the matrix from the (Al-Rakaez Building Materials in Amman)   Made in Egypt in the form of transparent viscous liquid at room temperature which is a thermally hardened polymers (Thermosets) with a density of (1.05 gm / cm^3). The mean of powder was used for Aluminum Oxide (1.914μm), Silicon Oxide (6.069μm) and Titanium Oxide (0.346μm), as shown in figure (2). All the required moulds for preparing the specimens were made from glass with dimensions of (150×150×5) mm. The inner face of the mould was covered with a layer of nylon (thermal paper) made from polyvinyl alcohol (PVA) so as to ensure no-adhesion of the resin with the mould.
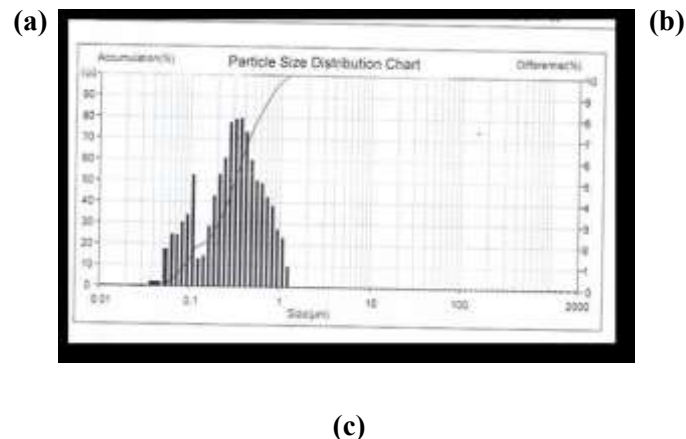
(a)   (b)

(c)
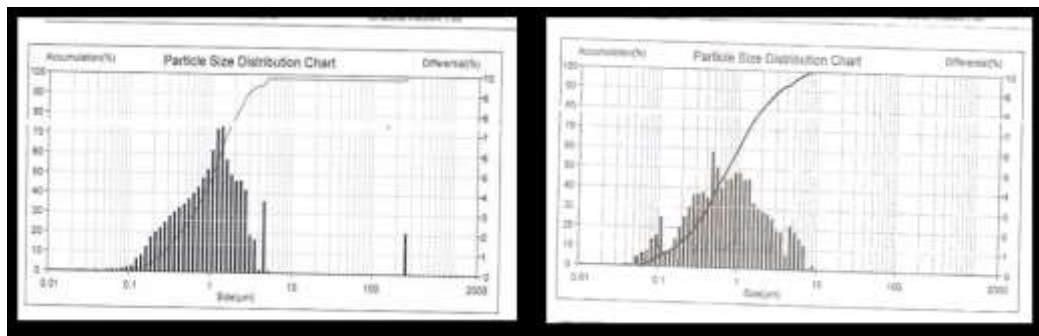
Figure (2) (a) Particle size of Aluminum oxide powder, (b) particle size of Silicon dioxide powder, (c) particle size of Titanium oxide powder

Raw Materials:

Table (1) show the properties of row materials that used in the research:

Table (1) show the properties of row materials [8]

Materials	Density (gm/cm^3)	Modulus of Elasticity(GP)	Tensile strength(MP)	Thermal conductivity(W/M.K)
Epoxy	1.05	2.41	27.6-90	0.19
E-glass fiber	2.58	72.5	3450	1.3
Al_2O_3	4.05	380	282-551	39
SiO_2	2.7	73	104	141
Tio_2	4.23	259	354	11.8

X-Ray Diffraction (XRD) used to find crystalline phases for the nano powder materials as shown in figure (3)
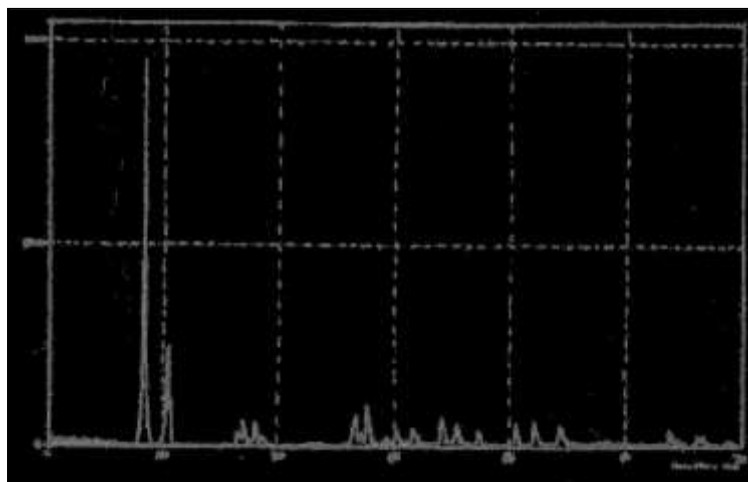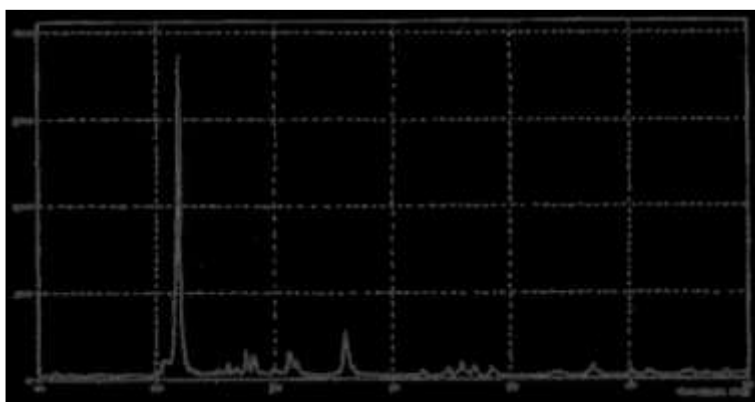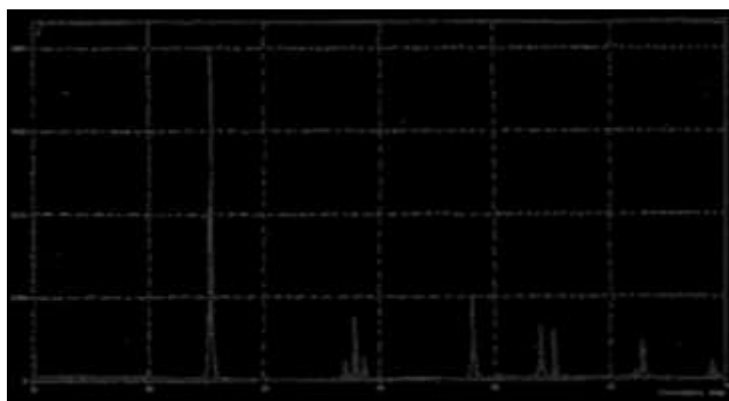
Figure (3) (a) the X- Ray Diffraction of Aluminum Oxide

Figure (3) (b) X- Ray Diffraction of Silicon dioxide powder

Figure (3) (C) X- Ray Diffraction of Titanium dioxide Powder

Figure (3-a) shows the X-Ray Diffraction pattern confirmed that (Aluminum Oxide powder) is mainly (monoclinic) structure.

Figure (3-b) shows the X- Ray Diffraction pattern confirmed that (tetragonal) structure.

Figure (3-c) shows the X-Ray Diffraction of Titanium dioxide powder, from the results, it is obviously that (tetragonal) structure.

Preparation of Composites

The method used in the preparation of the samples, in this research is the (Hand lay-Up Molding) composites are prepared according to the following steps:

1- Preparation of glass fibers woven of dimensions (150 × 150) mm according to the dimensions of the mould. The used volume fractions are (3%).

2- Weighing the reinforcing powder to specify a volume fraction of   (2%, 4% and 6%).

3- Weighing the epoxy depending on the volume fraction of reinforcement materials (fiber and powder), while taking into consideration the weight of hardener.

4- Mixing the epoxy with the hardener continuously and slowly by using a glass rod so as to avoid bubbles. The mixing is carried out at room temperature.

5- Adding the powder intermittently into the mixture and stirring it for a period of (10-15) minutes to obtain homogeneity. A rise in the temperature of the mixture will result as an indication to the beginning of the interaction process. It is very important that the mixture

must have a good viscosity for the purpose of protecting the particles from precipitation which may result in the heterogeneity of the mixture that leads to the agglomeration after hardening.

6- Pouring the mixture into the mould, then putting the glass fiber mat into the mould and continuing of mixture pouring until it covers the entire mat.

7- Pressing the mixture with an appropriate load.

8- For the purpose of completing the process of hardening, finally is leaving the sample in the mould for a period of (24) hour at room temperature. Samples are then extracted from the mould and then heat treated in an oven at (60Ċ) for a period of (60) minutes. This process is very important for the purpose of obtaining the best cross linking between polymeric chains, and to remove the stresses generated from the preparation process and complete the full hardening of the samples.

Mechanical Test

4.1. Hardness Test Measurement

This test is performed by using hardness (Shore D) and according to (ASTM DI-2240) standard at room temperature. Samples have been cut into a diameter of (40mm) and a thickness of (5mm). Figure (4) shows standard specimens for this test [9]. Figure (10) shows hardness device used in this research. For each specimen five hardness measurements were taken and the average hardness is calculated.
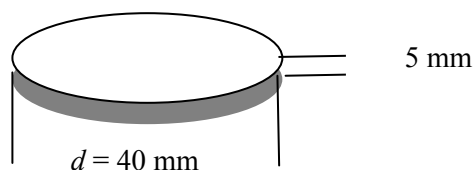

$d = 40$ mm
5 mm

Figure (4): Hardness (Shore D) standard specimens [9]

Figure (5): Hardness device.

Flexural Strength test

This test is performed according to (ASTM D790) at room temperature. Samples have been cut into the dimensions (80*3.2*2.5) mm. Figure (6) shows standard specimens for this test [10]. Figure (7) shows flexural strength device used in this research. The flexural strength are calculated according to the equations [11].

$$F.S = \frac{3PL}{2bd^2} \quad \text{..................} (1)$$

Where
  F.S: flexural strength (MPa).

P: force at fracture (N).

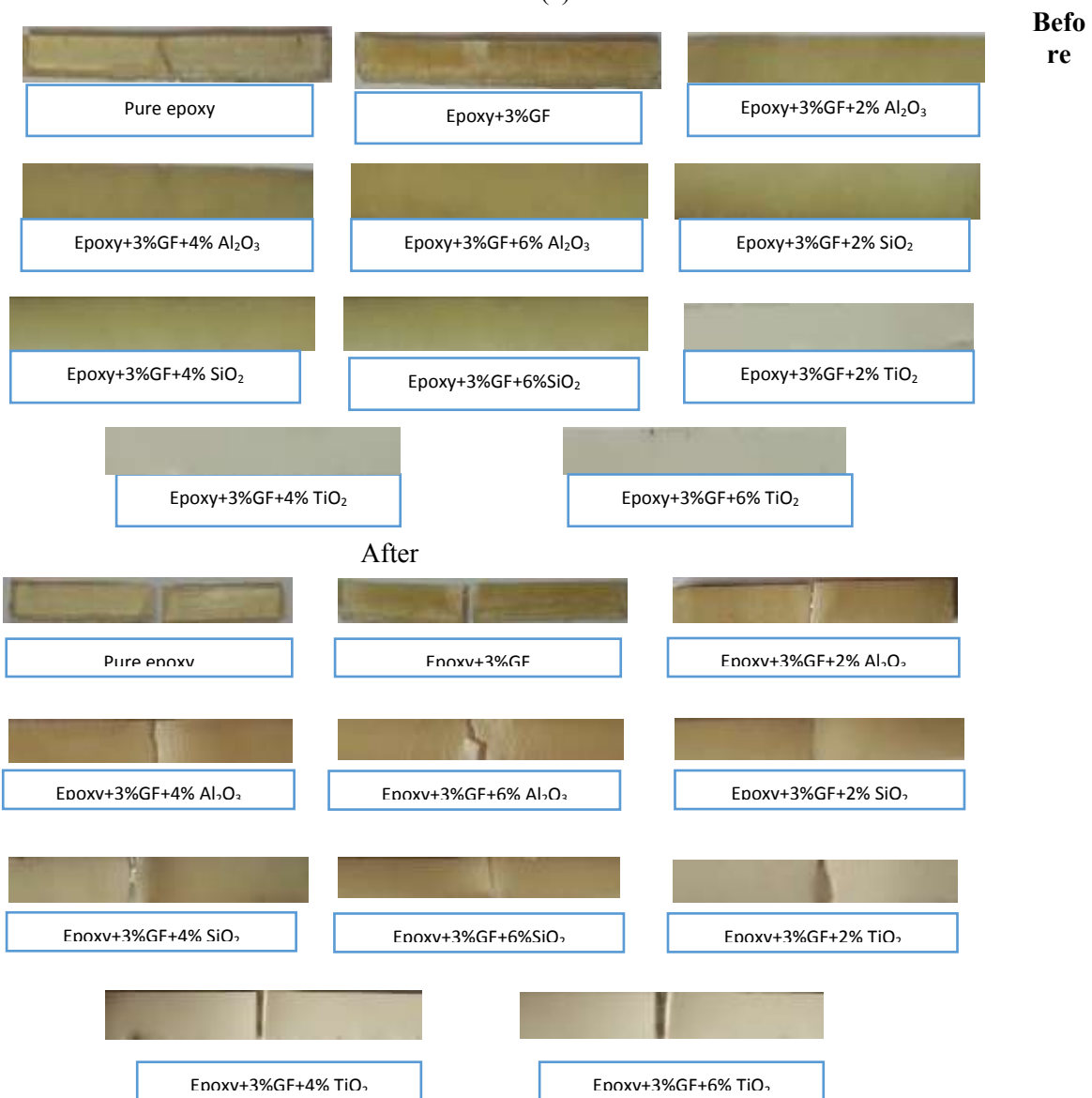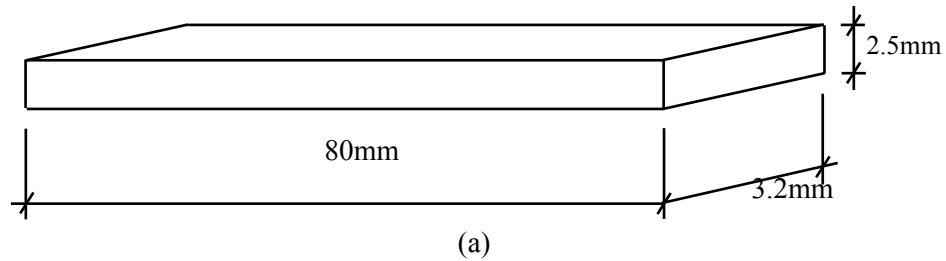L: length of the sample between Predicate (mm).
b:thikness(mm).
d:width(mm).

2.5mm

80mm

3.2mm

(a)

Before

| Pure epoxy | Epoxy+3%GF | Epoxy+3%GF+2% Al_2O_3 |

| Epoxy+3%GF+4% Al_2O_3 | Epoxy+3%GF+6% Al_2O_3 | Epoxy+3%GF+2% SiO_2 |

| Epoxy+3%GF+4% SiO_2 | Epoxy+3%GF+6%SiO_2 | Epoxy+3%GF+2% TiO_2 |

| Epoxy+3%GF+4% TiO_2 | Epoxy+3%GF+6% TiO_2 |

After

| Pure epoxy | Epoxy+3%GF | Epoxy+3%GF+2% Al_2O_3 |

| Epoxy+3%GF+4% Al_2O_3 | Epoxy+3%GF+6% Al_2O_3 | Epoxy+3%GF+2% SiO_2 |

| Epoxy+3%GF+4% SiO_2 | Epoxy+3%GF+6%SiO_2 | Epoxy+3%GF+2% TiO_2 |

| Epoxy+3%GF+4% TiO_2 | Epoxy+3%GF+6% TiO_2 |

Figure (6): (a) Flexural strength standard specimen, (b) Experimental specimens before & after test.

Figure (7) Flexural Strength device

Physical Tests
Measured Density
   This test is performed according to (ASTM D792) standard at the room temperature( ).The samples were cut into a diameter of 40 mm and a thickness of 5 mm the measured density (ρ_t) is calculated from the method of immersion in water using the following relationship [12]. Figure (8) shows the stander specimens for this test [13].
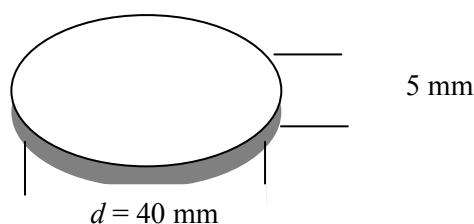

d = 40 mm

Figure (8): Standard specimens [13]

$$\rho t = (Wd / Ws - Wn) * D \quad ………(2)$$

Where:
ρt: Measured density or bulk density (gm/cm^3).
D: Density of distilled water (1 gm/cm^3).
Wd: dry weight of sample (gm).
Wn: weight of the sample, a commentator and submerged with water (gm).
Ws: weight of the sample is saturated with water (gm).

Water Absorption
   This test is performed according to (ASTM D 570) standard at room temperature [14]. Samples have been cut into a diameter of (40mm) and a thickness of (5mm). The mechanism of water absorption is explained to be the direct uptake and flow of water by capillary and transport along the reinforcement-matrix interface [15]. Water absorption percentage is calculated using (Archimedes base) according the following formula [16], Figure (9) shows standard specimens for this test.
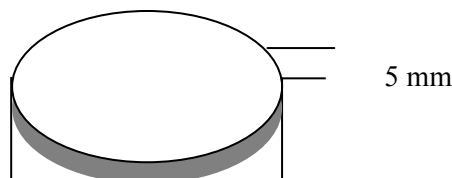
$$M\ (\%) = \frac{(mt - m_o)}{m_o} \times 100 \ \dots\dots\dots\dots\dots (3)$$

Where

M (%): water absorption percentage.

m_o : mass of specimen   before immersion (g).

m_t: mass of specimen after immersion for seven days (g).

5 mm

Figure (9): Standard specimens

Results and Discussion

Hardness shore (D)

Hardness test type (Shore (D)) has been carried out on pure Epoxy before and after glass fiber and powder fillers were added and the average of five readings in each case was taken to obtain higher accuracy results. Table (2) shows the values of hardness for the prepared (Pure Epoxy, Epoxy +3% glass fiber and micro) composites.

From figure (10) it is that there is clear that there is a pronounced effect of the addition of 3% glass fiber volume fraction percents on the hardness of the material. Increase in fiber content leads to an increase in the hardness, this may be due to the fact that the hardness is generally considered to be a property of the surface therefore this behavior of hardness is expected. The addition of the fiber leads to an increase in the elasticity and a decrease in the matrix surface resistance to the indentation, thus specimen (Epoxy +3%G.F) have higher hardness than specimen (pure epoxy).And can be seen from figure a pronounced effect of the addition of 3% glass fiber with 2%, 4%and 6% volume fraction from (micro powder) percents on the hardness of the material. It can be seen that the hardness increases with increasing volume fraction. Adding the filler particles will raise the materials hardness due to increasing in material resistance against the plastic deformation.

Result had revealed that the hardness of pure epoxy alone was (77 shore D) compared to maximum value (83.2) at volume fraction of (6% TiO_2) with particle size is (0.346µm).The reason of the increase in hardness is that TiO_2 have particle size smaller than ($Al_2O_3$and SiO_2).

Table (2): (D) of micro   **Hardness shore composites**

Types of composite	Hardness Shore (D
Epoxy	77
Epoxy +3% Glass fiber	78
(Micro Composites)	
Epoxy+3%GF+2% SiO_2	79
Epoxy+3%GF+4% SiO_2	80
Epoxy+3%GF+6% SiO_2	80.5
Epoxy+3%GF+2% Al_2O_3	81
Epoxy+3%GF+4% Al_2O_3	81.5

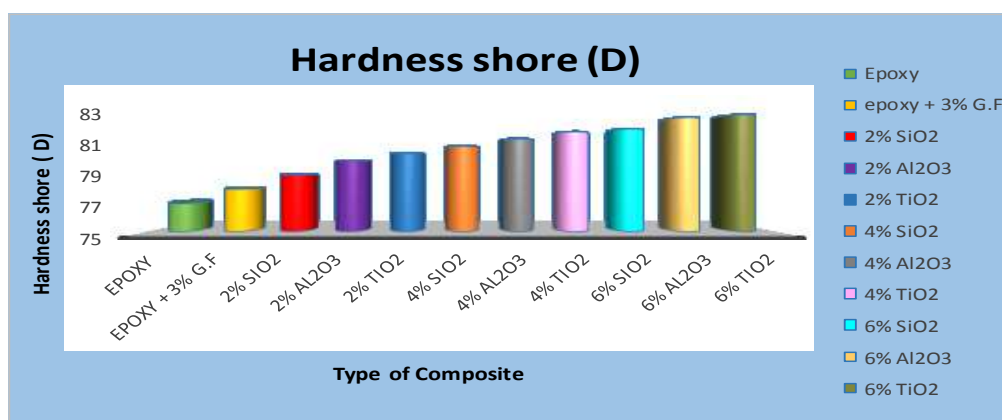|	
Epoxy+3%GF+6% Al$_2$O$_3$	82
Epoxy+3%GF+2% TiO$_2$	82.2
Epoxy+3%GF+4% TiO$_2$	83
Epoxy+3%GF+6% TiO$_2$	83.2

Figure (10) Hardness shore (D) of micro Composites

Flexural strength

   Table (3) shows the values of flexural strength for the prepared (pure epoxy, epoxy +3% glass fiber and micro) composites.

From figure (11) it is clear that there is a pronounced effect of the addition of 3% glass fiber volume fraction percent on the flexural strength, where specimen (epoxy +3%glass fiber) have higher flexural strength than specimen (pure epoxy) due the addition of 3% volume fraction of glass fiber. And can be seen from figure there is clear a pronounced effect of the addition of 3% glass fiber with 2%, 4%and 6% volume fraction from (micro powder) percents on the flexural strength of the material. It can be seen that the flexural strength increases with increasing volume fraction and decreasing of the particle size. Flexural strength of pure reference epoxy was (149 MPa) then an increasing had observed with increasing in volume fraction till it reached to its maximum value of (233MPa) by the addition of (3% glass fiber) and volume fraction of (6%TiO$_2$) with particle size (0.346μm), these results become match with our work because the TiO$_2$ have particle size smaller than (Al$_2$O$_3$ and SiO$_2$), and that consider with our suggestion in that bonding strength between epoxy and reinforcing material is high. This is attributed to the homogenous dispersibility and reinforcing-ability of the TiO$_2$ powder in the epoxy/glass fiber composites.

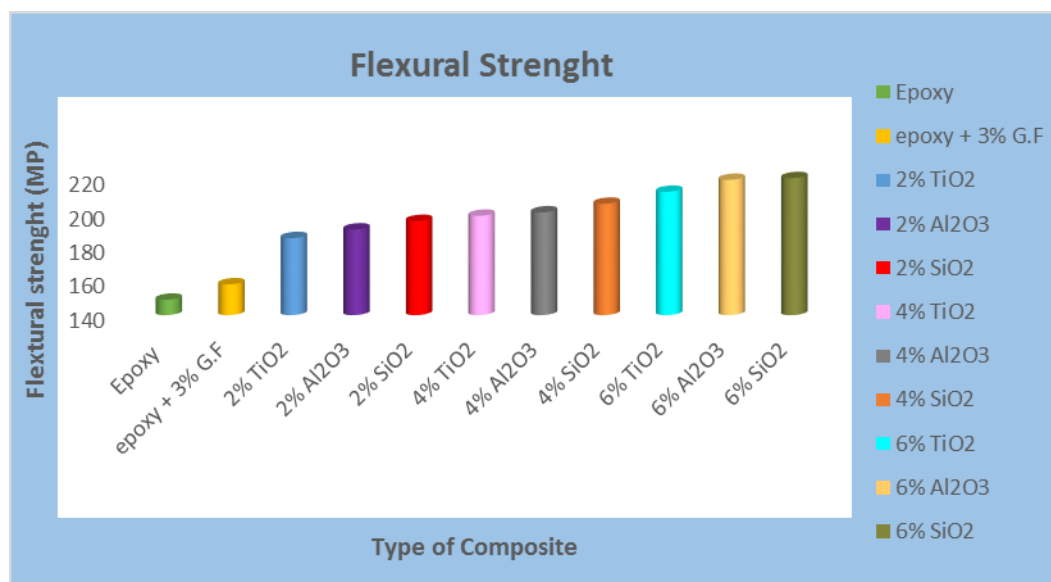**Table (3): flexural strength micro composites**

Figure (11): flexural strength micro composites

Density

Table (4) shows the values of density for the prepared (pure epoxy, epoxy +3% glass fiber and micro powder) composites.

Figure (12) show the measured density for (epoxy +3%glass fiber) with pure epoxy composites. *In Figure (12)* can be seen the higher measured density has been found to be for the specimen (epoxy +3%G.F+6%TiO$_2$) due the TiO$_2$ powder have density higher than individual density when compared with composite (Al$_2$O$_3$ and SiO$_2$) micro Powder. Where the density of TiO$_2$ (4.25 gm/cm^3), gm/cm^3) and gm/cm^3) .When value of micro composite density of (pure seen higher than additions of (TiO$_2$, Al$_2$O$_3$, powder that have than matrix (pure density of pure gm/cm^3). Al$_2$O$_3$ (4.05 SiO$_2$ (2.7 comparing the measured density with measured Epoxy) can be because the reinforcement SiO$_2$) micro higher density epoxy).where epoxy (1.05

Types of composite	Measured density (gm/cm^3)
Epoxy	1.05
Types of composite	**Flexural strength(MP)**
Epoxy	149
Epoxy +3% Glass fiber	158
(Micro Composites)	
Epoxy+3%GF+2% SiO$_2$	185
Epoxy+3%GF+4% SiO$_2$	198
Epoxy+3%GF+6% SiO$_2$	212
Epoxy+3%GF+2% Al$_2$O$_3$	190
Epoxy+3%GF+4% Al$_2$O$_3$	200
Epoxy+3%GF+6% Al$_2$O$_3$	219
Epoxy+3%GF+2% TiO$_2$	196
Epoxy+3%GF+4% TiO$_2$	205
Epoxy+3%GF+6% TiO$_2$	223

Table (4): micro   **Density of the composites**

Epoxy +6% Glass fiber	1.080
(Micro Composites)	
Epoxy+3%GF+2% SiO_2	1.140
Epoxy+3%GF+4% SiO_2	1.201
Epoxy+3%GF+6% SiO_2	1.245
Epoxy+3%GF+2% Al_2O_3	1.165
Epoxy+3%GF+4% Al_2O_3	1.220
Epoxy+3%GF+6% Al_2O_3	1.259
Epoxy+3%GF+2% TiO_2	1.180
Epoxy+3%GF+4% TiO_2	1.235
Epoxy+3%GF+6% TiO_2	1.265
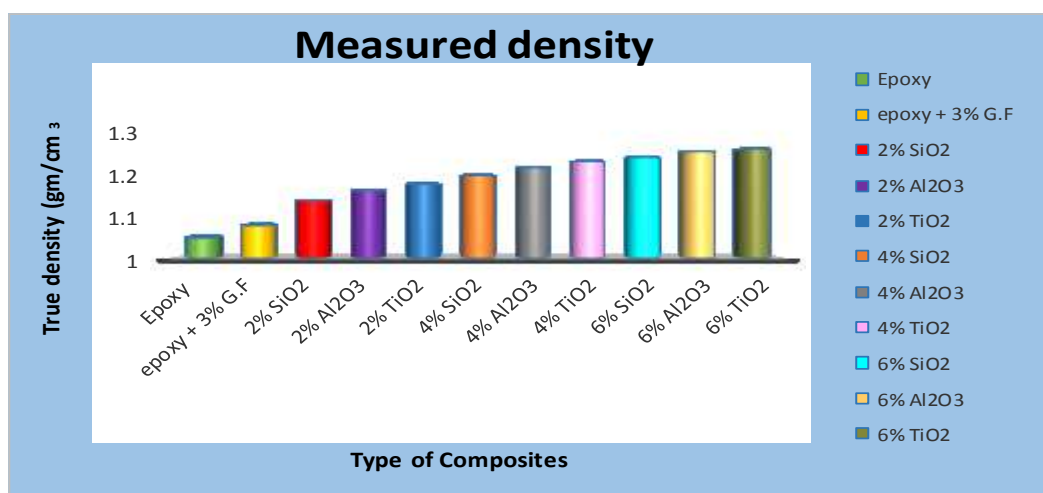
Figure (12): Density of the micro composites

Water Absorption

In figure (13) shows the water absorption of micro composites) can be seen the specimen (epoxy +3%G.F) have higher water absorption than specimen (pure epoxy), the increasing water absorption percentage with increasing volume fraction of fiber depends on the rule of mixture theory where fiber have a higher water absorption percentage than specimen pure epoxy [17]. The water absorption attacked the fiber-matrix interface, causing de-bonding of the fiber and the matrix. The failures of the composite materials were due to voids [18]. And micro composites given higher water absorption percentage than specimen pure epoxy and specimen (epoxy +3%glass fiber) composites. the higher water absorption percentage of micro composite has been found to specimen (epoxy +3%G.F+6%SiO_2) while the specimen (epoxy + 3%G.F+6% TiO_2) have lower than specimen of (Al_2O_3 and SiO_2) at the volume fraction (3%) of glass fiber and (6%) volume fraction of filler particles (micro powder), and the specimens (epoxy

+3%G.F+2% SiO$_2$), (epoxy +3%G.F+4% SiO$_2$)have higher water absorption percentage while the specimen 3%G.F+2% 3%G.F+4% than specimen of at the volume glass fiber and fraction of filler powder).

In this work the composite with larger higher water percentage when the specimens of material filled (epoxy + TiO$_2$), (epoxy + TiO$_2$) have lower (Al$_2$O$_3$ and SiO$_2$) fraction (3%) of (2%,4%) volume particles (micro

specimens of material filled particles show a absorption compared with composite with small

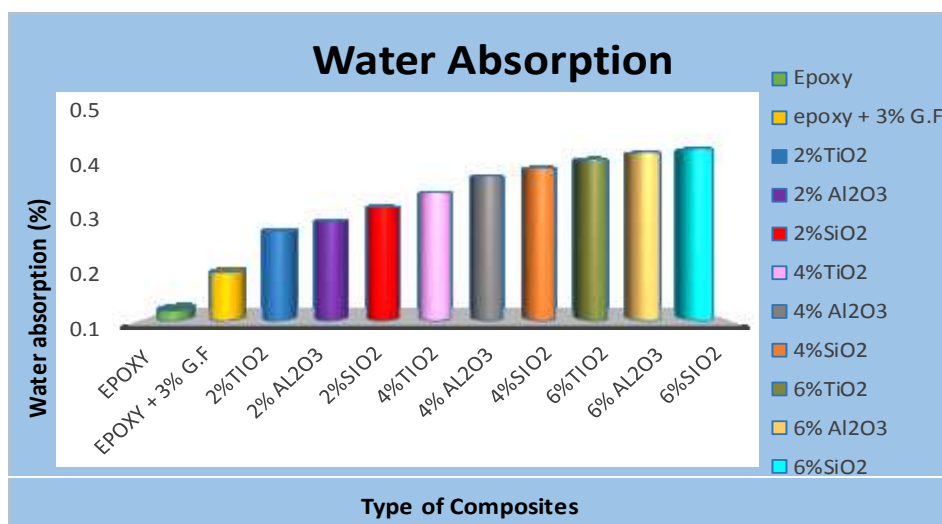| Types of composite	Water absorption %
Epoxy	0.120
Epoxy +3% Glass fiber	0.195
(Micro Composites)	
Epoxy+3%GF+2% SiO$_2$	0.322
Epoxy+3%GF+4% SiO$_2$	0.398
Epoxy+3%GF+6% SiO$_2$	0.435
Epoxy+3%GF+2% Al$_2$O$_3$	0.295
Epoxy+3%GF+4% Al$_2$O$_3$	0.382
Epoxy+3%GF+6% Al$_2$O$_3$	0.428
Epoxy+3%GF+2% TiO$_2$	0.275
Epoxy+3%GF+4% TiO$_2$	0.350
Epoxy+3%GF+6% TiO$_2$	0.415

particles because the saturation level of fillers matrix composition influenced by agglomeration that will affect the water absorption percentage of the composite material. The mean particle size of the Aluminum Oxide powder is (1.914μm) while mean particle size of the Silicon Oxide powder is (6.069μm) and mean particle size of the Titanium Oxide powder is (0.346μm).

Table (5): water absorption of the micro composites

Figure (13) Water absorption of micro Composites

CONCLUSIONS

Non –reinforced pure Epoxy has lower physical and mechanical properties than (epoxy+3%glass fiber) composites and micro composites. The values of measured density are lower than micro composite with 3% glass fiber and 6% micro powder have the higher density when compared with other composites. Micro composite with (epoxy +3% glass fiber +6%TiO$_2$) has the maximum density of (1.265) (gm/cm3) when compared with other composites. The values of water absorption of specimen (pure epoxy) lower than specimen (epoxy +3% glass fibers). Micro composite with 3% glass fiber and 6% micro powder have the higher water absorption when compared with specimen (pure epoxy) and specimen (epoxy +3% glass fibers) composites. Micro composite with (epoxy +3% glass fibers +6%SiO$_2$) has the maximum water absorption of (0.435%) Result shows that the best hardness value was (83.2 shore D), Flexural strength (223MPa) at volume fraction of (3%glass fiber) with (6% TiO$_2$).

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