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Preparation Nano Composite Material Used in Oil and Gas Pipelines

Abstract- In this research a polymeric composites have been prepared using epoxy resin reinforced with fiber glass and two nano fillers silicon oxide SiO_2 particle size around 35 nm and titanium dioxide TiO_2 particle size around (100-150 nm). These composites have been prepared in square slices (25x25) cm and 8mm thickness, by saturating fiber glass mattresses in the liquid epoxy resin with nano fillers and then cutting the product in suitable shapes to conduct the mechanical tests highest properties (tensile strength, elongation, Flexural strength, hardness strength, and impact strength. were (116.1 N/mm², 9.98%, 396 N/mm², 83.8 shore-D and 5.8 J). The aim of this research is to prepare polymeric materials with high mechanical properties that can be used in many industrial applications especially in the manufacture of oil and gas pipe systems and crude oil pipelines.

Keywords Nano Fillers, Fiber Glass, Polymeric Composites and Mechanical Properties .

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1.Introduction

Fiber glass widely is used in reinforcing the polymeric materials to improve the efficiency of the polymeric systems performance [1] which are used especially in cars, aircrafts, missiles, satellites, ships and structures for construction and in the oil industry plants particularly in the manufacturing of PVC pipes reinforced with fibers glass which are used in the oil fields and in gas distribution systems and these materials called composite materials [2]. The concept of composite material is the combination of two or more materials, one of them is reinforcing fibers and the other works as a binding agent for these fibers to give this union a new developed characteristics and specifications composite that formed from , and the most prominent examples of composite materials are: the fibers reinforced plastics (F.R.P), especially fiberglass reinforced plastics and the most important fibers used in the composite materials are fiberglass, carbon [3], boron, nylon fibers, linear polyester, jute and rayon. The most important bonding materials used in the preparation of composite materials may be form from organic origin like thermosets and the most famous thermosets are polyurethane resins, unsaturated ester [4] and epoxy and phenol formaldehyde and thermoplastic like nylon and other engineering plastic. There are other types of additives such as fillers that are added to the resin for the purpose of developing and modulating the physical and mechanical properties of the

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composite resin [5] and the efficiency of fillers depend on their physical properties such as the size of the particles and its combination and the ratios that used in the preparation [6]. These fillers have many important benefits including: reduce the cost of the product, reduce the shrinkage that occurs during the curing process , reducing the emitted temperature, changing certain characteristics of the polymer (electrical and mechanical) and increasing the strength hardness of the product [7]. The most prominent bonding material used in this research are epoxy resins which its discovery dates back to 1927 and launched the first industrial production in 1934, in which they were produced as a low molecular weight resins, then turned into a rigid thermoset resins by adding hardener .The hardener consists of poly amine monomers, for exp. (TETA)triethylenetetramine. When these compounds are mixed together, the amine groups react with the epoxy rings to form a covalent bond. Each NH group can react with an epoxy ring, so that the resulting epoxy is heavily cross linked, and is thus strong and rigid [8]. Epoxy resin has high strength, good insulating behaviour, high mechanical properties, high electrical insulation and low cost. Fibre glass is the most widely used synthetic fibre used in epoxy composites. Natural fibres have the advantages of low density and low cost. Glass is the most widely used synthetic fibre used in epoxy composites. Its advantages include its high strength, high chemical resistance, low cost and good insulating behaviour.

The type of glass fibre used as reinforcement in this study is E-glass fibre [9]. Polymer nano composites offer significant potential in the development of advanced materials for numerous applications. This new material improves bonding between filler particles and polymer chains. The properties of particulate filled polymer composites depend on the particles size, dispersion, interfacial bonding, loading, shape and surface treatment of the fillers. The dispersion of fillers in epoxy resin has influence on the physical, thermal and mechanical properties of epoxy resin. Nano fillers like silicon oxide (SiO_2) and titanium dioxide (TiO_2) are best used as protect the polymer matrix from degradation [10], long pot life period, increase thermal stability and increase mechanical properties of epoxy resin.

2. Experimental Part

I. Materials Used.

The type of epoxy resin used is (EPLV) Nitocote, from Fosroc company. The amine hardener, (Ethylene diamine) was used as a cross-linker for the epoxy. The mixing ratio was 3:1, specific gravity 1.04, mixed viscosity 1.32 poise at 35°C and fully cured at 35°C in 7 days. Two types of nanoparticles fillers were used with epoxy resin, nano silicon oxide SiO_2 particle size was about 35 nm and nano titanium dioxide TiO_2 particle size was about (100-150 nm), was obtained from Riedel Haen (product code: 14027), Germany. While the purity of titanium nano particles was $\geq 99.5\%$. E-glass fiber was purchased from Saint-Gobain Vetrotex (USA). The specific gravity of the fiber glass is about 2.7. Fiber glass was in the form of mats type woven roven. The mixture was impregnated into 8 layers of fiber glass by using hand lay-up technique. The laminates were then thermally cured in oven at 80°C [11].

II. Samples preparation method

Epoxy resin and amine hardener (Ethylene diamine) were used in this research in a ratio of 3:1. The method used in the preparation of samples of reinforced plastic was manual brushing where three samples of polymer composites have been prepared as follows; Epoxy resin and fiber glass to form the composite material sample (1). Epoxy resin, nano SiO_2 (35 nm) and fiber glass to form nano SiO_2 composite sample (2) [12]. Epoxy resin, nano TiO_2 (100-150 nm) and fiber glass to form nano TiO_2 composite sample (3) [13]. Careful mixing of epoxy resin with TiO_2 , SiO_2 and the composite then was mixed at 35°C using a high speed magnetic stirrer for one hour. The hardener was added and used as a cross-linker for

the epoxy resin. The method used in the preparation of three samples was as follows;

- 1- 25x 25 cm molds were prepared with a thickness of 8 mm in the shape of a frame fixed on a glass base both the mold and the base were coated with lubricating material for easy extraction from the mold after hardening.
- 2- The fiber glass has been cut into suitable pieces according to the dimensions of the desired mold.
- 3- Both liquid epoxy resin and filler weighed with appropriate percentage and mixed properly until homogenize, finally the addition of the hardener in a ratio 3:1.
- 4- The weight percentage used for nano titanium dioxide (TiO_2) was 5% and the ratio of nano silicon oxide (SiO_2) was 3%.
- 5- A piece of the reinforcing material was placed on top of the mold coated with the lubricating material then the binding material with the filler which has been prepared was added and brushed by a certain roll brush for the purpose of saturating the resin with the fiber glass, the addition to be continued until it reach the limit where the fibers became saturated, this was observed when the fiber changed its color, after that the air gaps are expelled by a saturated roll brush and then another layer of fiber is placed in the same way one after the other until the mold is completed.
- 6- The mold has been left to cure until the next day and then put in an oven for three hours at a temperature of (80°C) to reach full curing.

III. Characterization

All samples of epoxy resin and fiber glass sample (1), epoxy resin/ nano SiO_2 (35 nm) and fiber glass sample (2), epoxy resin / nano TiO_2 (100-150 nm) and fiber glass sample (3) were subjected to the following mechanical tests; The tensile and elongation properties were tested on Jianqiao testing machine (Tinius Olsen, Co.) Model H 50 KT (UK) at a cross-head speed of 10 mm /min. Tensile measurements were performed according to ASTM D 638-89, Uni. of Technology. Rectangular specimens of $2 \times 10 \times 8$ mm. The flexural measurements were performed according to ASTM D790 [14]. The cross-head speed was 10 mm / minute. The measurements were taken with a universal testing machine (Tinius Olsen Co) Model H 50 KT (UK) at a cross-head speed of 10 mm/min. Rectangular specimens of $2 \times 10 \times 8$ mm³ were used. The flexural modulus was calculated from the slope of the initial portion of the flexural stress-strain curve. Impact strength device was used for measuring Charpy impact test achieved by using machines testing. Inc., Amityville, ISO-179, New York. Uni. of Tech. The dimensions of the specimens were approximately $2 \times 10 \times 8$

mm³. Shore-D was used for measuring surface hardness of epoxy and polymer matrix composite fabricated by TIME GROUP INC Company, model TH 210, ITALY. University of technology [15]. Scanning electron microscopy (SEM) was performed by AIS 2300 C Angstrom Advanced Inc. New York.

3. Results and Discussion

The epoxy resin and hardener (EDA) were mixed in a ratio of 3:1 then the fiber glass was added to the mixture to form sample (1), mechanical properties for this sample were listed in table (1). The test results for tensile strength and moduli were shown in table (1). It was seen that in all the samples irrespective of the filler material the tensile strength of the composite vary with the change in types of filler of the composites. The unfilled glass epoxy composite has a strength of 97.4 N/mm² in tension and it can be seen from table (1) that the tensile strength value drops from 97.4 N/mm² to 52.7. N/mm² with the addition of nano SiO₂ filler material sample (2). The reason for the decline in the strength properties of these particulate filled composites compared to the unfilled one was that the distribution points of the irregular shaped particles SiO₂ result in stress concentration in the epoxy matrix without distributing it. We find that the highest stress strain obtained by sample (3) 116.1 N/mm² when using nano TiO₂ filler where the value increased from (97.4 → 116.1) N/mm² and this increase due to the use of this filler due to its distinctive properties in increasing the strength and durability of the composite material to be prepared as well as the partial size of the filler has a significant impact on this increase. Figure (1) shows the effect of adding fillers on the value of tensile strength. The highest value for elongation and flexural tests were measured for composite sample (1), when using epoxy and fiber glass, without adding any filler this is shown in figures (2 and 3), the highest value of the hardness test was in the composite sample (3) when using nano titanium dioxide with fiber glass and epoxy, as shown in figure (4) and the highest value of the impact strength test was in the composite samples (2 and 3) where both gave the same results as shown in figure (5). According to the obtained results the use of nano titanium dioxide filler with fiber glass and epoxy increases most of the mechanical properties of tensile strength, hardness and impact strength so composite sample (3) gave the best results (where nanoparticles help in adhesion of polar force of nano TiO₂ particles and Van der-waals bonding) and good space distribution of nano TiO₂ particles in nano composite. Figures (6 and 7) show the

scanning electron microscopic (SEM) test of nano (SiO₂ and TiO₂) particles. SEM test were recorded of nano-SiO₂ with average particle size around (35) nm and nano-TiO₂ with average particle size around (100-150) nm. Figure (8) shows the picture of the surface of samples (1), (2) and (3) under microscope before tensile test, where the surfaces are satin without any cracks. Figure (9) shows the picture for vertical view for samples (1), (2) and (3) under microscope before tensile test, where shows the uniform arrangement of fiber glass layers that been combined the epoxy resin. Figure (10) shows the picture of the surface the samples (1), (2) and (3) under microscopic after tensile test and the image shows cracks in the surface caused by a large force of tension. Figure (11) shows the picture for vertical view the samples (1), (2) and (3) under microscopic after tensile test shows the crashed and Irregular layers of fiber glass because of exposure the samples to the cutting force and large tension.

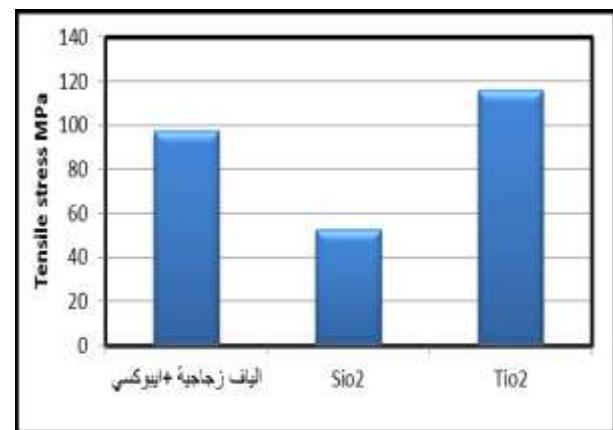


Figure (1): The effect of adding fillers on the value of tensile stress

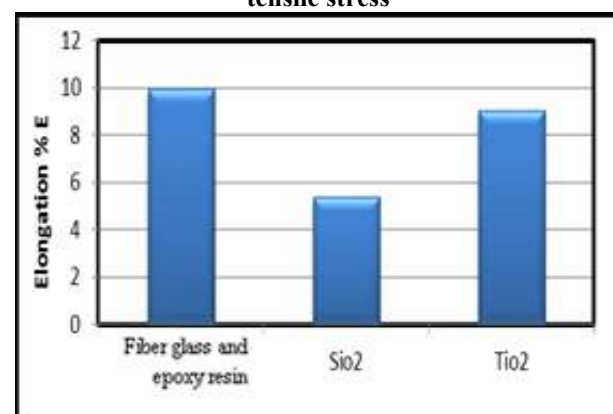


Figure (2): The effect of adding fillers on elongation test

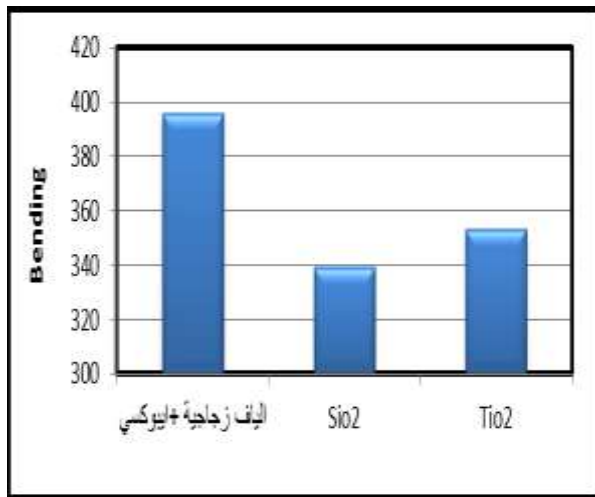


Figure (3): The effect of adding fillers on flexural test

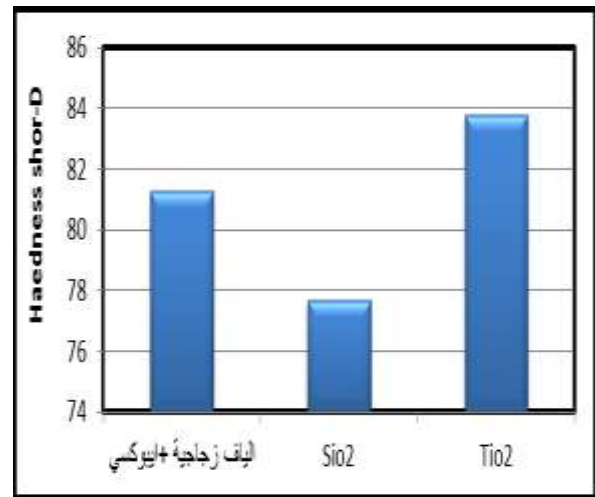


Figure (4): The effect of adding fillers on hardness tes

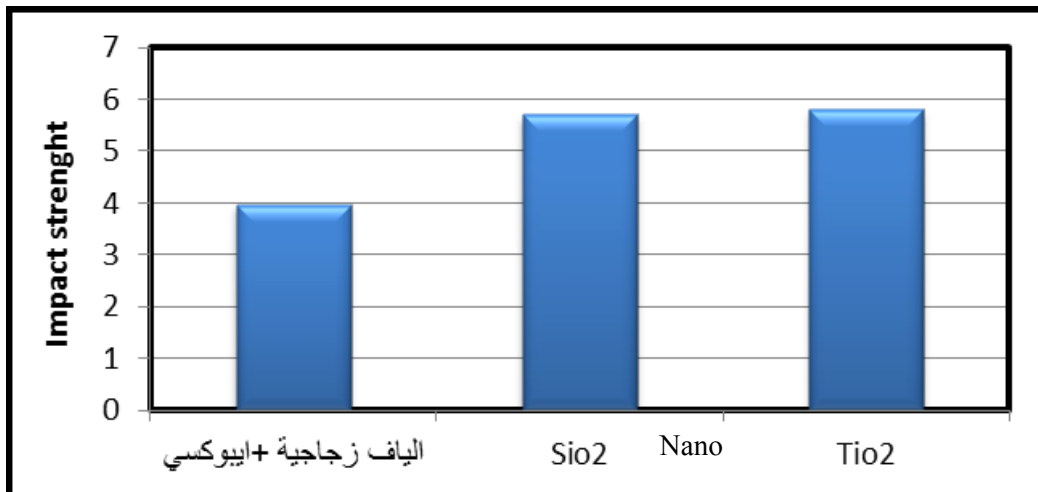


Figure (5): The effect of adding fillers on impact test

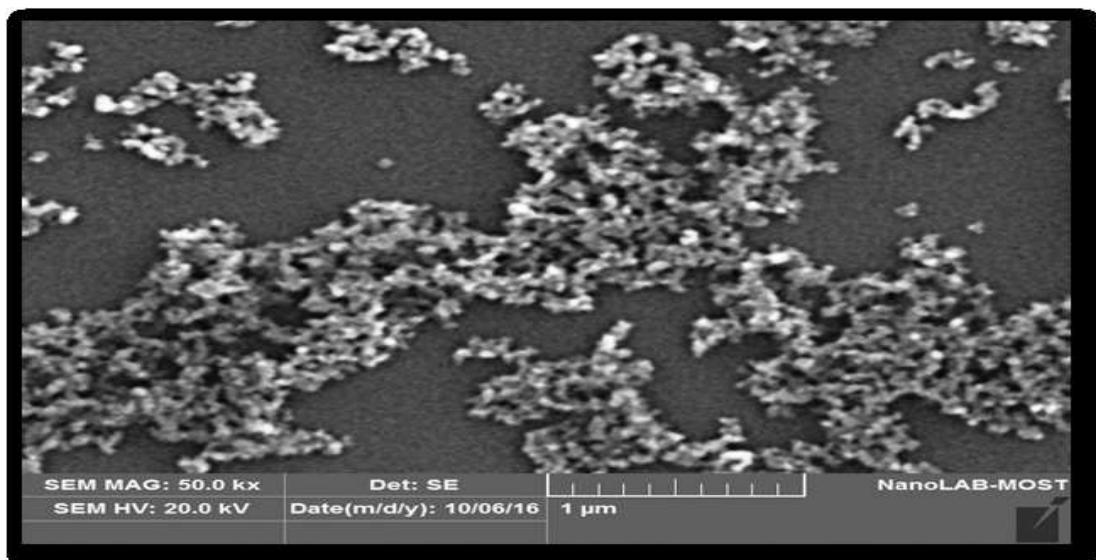


Figure (6) : Scanning electron microscopic (SEM) of nano - SiO₂

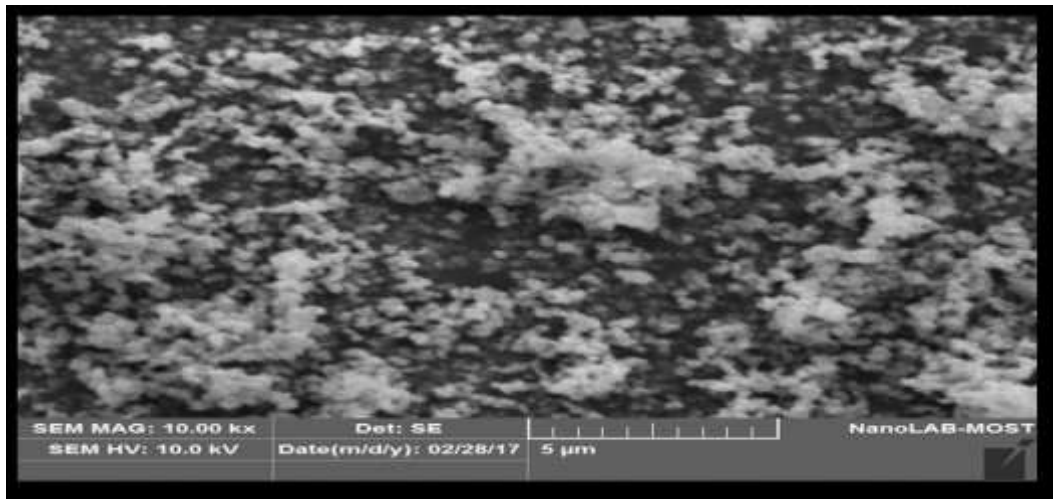


Figure (7) : Scanning electron microscopic (SEM) of nano -TiO₂

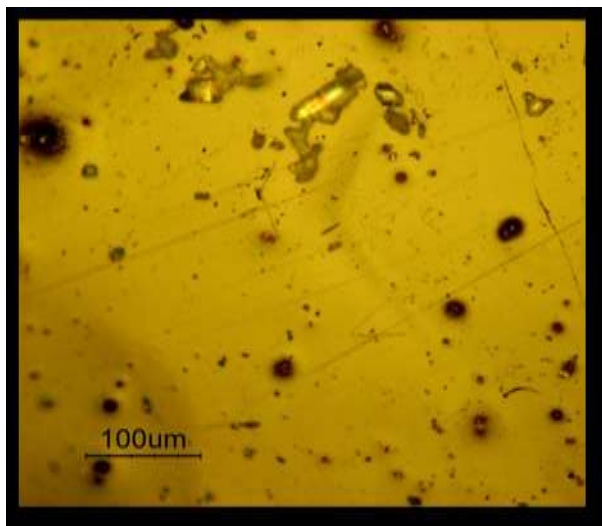


Figure (8)

Picture of the surface the sample (1) under microscope before tensile test

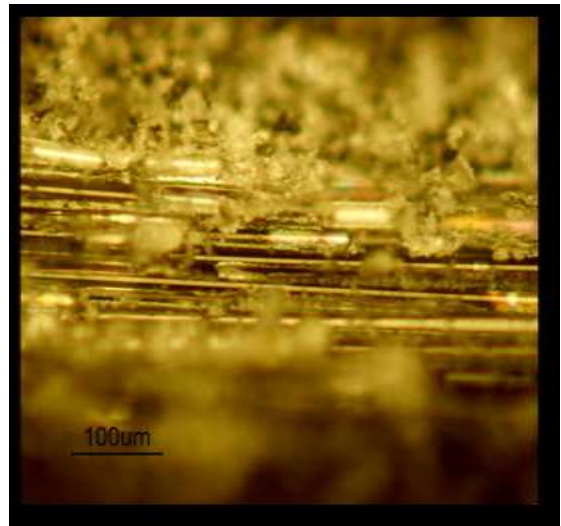


Figure (9)

A photograph for vertical view for sample (1) under microscope before tensile test

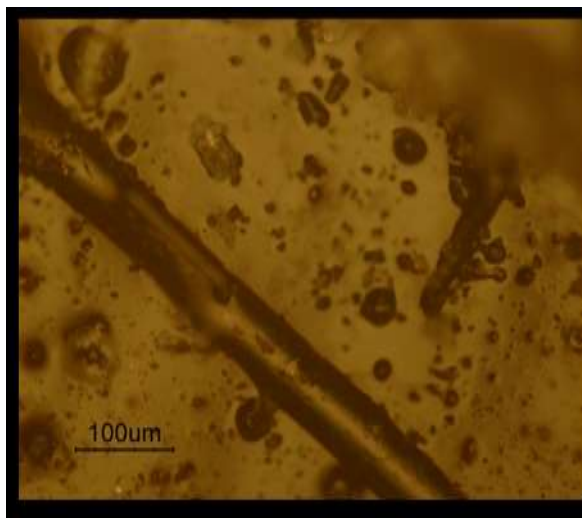


Figure (10)

Picture of the surface the sample (1) under microscopic after tensile test



Figure (11)

A photograph for vertical view for sample (1) under microscope after tensile test

Table (1) shows the results of the mechanical tests of the composites

N0.	Sample	Tensile Strength N/mm ²	Elongatio n %	Flexural Strength N/mm ²	Hardness shore D	Impact Strength J
[1]	Fiber glass /Epoxy	97.4	9.98	396	81.3	3.97
[2]	Fiber glass and nano SiO ₂ /Epoxy	92.7	9.42	339.6	77.7	9.7
[3]	Fiber glass and nano TiO ₂ /Epoxy	116.1	9.00	353.3	83.8	9.8

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