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The Influence of Immersion in Different Chemical Solutions on the Mechanical and Physical properties of (Epoxy/Styrene-Butadiene Rubber) Blend Reinforced with Nano Copper Oxide

Abstract- The aim of this work was to evaluate some mechanical and physical properties of a composite material which consists of (epoxy/styrene-butadiene rubber) blend as a matrix, reinforced with copper oxide (CuO) with a weight fraction 3%, and the composite material was manufactured by hand lay-up. The optimum mixing ratio was (75:25) % of epoxy and (SBR) was chosen to accomplish the work due to its highest impact strength (2.1KJ/m^2). The tests that were performed on the material were: tensile test, impact test, thermal conductivity test, and the absorption test, in addition to the microscopic imaging using scanning electron microscope (SEM), to determine the surface morphology of the specimens. Sodium hydroxide (NaOH) and hydrochloric acid (HCl), both (0.1) normal concentration solutions, were used for the immersion. The results showed that CuO nanofiller improved tensile and impact properties of the blend, besides increasing resistance to diffusion of chemicals into the material. The results showed that the immersion in HCl solution increased the impact strength of the composite from (2.27KJ/m^2) to (3.38KJ/m^2), and also increased the tensile strength from (9.2MPa) to (9.8 MPa), while immersion in NaOH solution decreased the tensile strength to (7.3MPa), but increased the impact strength to (2.42KJ/m^2). Thermal conductivity was ($0.21\text{W/m.}^\circ\text{C}$) before immersion in solutions, but changed to ($0.27\text{W/m.}^\circ\text{C}$) and ($0.24\text{W/m.}^\circ\text{C}$) after immersion in HCl and NaOH respectively. The weight gain after immersion in NaOH was higher than weight gain after immersion in HCl.

Keywords- (Epoxy/SBR) blend, nano filler, Optimum mixing ratio, tensile test, impact test, thermal conductivity.

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1. Introduction

Polymer Blending is a common practice to obtain properties that don't exist in the original polymeric constituents, like adding an elastomer to a rigid polymer, so the resulting blend can be more ductile and high impact resistant. Nano structured composite materials have acquired interest in the last few decades as that offer a wide range of potentials and properties [1].

Epoxy resins are thermosets that are widely used in industrial and engineering applications, and styrene-butadiene rubber (SBR) is a synthetic rubber that can be used as an ingredient in many industries [2]. Copper oxide is a transient metal oxide, which is used as a nano additive for polymers to get some desired properties due to its high surface area compared to its bulk form, resulting in a more homogeneous and an easier to process composite [1].

Rana [3] studied some mechanical properties of composite materials consisting from a polymer blend of (epoxy/ NBR) as a matrix, which were reinforced with a 35% volume fraction of reinforcement of glass and carbon fibers. The

researcher also studied the effect of weather factors and ultra violet light on these properties. The results obtained in the study showed that the composite reinforced with carbon fibers maintained their properties against weather factors and U.V. light, better than the composite which was reinforced with glass fibers, and that water had a milder effect than KOH solution on the mechanical properties.

Dheya and Jabbar [4] studied the mechanical properties of a particulate polymer blend composite, consisting of (epoxy/NBR) blend as a matrix, reinforced with silica (SiO_2), and alumina (Al_2O_3). The researchers also studied the effect of immersion in solutions on the properties, and found that acid solutions had a stronger effect on water, and that impact strength, hardness and wear resistance decreased after immersion.

2. Experimental Work

I. Matrix materials

Epoxy resin used in this work was (sikadur -105); manufactured by Sika™ corporation (United

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States) is a low viscosity, clear or amber color, two component system, which is hardened by the addition of a hardener in a ratio (2:1) of resin and hardener respectively. The styrene butadiene rubber (SBR) used in this work was manufactured by Robinson™, in the United States, this material is a synthetic rubber, with a pale white colour, and has excellent tensile and impact properties, together with chemical and abrasion resistance.

II. Reinforcement Material

Nano Copper Oxide (nano CuO): this nano powder was manufactured by Shanghai Hanghong Chemicals™ Co. Ltd. (China) and was used as reinforcement for the (Epoxy/SBR) blend. It comes in the form of a black powder with a grain size of (40 nm), and a purity of (99%), and a chemical composition of (79.87%) copper and (20.10%) oxygen. The nano CuO was added in a weight fraction of (4%).

III. Method of Preparation

Hand lay-up technique was used to prepare the specimens, and the process involves mixing epoxy with its hardener in a ratio of (2:1) respectively. The SBR was added to epoxy and its hardener while it's still in the liquid state. The optimum mixing ratio was decided upon mixing different ratios of epoxy and SBR, and then the impact test was carried out on each specimen to evaluate the blend ratio that has the highest impact strength. The blend ratios of (EP:SBR) were (95:5)%, (90:10)%, (85:15)%, (80:20)%, and (75:25)%, respectively. The optimum mixing ratio (OMR) was decided upon the results of impact test, thus the blend with the highest impact strength (i.e. (75:25) % of (EP:SBR) respectively, was chosen to accomplish the work. The liquid mixture was poured into a flat mold, and left to harden overnight at room temperature, then the solid mold was put in an oven for further curing at (50-60) °C for 4 days, 4 hours a day, before cutting it into specimens according to standard specifications.

3. Characterization and Measurements

I. Impact Test

Charpy impact test was used to measure the impact strength of the material. The instrument was manufactured by Testing machines™, AMITYVILLE Inc., New York, USA, and according to the standard specification (ISO-179) [5]. The impact strength can be calculated from the following equation [5]:

$$\text{Impact strength} = \frac{E}{A} \quad (1)$$

where E is the fracture energy (KJ), and A is the cross sectional area of the specimen (m^2), The test was carried out before and after immersion in chemical solutions of sodium hydroxide (NaOH) and hydrochloric acid (HCl), both with a (0.1) normal concentration for one month. Figure 1 shows the impact test specimen.

II. Tensile Test

In this test, the specimen is subjected to two equal forces acting in different directions. The tensile test was done according to the standard specification ASTM-D 638 [6]. The stress can be obtained from the following equation [6]:

$$\sigma = \frac{F}{A} \quad (2)$$

Where σ is tensile stress (N/mm^2), F is the applied force (N), A is the cross sectional area (mm^2), the specimen is shown in Figure (2) [6] and the test was carried out using a computer controlled electronic Laryee machine, by Universal Testing Machine™ (WDW-50), with a loading rate (5 mm/min.). As with impact test, tensile test specimens were immersed for 1 month in HCl and NaOH solutions to evaluate the effect of these solutions on tensile strength. Figure 2 shows the tensile test specimen.

III. Thermal Conductivity

Thermal conductivity was measured using Lee's disc method, which consists of three brass discs, and a heater is placed between the first and the second discs, while the specimen is placed between the second and third discs. Thermal conductivity can be expressed by the following equation [7]:



Figure 1: Impact test specimen [5]

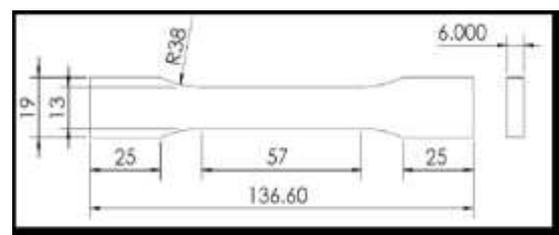


Figure 2: Tensile test specimen [7]

$$q = -k \frac{dT}{dX} \quad (3)$$

Where q is the heat flux per unit time per unit area (the area perpendicular to the heat flow direction) (W/m^2), k is the thermal conductivity ($W/m.°C$), dT/dX is the temperature gradient through the conducting medium. Thermal conductivity for a material can be measured by Lee's disc method through the following equation [7]:

$$K = \frac{t_2 - t_1}{d_1} = e \left[t_1 + \frac{2}{r} \left(d_1 + \frac{1}{4} d_s \right) + \frac{1}{2r} ds t_2 \right] \quad (4)$$

Where the value of (e) can be calculated from the following formula [7]:

$$H = IV = \pi r^2 e (t_1 + t_2) + 2\pi r e \left[d_1 t_1 + \frac{1}{2} d_s (t_1 + t_2) + d_2 t_2 + d_1 t_1 \right] \quad (5)$$

Where e is the heat loss for one second for one square metre, H is the supplied power to heat coil at steady state, d is the thickness of disc (m), r is the radius of disc (m), t is the temperature increase ($°C$). Figure 3 shows the thermal conductivity specimen.

IV. Absorption Test

The absorption test was carried out using square shaped specimens (10×10) mm^2 , according to the standard specification ASTM D-570 [8], and these specimens were weighed before immersion and each week after immersion in (0.1) normal concentration solutions of NaOH and HCl. The weight was taken using a sensitive 4-digit scale (ae ADAM)[®], and the weight gain was obtained as function of time, and was calculated by the following equation [8]:

$$Weight\ gain\ \% = \frac{W_2 - W_1}{W_1} \times 100 \quad (6)$$

Where W_1 , W_2 are the weights of specimen before and after immersion respectively [8]. Figure 4 shows the absorption test specimen.

V. Scanning Electron Microscope Imaging

Scanning electron microscope (SEM) model (Inspect- S50) by FEI[™], Netherland, was used to compare the specimen surface morphology after immersion in HCl and NaOH. Figure 5 shows the scanning electron microscope used in this work.

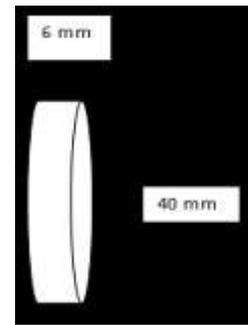


Figure 3: Thermal conductivity test specimen [7]

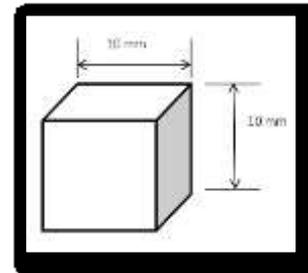


Figure 4: Absorption specimen [8]



Figure 5: Scanning electron microscope used in the study

3.4. Results and Discussion

I. Impact Strength

The results can be noticed in Figure 6. Epoxy gained toughness through the addition of a rubber phase (SBR). The rubber helps to disperse the impact energy and transform it into a plastic deformation [3], thus the addition of CuO nanoparticles have also helped to further improve the impact strength of the composite (2.32 KJ/m^2), since these particles act as crack-inhibitors in the way of the growing micro cracks [9]. It is noticed that the impact strength had increased to some extent after immersion in chemical solutions, with the specimens immersed in HCl having the highest impact strength (3.38 KJ/m^2), followed by the specimens immersed in NaOH with impact strength value (2.42 KJ/m^2). This can be attributed to the fact that the chemical solution penetrated into the matrix (epoxy/SBR blend) and the specimens

immersed in HCl gave the highest impact strength compared to NaOH is due to the fact that the molecular weight of HCl (36.4 g/mol.) is less than the molecular weight of NaOH with a molecular weight (39.9 g/mol.), thus the elasticity increased due to weakening of chemical bonds between the polymer chains, this consequently led to a higher amount of energy absorption to fracture the material [10].

II. Tensile test

The results show that the specimens varied only slightly at the beginning of the tensile test, but as the test proceeded, the specimens that were immersed in HCl and NaOH revealed a higher extension than that of the original specimens, with the one that was immersed in NaOH having the highest extension. The tensile behavior of the specimens is shown in Figure 7.

The addition of CuO nanoparticles creates a high surface area, which is beneficial since the external load applied to the matrix will be transferred more efficiently to the particles, thus the reinforcement will be effectively absorbing the external load, and its contribution to the overall strength of the composite material will be greater [11]. NaOH solution clearly showed that it had a higher effect in reducing the tensile strength of the material, so the tensile strength decreased from (9.2 MPa) before immersion to (7.3 MPa) after immersion in NaOH, at the same time increasing elongation due to increased elasticity, thus the elongation% after immersion in NaOH was (8.3%) compared with (6.4%) before immersion. The immersion in HCl increased tensile strength to (9.8 MPa), and the elongation increased to (7.9%). The solution penetrates first into the matrix, making use of the existing flaws that originate during preparation of the material, and thus facilitating the way for the liquid to penetrate into the interface, decreasing the bonding between the reinforcement and the matrix, and this effect is noticed mainly when polymers are subjected to basic solutions [3].

III. Thermal Conductivity

The CuO nanoparticles had a positive effect by increasing thermal conductivity of the material to (0.21 W/m.°C), and this may be caused by particle-to-particle interaction, which makes the whole system conduct heat more effectively because of the contact that takes place between the nanoparticles, i.e.: adding other methods of heat transfer (conduction, as CuO is a conductive metallic material) [12]. The thermal conductivity of the samples increased to (0.27 W/m.°C) and (0.24 W/m.°C) after immersion in HCl and NaOH

respectively. This may be caused by the penetration of chemical solution into the blend. Thus, the mobility of the chains increase, and the links between them weakness, leading to an increase in the ability to conduct heat. The acidic solution dissolves the matrix hydrolytically and thus its effect is more obvious than the alkaline solution [13]. Figure 8 shows thermal conductivity values of the specimens.

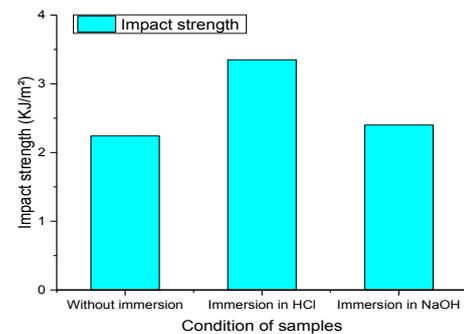


Figure 6: Relation between impact strength and condition of composite.

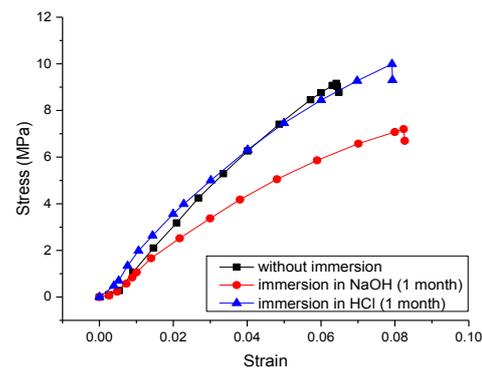


Figure 7: Relation between stress-strain of the composite.

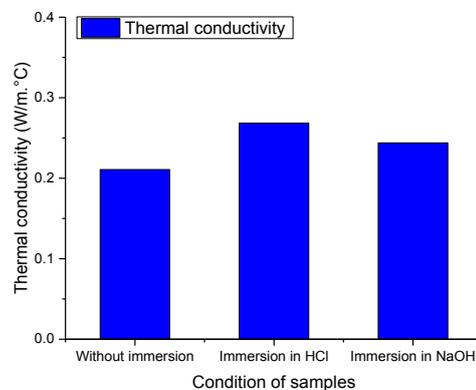


Figure 8: Relation between thermal conductivity and condition of composite.

IV. Absorption Test

Figure 9 shows the weight change for the studied blend after immersion in chemical solutions. The two specimens showed a weight gain after immersion in chemical solutions for 8 weeks, and this phenomenon may have resulted from the presence of discontinuities and voids that originate during preparation of the composite, as these voids tend to be weak points towards the attack of solutions, leading eventually to weight gain [3]. The specimen immersed in NaOH was the one which was most affected by the attacking nature of NaOH. The movement of the solutions into the polymer structure is driven by the difference in concentration between the two mediums, where the concentration gradient tries to balance on both sides of the mediums, thus the molecules move from high concentration towards low concentration [14]. The weight gain was higher in the specimens immersed in HCl in the early stages of immersion, while for the specimens immersed in NaOH, the weight gain was higher at the later stages of immersion, which may be attributed to the fact that the HCl has a lower molecular weight, hence more ability to penetrate into the material than the NaOH solution [10]. In the case when filler is added to the polymer in a composite, the free volume that exists in the polymer matrix will be occupied with the filler particles, hence the penetrant molecules will not be able to move easily towards the polymer, thus the addition of the filler will contribute positively to the stability of the polymer matrix [14].

V. Scanning Electron Microscope Imaging

SEM technique was used to determine the surface morphology of the specimens and the extent of the effect done by the solutions on the material, Figures 10 and 11 show different patterns of chemical attack for each solution, so the specimens immersed in NaOH showed polymeric components exposed in a network structure of (SBR) exposed through the larger component (epoxy), while the specimens immersed in HCl showed a pitting pattern distributed all over the structure, so the solution didn't preferably attack a single component [15].

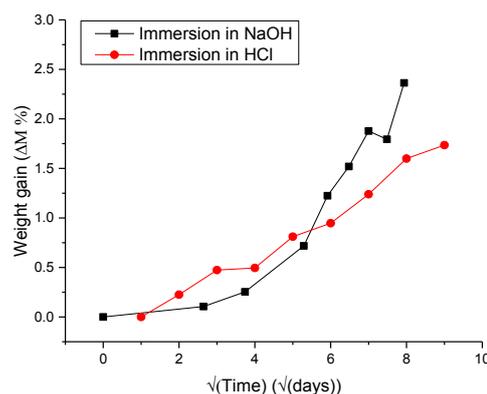


Figure 9: Relation between weight gain vs. $\sqrt{\text{time}}$ (days) of composite

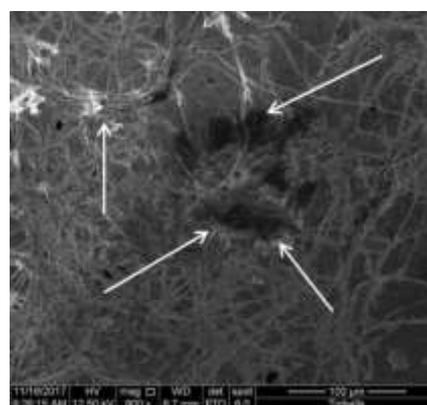


Figure 10: Surface morphology after immersion in NaOH.

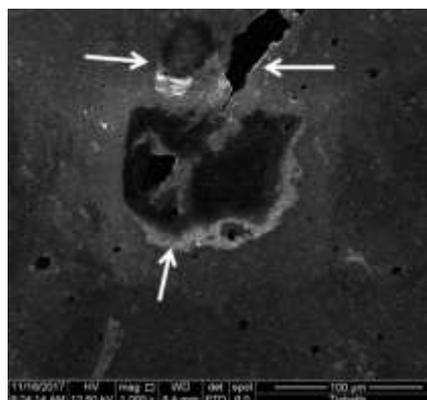


Figure 11: Surface morphology after immersion in HCl.

4. Conclusions

1. The O.M.R of (epoxy/SBR) blend gave an impact strength (2.1 KJ/m^2), and the addition of nano CuO increased this value to (2.32 KJ/m^2).
2. The addition of nano copper oxide contributed positively to mechanical properties, so the tensile strength became (9.2 MPa), with elongation (6.4%).

3. Immersion in HCl solution increased the tensile strength from (9.2 MPa) to (9.8 MPa), while NaOH decreased tensile strength to (7.3 MPa). On the other hand, elongation after immersion in both solutions increased, and elongation became (8.3%) and (7.9%) with NaOH and HCl respectively.

4. Immersion in HCl solution increased impact strength from (2.32KJ/m²) to (3.38 KJ/m²), while impact strength increased to (2.42KJ/m²) after immersion in NaOH.

5. Thermal conductivity was (0.21 W/m.°C) before immersion, and changed to (0.27 W/m.°C) and (0.24 W/m.°C) after immersion in HCl and NaOH respectively.

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