A Study of the Chemical Resistance and Hardness of Epoxy Reinforced by Magnesium oxide and charcoal l activated Particles

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ABSTRACT

In this research, some samples from Epoxy with Magnesium oxide and charcoal activated Particles as filler in different weight percent (1, 3, and 6) wt. % have been prepared. Chemical resistance test has been done using simple immersion test in different periods of time and the immersion done in different solutions (1N NaOH, 1N HCL and 25 g of NaCl per litter of distilled water). Shore (A) hardness tests were carried out on the prepared samples before and after the immersion into distilled water, (1N HCl) acid, (1N NaOH) and 25 g of NaCl per litter of distilled water with normality (1). The results exhibited increase in chemical resistance and hardness values increase with increasing magnesium oxide and charcoal activated ratio. The maximum hardness was observed in the case of epoxy -6wt% MgO composites i.e. 108.8 and least chemical resistance in the case of pure epoxy being 4.885% in NaOH.

Keywords: Epoxy, Percent weight loss, Hardness.

INTRODUCTION

Many types of thermoplastic elastomers are used in outdoor applications and a common concern of these materials is the durability of the materials in industrial applications. During long periods of service, most of the polymer products gradually lose their properties due to the macromolecular chain degradation. Normally, polymers in the state of their end-use are not pure materials. In many cases there are added substances which alter the engineering and/or chemical properties of the polymer in a useful way. The polymer also may contain small amounts of monomer, entrapped during the polymerization process. Such additives and impurities may participate in the slow chemical degradation of the polymer and, of course, add to the general physical complexity of the polymer [1] and [2]. If the polymer is attacked by the environment, the performance of the material in service will be adversely affected. Degradation of the performance of the polymer may lead to premature failure of the material, resulting in increased downtime for the system, and requiring costly maintenance procedures. The study of dimensional stability and mechanical properties of polymeric materials under different environmental conditions such as humidity changes or changes in temperature, solvent, mechanical load, radiation, deserves much importance. This is because materials with superior ageing resistance can be satisfactory durable, [3].

Polymer matrix composites (PMCs) have found increasing use in the aerospace industry due to potential benefits that they offer when compared to metals, such as reduced part count, lower weight and improved fatigue performance. Epoxy resins modified with inorganic particles such as, titanium dioxide, silica, alumina, fly ash, clay and so on have shown improved performances. For inorganic/organic composites, the size of particles and the interfacial adhesion have great effect on the properties of the resin matrix [4] and [5].

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However the main drawbacks are poor mechanical properties and high moisture absorption. The latter is owing to its hydrophilic nature that produces a harmful effect on almost all properties, including the dimensional stability .This problem can be imperative if the composite is used in high moisture environments. Moisture uptake in polymer matrix composites has a deleterious effect on their mechanical properties [6] and [7].

Several researchers studied the effect of water absorption on the behavior of Epoxy particle Composite as, Randa K.Hussain et al [6], have studied the effect of Particle Weight Fraction on Bending of Epoxy particle Composite .The filler particles content (Al_2O_3 and SiO_2) was varied from 2%, 4%, 6% and 8% by weight of total matrix in the composites were subjected to bending tests. The composites showed improvements in bending properties with increased filler content, composite materials have significantly higher modulus of elasticity than the matrix material. Faik H. Anter and Hind S. Hasan [7], investigated the effect of Water Absorption on Hardness Property for Epoxy Reinforced by Glass Fibers. The results showed that value of hardness done at room temperature decreases with increasing time of immersion in water. The weight of absorbed water increases with increasing time of immersion in water specially in the first week .The quantity of absorbed water decreases or increases also according to kind and number of reinforcing layers of glass fiber . E. Mboungou et al [8], investigated the Impact of Water Content on the Electrical Behavior of Epoxy Insulators, Experimental results suggested that the water absorption rate is independent of the roughness of the sample, but is influenced by the kind of filler used; a high humidity level leads to an increase of the leakage current in smooth silica filled samples. Vignesh .C and Kumareesan .K [9], have studied ageing of SiC filled reinforced epoxy hybrid composites. Experimental results showed chemical resistance test when materials are immersed in HCL and NaOH solutions, the materials chemical resistance is found higher than the 10wt% Sic filled material. Its chemical resistivity is nearly 0.8 percent higher than other materials. In 1990 Jatin R. , Ravji D. , Ranjan G. [10], showed that the harness values measured on the glass fiber reinforced composites after immersion in (H2O, Acetone , 10% NaOH , 10% HCl) for seven days fell within the small range . This indicated no appreciable influence of the regents on the harness.

The objective of this study is to compare the effect of MgO and charcoal activated content in epoxy composite on chemical resistance and hardness as function of immersion time in NaCl, NaOH and HCl solutions.

Experimental Method

Materials

For the present study, a commercial available epoxy resin TOPSEAL EP 70 Part A was used as the polymer matrix. TOPSEAL EP 70 Part B was used as the hardener for epoxy resin produced from Specialty Building Industries UAE. Epoxy resin is primarily of low viscosity (high fluid), which penetrates in to the smallest capillaries and pores. The hardener material was added with ratio 1:4 of resin. TOPSEAL EP 70 is an anti-corrosive coating provides protection to concrete and metal strictures against corrosion from aggressive environments. The two types of reinforcement materials (Magnesium oxide and charcoal activated) were used to string the epoxy rating. Magnesium oxide (MgO) which manufacture by B.D.H , Ltd (British). It is a white powder with a specific gravity of 3.58 g/cm^3 and particle size of 100μm. Charcoal Activated which manufacture by Latha Chemical Company, it is black powder with specific gravity of 1.1533 g/cm^3. In addition hydrochloric acid (HCL) and sodium hydroxide (NaOH*)* supplied by BDH, England.

Preparation OF Composite Specimen

The Magnesium oxide and Charcoal Activated materials are reinforced with epoxy resin matrix material and curing hardener. The seven specimens were manufactured for ageing of materials and hardness studies. The manufactured seven specimens are pure epoxy, 1wt%

MgO– epoxy resin, 3wt% MgO – epoxy resin, 6wt% MgO – epoxy resin, 1wt% charcoal – epoxy resin, 3wt% charcoal – epoxy resin, 6 wt% charcoal – epoxy resin. The materials were manufactured by hand layup method and then used vacuum chamber to remove micro bubble that forming during mixing materials. The specimens were completely submerged in 1N hydrochloric acid (HCl) ,1N sodium hydroxide (NaOH) and 25 g of NaCl per litter of distilled water.

Chemical Resistance Test

For the chemical resistance test, the dried specimens (20 mm x 20 mm x 3 mm) were immersed in 100 ml of 1 N NaOH ,1N HCl and 25g NaCL per litter of water for different intervals of time (2,7 ,18 and 31 days in NaOH and Hcl) and (4,11,18 and 31 days) in 25 g of NaCl per litter of distilled water. After this, the samples were filtered out, dried and weighed. The percent chemical resistance (Pcr) was calculated in terms of weight loss in the following manner [9]:

$$\text{Percent weight loss \% (Pwl)} = \frac{\text{Wi} - \text{Wf}}{\text{Wi}} * 100 \qquad \ldots(1)$$

Where: Wi is the initial weight of specimen and Wt is final weight of specimen from acid, alkali and salt solution.

Weight of sample

Electronic sensitive balance (Type AS 220/C/2, 4 digits) was used to weight the samples before and after immersion in solution.

Hardness Test

Hardness is a measure of how resistant solid matter is to various kinds of permanent shape change when a compressive force is applied [7]. Shore A hardness instrument (Type HT-6510 A) shown in Figure (1) was used to carry out the hardness test by using pointed dibbing tool. The pointed dibbing tool penetrates the material surface by the pressure applied to the instrument where the dibbing tool head touches quite the surface of the samples then the hardness values of the samples are measured. Hardness test has been carried out for as for the standard test method ASTM D 224-00 [11]

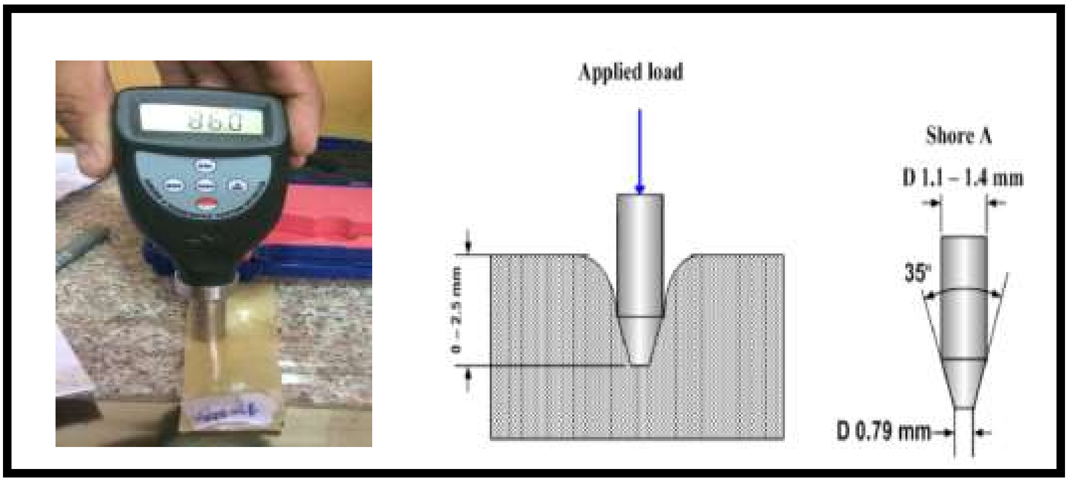


Figure (1): Durometer Hardness instrument of type (Shore A)

Results and Discussion

Alkali, Acid and Salt solution exposure

To outline the main point the weight loss capability of the composite immersed in the three different kind of solution is shown in figures (2, 3, and 4). It can be seen from these figures that weight loss increased with time as well as decreased with increased particle reinforced. The maximum weight loss was found to be 0 wt% epoxy. The least weight loss was in the case 6 wt% charcoal activated – epoxy resin and 6 wt% MgO – epoxy resin owing to hydrophobic nature of MgO and charcoal that repels the water molecules and prevent solution penetration into composite. Furthermore the change in percent weight loss as function of increased percent particles reinforced is shown in Figures (2,3and 4) the specimen have shown decrease in percent weight loss with increase in percent of particles this due to decreased moisture absorption and diffusion coefficient with increased percent of particles .According to Figure 2 and 3, in the first stage, the curve obeys the Fick's law, weight gain went up gradually over time until reached the balance the weight increase of specimen was due to the water absorbed through the new pathway. In the second stage the Percent weight loss of the composite in all three conditions increased due to the interaction between particles and solution (H_2O, HCL and NaOH) which gave the chemical reactions shown in Equations (2) and (3). The weight decrease of specimen was due to the magnesium hydroxide Mg $(OH)_2$ and magnesium chloride $MgCl_2$, which were separated out from the material, besides, dissolved the molecules in the surface of epoxy. And also in the second stage, weight gain in the alkaline solution was higher compared to the Acid solution. It's due to epoxy being sensitive to the alkali environment that might be primary due to the reaction between particles and the alkali substance. Even more the NaCl particles were trapped on the surface of composite, becoming a coating, which played role in separating the MgO and Charcoal from the moisture [12].

Chemical reaction between MgO and NaOH, are explain in Equation (3).moreover, the surface of the particles was damaged, and the pits on the rough surface were filled with water. This could be a new pathway for water absorption. Yet, absorbed water might have adverse effect on the performance of the materials. The moisture will cause progression of delamination between molecules.

$$MgO + H_2O \longrightarrow Mg(OH)_2 \quad \ldots(2)$$

$$MgO + 2\,HCl \longrightarrow MgCl_2 + H_2O \quad \ldots(3)$$

$$MgO + 2\,NaOH \longrightarrow Mg(OH)_2 + Na_2O \quad \ldots(4)$$

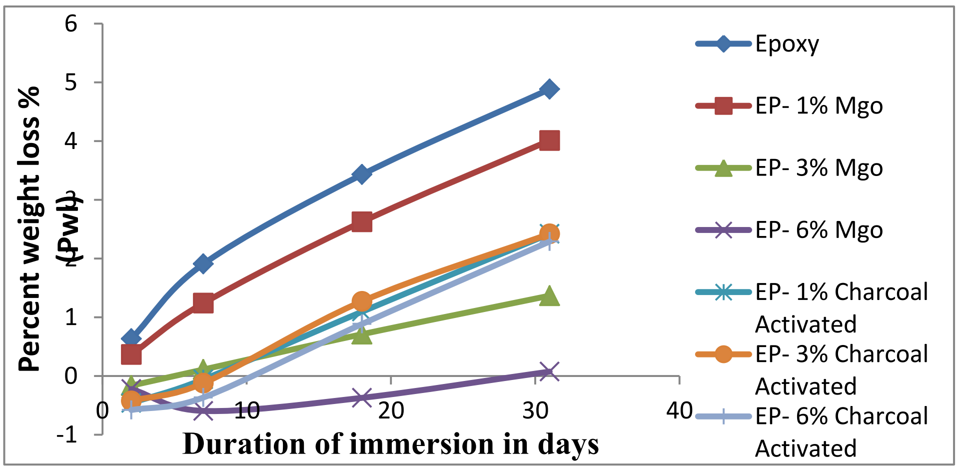
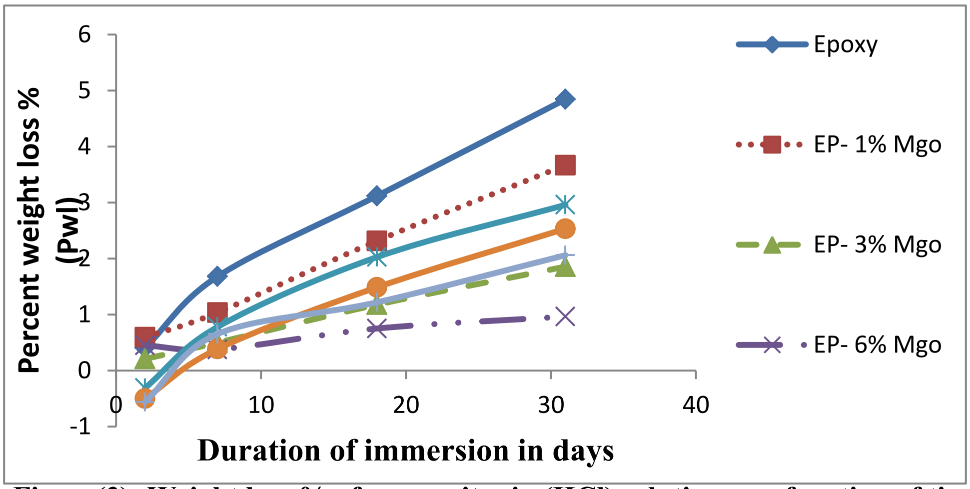
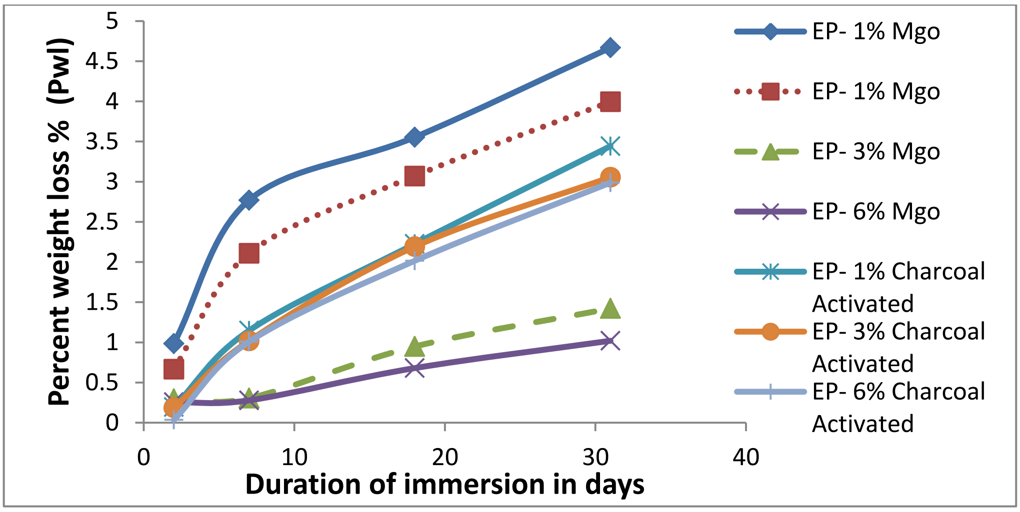


Figure (2):Weight loss % of composites in (NaOH) solution as a function of time



Figure(3): Weight loss % of composites in (HCl) solution as a function of time



Figure(4): Weight loss % of composites in (NaCl) solution as a function of time.

Table (1). weight loss % of composite in HCl solution as function of time

Duration of immersion in days	Pure Epoxy	EP- 1% Charcoal Activated	EP- 3% Charcoal Activated	EP- 6% Charcoal Activated	EP- 1% MgO	EP- 3% MgO	EP- 6% MgO
2	0.4051	-0.3118	-0.499	-0.562	0.594	0.2082	0.45
7	1.68	0.7796	0.3854	0.6533	1.036	0.4979	0.376
18	3.119	2.027	1.48678	1.2189	2.31629	1.177	0.7507
31	4.844	2.96	2.533	2.06	3.668	1.856	0.964

Table (2): weight loss% of composite in NaOH solution as function of time

Duration of immersion in days	Pure Epoxy	EP- 1% Charcoal Activated	EP- 3% Charcoal Activated	EP- 6% Charcoal Activated	EP- 1% MgO	EP- 3% MgO	EP- 6% MgO
2	0.6331	-0.46109	-0.42398	-0.5801	0.3646	-0.1641	-0.2233
7	1.908	-0.0576	-0.12114	-0.36641	1.2399	0.1094	-0.5956

18	3.431	1.0951	1.2719	0.8854	2.6258	0.7111	-0.3723
31	4.885	2.42	2.422	2.29	4.01	1.367	0.074

Table(3). weight loss % of composite in NaCl solution as function of time

Duration of immersion in days	Pure Epoxy	EP- 1% Charcoal Activated	EP- 3% Charcoal Activated	EP- 6% Charcoal Activated	EP- 1% MgO	EP- 3% MgO	EP- 6% MgO
4	0.744	0.194	0.1852	0.0347	0.66592	0.2952	0.255
11	2.77	1.147	1.01883	1.00764	2.108	0.30487	0.27812
18	3.554	2.2253	2.192	2.01528	3.07066	0.948	0.67985
31	4.668	3.44228	3.056	2.98818	3.99556	1.422	1.01977

Hardness Test Result

The hardness of epoxy reinforced by magnesium oxide and charcoal activated particles are shown in figures (5, 6 and 7)**.** In addition, hardness test results are given in Table 4, 5, and 6. Figures and tables show the value of hardness before and after immersion in acid, alkaline and salt solution for different days.

It is noted that all composite materials have significantly higher hardness than the matrix materials; these increase the hardness because the MgO and activated charcoal particles have very high hardness and the strengthening occurs due to the load –carrying of the particles.

In addition to that it is clearly evident that the increased hardness is a function of reinforcement materials content and that the hardness is directly proportional to the reinforcement content. The effect of MgO and activated charcoal content on the hardness is shown in Figures 5,6 and 7 .These figures indicate that the hardness increases at almost constant rate with increase in reinforcement materials content.

From tables 4, 5, and 6 and Figures 5, 6 and 7 show the hardness of all specimen decreased with increased the time of immersion in solutions. These results are because of solution penetration inside epoxy decreasing the connection between molecules of epoxy and then softening of manufactured material. Composite materials reinforced with activated charcoal had porosities which allowed for solution molecules to penetrate insides the materials and acting along the interface between epoxy and activated charcoal causing swells in the sample. Then the bonds between resin and particles will break.

Hardness of composite in the acid solution was higher compared to the alkali solution. It's due to epoxy is sensitive to the alkaline environment, that might be primary due to the reaction between particles and the alkali substance. Alkaline environment aging of polymers may create damage in the form of crazing, micro cracking and other types of morphology changes, thus allowing additional moisture absorption to occur [13]. Even more the NaCl particles were trapped on the surface of composite, becoming a coating, which played role in separating the MgO and Charcoal from the moisture [14].

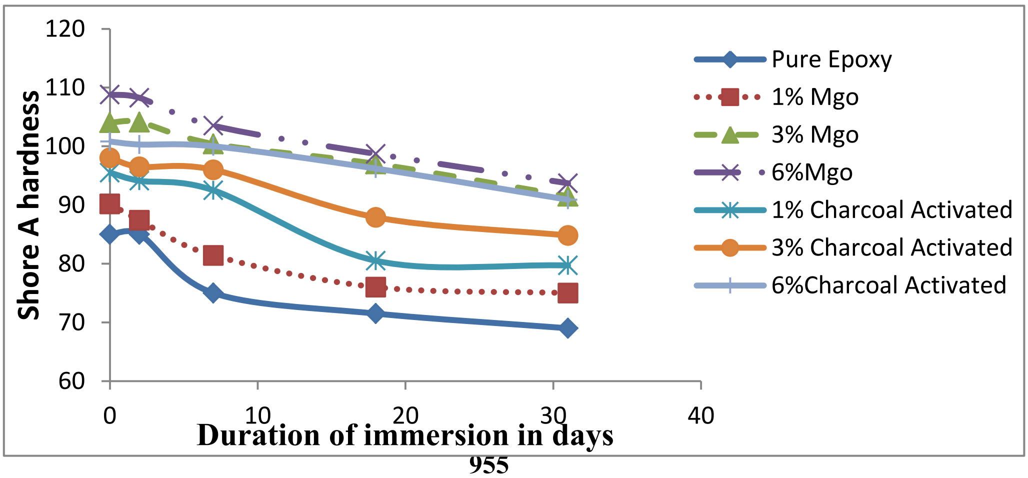


Figure (5): Shore A hardness of composite in HCl solution as a function of time.

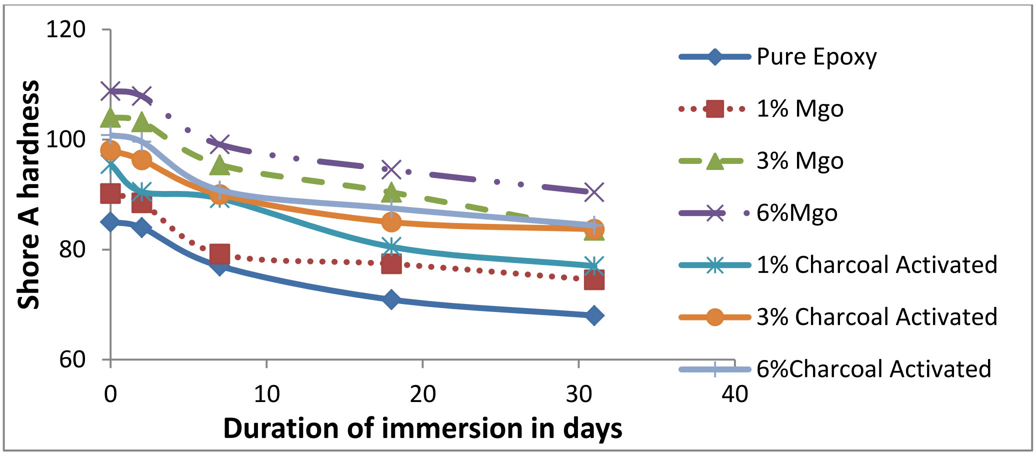


Figure (6): Shore A hardness of composite in NaOH solution as a function of time.

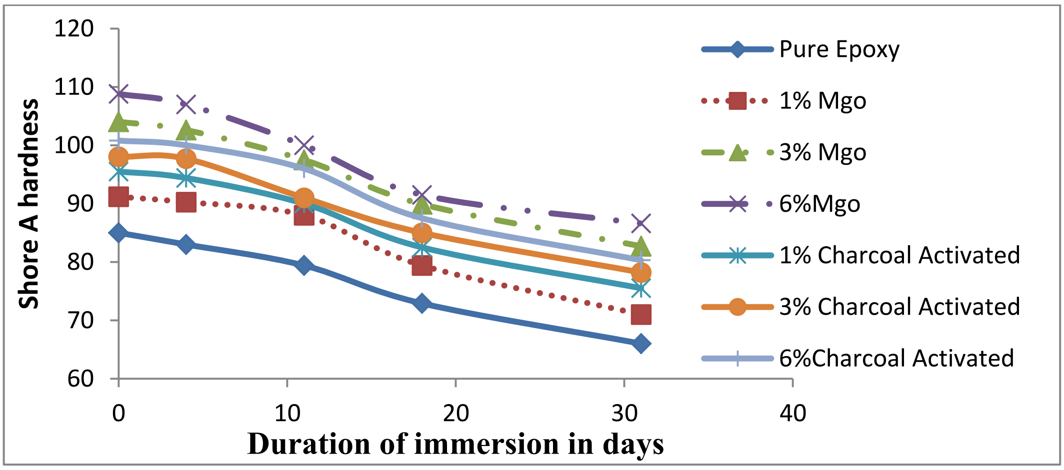


Figure (7): Shore A hardness of composite in NaCl solution as a function on time.

days	Pure Epoxy	1% Mgo	3% Mgo	6%Mgo	1% Charcoal Activated	3% Charcoal Activated	6%Charcoal Activated
0	85	90.18	104.03	108.8	95.5	98.03	100.8
2	85	87.4	104.1	108.25	94.12	96.5	99.6
7	71.5	81.4	100.4	103.1	92.5	96	100
18	72.0	76	97	98.7	80.5	87.9	96.2
31	69	75	91.5	93.7	79.7	84.8	90.9

Table (4). Shore A hardness of composite in HCl solution as function of time

Table(5). Shore A hardness of composite in NaOH solution as function of time

days	Epoxy	1% Mgo	3% Mgo	6%Mgo	1% Charcoal Activated	3% Charcoal Activated	6%Charcoal Activated
0	85	91.18	104.03	108.8	95.5	98.03	100.8
4	83	90.26	102.6	107	94.4	97.7	100
11	76.4	88	97.5	100	90	91	96

18	72.9	79.4	89.9	91.5	82.5	85	87.5
31	66	71	82.7	86.6	75.5	78.2	80.3

Table (6). Shore A hardness of composite in NaCl solution as function of time

days	Pure Epoxy	1% Mgo	3% Mgo	6%Mgo	1% Charcoal Activated	3% Charcoal Activated	6%Charcoal Activated
0	85	90.18	104.03	108.8	95.5	98.03	100.8
2	84.7	88.5	103.2	107.9	90.5	96.3	99.6
7	77	79.2	95.4	99.1	89.3	90	90.8
18	70.9	77.4	90.4	94.5	80.5	85	87.5
31	68	74.5	83.5	90.4	77	83.7	84.4

CONCLUSIONS

The following conclusions can be drawn from the present work:

1. Results of hardness test before immersion show that the samples of 6% MgO and 6% charcoal activated have the highest values of shore hardness (A) were 108.8 and 100.8 respectively .In addition, the result exhibit an increasing in hardness with increasing in particle concentration. This increase of hardness is because the particles have very high hardness and strength due to the load occurring of the particles.
2. Results of Percent weight loss % (pwl) shows that the highest values of Percent weight loss appear for pure epoxy when immersed in NaOH (4.885%) while lower values appear for 6% MgO when immersed in NaOH (0.074%). Furthermore, 6wt% MgO – epoxy resin has highest chemical resistance than 6 wt% charcoal – epoxy resin when immersed in chemicals solustion.

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