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Mechanical and Thermal Stability of Epoxy – TEOS Hybrids Materials using Sol-Gel Method

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Abstract

Sol-gel method is the simplest method and has to preparation of nano particles, nano fibers, and nano flaks. The preparation of hybrid epoxy thermosets enables their improvement in characteristics such as mechanical properties (bending,and hardness), thermal properties. The effect of water absorption on bending and hardness test for tetraethylorthosilicate (TEOS) and epoxy hybrid have been investigated. TEOS with (0%,2%,4%,6%and 8%) volume percentage mixed with epoxy to prepared sheets of hybrid, these sheets cut as samples for ASTM water absorption, bending and hardness tests.
The results show that, adding TEOS to epoxy will increase absorbance of water. bending test shows that there are increase in values of bending stress with increasing TEOS but after immersion in water all the values for bending stress will reduce. Hardnessvalues at least for the samples immersion in water from dry samples, occur the diffusion of molecules water in the polymer and hybrids leads to space molecular chains for each other and decrease the hardness values. Thermal stability of hybrids increase with increasing TEOS ratio, the glass and decomposition temperature were shift compare with epoxy pure.

Keywords: TEOS, Epoxy, water absorption, three point bending, hardness test, DTA analysis

الخلاصة

أستخدمت طريقة كيميائية هي طريقة الصل–جل وهي ابسط الطرق في تحضير جسيمات والياف ومتراكبات نانوية ، بعد تحضير المتراكب الهجين مع الايبوكسي لتحسين الخصائص الميكانيكية والخصائص الحرارية ودراسة تأثير امتصاص الماء على اختبار الانحناء والصلادة للمتراكب الهجيني TEOS مع الايبوكسي والايبوكسي المحضر بشكل متراكب هجيني وتقطيعه حسب المواصفات الامريكية لفحص امتصاص الماء والانحناء ثلاثي الابعاد والصلادة .

Introduction

New materials, organic and inorganic components can interpenetrate each other in scales ranging from a few micrometers to a few nanometers. In general, organic and inorganic structures are covalently bonded, more or less strongly [1]. Epoxy composites, which are hybrid organic-inorganic materials, have a wide range of engineering applications that require a high strength to weight ratio, low cost and ease of fabrication [2].this reduces the stable lifetime of these materials. Experiment the water diffuse in epoxy in two states, evenly distributed water molecules between the polymer chain and condensed water in fractures and cavities [3].An epoxy is a copolymer formed from two different chemicals referred as the resin and the hardener. the resin consists of monomers or short chains [4]. Hybrid organic/inorganic nanomaterials have attracted a great deal of attention in the field of polymer research as well as in industrial applications because their advanced properties like abrasion and scratch resistance, toughness, mechanical properties, thermal stability to the formation of the inorganic particles in the polymer matrix, while keeping transparency [5]. Sol-gel method is a low temperature processing technique that is used for the synthesis of the ceramic materials. A hybrid organic/inorganic has been thought of in order to achieve a homogeneous combination of both materials or somehow control their properties [6]. Sol-gel method products many shape different have various applications, such as thin films, protective coating, decorative coating, electro-optic components, composite, and hybrid materials [7, 8].

Mustafa *et al.* [9] studied the effect water absorption on wear test with epoxy / silica of sol-gel method and show the water absorption was found to be highly effective on the wear behavior of hybrids. Anteret *et al.* [10] studied effect of water absorption on hardness test for epoxy reinforced with glass fibers and show the values of hardness (shore D) at room temperature decreases with increasing the time of immersion in water. Water or any other fluid diffusion in a solid material is described by fick's two laws. Fick's first law states that in the steady state condition, the flux of water J(g/mm^2) through a solid is proportional to the gradient of the water concentration (g/mm^3) [11].

$$J = -D\frac{\partial \phi}{\partial x} \quad (1)$$

Φ= water concentration in the material, D=diffusion coefficient (mm^2/s). Fick's second law is given by:

$$\frac{\partial \phi}{\partial t} = D\frac{\partial 2y}{\partial x2} \quad (2)$$

Weight gain of the material, given by the formula below:

$$M=M(t)= \frac{W-Wd}{Wd} * 100 \quad (3)$$

Where W= weight of the sample at any given time,W_d= weight of the dry material, By putting $W=W_d+m$, and rearranging the terms we get:

$$M=G(M_m-M_i)+M_i \quad (4)$$

Where,M_i =initial moisture content,
M_m= moisture content at full saturation [11].

The aim of the work is study the effect of water absorption on some mechanical properties (bending and hardness) for inorganic (TEOS) with organic (Epoxy) with different ratio and study thermal stability of hybrids materials.

Materials and Methodologies

***Materials*:** TEOS was a silica precursor was produce from sigma-aldrich. Ethanol (EtOH), GCC/Gainland chemical company, C_2H_5OH Mw=46.07g/mol ,density= 0.785, purity= 99.9%. H_2ODeionized Water (H_2O), Mw=18g/mol high degree of purity/empty of additional ions. Epoxy (105) supplied from (Don), Amman–Jordan. The ratio of hardener to epoxy used in this study was approximately 3:1.. Hydrochloric Acid (HCl), BDH, Mw= 36.46 g/mol, density= 1.19.

***Preparation of Epoxy–Silica Hybrid Materials*:** Sol-gel method was used to prepare silica network. TEOS (5ml) and ethanol (5ml) and $H_2O$with HCL (PH=1) were stirred at 50 °C until it become homogenous solution. The composition of the matrix materials is epoxy and hardener the ratio (3:1). To add the alkoxysilane solution to the epoxy and stirred solution in a glass tube was prepared using different ratio (0, 2, 4, 6, 8) %.

Water Absorption Test:-The specimens were placed in a close tube containing water maintained at a temperature of approximately 24°C and the samples cut as ASTM D 570-98, in the beginning every two hours and after several days become every 24 hours, each specimen is removed from the water, wiped with a dry cloth free of surface moisture, and weighted. This procedure was repeated consecutively for 45 days until the composites were substantially saturated. The percentage water absorption was calculated by the difference between the weights of fully saturated specimens to the weight of dry specimens using the following equation (3).

***Bending Test*:** A three-point bending test was conducted according to ASTM D-790 of testing polymeric composites, the test was conducted in a direction perpendicular to the material. A test specimen was positioned on the surfaces of two cylindrical supports according to the span length, and the loading nose was aligned such that the axes of the cylindrical

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supports are parallel to each other and the loading nose is midway between the supports. A crosshead speed is 1mm/min.

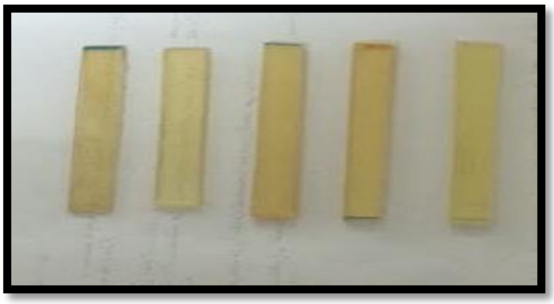

Figure 1: Samples of bending test

Hardness Test**:** Shore D hardness, the indenter was attached to a digital scale that is graduated from 0 to 100 units.

Differential Thermal Analysis (DTA)**:** Thermal analysis using a reference sample material (sample) is heated in one furnace. The difference of the temperature is recorded during programmed heating and cooling cycles

Results and discussion

Water absorption: The water absorption depends on many parameters like polarity and the structure for materials [12]. Figure (2) shows that pure epoxy has lower absorbance for water ,Molecular chains containing in epoxy the polarized groups help to movement and diffusion liquid polarized molecules (water). The process of water penetration in the epoxy is the effect of the presence of polarization between the polymeric chains, where water molecules diffusion between the polymeric chains until the state of saturation. When add to TEOS with epoxy and after the polymerization process will from a network of silica and increase these percentages will increase the weight gain of water absorption because the hydrophilic nature and porosity of silica structure which agree with Mustafa *et al.* [9].

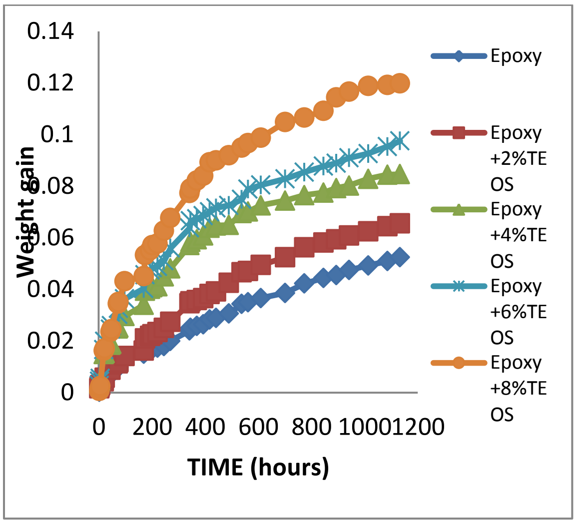

Figure 2: Water absorption as a function of time

Bending test**:** when adding TEOS after the polymerization process will from a network of silica with epoxy, the interpenetrating network with each other and that the of overlap between organic- inorganic causing obstruction movement these chains when it loaded straining vertical, leading to increase bending stress at ratio TEOS, it will lead to the formation of a network silica and this in turn wills pace the home chains of epoxy and reduce the density of penetrating network. When immersion the samples with water, occur the diffusion of molecules water in the polymer and hybrids leads to space molecular chains for each other and decrease the bending stress[13] shows in Fig (3,4).

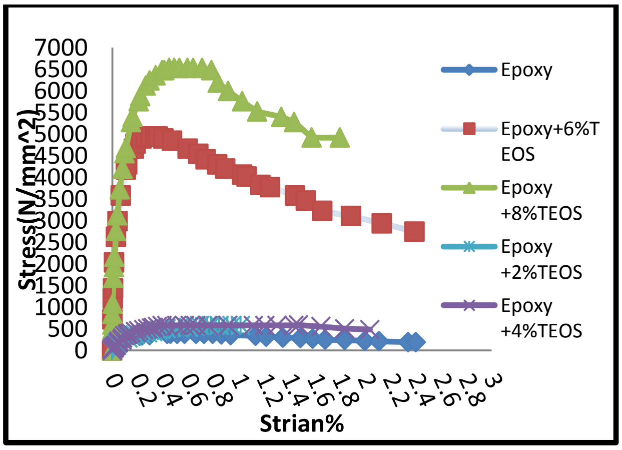

Figure 3: Stress-strain curve before immersion water

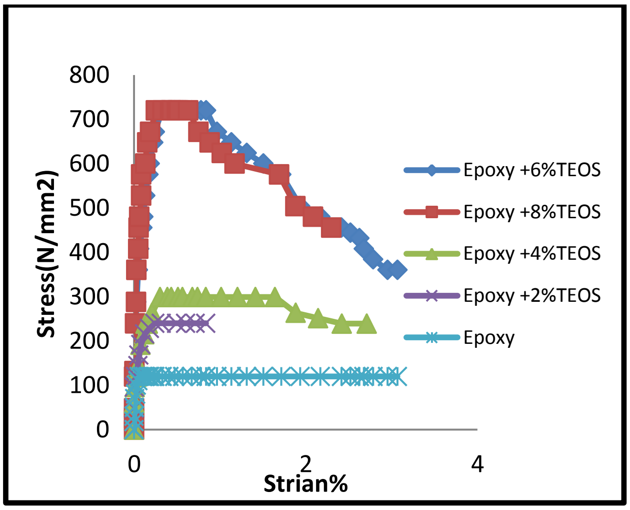

Figure 4: Stress- strain curve after immersion water

***Hardness test*:** Table (1) listed values for hardness, epoxy has value 61 because the indented will penetrate or moving the chains easily. The silica network will interpenetrated with epoxy chains, that mean the density of chain will increase so that when the indenter put on the surface for hybrids material the interpenetrating chains will resist the force. which act on indenter so that the hardness values will increase immersion the samples in water decrease the values of hardness because the molecules of water cause more spacing chains molecular and reduced resistance indenter.

Table (1) the values of hardness of dry samples and after immersion water

samples	Dry samples	After immersion water
Epoxy	61	58
Epoxy+2% TEOS	66	62
Epoxy+4% TEOS	74	70
Epoxy+6% TEOS	77	74
Epoxy +8%TEOS	77	73

***Differential Thermal Analysis (DTA)*:** All the degradation curves of the samples showed a similar shape, with two peaks the first peak the glass temperature and the second peak of the decomposition temperature that appear in Figure(5) in the mixed between Epoxy and TEOS consists the network between Epoxy and TEOS overlap between the chains to strong bonding leading to higher thermal stability of the final output, the glass and decomposition temperature was shift which agree with result the Figure (5 a, b, c, d, and e) and Table (2)

Table (2) values of glass and decomposition temperature

Samples	Tg (glass temp.)	Td (decomposition temp.)
epoxy	98	160
2% TEOS	100	168
4% TEOS	112	178
6%TEOS	118	182
8%TEOS	120	185

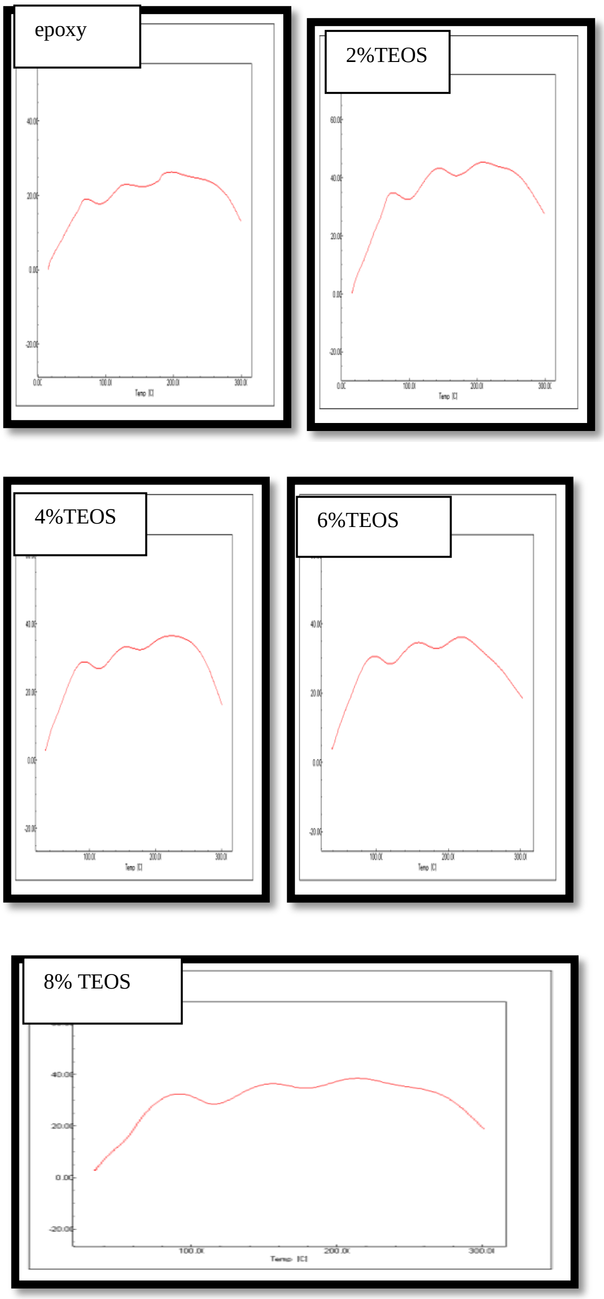

Figure 5: Curves of Differential Thermal Analysis

Conclusions

Weight gain of hybrid materials increases with the increasing of silica network ratio because the hydrophilic nature and porosity of silica structure. Bending stress and shore D hardness values increase with increasing TEOS ratio interpenetrating network between organic- inorganic causing obstruction movements these molecular chains. The glass and decomposition temp shift with ratio of TEOS due to the thermal stability increase with increasing ratio of TEOS.

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