

The Effect of Some Fillers on the Residue Weight of Novolac-Epoxy Hybride Composites

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KEYWORDS :

- Epoxy polymer (EP)
- Volume ratio (\emptyset)
- Gain Weight (ΔW)

ABSTRACT

In this work a hybrid composite materials were prepared containing matrix of polymer blend (Novolac 70% + Epoxy 30%) reinforced by different reinforcing materials (Alumina Powder + Moissanite Powder (SiC) + Asbestos short fiber) with values of volume ratio (35) %. The hybrid composite materials prepared are:

C1 = Blend + Al_2O_3 + A_s (35) %, C2 = Blend + SiC + A_s (35) % and

C3 = Blend + Al_2O_3 + SiC + A_s (35) %. All samples related to thermal tests were prepared by hand lay-up process. Thermal tests: thermogravimetric analysis (TGA). Thermal experimentations results were in favor of the samples (C3) with an obvious weakness of the samples (C1) and a decrease of these properties with a rise in temperature. These thermal properties of the samples which contain the mixture (Alumina Powder + Moissanite Powder + Asbestos short fiber) (C3) were of lower residue weight which is good heat isolation and fire resistance. For the (C1) samples there was high tendency to lose weight with high temperature and less as to the samples (C3).

The aim of the research is to know the resistance of the material to the amount of loss of weight with high temperatures and in any industrial application can be used.

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تأثير بعض الحشوات على فقدان الوزن الحاصل في المادة المترابطة الهجينة نوفولاك-إيبوكسي

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الكلمات المفتاحية:

- راتنج الايبوكسي (EP)
- النسبة الوزنية (\emptyset)
- الريح بالوزن (ΔW)

الخلاصة

تم في هذا البحث تحضير مواد مترابطة دقائق هجينة تحتوي على مادة اساس واحدة هي الخليط البوليمري المكون من (نوفولاك ٧٠ % + ايبوكسي ٣٠ %) مقواة بأنواع مختلفة من مواد التقوية الدقائقية (مسحوق الالومينا + مسحوق كاربيد السيليكون + الياق الاسبيستوس المقطعة) علما ان الكسر الحجمي لجميع المواد المترابطة الهجينة كان بمقدار ٣٥ % . المواد المترابطة الهجينة التي تم تحضيرها هي C3 = Blend + Al_2O_3 + SiC + A_s (35) % , C1 = Blend + Al_2O_3 + A_s (35) % , C2 = Blend + SiC + A_s (35) %

ان جميع العينات المستخدمة في الاختبار الحراري (فقدان الوزن) كانت قد حضرت بأستخدام طريقة القولبة اليدوية . الاختبار الحراري الذي تم اجراءه هو اختبار التحلل الحراري الوزني . اوضحت النتائج للاختبارات الحرارية ان العينة C3 ابدت قابلية فقدان بالوزن قليلة مع ارتفاع درجات الحرارة وهذا على العكس من العينات C1 التي ابدت قابلية على فقدان في الوزن بصورة اكبر من باقي العينات ويعود سبب فقدان الوزن بالنسبة الى العينات C3 الاقل مقارنة مع باقي العينات الى عملية التجانس الحاصل ما بين مواد التقوية الثلاث مجتمعة مما يدل على ان المادة C3 هي من المواد المترابطة المقاومة للحرق . ان الهدف من البحث هو معرفة مقاومة المادة الى مقدار فقدان بالوزن مع ارتفاع درجات الحرارة وفي اي تطبيق صناعي يمكن استخدامها .

1. Introduction

Composites are quite common today and are used in nearly every segment of civilian and military industry. The idea of reinforcement is not new. Over the centuries, natural fibers, such as grass or animal hair, have been used to improve the strength and to lessen shrinking of pottery prior to firing and increase the strength in mud houses. This idea in the present form has been exploited with the development of glass, carbon and later of aramid fibers [1].

Thermal Effect (Ablation test)

Thermal degradation is the scission of molecular chain at elevated temperatures, so some rubbers undergoes chemical reactions in which gaseous species are produced. These reactions are evidenced by a weight loss of material, polymers thermal stability is a measure of its resilience to this decomposition. Thermal stability is related primarily to the magnitude of the bonding energies between the various atomic constituents of the polymer.

Plastics can be protected from thermal degradation by incorporating stabilizers into them.

Fire performance characteristics of polymeric composites are dependent on the chemical nature and amount of the resin matrix used (resin) however needs extra attention. In general, resins are made fire retardant, by the use of additives and fillers like for example aluminum nitride. Fig. (1) explains the concepts of the theory for polymeric material and its composites after exposure to high temperature, which is classified into three regimes [2]:

1. Carbonized zone.
2. Pyrolyzed zone.
3. Virgin base material

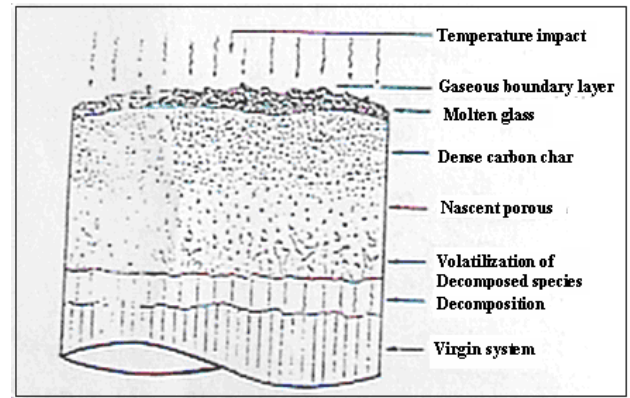


Fig (1): the ablation for elastic polymer reinforced with the particles [3].

The fire resistance of a polymer can be increased by adding fire retardant fillers. The degree of flammability of a polymer is a function of [3]:

- 1-Matrix type and amount (dominant)
- 2- Quantity of fire-retardant additives. (It is common practice to add this to hard polymers)
- 3-Type and amount of reinforcement (if any). (e.g. adding a lot of particles helps to improve fire resistance in hard polymers such as resin and polyester).

Fillers (particulate) improve thermal and thermal-oxidative stability of polymers by the formation of thermally stable chemical bonds between polymer and the filler. It is believed that formation of cross-linking bonds and of a three dimensional polymer-filler network at elevated temperatures increases the thermal stability of the polymer. Besides, chemically active filler may promote the decomposition of thermally unstable groups present in the polymer to form more thermally stable bonds or these filler may play an important role in retarding the kinetics of radical decomposition. Also, relatively higher thermal conductivity and heat capacity of filler is supposed to dissipate the heat energy, and improve the thermal stability of the polymer-filler composite [4].

The degree of the thermal erosion rate is determined by the following relation: [3]

$$E_r = x/t_r \quad \dots\dots\dots (1)$$

Where, E_r is erosion rate (mm/sec), x is specimen's thickness; t_r is the burning time (sec).

2. Materials

2.1 Phenol Formaldehyde polymer (novolac type)

Phenol Formaldehyde (novolac type) is most widely utilized since it is cheap polymer resin. This matrix material is used primarily with carbon fiber, glass fiber composites, and alumina, silica powder.

Commercial phenolic resins provided from Iran novolac type, mixed with weight fraction of (12-14) % hexamethylenetetramine (H.M.T.A) of yellow color powder of density 0.921 g/cm^3 are used.

2.2 Epoxy polymer (EP)

Epoxy resin (type Conbextra EP10) was used in this research; it is a liquid with moderate viscosity and capable to be converted to solid state by adding the solution (Metaphenylene Diamine, MPDA) as hardener. This hardener is a light liquid with yellowish color, the ratio of this hardener to the epoxy is about (1:3). This resin also has applicable technical specification such as, high adhesion to fibers and low shrinkage during solidification.

2.3 Asbestos short fibers

Chrysotile known as white asbestos was used, Chrysotile is hydrated silicates are found in certain types of rocks, known for its snake-like, curly appearance, soft, flexible, strong, durable, and resistant to heat and fire, its density is 2.4 g/cm^3 [5].

2.4 Alumina particles

A white powder Al_2O_3 of density (3.89 g/cm^3) and of particle size less than 250μ was used. It is useful at high temperature and has a high dielectric strength, excellent electrical resistance [5].

2.5 Silica carbide particles

After carbide, silicon is the most plentiful element on the earth's crust. It occurs as its carbide either free or combined with metallic oxides as silicates.

Silica crystallizes in different forms at different temperatures, but as the changes are slow, the unstable form occurs naturally, as well as the far more common stable form which is γ -phase [5].

Silica carbide materials are basic fundamental materials for much of the ceramic application. Silica carbide is a raw materials, which provide strength to the dried and fired wares [5]. It is widely used because it is inexpensive, hard, chemically stable and relatively infusible.

2.6 Preparation steps for composites materials

1 - The novolac was mixed with methanol (1/2 weight of solvent to novolac) [5].

2 - The novolac liquid mixed with (H.M.T.A) hardener (12-14) % powder [5].

3 - Epoxy resin mixed with (33) % hardener.

4 - The mixture in the step (2) mixed with the mixture in the step (3).

(70% Novolac + 30% Epoxy) in order to prepare the polymer blend (Inter penetrating polymer network) [5].

5 - The polymer blend in the step (4) reinforced by different types of particles (Al_2O_3 , SiC) and asbestos fibers with values of volume fraction (35) %.

6 - Three hybrids composites materials prepared:

C1 = Blend + Al_2O_3 + As (35) %

C2 = Blend + SiC + As (35) %

C3 = Blend + Al_2O_3 + SiC + As (35) %

7 - For all cases, this was calculated by applying the relationship [5]:

1

$$\Phi = \frac{1}{1 + ((1 - \psi) / \psi) \times (\rho_f / \rho_m)} \dots (2)$$

Where Φ , ψ are the volume and weight ratio of the reinforcements respectively.

ρ_f , ρ_m are the density of reinforcements and matrix respectively.

The density of the prepared hybrids was determined from the equation:

$$\rho_m = x_1 \rho_1 + x_2 \rho_2 \dots \dots \dots (\text{rule of mixtures}) [5]$$

Where ρ_m : the density of the matrix (polymer blend).

ρ_1 , ρ_2 : the density of the first polymer and the second respectively.

x_1 , x_2 : the percentages of the first polymer and the second respectively.

Thermogravimetric Analysis (TGA)

This analysis shows how the prepared hybrid behaves as temperature increases from low to high temperature gradually. The thermal oxidative degradation pathways are studied using computerized digital oven Model (Gallenham Program Rapid), and the specimens (of weight 100 gm) were put in a porcelain vessel and placed in oven at heating rate 50°C/min in air to various temperature from 25 up to 500 °C. The temperature was measured with a sensitive ballistic system Model (four digits), as the temperature increased the weight, loss occurred.

3. Thermo gravimetric Analysis (TGA) Test

Figures (2) and represent the relationship between increasing the temperature and the residue weight for hybrids composites materials and from this figure one can conclude the following results:

1 – C1 has high ability to residue weight with increasing temperature while C3 has low

ability to residue weight with increasing temperature.

2– Specimens that contain only (SiC) give (Residue weight) lower than the (Residue weight) of the Specimens that contain only (Al₂O₃).

$$C1 > C2.$$

3 – Specimens that contain both (Al₂O₃ + SiC) give (Residue weight) lower than the Specimens that contain only (Al₂O₃) or Specimens that contain only (SiC).

$$C1 > C3, C2 > C3.$$

The polymeric material undergoes many changes when it is heated gradually from low to high temperature at constant rate, during which the material emits gases and liquid, changes occur in shape, color and molecular weight. The ability of polymer to resist these changes at high temperature is called thermal stability [6].

Therefore TGA measurement is employed to study the thermal- oxidative degradation of prepared hybrids in air. Fig. (2) and Table (1) represent the TGA curves for all samples. These figures show three stages for pyrolysis degradation.

1 – First stage is at temperature range from room temperature up to 300 °C during this interval hybrids releases little gaseous components, therefore the loss in weight in this stage is very small [7].

2 – Second stage is at temperature range from (200-350) °C, heating causes the release vapors of water, CO, phenol. And weight loss increases at rate more than that at first stage, in this stage thermal degradation occurs with less degradation in density with out shrinkage [8]. This stage also includes broken of molecular chain of polymer.

3 - Third stage is at temperature range from 350 °C and so on, in this stage there is shrinkage with large decrease in weight, heating in this

stage causes the release of phenol, H_2O , CH_4 , CO_2 , Benzene [9].

Results also show that the thermal stability increases in (C3) compared with other samples, depending on the type and loading ratio of the reinforcements in the hybrids composites. As the loading ratio of (Al_2O_3 + SiC) increases the hybrids become more stable compared with hybrids as loading ratio of (SiC) or Al_2O_3 increases as shown in Fig (2) .

4. Conclusions

This work has reached to the following conclusions: Specimens that contain both (Al_2O_3 + SiC) give (Residue weight) lower than the Specimens that contain only (Al_2O_3) or Specimens that contain only (SiC) . Specimens that contain only (SiC) give (Residue weight)

lower than the (Residue weight) of the Specimens that contain only (Al_2O_3).

3 – C1 has high ability to residue weight with increasing temperature while C3 has low ability to residue weight with increasing temperature

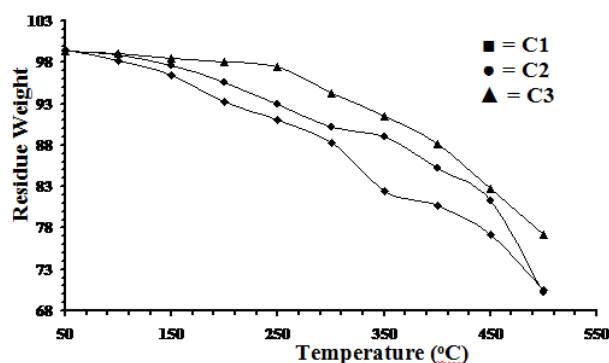


Figure (2): Show the values of residue weight for prepared hybrids composites as elevated temperature in TGA test.

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