

## Study some thermal properties for hybrid composite reinforced with particales

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**Dr. Ali Hassan.R.H Al-Azzawi**

Email: Dr.alhathal@yahoo.com

Faculty of education, University of mustansiriyah , Baghdad.

**Dr. Balkees M. D.AL-Dabbagh**

Department of Applied Science, University of Technology, Baghdad.

**Dr.Sana Ali .j**

Faculty of education, University of mustansiriyah , Baghdad.

### Abstract

In this work a hybrid composite materials were prepared containing matrix of polymer blend (Novolac 80% + Epoxy 20%) reinforced by different reinforcing materials (Alumina Powder (type  $\alpha$ ) + Silica Powder + Asbestos short fiber) with two values of volume fraction (30, 40) %.

### The hybrid composite materials prepared are:

- $H_1 = \text{Blend} + \text{Al}_2\text{O}_3(\alpha) + \text{AS} (30) \%$
- $H_2 = \text{Blend} + \text{SiO}_2 + \text{AS} (30) \%$
- $H_3 = \text{Blend} + \text{Al}_2\text{O}_3(\alpha) + \text{SiO}_2 + \text{AS} (30) \%$
- $H_4 = \text{Blend} + \text{Al}_2\text{O}_3(\alpha) + \text{AS} (40) \%$
- $H_5 = \text{Blend} + \text{SiO}_2 + \text{AS} (40) \%$
- $H_6 = \text{Blend} + \text{Al}_2\text{O}_3(\alpha) + \text{SiO}_2 + \text{AS} (40) \%$

All samples related to mechanical, thermal, electrical and physical tests were prepared by hand lay up process. The tests can be classified into four groups: For the ( $H_1$ ) samples, there was high tendency to lose weight with high temperature and less as to the samples ( $H_6$ ).

**Keyword:** Thermogravimetric analysis (TGA), Alumina ( $\text{Al}_2\text{O}_3$ ), Silica ( $\text{SiO}_2$ ), asbestos short fiber (As).

### دراسة بعض الخواص الحرارية لمتراكبات هجينة مقواة بالدقائق

#### الخلاصة

تم في هذا البحث تحضير مواد متراكبة هجينة مكونة من مادة أساس بوليميرية هي عبارة عن خليط بوليميري (Novolac 80% + Epoxy 20%) ومقواة بأنواع مختلفة من مواد التقوية (مسحوق الألومينا نوع الفا + مسحوق السيليكا + ألياف الاسبستوس المقطعة العشوائية) وبكسرين حجمين (30%, 40%).

المواد المتراكبة الهجينة التي تم تحضيرها هي :

- عينات مدعمة بالألومينا والاسبستوس وبكسر حجمي 30% ورمزت ( $H_1$ ).
- عينات مدعمة بالسيليكا والاسبستوس وبكسر حجمي 30% ورمزت ( $H_2$ ).
- عينات مدعمة بالألومينا، بالسيليكا والاسبستوس وبكسر حجمي 30% ورمزت ( $H_3$ ).

- عينات مدعمة بالالومينا والاسيستوس وبكسر حجمي 40% ورمزت (H<sub>4</sub>) .
  - عينات مدعمة بالسيليكا والاسيستوس وبكسر حجمي 40% ورمزت (H<sub>5</sub>) .
  - عينات مدعمة بالالومينا ،السيليكا والاسيستوس وبكسر حجمي 40% ورمزت (H<sub>6</sub>) .
- تم استخدام طريقة القولة اليدوية في تحضير العينات المستخدمة في الاختبار للعينة H<sub>1</sub> قابلية على فقدان في الوزن عند درجات الحرارة العالية مقارنة مع العينة H<sub>6</sub> .
- الكلمات المفتاحية :** التحلل الحراري التفاضلي ، مسحوق الالومينا ، مسحوق السيليكا، اليف الاسيستوس القصيرة .

## INTRODECTION

Composite materials 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 [1,2].

In 2006, Das [3] prepared novolac composites from Bamboo strips, using bamboo strips that were treat with varying concentrations of sodium hydroxide solution. Mechanical properties of various composites (flexural modulus, toughness, tensile strength, and elastic modulus) were determined. The physical characteristics, such as the wetting ability of the alkali treated reinforcements, were increas because of alkali treatment, with increasing concentrations of alkali. The mechanical properties were increas with increasing mercerizing strength. Maximum improvement in properties was achiev with 16-20% of caustic treated reinforcements.

## Matreiales

### Phenol Formaldehyde Resin (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 (Bazerkane), mixed with weight fraction of (11-13) % hexamethelitramine (HMTA) of yellow color powder of density 0.91g/cm<sup>3</sup> are used.

### Epoxy Resin (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.

## Asbestos

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<sup>3</sup> [4,5] .

**Alumina powder**

A white powder Al<sub>2</sub>O<sub>3</sub> of density (3.89) g/cm<sup>3</sup>. It is useful at high temperature and has a high dielectric strength, excellent electrical resistance [6, 7].

**Silica powder**

After oxygen, silicon is the most plentiful element on the earth's crust. It occurs as its oxide 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 occur naturally, [8, 9].

Silicate materials are basic raw materials for much of the ceramic industry. Silica is non – plastic raw materials, which provide strength to the dried and fired wares [10, 11]. It is widely used because it is inexpensive, hard, chemically stable and relatively infusible.

**Preparation methods for hybrids composites materials**

- 1 - The novolac was mixed with methanol (1/2 weight of solvent to novolac) [12].
- 2 – The novolac liquid mixed with (HMTA) hardener (11-13) % powder [13].
- 3 - Epoxy resin mixed with (33) % hardener.
- 4 – The mixture in the step (2) mixed with the mixture in the step (3). (80% Novolac + 20% Epoxy) in order to prepare the polymer blend (Inter penetrating polymer net work) [14].
- 5 – The polymer blend in the step (4) reinforced by different types of particles (Al<sub>2</sub>O<sub>3</sub>, SiO<sub>2</sub>) and asbestos fibers with two values of volume fraction (30, 40) %.
- 6 – Six hybrids composites materials prepared:  
 H<sub>1</sub> = Blend + Al<sub>2</sub>O<sub>3</sub> + As (30) %  
 H<sub>2</sub> = Blend + SiO<sub>2</sub> + As (30) %  
 H<sub>3</sub> = Blend + Al<sub>2</sub>O<sub>3</sub> + SiO<sub>2</sub>+ As (30) %  
 H<sub>4</sub> = Blend + Al<sub>2</sub>O<sub>3</sub> + As (40) %  
 H<sub>5</sub> = Blend + SiO<sub>2</sub> + As(40) %  
 H<sub>6</sub> = Blend + Al<sub>2</sub>O<sub>3</sub> + SiO<sub>2</sub>+ As (40) %
- 7 – For all cases, this was calculatby applying the relation ship:

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

Where:

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

ρ<sub>f</sub>, ρ<sub>m</sub> are the density of reinforcements and matrix respectively.

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

$$\rho_m = x_1\rho_1 + x_2\rho_2 \dots\dots\dots \text{(Rule of mixtures)}$$

Where ρ<sub>m</sub> : the density of the matrix (polymer blend).

ρ<sub>1</sub>, ρ<sub>2</sub> : the density of the first polymer and the second respectively.

x<sub>1</sub>, x<sub>2</sub> : the percentages of the first polymer and the second respectively.

8 – The metal mould was clean and used for casting the sheet of hybrids composite material.

9 – The fablon was fix on the inner mould faces before casting to facilitate the releasing of casting hybrids and having smooth faces.

10 – Cover plate, with identical dimension of the mould face, was used to apply appropriate load on the casting sheet for releasing voids, bubbles, to have a specified thickness and smooth face.

11 – Casting sheet was leftn inside the mould at room temperature about (24h).

12 – After solidification, the casting sheets were released from the mould and placed in an oven with (50°C setting temperature) for (3h) to post cure the considered sheets.

13 –The testing samples were obtained by cutting the casting sheets.

### Themogravimetric 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 (Gallenhamp 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 1000 ° C. The temperature was measur with a sensitive ballistic system Model (four digits), as the temperature increased the weight, loss occurred. .

### Results and Discussions

Figures (1) and table (1) 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 – H<sub>1</sub> has high ability to residue weight with increasing temperature while H<sub>6</sub> has low ability to residue weight with increasing temperature.

2 - The values of (Residue weight) decrease with increasing the volume fraction.

**H<sub>3</sub> > H<sub>6</sub> , H<sub>2</sub> > H<sub>5</sub> , H<sub>1</sub> > H<sub>4</sub>.**

3– Specimens that contain only (SiO<sub>2</sub>) give (Residue weight) lower than the (Residue weight) of the Specimens that contain only (Al<sub>2</sub>O<sub>3</sub>).

**H<sub>1</sub> > H<sub>2</sub> , H<sub>4</sub> > H<sub>5</sub>.**

4 – Specimens that contain both (Al<sub>2</sub>O<sub>3</sub> + SiO<sub>2</sub>) give (Residue weight) lower than the Specimens that contain only (Al<sub>2</sub>O<sub>3</sub>) or Specimens that contain only (SiO<sub>2</sub>) .

**H<sub>1</sub> > H<sub>3</sub> , H<sub>2</sub> > H<sub>3</sub> , H<sub>4</sub> > H<sub>6</sub> , H<sub>5</sub> > H<sub>6</sub> .**

The polymeric material undergoes many changes when it is heat 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 was call thermal stability [15, 16].

Therefore, TGA measurement is employ to study the thermal- oxidative degradation of prepared hybrids in air. Fig. (1) 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 [17].

2 – Second stage is at temperature range from (300-600)°C, heating causes the release vapors of water, CO, phenol. Moreover, 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 [18, 19]. This stage also includes broken of molecular chain of polymer.

3 - Third stage is at temperature range from 600 °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<sub>2</sub>O, CH<sub>4</sub>, CO<sub>2</sub>, Benzene [19].

Results also show that the thermal stability increases in (H<sub>6</sub>) compared with other samples, depending on the type and loading ratio of the reinforcements in the hybrids composites. As the loading ratio of (Al<sub>2</sub>O<sub>3</sub> + SiO<sub>2</sub>) increases the hybrids become more stable compared with hybrids as loading ratio of (SiO<sub>2</sub>) or (Al<sub>2</sub>O<sub>3</sub>) increases as shown in Fig (1) .

**Conclusions**

This work has reached to the following conclusions:

- 1 - The results show the hybrid (H<sub>1</sub>) gives high ability to weight loss with increasing temperature while the hybrid (H<sub>6</sub>) gives low ability to weight loss with increasing temperature.
- 2 - The results show that as the values of volume fraction increases the ability to weight loss with all hybrids decrease also.

**Table (1) gives the values of residue weight (%) for prepared hybrids composites as elevated temperature in TGA test.**

TGA						
Temp(°C)	H <sub>1</sub>	H <sub>2</sub>	H <sub>3</sub>	H <sub>4</sub>	H <sub>5</sub>	H <sub>6</sub>
25	100	100	100	100	100	100
50	99.347	99.401	99.498	99.398	99.514	99.637
100	99.108	98.905	98.137	99.006	98.672	99.0086
150	98.479	97.553	96.442	98.751	98.110	98.729
200	98.005	95.617	93.175	98.151	97.327	98.197
250	97.425	92.881	91.094	95.579	94.285	97.278
300	94.271	90.134	88.256	91.801	93.357	96.019
350	91.487	89.002	82.508	88.925	91.044	95.331
400	88.165	85.146	80.739	75.153	87.316	93.402
450	82.751	81.329	77.197	70.284	86.599	92.555
500	77.239	70.247	70.529	62.706	82.287	90.047
550	70.934	61.818	67.190	53.183	73.444	82.329
600	62.862	50.973	61.808	40.279	60.393	70.518
650	50.741	44.892	55.729	26.372	53.478	67.397
700	41.113	39.336	52.113	22.193	40.511	64.428
750	28.513	30.776	49.327	20.829	32.802	60.906
800	16.432	26.021	45.319	17.305	25.767	57.782
850	10.576	21.247	42.875	16.718	21.093	50.079
900	3.947	14.419	40.701	14.070	20.814	47.818
950	0.972	10.058	35.697	13.129	18.055	44.509
1000	0.713	9.410	32.782	12.517	14.370	40.151

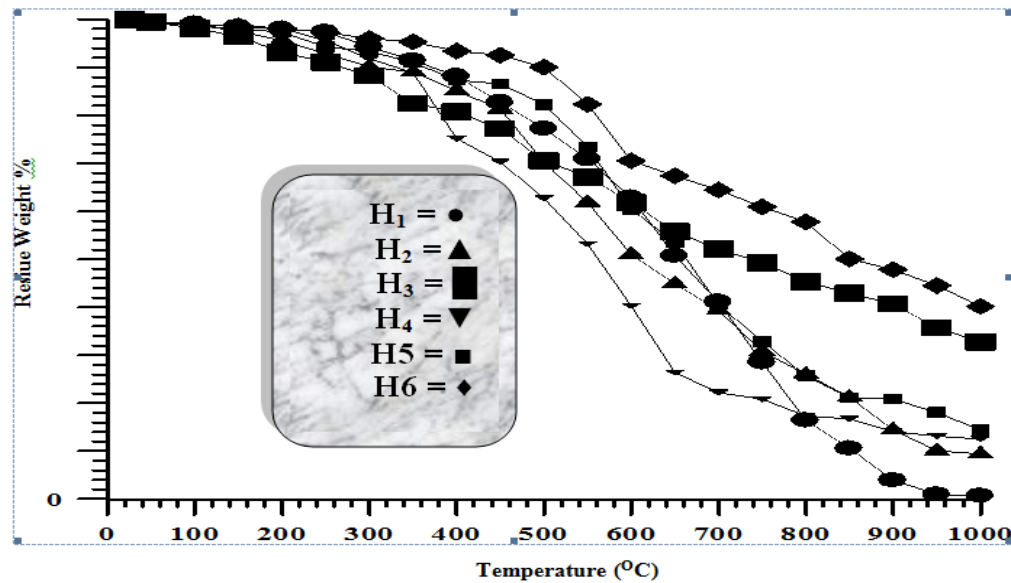


Figure (1) TGA thermal oxidative degradation curves at elevated temperature for all prepared hybrids .

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