The Inhibition Combustion of Polyester and Epoxy Resins by Using Some of Inorganic Phosphorus Salts

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الخلاصة

في هذا العمل، درس تأثير اربع انواع من املاح الفسفور اللاعضوية على تثبيط اللهوبية لراتنجي البولي استر والايبوكسي، وبنسب مئوية وزنية (0.5, 1.5, 2.5 و3.5%) بواسطة تحضير شرائح بقياس (130×130×3) ملم^3.

استخدمت ثلاث طرائق قياسية لاختبار تثبيط اللهوبية وهي: الطريقة القياسية ASTM: D-2863، الطريقة القياسية ASTM: D-635 والطريقة القياسية ASTM: D-3014. أن النتائج المستحصل عليها تشير الى ان المضاف IV يمتلك تأثيرا كبيرا كمثبط لهب، حيث يحدث الأطفاء الذاتي (S.E.) عند النسبة 0.5% لراتنج البولي أستر غير المشبع وكذلك حدوثه عند النسبة 1.5% لراتنج الأيبوكسي. كذلك يحدث عدم الأشتعال للعينة (N.B.) عند النسبة 2.5% لراتنج البولي أستر غير المشبع وعند النسبة 3.5% لراتنج الأيبوكسي. أما المضافان I وII، فقد أظهرا تأثيرا قليلا على تثبيط اللهوبية في كلا الراتنجين.

الكلمات المفتاحية

الراتنجي، البولي استر والايبوكسي، تثبيط اللهوبية.

Abstract

In this work, the effect of four types of inorganic phosphorus salts on flammability for polyester and epoxy resins has been studied in the weight ratios of (0.5, 1.5, 2.5 & 3.5%) by preparing films of (130×130×3) mm^3 in diameter, Three standard tests method were used to measure the flame retardation which are; ASTM: D-2863, ASTM: D-635 & ASTM: D-3014.

The results obtained from these tests indicated that additive IV has high efficiency as a flame retardant, self-extinguishing (S.E), occurred at the percentage 0.5% for unsaturated polyester resin and the percentage 1.5% for epoxy resin. Non-burning (N.B), occurred at the percentage 2.5% for unsaturated polyester resin and at the percentage 3.5% for epoxy resin. Additives I and II show low effect on retard combustion in both resins.

Keywords

Polymers, Polyester resin and Epoxy resin, Fire-retardant.

1. Introduction

Flame retardation is essentially an interruption of the burning process [1]. There are two distinct types of flame retardant to be recognized [2]:

a. Reactive flame retardants are compounds usually containing hetero elements which can also be chemically incorporated in smaller proportions, usually during the polymerization process.

b. Additives of flame retardants are incorporated into polymers by physically mixing with the polymer, normally after the polymerization complete.

A good flame – retardant additive must meet the following requirements [2, 3]: thermally stable up to the processing temperature of the polymer and stable to light, not interact with main chain of the polymer, should not be poisonous and should not inversely affect physical properties of polymer.

Many inorganic phosphorus compounds are used as fire retardants in polymer compositions, the flame retardant action of which is understood less than other materials which are used flame – retardants. [4] These compounds are often used synergistically with other elements especially nitrogen and halogens [3,5]. Phosphorus compounds, especially phosphate esters are the more widely used for high hydroxylated polymers and the mechanism for their fire retardant action is in condensed phase by reducing the production of combustible gasses and increasing char formation.[1,6,7]. The inorganic phosphate salt compounds, which shows high efficiency in flame – redundancy of unsaturated polyester and epoxy resins [8,11]. These resins are used in many important applications [9, 10], so that, in this work the influence of increasing the ratio of additives as flame-retardant on unsaturated polyester and epoxy resins were studied.

2. Experimental part

2.1. Materials

a. polymers:

(1) Unsaturated polyester resin, hardener type (MEKP), imported from United Arab Emirate (U.A.E.).

(2) Epoxy resin, type (CY223), hardener type (HY956), imported from Ciba-Geigy Co.

b. Flame-retardant Materials:

Mono ammonium phosphate, with purity 99%, (additive I); Di ammonium phosphate with purity 99% (additive II); Tri ammonium phosphate with purity 99.5%, (additive III) and Poly ammonium phosphate with purity 99%, (additive IV); in powder form, imported from MERCK Co.

2.2. Tests

a. ASTM: D-2863: The measurement of Limiting Oxygen Index (LOI), is widely used for measuring flammability of polymers, the (LOI) is the minimum concentration of oxygen and nitrogen, that supports a Candle-Like combustion of specimen [12].

b. ASTM: D-635: Measurement of rate of burning (R.B.), extent of burning (E.B.) and time of burning (T, B) for self-supporting plastic in a horizontal position [13, 14].

c. ASTM: D-3014: Measurement of maximum flame height (H) [15].

2.3. Preparation of samples

The samples were prepared in the dimensions of (130×130×3) mm^3, three sheets of each unsaturated polyester and epoxy resins were

prepared for each percentage weight (0.5, 1.5, 2.5 & 3.5%) with the additives I, II, III, and IV. These sheets were cut as a sample according to ASTM: D-2863, ASTM: D-635 and ASTM: D-3014.

3. Result and Discussion

1. Measurements of (LOI) according to the ASTM: D-2863:

Table-1 & 2 indicate that, also Figs.(1&2). The limiting oxygen Index (LOI) for unsaturated polyester resin without additives is 20.4, and for epoxy resin is 19.7.

The oxygen concentration required to support a candle – like of unsaturated polyester and epoxy resin samples was increased with increasing the weight percentages of additives. The efficiency of I, II, III and IV additives is in the following order:

IV> III > II > I

2. Measurement of rate of burning (R.B.), according to the ASTM: D-635:

The results obtained from these tests showed that the rate of burning (R.B.) of the unsaturated polyester and epoxy resins with the additives has a continuous reduction with increasing the percentage weight of additives, as in Table (3 and 4) respectively.

Fig.(3) and (4) show the flame speed curves of flame – retardation for resins. This result indicated that the additive IV has the highest efficiency on self-extinguishing of resins, especially in percentage 0.5% for unsaturated polyester resin and in percentage 1.5% for epoxy resin. Non-burning occurs in percentage 2.5% for unsaturated polyester resin and in percentage 3.5% for epoxy resin.

3. Measurement of flame high (H), according to the ASTM: D-3014:

Figs.(5) and (6) showed that the maximum flame high (H) decreases with increasing the percentage of additives (inversely proportional), as listed in Table (5) and Table (6). The flame high was 4.0 cm in percentage 1.5% for unsaturated polyester resin and it was 5.5 (cm) in percentage 2.5% for epoxy resin.

The results indicate that additive IV has the best efficiency on retard combustion. This high efficiency depends in basically on the structure of this material (Poly ammonium phosphate), its contain in their structure on phosphor element and nitrogen which have high effect on retard combustion. The free radicals were formed from decomposition of material ($P^{\cdot}$,$N^{\cdot}$) will reactrapidly with the free radicals of flame chain, such as ($H^{\cdot}$,$O^{\cdot}$, $^{\cdot}OOH$, …,etc.) to form inert compounds like (HPO, NH4, …,etc.) and work on inhibition of thermal decomposition will occur in flame front, because decreases of amount of generation heat and to form a group from the non-flammable gases, such as (CO, CO_2, H_2O,…, etc.) thus will decrease from volatile materials flammable. The char will form as a result from the thermal decomposition of the specimen, it covered the polymer roof.

4. Conclusion

1. The limiting oxygen index (LOI) increases with increasing the weight percentages of the additives, but the rate of burning (R.B.) and the flame height (H) decrease with increasing the weight percentages of the additives.

2. The flame-retardancy efficiency of the additives I, II, III and IV appear to follow the order:

IV > III > II > I

3. Poly ammonium phosphate has a good effect to retard the flammability and combustion of both resins.

4. Self-extinguishing (S.E), occurred at the percentage 0.5% of the additives (III and IV) for unsaturated polyester resin, but in epoxy resin occurred at the percentage 1.5% of the additives (III and IV).

5. Non-burning (N.B) occurred at the percentage 2.5% of the additives (IV) for unsaturated polyester resin, but in epoxy resin it occurred at the percentage 3.5% of the additive IV.

Finally, the combustion products like; free radicals (˙CL, ˙OH), chare,…, etc.; will form allayer to prevent burning and displacing oxygen that help continue burning of polymers.

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Table (1): Limiting oxygen index (LOI) for polyester resin with additives.

% Additives	(LOI)				
	Non	0.5	1.5	2.5	3.5
I	20.4	21.20	22.35	22.94	23.95
II	20.4	21.47	22.82	23.35	24.17
III	20.4	21.69	23.75	24.37	25.44
IV	20.4	21.93	23.92	24.81	25.83

Table (2): Limiting oxygen index (LOI) for epoxy resin with additives.

% Additives	(LOI)				
	Non	0.5	1.5	2.5	3.5
I	19.7	20.37	21.70	22.27	22.84
II	19.7	20.51	22.38	22.91	23.43
III	19.7	20.99	22.94	23.67	24.48
IV	19.7	21.23	23.22	24.11	25.0

Table (3): Rate of burning (R.B.) for unsaturated polyester resin with additives.

% Test	Non	0.5	1.5	2.5	3.5	Additives
AEB (cm)	10	10	10	8.8	6.5	I
	10	10	9.8	7.4	5.5	II
	10	10	8.1	5.2	-	III
	10	9.3	6.5	-	-	IV
ATB (min)	6.92	7.35	8.77	8.71	9.28	I
	6.92	8.95	9.64	9.80	8.87	II
	6.92	9.70	10.12	8.66	-	III
	6.92	9.2	8.90	-	-	IV
R.B (cm/min)	1.44	1.36	1.14	1.01	0.70	I
	1.44	1.12	1.01	0.76	0.62	II
	1.44	1.03	0.80	0.60	-	III
	1.44	1.01	0.73	-	-	IV
S.E	-	-	-	yes	yes	I
	-	-	yes	yes	yes	II
	-	yes	yes	yes	yes	III
	-	yes	yes	yes	yes	IV
N.B	-	-	-	-	-	I
	-	-	-	-	-	II
	-	-	-	-	yes	III
	-	-	-	yes	yes	IV

Note:

AEB: Average extent of burning.

ATB: Average time of burning.

R.B: Rate of burning.

S.E: Self-Extinguishing.

N.B: Non-Burning.

Table (4): Rate of burning (R.B) for epoxy resin with additives.

% Test	Non	0.5	1.5	2.5	3.5	Additives
AEB (cm)	10 10 10 10	10 10 10 10	10 10 9.6 8.0	9.8 8.3 7.1 5.7	7.5 5.2 3.8 -	I II III IV
ATB (min)	5.12 5.12 5.12 5.12	5.81 6.84 7.29 7.75	6.66 8.33 8.57 8.88	7.71 9.23 8.65 10.75	7.14 8.50 7.16 -	I II III IV
R.B (cm/min)	1.95 1.95 1.95 1.95	1.72 1.46 1.37 1.29	1.50 1.20 1.12 0.90	1.27 0.89 0.82 0.53	1.05 0.61 0.53 -	I II III IV
S.E	- - - -	- - - -	- - yes yes	- yes yes yes	yes yes yes yes	I II III IV
N.B	- - - -	- - - -	- - - -	- - - -	- - - yes	I II III IV

Table (5): Flame height (H) for unsaturated polyester resin with additives.

% Test	Non	0.5	1.5	2.5	3.5	Additives
W1	5.63 5.63 5.63 5.63	6.10 6.11 6.13 6.15	6.13 6.15 6.17 6.34	6.17 6.19 6.20 6.54	6.21 6.23 6.24 6.64	I II III IV
W2	2.57 2.57 2.57 2.57	2.275 2.335 2.40 2.23	2.325 2.365 2.43 2.30	2.375 2.395 2.46 2.38	2.425 2.43 - -	I II III IV
PWR	54.35 54.35 54.35 54.35	62.70 61.78 60.84 63.73	62.07 61.54 60.61 63.72	61.50 61.31 60.32 -	60.95 60.99 - -	I II III IV
H	14.0 14.0 14.0 14.0	13.0 12.5 11.0 8.0	11.0 10.5 7.0 4.0	9.5 9.0 4.0 -	8.0 7.5 - -	I II III IV

Note:

W_1: weight of sample before burning.

W_2: Weight the loss from sample after burning.

PWR: The percentage of weight.

H: Flame height (cm).

Table (6): Flame height (H) for epoxy resin with additives.

% Test	Non	0.5	1.5	2.5	3.5	Additives
W_1	4.52 4.52 4.52 4.52	4.99 5.0 5.03 5.05	5.03 5.04 5.07 5.09	5.07 5.08 5.10 5.12	5.11 5.12 5.13 5.16	I II III IV
W_2	1.43 1.43 1.43 1.43	1.135 1.19 1.21 1.22	1.185 1.23 1.25 1.28	1.235 1.27 1.29 1.32	1.285 1.31 1.33 -	I II III IV
PWR	68.36 68.36 68.36 68.36	77.25 76.20 75.94 75.84	76.44 75.59 75.34 74.85	75.64 75.0 74.70 74.21	74.85 74.41 74.07 -	I II III IV
H	12.0 12.0 12.0 12.0	11.0 10.5 9.5 9.0	10.0 9.5 8.5 7.5	9.0 8.5 7.0 5.5	7.5 7.0 5.0 -	I II III IV

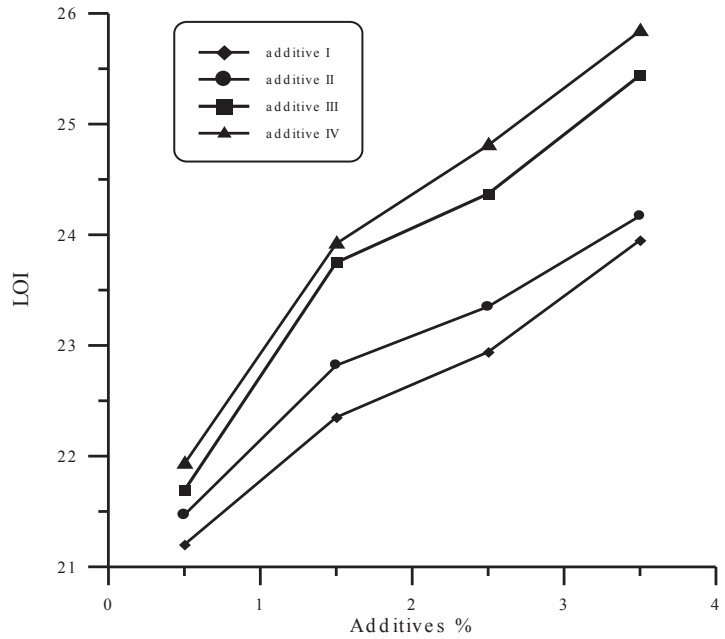

Fig. (1): (LOI) of the unsaturated polyester resin with additives.

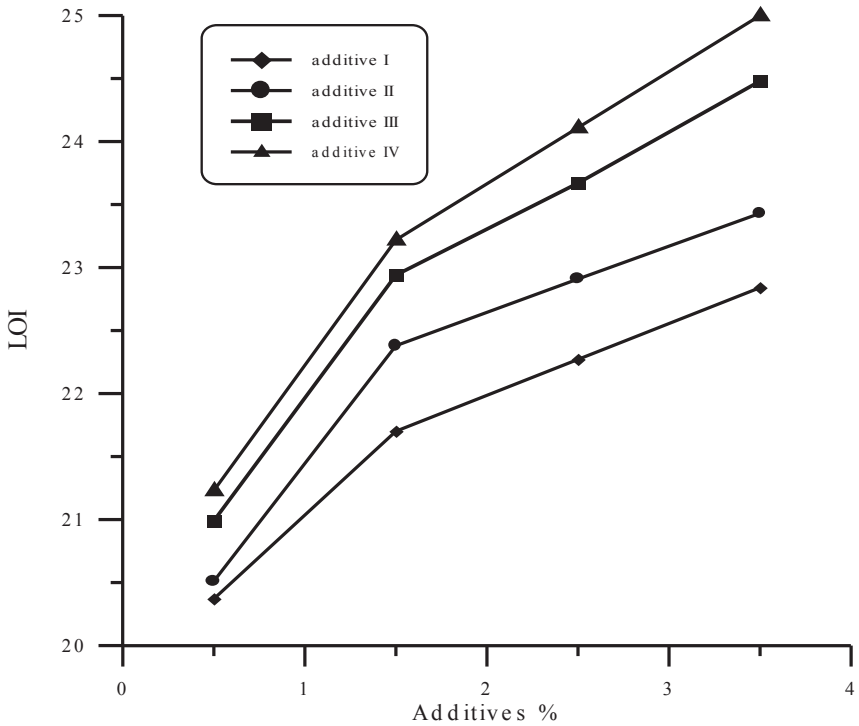

Fig. (2): (LOI) of the epoxy resin with additives.

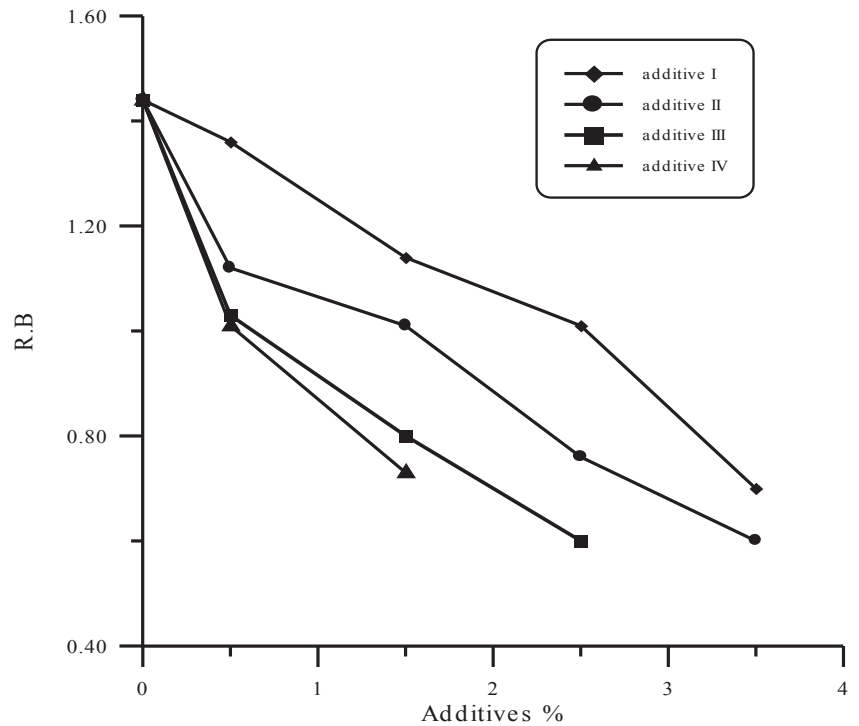

Fig. (3): Rate of burning (R.B) of the unsaturated polyester resin with additives.

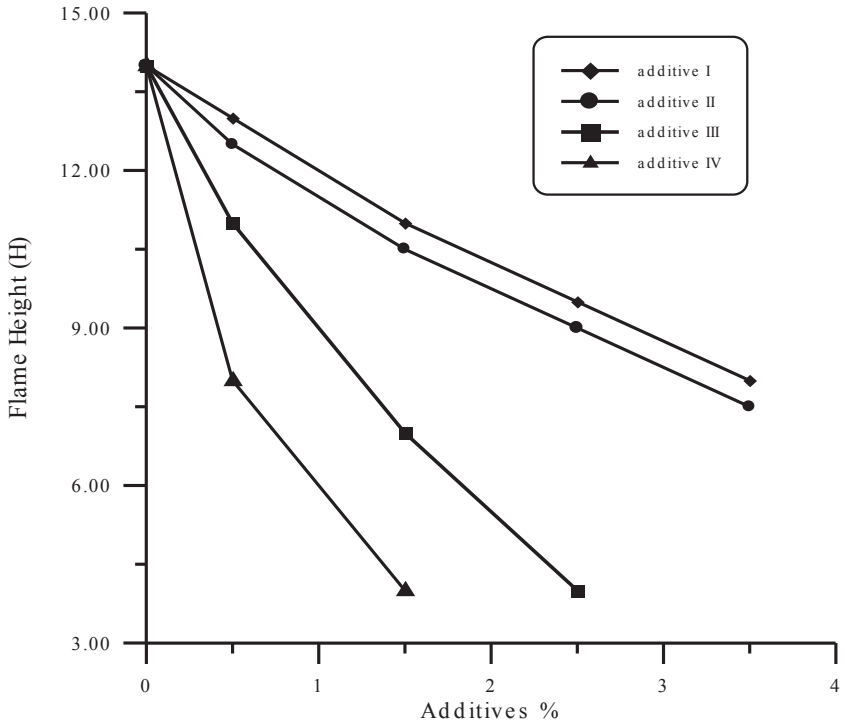

Fig. (4): Rate of burning(R.B) of the epoxy resin with additives.

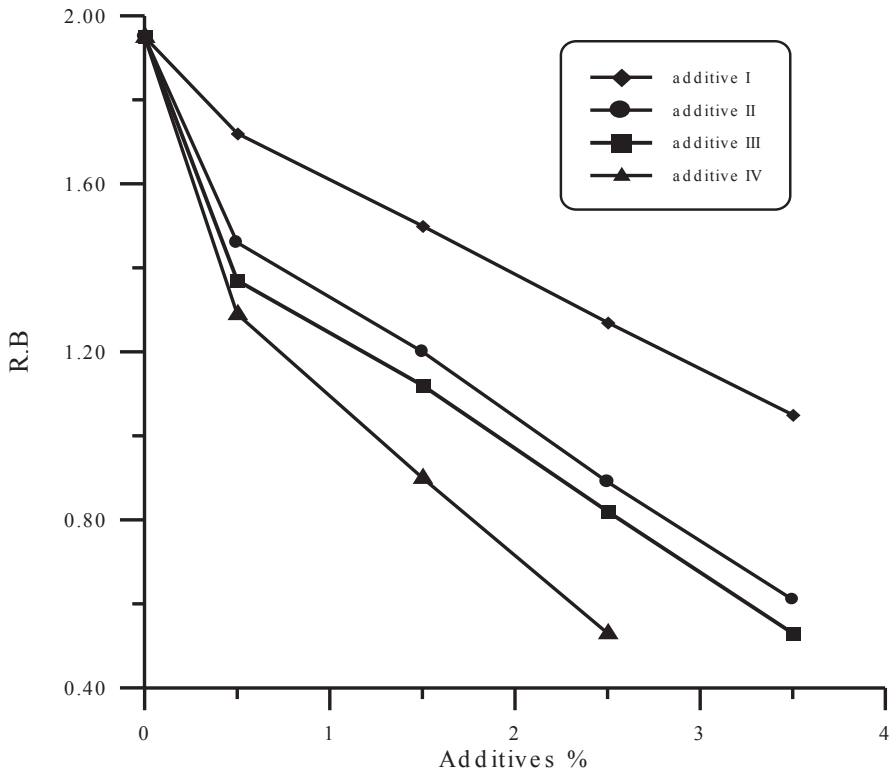

Fig. (5): Flame high(H) of the unsaturated polyester resin with additives.

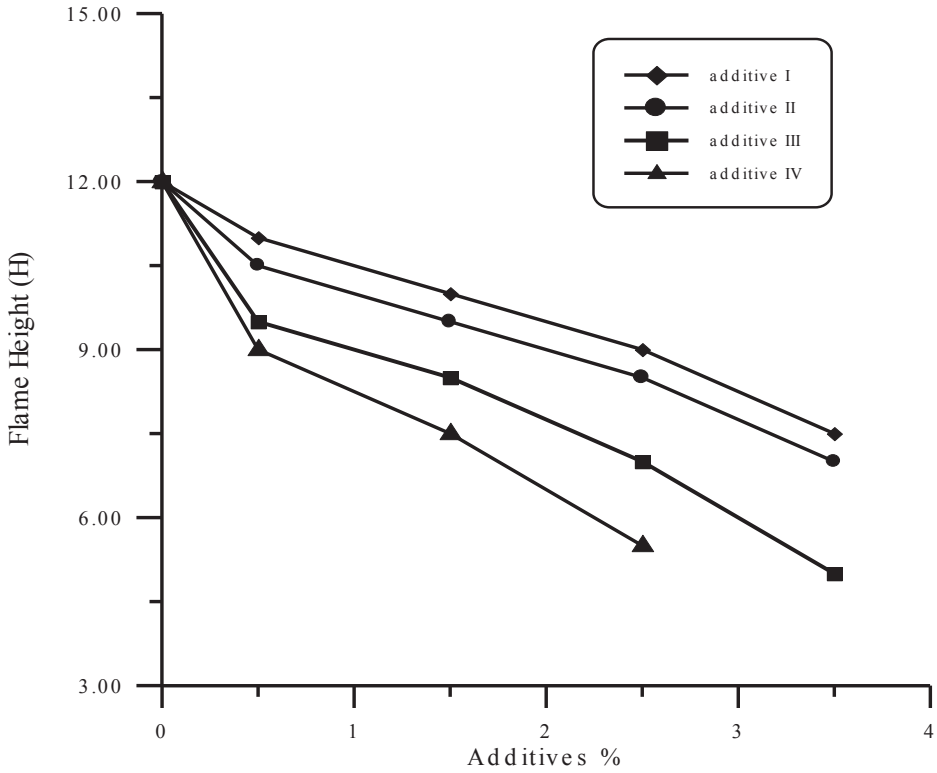

Fig. (6): Flame high(H) of the epoxy resin with additives.