



Evaluation of the thermal stability, flammability and toxicity properties of new back coated textile samples

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ABSTRACT

New flame retardant compounds containing either phosphate groups or phosphate boron derivatives were prepared and the structures were proved by Infrared spectroscopy and mass spectrometry. The new back coating system containing the new flame retardant compounds improved the flammability and thermal stability of cotton and blend textile samples. The toxicity of gas products evolved during the combustion of cotton and blend textile samples were evaluated according to ASTM 1678 and NFPA 269 standards. The new back coating system succeeded in reducing the toxicity effect of burned textile on the surrounding environment especially human been.

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Introduction

The essential reason for incorporating fire retardants into materials is to reduce the hazard to life from fire. Fire retardant strategies aim to reduce the ignitability or the rate of heat release of burning items. However, fire hazard is a combination of both the flammability and the fire gas toxicity [1].

Fires in dwellings constitute about 60% of the total number of fires in buildings [2,3]. Flammable interior furnishings and decorative materials (particularly textiles) are among the main fire hazards in dwellings. In fires, they can constitute the so called first ignited material and contribute to the development of fire [4-6]. These textiles are responsible for about 50% of deaths in these fires. When studying the reaction of people to fire conditions, it was found that there is a common fear of smoke and a reluctance to run even a small distance in places filled with smoke. It results from the fact that the evolved smoke reduces the visibility and also makes people lose their orientation due to prolonged exposure to toxic substances, which results in an inability to escape a hazardous place, even if it is quite distant from direct contact with flames [7-9]. Thus, the number of deaths increases, often among young, able people. The fire statistics confirm that usually 50%–75% of fire deaths, is not due to direct contact with flames, but as a result of inhalation of toxic gases (asphyxiant, irritant, narcotic gases) contained in the smoke [10-14].

To improve the behaviour of fabrics in fire conditions, better and better flame retardants are being developed, which improve the thermal resistance of materials to a very great extent, increasing their ignition temperature, reducing the combustion rate and decreasing the amount of heat released [15]. Such actions enable the classification of fabrics according to appropriate classes that according to the requirements and rules of fire protection can be used in public buildings. For that they are important because they decrease the fire hazard.

However, from the point of view of toxicity, the application of fire retardants can result in a considerable unfavorable alteration of the gas composition emitted during the combustion of fabrics [16]. Assessment of toxic hazard is increasingly being recognized as an important factor in the assessment of fire hazard. Prediction of toxic fire hazard depends on two parameters [17]:

1. Time-concentration profiles for major products. These depend on the fire growth curve and the yields of toxic products.
2. Toxic potency of the products, based on estimates of doses likely to impair escape efficiency, cause incapacitation, or death.

There is an increasing tendency to use chemical analysis methods [18] to calculate toxic potency values using equations such as those of ISO 13344 [19] and ISO TS 13571 [20]. These methods calculate the toxic potency of fire effluents in terms of the narcotic gases, carbon dioxide (CO₂), carbon monoxide (CO), hydrogen cyanide (HCN) and oxygen depletion (DO₂), and irritant gases including hydrogen chloride (HCl), hydrogen bromide (HBr), hydrogen fluoride (HF), sulphur dioxide (SO₂), nitrogen oxides (NO_x), formaldehyde, acrolein and other organo-irritants. In current ISO documents, all oxides of nitrogen (NO_x) are considered to be nitrogen dioxide (NO₂) [21].

Certain bench-scale determinations of toxic product yield apply only to well-ventilated burning, such as the cone calorimeter [22] or ASTM E1678 [23].

The aim of this work is to study the effect of new back coating flame retardant system on the flammability and toxicity of thermal decomposed textile samples during fire.

Experimental

Materials

Natural cotton fabric and synthetic polyester polyacrylic (80/20) blend was supplied by Texmar Company, Egypt. Commercial binder and thickeners, were used to form back coating formulations which illustrated in Table(1).

Diethylmalonate was supplied from Merck and Alfa Aesar, Germany, with purity 99%. Phosphoric acid was supplied from Fine Chem., India, with purity 99%. Phosphorus oxychloride was supplied from PVT.LTD., India. Boric acid made in Hungary.

Preparation

Preparation of Trimalonyl phosphate phosphine oxide (A₁₁)

30 ml (0.1M) of malonyl phosphate (A₁) [24] was mixed with 5.8 ml (0.033 M) of phosphorus oxychloride in 250 ml beaker. The Trimalonyl phosphate phosphine oxide (A₁₁) was formed after good stirring. The structure of A₁₁ has been proven by IR and MS analysis as in Fig.1(A).

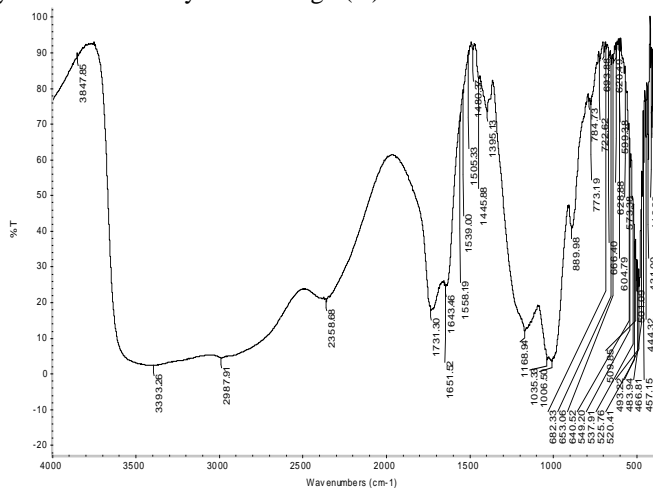


Fig.1 (A) FTIR of A₁₁

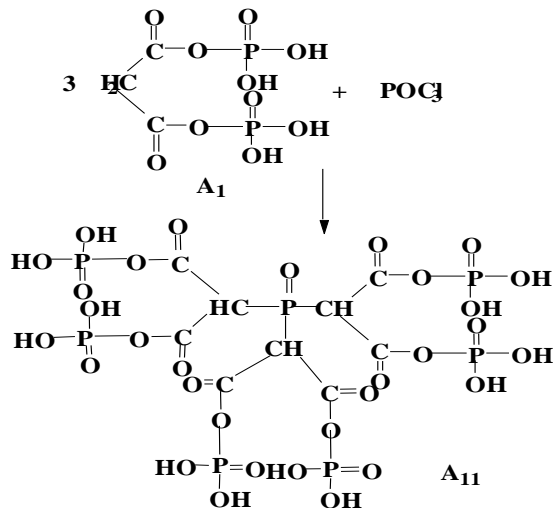
The IR spectrum and mass fragmentation of A₁₁ could be summarized as following:

IR

- 3393.26 cm⁻¹ for the O-H stretching absorption;
- 1731.13 cm⁻¹ for the C=O stretching absorption;
- 1168.94 cm⁻¹ for the P=O stretching absorption;
- 1006.5 cm⁻¹ for the P-O-C stretching absorption.

MS

- m/z 678 [M⁺ - 159] CO₂PO(OH)₂, 2 (OH) ;
- m/z 586 [M⁺ - 250] 2 CO₂PO(OH)₂;
- m/z 462 [M⁺ - 375] 3 CO₂PO(OH)₂ ;
- m/z 331 [M⁺ - 500] 4 CO₂PO(OH)₂ ;
- m/z 312 [M⁺ - 518] 4 CO₂PO(OH)₂, H₂O;
- m/z 178 [PO₃COPO(CH₂)⁺²] base peak .



Scheme (1) the reaction of 3 A₁ with phosphorus oxychloride.

Preparation of malonyl phosphoroborate A₁₆

Add into a round flask 80 ml (0.3M) of A₁ and 18.7 gm (0.3M) of boric acid with checking the mixture well. Then reflux the resultant mixture on hot plate at 110-120 °C for six hours. The white precipitate was formed then washed by ethanol and dried in oven at 70 °C. The structure of A₁₆ has been proven by IR and MS analysis which is graphically represented in Fig.1(B).

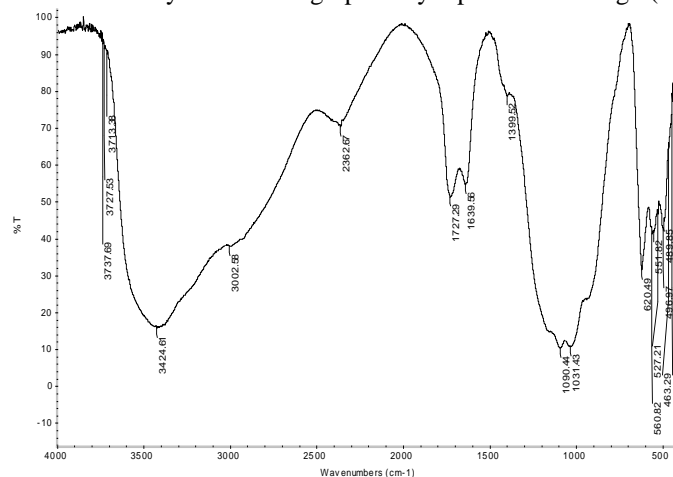


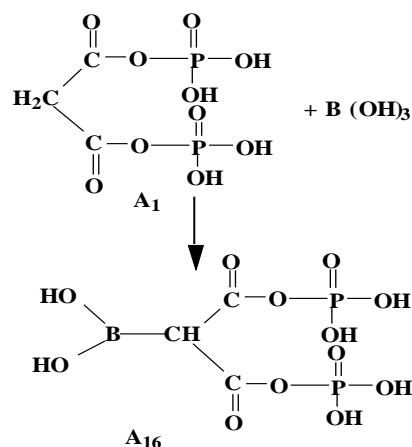
Fig.1 (B) FTIR of A₁₆

The IR spectrum and MS of A₁₆ showed as following peaks:

- IR
- 3424.61 cm⁻¹ for the O-H stretching absorption;
 - 1727.29 cm⁻¹ for the C=O stretching absorption;
 - 1090.44 cm⁻¹ for the P=O stretching absorption;
 - 1031.43 cm⁻¹ for the P-O-C stretching absorption.

MS

- m/z 256.8 [M⁺ - 51] 3(OH),
- m/z 246 [M⁺ - 51 - 10.8] B(OH)₃,
- m/z 229.2 [M⁺ - 61.8 - 18] B(OH)₃, OH,
- m/z 139.2 [M⁺ - 168.8] CO₂PO₃HB(OH)₂,
- m/z 57 [CH₂CO₂]⁺² base peak.



Scheme (2) the reaction of A₁ with boric acid.

Characterization

Mass spectrometry (MS) were conducted on a Shimadzu QP- 2010 plus. FTIR spectroscopy analyses were performed using Nicolet 380 spectrophotometer in spectral range 4004-400 cm⁻¹. Chromatography (IC) manufactured by DIONEX, model ICS-1000.

Differential Scanning Calorimeter (DSC) experiments was conducted by a Shimadzu DSC-50 with a heating rate of 10°C/min under nitrogen atmosphere with flow rate 30 ml/min, samples weight 5-10 mg. Thermogravimetric analysis (TGA)

experiments were conducted by a Shimadzu- TGA-50 with heating rate 10 °C/min under nitrogen atmosphere with flow rate 20 ml/min.

Limiting Oxygen Index (LOI) test was done according to ISO 4589[25]. The apparatus was manufactured by Stanton Rheometric Scientific Ltd, UK. Flame spread (UL-94) test was done according to ISO 3795[26]. UL-94 Flame Chamber manufactured by Stanton Rheometric Scientific Ltd, UK. Specific optical density of smoke was tested by smoke box chamber manufactured by Stanton Rheometric Scientific Ltd, UK, according to ASTM E 662-06 [27].

Toxicity test was done according to American National Standard E 1678[23], using toxicity apparatus, from fire testing Technology Limited.

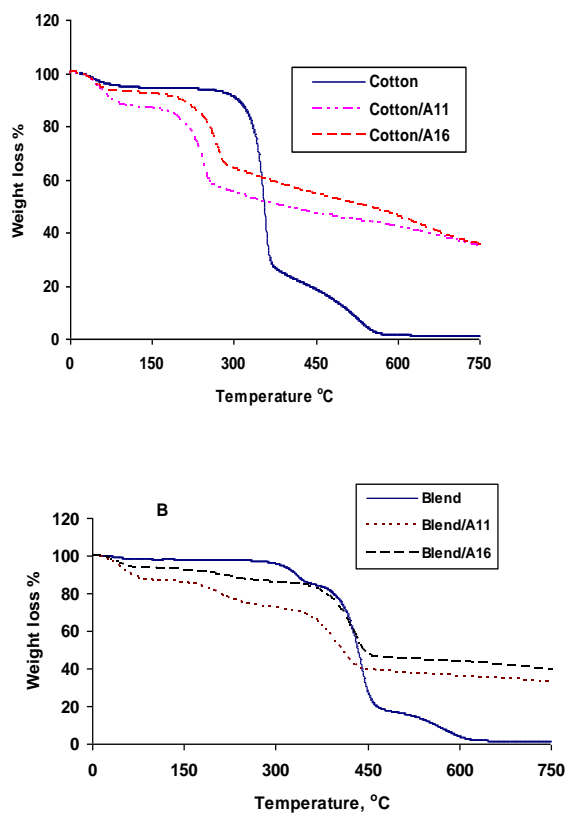


Fig. 2 (A) TGA of uncoated and back coated cotton fabrics, (B) TGA of uncoated and back coated blend fabrics.

Results and discussion

Thermal analysis

Thermo gravimetric analysis (TGA)

The TGA data was written in table (2) and represented graphically in Fig.2(A). The data in table showed that cotton fabric sample was totally decomposed in three stages with total weight loss percentage 99%.

The first region was located between 24-45 °C the weight loss in this region is primarily attributed to adsorbed water molecules[28]. The second region was the main decomposition stage lies in the temperature range 334-366 °C with weight loss percentage 66%. The third region was located between 494-535 °C. Complete decomposition of the fiber occurred at 650 °C.

The back coating formulation containing the flame retardant A₁₁ has changed the way of thermal decomposition of cotton sample.

The back coated cotton was totally decomposed in four stages and its decomposition temperature starting at 162 °C. This means that back coated cotton fabric has lower decomposition

temperature comparing with uncoated cotton because of a catalytic dehydration of cellulose by flame retardants [28]. In general the thermal stability of textile cotton sample is improved; this can be seen from the total weight loss which is decreased to 77 % instead of 99 % for pure cotton sample. Also the main decomposition stage of cotton sample is totally disappeared and no weight loss is detected within the temperature range 334-366 °C.

The back coating formulation containing the flame retardant A₁₆ has increased the thermal stability of cotton sample. The total weight loss is decreased to 69.5 % the main decomposition stage was lied within the temperature range 206-245 °C with weight loss percentage 29%.

TGA data in table (3) and graphically represented in Fig.2(B) showed that the blend sample is decomposed in four stages with 98.4% weight loss. The main loss percentage is happened within the temperature range 411-454 °C.

The new back coating formulation which containing the new flame retardant A₁₁, A₁₆ have increased the thermal stability of textile blend samples.

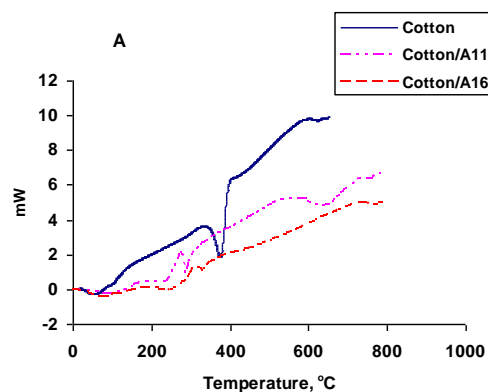
The increased thermal stability is clearly seen from the shifting in main decomposition stage to lower weight loss percentages 33.5 % and 39 % for A₁₁ and A₁₆ respectively. The second indication for improvement of thermal stability is the total weight loss percentage values which decreased to 73 and 66 for A₁₁, A₁₆ respectively.

Differential Scanning Calorimeter (DSC)

DSC is an important tool for predicting the fire behavior of textile samples. This is clearly seen from comparing the results of DSC and flammability test. If the main decomposition peak is disappeared after modification by back coating system, that mean it will achieve higher class in flame spread test[29].

The DSC data in tables (4), (5) for cotton and blend textile sample with and without back coating flame retardant system showed that there is a noticeable improve in the thermal decomposition behavior after applying the new back coating system.

For cotton sample it seen that the decomposition behavior is pass through three endothermic stages. The main endothermic peak at 373 °C needs 76 J/g to be completed. After applying the flame retardant back coating systems which contain A₁₁ and A₁₆, this endothermic peak was disappeared. This mean that protective group was formed above the textile samples prevent completely the main decomposition stage of cotton sample alone as in fig. 3(A).



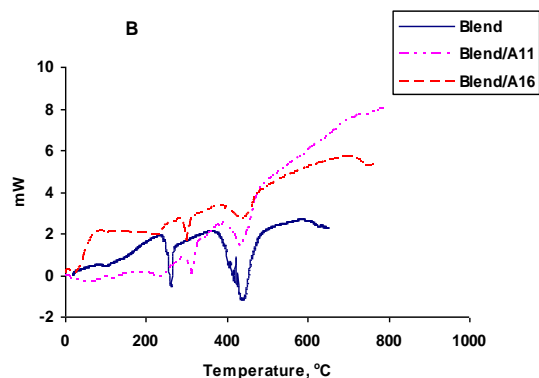


Fig. 3 (A) DSC of uncoated and back coated cotton fabrics, (B) DSC of uncoated and back coated blend fabrics.

In fig.3 (B) the new back coating system also protect blend sample from complete decomposition and increase its thermal stability. The main decomposition peak for blend sample is exothermic with 33.7 J/g heat of decomposition, this peak is totally disappeared for back coated sample.

Flammability properties

Limiting Oxygen Index (LOI) and flame spread

The LOI and UL-94 test results of cotton and blend samples back coated by A₁₁ and A₁₆ are tabulated in Table (6). A₁₁ and A₁₆ flame retardants have succeeded in acquiring textile samples good fire resistant properties especially with cotton in LOI test.

A₁₁ and A₁₆ flame retardants have succeeded to make cotton and blend achieve first class in flame spread test.

Smoke measurements

The smoke released from the cotton and blend samples back coated by A₁₁ and A₁₆ including the blank were tabulated in Table (6).

The cotton and blend samples produce small amount of smoke with specific optical density 49.4 and 82.4 respectively. The flame retardant back coating system decreased the smoke intensity in case of cotton sample and it increased in case of blend sample but in general it is of low smoke emission.

Toxicity

The lethal toxic potency LC₅₀ of the test specimen shall be predicted from the combustion atmosphere analytical data for CO, CO₂, O₂, and, if present, HCN, HCl, and HBr. This shall be determined for a given specimen mass loss by first calculating the total FED for the test. The total 0-30 minutes FED for a given specimen mass loss shall be determined from Eq. (1) [19,23,30].

$$FED = \frac{m[CO]}{[CO_2] - b} + \frac{21 - [O_2]}{21 - LC_{50,O_2}} + \frac{[HCN]}{LC_{50,HCN}} + \frac{[HCl]}{LC_{50,HCl}} + \frac{[HBr]}{LC_{50,HBr}} \quad (1)$$

All values of the toxic gases in above equation are in ppm, except O₂, which is expressed as a percentage. The values for m and b depend on the concentration of CO₂. If CO₂ < 5 %, m = -18 and b = 122 000. If CO₂ > 5 %, m = 23 and b = - 38 600. When the FED reaches a value of one, the gas concentration would be lethal to 50% of the population in a 30-min exposure [20].

For each individual toxicant, the LC₅₀ values shown have been statistically determined from independent experimental data to produce lethality in 50 % of test animals within a 30-minute exposure plus 14 days post exposure [23,29]. CO, CO₂, O₂ were analyzed by gas analyzer, where the remaining gases were analyzed by ion chromatography (IC) according to ISO 19701[31].

$$LC_{50} = \frac{M}{FED \times V} \quad (2)$$

Where the M is specimen mass loss is in g, the V chamber volume is 0.2 m³, and the resulting LC₅₀ in g.m⁻³.

The toxic gases emissions which were evaluated from samples under study were CO, CO₂, NO_x and HCl and tabulated in table (7).

The results showed that blank cotton when gets fire produce CO, CO₂ and HCl. The new back coating systems increase the emission of CO and reduce the HCl emission and prevent any CO₂ and emissions. On the other hand the back coated system with blend reduce the toxicity gases evolved comparing with the blend alone. Except the increasing emission of CO from A₁₁ and A₁₆ with fabric due to the concentration of CO₂ increases (up to 5%), the toxicity of CO increases. Above 5%, the toxicity of CO starts to decrease again [32]. A₁₆ with blend reduce CO₂ to 50 % and prevent NO_x in comparison with blank, but in the same time increase HCl emission due to antagonistic effect of back coated FR on blend.

The new back coating formulation containing A₁₁ was succeeded in decreasing the FED for cotton samples and increased the LC₅₀ as showed in fig. 4(A),(B) and this man that this flame retardant reduced the toxicity of different textiles. While the back coating system containing A₁₆ increased the FED of blend sample due to the increased emission of HCl. But this system succeeded in increasing LC₅₀ and decreasing the toxicity of treated fabrics.

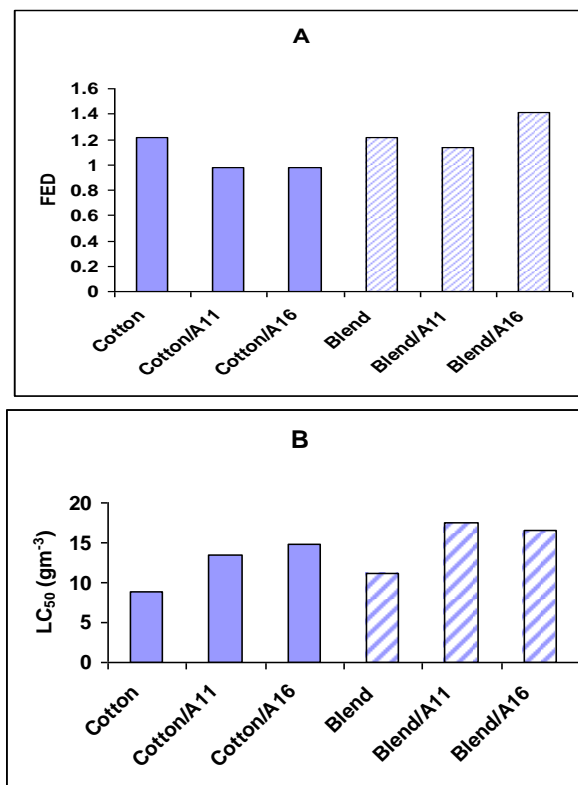


Fig. 4 (A) FED of uncoated and back coated fabrics, (B) LC₅₀ of uncoated and back coated fabrics.

Conclusion

The back coating system containing multi phosphate groups or simple phosphate groups and boric acid have succeeded in improving the flammability properties of cotton and blend textiles.

The back coating system containing A₁₆ has better effect on the flammability and thermal stability of cotton and blend textile

than containing A₁₁. Also flame retardant reduces the gas toxicity evolved during the thermal decomposition of textiles.

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Table (1) The Back-coating formulations of flame retardants compounds

Back coating name	Flame retardant concentration (%)	Binder concentration (%)	Thickener concentration (%)
A ₁₁	50	44	6
A ₁₆	64	32	4

Table (2) TGA data of uncoated cotton and back coated cotton

Sample name	Stages	Decomposition temperature range			Weight loss % at each stage	Total weight loss %
		Onset °C	Peak °C	Endset °C		
Cotton	1	23.7	39	44.6	3.5	99
	2	334	351	366	66	
	3	494	525	553.5	11.7	
Cotton/A ₁₁	1	33	57	69	12	77
	2	91.7	101.8	115.6	1.2	
	3	162	172.9	172	4.5	
	4	196	209.5	220.5	25.8	
Cotton/A ₁₆	1	28	47	58	7	69.5
	2	206	228	245	28.7	
	3	494	551	581	12	

Table (3) TGA data of blend and back coated blend.

Sample name	Stages	Decomposition temperature range			Weight loss % at each stage	Total weight loss %
		Onset °C	Peak °C	Endset °C		
Blend	1	73	45	141	2	98
	2	316.5	331	342	12	
	3	411	434	454	67	
	4	529.6	570.6	603.6	13.7	
Blend/A ₁₁	1	31	56.7	78	13.5	73
	2	147	160.5	130	4	
	3	177	199.5	208.8	9	
	4	314	345	372	33.5	
Blend/A ₁₆	1	28	48	63	6.7	66
	2	15	194	222.5	5.7	
	3	332.6	363	387	39	

Table (4) DSC data of cotton and back coated cotton samples by A₁₁,A₁₆ formulations

Sample name	Peaks	Decomposition temperature range			Heat J/g
		Onset °C	Peak °C	Endset °C	
Cotton	Endothermic 1	21	51	133	55.7
	Endothermic 2	305.5	326.6	360	1.6
	Endothermic 3	343	373	392.7	76
Cotton/A ₁₁	Endothermic 1	10.7	66	111.56	12.8
	Endothermic 2	223	230	241	12.4
	Endothermic 3	487	517.6	566.6	29
Cotton/A ₁₆	Endothermic 1	43.6	59	113.7	8
	Endothermic 2	158.6	193	226	14
	Exothermic 3	234	291.6	293	7
	Endothermic 4	605	621	648	2.8

Table (5) DSC data of blend and back coated blend samples by A₁₁,A₁₆ formulations

Sample name	Peaks	Decomposition temperature range			Heat J/g
		Onset °C	Peak °C	Endset °C	
Blend	Endothermic 1	77	98	114	2
	Endothermic 2	242	260.5	275	29
	Endothermic 3	354	362	415.5	27
	Exothermic 4	424	438	462	33.7
Blend/A ₁₁	Endothermic 1	20	52	118	13.8
	Endothermic 2	160	189.6	220	13.8
	Endothermic 3	242.5	254.8	265.5	12.8
	Endothermic 4	331	356	392	50.7
Blend/A ₁₆	Endothermic 1	31	38	58	5
	Endothermic 2	247	255	267	10
	Endothermic 3	335	369.7	402.8	38.5

Table (6) the flammability properties of uncoated fabrics and back coated fabrics

Sample	^a LOI %	Flame spread (mm/min)	Specific optical density
Cotton	20.9	76.4	49.4
Blend	21.3	98.8	62.4
Cotton/A ₁₁	73.5	Higher class (I)	25.97
Blend/A ₁₁	37.1	Higher class (I)	151.95
Cotton/A ₁₆	85.2	Higher class (I)	15.66
Blend/A ₁₆	40.6	Higher class (I)	86.2

^a Limiting Oxygen Index**Table (7) the concentration of evolved gases from uncoated and coated textiles.**

Sample name	The concentration of evolved gases by ppm			
	CO	CO ₂	NO _x	HCL
Cotton	428.5	6000	0.6	4036
Cotton/A ₁₁	978.9	0	0	2925
Cotton/A ₁₆	1083.4	0	0	2885
Blend	854.75	7025	0.7025	3769.9
Blend/A ₁₁	1084.95	7350	0.735	3310.9
Blend/A ₁₆	1004.2	4766	0.4766	4545