Fire retardant intumescent coatings

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ABSTRACT

A few useful fire retardant intumescent coatings incorporating a copolymer of vinyl acetate and 2-ethyl hexyl acrylate, a vinyl acetate acrylate copolymer emulsion and an amino resin have been developed. The fire performance of the coatings has been evaluated employing standard techniques and the results were found well within the limits. The samples which were treated with these coatings neither showed any surface spread of flame nor any glow on exposure. On the other hand the paint films swelled to form spongy, cellular insulating foams which acted as effective barriers against the conduction of heat.

INTRODUCTION

CELLULOSE is one of the most common materials used in a variety of forms for diverse applications in buildings such as for doors, windows, frames and partitions. Lining materials are being used for thermal insulation, acoustical treatment as well as for decorating purposes. However cellulosic materials are highly combustible in nature. When fire starts at a point, these materials generally help it spread and grow faster and thus pose a fire hazard

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If these materials are treated suitably for fire retardance, they will not only increase the growth period but will also reduce the spread of flames. Thus the fire hazard would be reduced and consequently loss of property and life especially in buildings can be minimised. Several methods have been employed for rendering cellulosic materials fire retardant i.e. chemical impregnation, spray method, surface treatment and incorporation method.[1-4]

The present communication concerns with the results of investigations carried out on a few intumescent fire retardant surface coatings.

EXPERIMENTAL

Materials and Method

The binders used in these investigations were supplied by Calico Chemicals Ltd., Bombay, Parekh Dyechem Industries Pvt. Ltd., Bombay and Synthetic and Polymer Industries, Ahmedabad.

Twelve formulations of fire retardant intumescent coatings were prepared. Their compositions are given in Table 1. For the preparation of different compositions of paints the following procedure was adopted:

TABLE 1 — COMPOSITION OF FIRE RETARDANT INTUMESCENT COATINGS

Ingredients	Parts by weight for formulation Nos. 1-12											
	1	2	3	4	5	6	7	8	9	10	11	12
1. Ammonium phosphate	21	15	21	24	25	25	25	30	35	25	25	24
2. Cyaniguanidine	7	14	14	14	20	—	—	—	—	20	20	14
3. 2:2 bishydroxymethyl-1- penta-erythritol 3-propanediol (polyol)	—	7	7	10	5	10	10	10	10	10	10	10
4. Starch	21	—	—	—	5	-	—	7	—	—	—	—
5. Melamine	—	—	—	—	—	15	—	—	—	—	—	—
6. Urea	—	—	—	—	—	-	20	8	15	—	—	—
7. Paraformaldehyde	—	5	4	3	—	5	3	3	3	3	3	3
8. Titanium dioxide	5	4	4	3.2	3	3	3	3	3	3	3	3.2
9. Sodium salt of d-mannuronic acid 2% soln.	21	22	22	22	22	22	22	22	22	20	20	22
10. Copolymer of vinyl acetate 2-ethylhexyl acrylate	15	14	16	16	17	17	18	18	20	—	—	16
11. Vinyl acetate acrylic emulsion	—	—	—	—	—	—	—	—	—	19	—	
12. Amino resin	—	—	—	—	—	—	—	—	—	—	17	1
13. Water	10	9	12	7.8	8	8	9	9	10	10	7	7.8

TABLE 2 — RESULTS OF BURNING TEST ON MANGO WOOD

Formulation Nos.	Time of flame spread sec	After glow sec	Weight loss gm	Char index C.C.	Height of intumescence mm
1.	0	0	5.28	6.75	9
2.	0	0	5.08	6.30	9
3.	0	0	3.57	0	14
4.	0	0	2.25	0	16
5.	0	0	3.98,	5.25	15
6.	0	0	2.42	0	16
7.	0	0	3.72	4.92	9
8.	0	0	4.18	5.44	10
9.	0	0	2.30	0	8
10.	0	0	2.30	0	16
11.	0	0	2.24	0	12
12.	0	0	2.06	0	12
Control	42	continues	21.12	50.40	—

TABLE 3 — PERFORMANCE OF COATINGS AS PER B.S. 476 Pt 7 (LARGE SCALE TEST)

Material		Thickness mm	Flame spread at 1½ min mm	Final flame spread at 10 min mm	Classification
MANGO WOOD					
Coated with Paint Formulation	4	12	0	0	Class-1
	6	12	0	0	Class-1
	9	12	0	0	Class-1
	10	12	0	0	Class-1
	11	12	0	0	Class-1
	12	12	0	0	Class-1
Uncoated Specimen	—	12	230	715	Class-3
PLYWOOD					
Coated with Paint Formulation	4	6	0	0	Class-1
	6	6	0	0	Class-1
	9	6	0	0	Class-1
	10	6	0	0	Class-1
	11	6	0	0	Class-1
	12	6	0	0	Class-1
Uncoated specimen	—	6	250	740	Class-3
CELOTEX BOARD					
Coated with Paint Formulation	4	18	0	0	Class-1
	6	18	0	0	Class-1
	9	18	0	0	Class-1
	10	18	0	0	Class-1
	11	18	0	0	Class-1
	12	18	0	0	Class-1
Uncoated specimen	—	18	325	900	Class-4

Class 1 — surfaces of a 'very low' flame spread
Class 3 — surfaces of 'medium' flame spread
Class 4 — surfaces of 'rapid' flame spread

The ingredients excepting the binder were ground in a ball mill to a 325-400 mesh size and to it, a 2% aqueous solution of the sodium salt of d-mannuronic acid was added. The required quantity of the binder was added to the above contents. To this homogeneous mixture, enough water was added in order to obtain brush consistency. The intumescent paints were then stored in an air tight container and were applied with brush on combustible surfaces of plywood, wood, soft board, hard board, and acoustical boards etc. The thickness of the paint film was maintained at 10 to 12 mil for obtaining effective fire retardancy.

Testing and Evaluation of the Fire Retardant Coatings

Flame spread rate, after glow time, char index and weight loss under five exposure are the main criteria of the effectiveness of the paint. Different standard tests such as B.S. 476 Pt 7-1971- surface spread of flame test for materials, B.S. 476 Pt 6-1968- fire propagation test for materials and A.S.T.M. D-1360-1959 fire retardancy of paints (cabinet method) were employed.

The data obtained for specimens with and without fire retardant intumescent paint provided a comparative performance value for particular coating under test.

(a) Fire Performance as per ASTM D-1360 19595 (Cabinet Method)

The test panels with smooth surfaces of size $\frac{1}{4} \times 6 \times 12$ inch ($6 \times 150 \times 305$ mm) free from knots and other imperfections were used. Absolute ethyl alcohol (5 ml) was taken as an ignition fuel. The burning test data for the controlled and the painted specimens are given in table-2.

(b) Fire performance as per B.S. 476 Pt 7-1971 (Large Scale Surface Spread of Flame Tests for Materials)

The specimens were subjected to gas fired radiant panel of approximately 900 sq.m/ and time of spread of the flame front for measured distances along the specimen was recorded, until the flame have died out or for 10 minutes whichever is longer.

The fire performance was evaluated by ASTM D-1360 method and the results indicated that all the formulations were quite effective. Since formulations 4, 6, 9, 10, 11 and 12 gave satisfactory results, they were selected for further studies. Specimens of mango wood of size 230x900x12 mm, ploywood of size 230x900x6 mm and celotex board of size 230x900x18 mm were tested. The results are recorded in Table 3.

(c) Fire Performance as per B.S. 476 Pt 6-1968 (Fire propagation test for materials)[7]

The specimens were subjected to heat in an enclosed space under prescribed conditions and the rate and the amount of heat evolved by them were determined. Plywood specimens of size 228x228x4 mm and celotex board of size 228x228x18 mm were coated with paint formulations and their fire propagation characteristics were evaluated. The results are given in Table 4.

TABLE 4 — PERFORMANCE OF COATINGS AS PER B.S. 476 Pt. 6-1968

Material	Results	
	i_1	I
I.C. No. 4 on 4 mm plywood	2.002	12.775
I.C. No. 6 on 4 mm plywood	2.013	12.842
I.C. No. 9 on 4 mm plywood	2.119	13.115
I.C. No. 10 on 4 mm plywoid	2.056	12.869
I.C. No. 11 on 4 mm plywood	1.983	12.479
I.C. No. 12 on 4 mm plywood	2.046	13.011
Uncoated plywood 4 mm	10.342	30.591
I.C. No. 4 in 18 mm celotex board	2.334	15.130
I.C. No. 6 on 18 mm celotex board	2.339	15.278
I.C. No. 9 on 18 mm celotex board	2.456	15.847
I.C. No. 10 on 18 mm celotex board	2.352	15.298
I.C. No. 11 on 18 mm celotex board	2.310	15.111
I.C. No. 12 on 18 mm celotex board	2.433	15.572
Uncoated celotex board 18 mm	37.291	69.235

i_1 = sub index (initial burning only). The lower the numerical value of the index the better is the material

I = index of performance

I.C. intumescent coating

DISCUSSION

From the results of burning tests, it is evident that different paint formulations are effective fire retardants. The samples which were coated with these paints neither show any surface spread of flame nor after glow or smouldering on exposure. It is also concluded from the burning tests that combinations of pentaerythritol, dicyandiamide and ammonium phosphate produced instant intumescence on exposure and showed minimum weight loss when used in the ratio of 1:1.4:2.4 parts by weight (formulation No. 4), 1:1.5:2.5 (formulation 6), 1:2:2.5 (formulation 10).

When melamine was used instead of dicyandiamide, the performance was found almost to be the same. A satisfactory fire performance was also achieved by using urea instead of dicyandiamide or melamine. However, the required ratio of pentaerythritol, urea and ammonium phosphate was found to be 1:1.5:5:3.5 (formulation 9).

The main constituents of fire retardant intumescent coatings under study are pentaerythritol, ammonium phosphate and dicyandiamide. When this combination is exposed to fire, ammonium phosphate begins to decompose to produce polyphosphoric acid. Pentaerythritol reacts[8] with this acid and evolves a large amount of carbonaceous char which produces a non-combustible barrier for protecting the substrate. Dicyandiamide gives off non-flammable gases causing the foamable carbon to produce a honeycomb blanket resulting in a highly effective insulation. The binder on softening forms an expandable skin over the carbonaceous char to resist the escape of gases produced by dicyandiamide. All these reactions take place within the coating, thereby protecting the material from heat. The above mechanism may be expressed by the following equations:

$$x(NH_4)_2HPO_4 \xrightarrow{heat} HO\left[-\underset{\underset{H}{|}\atop{O}}{\overset{\overset{\vdots}{O}}{\overset{|}{P}}}-O-\right]_x H + 2x\,NH_3 + (x\text{-}1)\,H_2O \quad \ldots (1)$$

Polyphosphoric acid

$$HO\left[-\overset{\overset{\vdots}{O}}{\underset{\underset{H}{O}}{P}}-O-\right]_{x}H + R\,CH_2CH_2OH \rightarrow R\,CH_2CH_2OPO_3H_2 + HO\left[-\overset{\overset{\vdots}{O}}{\underset{\underset{H}{O}}{P}}-O-\right]_{x-1}H \quad \ldots (2)$$

$$R\,CH_2CH_2OPO_3H_2 \xrightarrow[\text{heat}]{\text{more}} R\,CH = CH_2 + H_3PO_4 \quad \ldots (3)$$

$$xH_3PO_4 \xrightarrow{\text{heat}} HO\left[-\overset{\overset{\vdots}{O}}{\underset{\underset{H}{O}}{P}}-O-\right]_{x}H + (x - 1)H_2O \quad \ldots (4)$$

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