Synthesis and performance of new modified reactive flame-retardant alkyd resin based on tetrabromophthalic anhydride as varnish for surface coatings

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Abstract There exist two distinct approaches to achieving flame retardancy in polymers, namely the additive type and the reactive type. In this case, new modified reactive flame-retardant (FR) alkyd resins (short, medium, and long oil alkyd) were produced by means of a condensation polymerization reaction between a linseed oil fatty acid and glycerol, to produce the monoglyceride as the ingredient source of the polyol used. This then reacted with phthalic anhydride, which was partially replaced with tetrabromophthalic anhydride (TBPA), as the ingredient source of the dibasic acid. The resulting resin was characterized using FTIR and ¹H NMR spectral studies. The coatings of $50 \pm 5 \,\mu\text{m}$ thickness were applied to the surface of wood, glass panels, and mild steel strips by means of a brush. The fire retardant capacity of the modified reactive FR alkyd resins was assessed using the limiting oxygen index (LOI) test. The physical, mechanical, and chemical properties were also examined to evaluate any drawbacks associated with the modification. The results of the LOI test indicate that the modified reactive FR alkyd resins exhibit an improved flame retardancy effect following

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Color Science, Analytical Department of Color Science School of Chemistry, Leeds University, Leeds, UK an increase in the TBPA content within the alkyd resin backbone structure. Improvement to the mechanical properties of the modified resins was also observed, but chemical resistance was not significantly changed. Finally, the prepared reactive FR alkyd resins were observed to overcome a range of problems that weaken the attraction of additive FRs namely, poor compatibility, leaching, and the reduction in mechanical properties.

Keywords Flame-retardant coatings, Reactive flameretardant alkyd, Halogenated flame-retardant, Tetrabromophthalic anhydride, Limiting oxygen index

Introduction

Flame retardants (FRs) can be divided into two distinct classes depending upon their use, namely, additive and reactive. The additive compounds dominate the FR market due to their lower processing cost. This is due to the fact that they are physically mixed with, or dissolved in, the material during processing and not chemically reacted. This of course means that the FR can more easily migrate out of the product under certain conditions. The reactive compounds are polymerized with a resin during processing to enable integration into the molecular network structure, and therefore they are far less likely to leach out into the environment until the product is decomposed or burnt.^{1–4}

FRs can be classified as organic material, like phosphorus and halogenated compounds, or inorganic materials like metal oxides, metal hydroxides, and metal borates. Although inorganic FRs offer low cost and a lack of toxic by-products, their effectiveness is relatively poor. Thus, high levels of additives are required to enable this type of retardant to reach the desired level of performance. The addition of additives can cause detrimental effects to key properties of the polymer.⁵⁻⁹ Lu and Hamerton¹⁰ provide an excellent summary of common types of FRs and their respective fire suppressing mechanisms. Shuyu et al.¹¹ also provided a review that focuses on various approaches in development of FR coatings on various substrates in the last decade.

Reactive fire retardants are mainly based on halogen, phosphorus, inorganic materials, and melamine compounds. The application of reactive FRs involves either the design of new intrinsically flame retarding polymers, or modification of existing polymers through copolymerization with a flame retarding unit within the chain, or as a pendent group. The chemical modification of polymers using organohalogen compounds is one of the more common and effective methods for reducing the flammability of composite materials.^{12–15}

Organohalogen compounds are very effective in trapping free radicals, hence removing the capability of the flame to propagate. However, not all of the organohalogen compounds are suitable FRs. Fluorinated compounds are very stable and decomposed at much higher temperature than the polymers burn. On the other hand, iodinated compounds are not very stable and decompose at slightly elevated temperatures. Consequently, only organochlorine and organobromine compounds are suitable as FRs.^{16,17} Chlorine compounds have been studied extensively as flame retarders, with chlorinated polymers found to be in general less FR than brominated polymers, for equivalent halogen content. Therefore, during the last decade, interest has shifted from chlorine flame retarders to bromine flame retarders.¹⁸⁻²⁰ Organic bromine compounds can be either aromatic, aliphatic, or cycloaliphatic compounds and can contain between 50% and 85% bromine by weight.²¹ The incorporation of bromine into the molecular structure of a polymer is the most common method used for improving the flammability resistance of thermoset resins and thermoplastics.

Cost and effectiveness have elevated brominated compounds to the fore with respect to the modern day FR market. For example, in the United States of America between 40% and 50% of the market is taken up by brominated flame-retardants (BFRs).²³ Tetrabromophthalic anhydride (TBPA) is a white to pale yellow powder, or crystalline solid that is mainly used as a FR in plastics, paper, and resins. TBPA is a reactive FR that has been incorporated into the construction of FR polyesters, epoxy resins,²⁴ and urethane foams,²⁵ since the late 1960s. Its high bromine content (68%) enables it to impart a high degree of flame retardancy to the resins.

So for this work we studied the incorporation of TBPA, a powerful FR, into the construction of an FR alkyd resin by means of the partial replacement of phthalic anhydride (PA), as the ingredient source of the dibasic acid. This was undertaken in an attempt to improve the resulting alkyd's flame retardancy properties.

Experimental

Methods and materials

Materials

The linseed oil fatty acid (LOFA) used was supplied by Akzo Nobel based in the Netherlands. The PA and TBPA compounds used were produced by Avondale Laboratories in England. The glycerol (G), sulfuric acid, anhydrous sodium carbonate, and benzene were all supplied by the EINasr Pharmaceutical Company in Egypt. The xylene and mineral turpentine solvents were supplied by the Misr Petroleum Company in Egypt.

Methods and techniques

Synthesis of new modified reactive flame-retardant alkyd resin

Alkvd resins are the reaction products of dibasic acid and polyol. The synthesis of the new modified reactive FR alkyd resins was prepared through a condensation polymerization reaction via a solvent process, in a single-step reaction. Thus, a mixture of a calculated amount of LOFA, phthalic anhdride (PA) [replaced partially with TBPA as the source of polybasic acid ingredient], and anhydrous glycerol (G) as the source of the polyol, was added to a 250-mL round-bottom flask fitted with a Dean-Stark trap and refluxed in the presence of 10% xylene solvent. The course of esterification was followed by observing the theoretical amount of water liberated. The resins were prepared, covering a wide range of oil lengths and hydroxyl content (0%, 10%, 20%, and 30% excess-OH). It should be noted that within each set of formulations, the total number of acid and hydroxyl equivalent were kept constant for each of the individual runs.²⁶ The resin characteristic constant and weight changes for the various formulations are illustrated in Table 1. Alkyd calculations can be used to predict the completed formulation, properties of the resin, the amount of reaction water liberated, and the probability of gelation. The calculation of water evolved is also useful, both as a tool for following the course of the esterification reaction, and as a means to find the theoretical vield. The solid content (ASTM D1644-01) was adjusted by means of thinning with mineral turpentine to 50% solids for the 0%, 10%, and 20% excess-OH samples. However, xylene was used for thinning the 30% excess-OH samples. The resin samples were then filtered and the drier combination of cobalt, zirconium, and calcium octoates, 0.04%, 1.0%, and 0.05%, based on metal/solid resin, respectively, were then added.

Measurements

Chemical structure of the prepared alkyd resin was confirmed using Fourier transform infrared spectro-

Resin no.	Ingredient	eo	e _A	e _B	Е	F	$w = Ee_{o}$	$m_{\rm o} = e_{\rm o}/{\rm F}$	$R = e_{\rm B}/e_{\rm A}$	$K = m_{\rm o}/e_{\rm A}$	H ₂ O off (mL)
la	LOFA	0.111	0.111		280	1	31.08	0.111	1	1.05	2.00
	PA	0.148	0.148		74.1	2	10.97	0.074			1.33
	TBPA										
	G	0.260		0.260	30.7	3	7.98	0.087			
	Total	0.519	0.259	0.260			50.03	0.272			3.33
lb	LOFA	0.111	0.111		280	1	31.08	0.111	1	1.05	2.00
	PA (0.9)	0.133	0.133		74.1	2	9.86	0.066			1.20
	TBPA (0.1)	0.015	0.015		231.86	2	3.48	0.008			0.13
	G	0.260	0.050	0.260	30.7	3	7.98	0.087			0.00
1.	Total	0.519	0.259	0.260	000		52.40	0.272		1.05	3.33
lc		0.111	0.111		280	1	31.08	0.111	1	1.05	2.00
	PA (0.7)	0.104	0.104		74.1 231.86	2	7.71	0.052			0.93
	TBPA (0.3)	0.044 0.260	0.044	0.060		2 3	10.20 7.98	0.022			0.40
	G Total	0.200	0.259	0.260 0.260	30.7	3	7.98 56.97	0.087 0.272			3.33
ld	LOFA	0.111	0.233	0.200	280	1	31.08	0.272	1	1.05	2.00
iu	PA (0.5)	0.074	0.074		200 74.1	2	5.48	0.037	1	1.05	0.67
	TBPA (0.5)	0.074	0.074		231.86	2	17.16	0.037			0.67
	G	0.260	0.074	0.260	30.7	3	7.98	0.087			0.07
	Total	0.519	0.259	0.260	00.7	0	61.70	0.272			3.33
lla	LOFA	0.100	0.100	0.200	280	1	28	0.100	1.10	1.05	1.80
na	PA	0.173	0.173		74.1	2	12.82	0.087	1.10	1.00	1.56
	TBPA	0.175	0.175		/ 4. 1	2	12.02	0.007			1.50
	G	0.300		0.300	30.7	3	9.21	0.100			
	Total	0.573	0.273	0.300	00.7	0	50.03	0.287			3.36
llb	LOFA	0.100	0.100	0.000	280	1	28	0.100	1.10	1.05	1.80
110	PA (0.9)	0.156	0.156		74.1	2	11.56	0.078	1.10	1.00	1.41
	TBPA (0.1)	0.017	0.017		231.86	2	3.94	0.009			0.15
	G	0.300	0.017	0.300	30.7	3	9.21	0.100			0.10
	Total	0.573	0.273	0.300	0011	Ũ	52.71	0.287			3.36
llc	LOFA	0.100	0.100	0.000	280	1	28	0.100	1.10	1.05	1.80
	PA (0.7)	0.121	0.121		74.1	2	8.97	0.061			1.09
	TBPA (0.3)	0.052	0.052		231.86	2	12.06	0.026			0.47
	G	0.300		0.300	30.7	3	9.21	0.100			
	Total	0.573	0.273	0.300		-	58.24	0.287			3.36
lld	LOFA	0.100	0.100		280	1	28	0.100	1.10	1.05	1.80
	PA (0.5)	0.087	0.087		74.1	2	6.45	0.044			0.79
	TBPA (0.5)	0.086	0.086		231.86	2	19.94	0.043			0.77
	G	0.300		0.300	30.7	3	9.21	0.100			
	Total	0.573	0.273	0.300			63.6	0.287			3.36
Illa	LOFA	0.082	0.082		280	1	22.96	0.082	1.2	1.04	1.48
	PA	0.216	0.216		74.1	2	16.01	0.108			1.94
	TBPA										
	G	0.358		0.358	30.7	3	10.99	0.119			
	Total	0.656	0.298	0.358			49.96	0.309			3.42
IIIb	LOFA	0.082	0.082		280	1	22.96	0.082	1.2	1.04	1.48
	PA (0.9)	0.194	0.194		74.1	2	14.38	0.097			1.75
	TBPA (0.1)	0.022	0.022		231.86	2	5.10	0.011			0.19
	G	0.358		0.358	30.7	3	10.99	0.119			
	Total	0.656	0.298	0.358			53.43	0.309			3.42
IIIc	LOFA	0.082	0.082		280	1	22.96	0.082	1.2	1.04	1.48
	PA (0.7)	0.151	0.151		74.1	2	11.19	0.076			1.36
	TBPA (0.3)	0.065	0.065		231.86	2	15.07	0.032			0.58
	G	0.358		0.358	30.7	3	10.99	0.119			
	Total	0.656	0.298	0.358			60.21	0.309			3.42

Table 1: Formulation calculation of new modified (TBPA) reactive flame-retardant alkyd resins

Table 1 Continued

Resin no.	Ingredient	$e_{\rm o}$	e _A	$e_{\rm B}$	Е	F	$w = Ee_{\rm o}$	$m_{\rm o} = e_{\rm o}/{\rm F}$	$R = e_{\rm B}/e_{\rm A}$	$K = m_{\rm o}/e_{\rm A}$	H ₂ O off (mL)
llld	LOFA	0.082	0.082		280	1	22.96	0.082	1.2	1.04	1.48
	PA (0.5)	0.108	0.108		74.1	2	8.00	0.054			0.97
	TBPA (0.5)	0.108	0.108		231.86	2	25.04	0.054			0.97
	G	0.358		0.358	30.7	3	10.99	0.119			
	Total	0.656	0.298	0.358			66.99	0.309			3.42
IVa	LOFA	0.055	0.055		280	1	15.40	0.055	1.3	1.01	0.99
	PA	0.284	0.284		74.1	2	21.04	0.142			2.56
	TBPA										
	G	0.439		0.439	30.7	3	13.48	0.146			
	Total	0.778	0.339	0.439			49.92	0.343			3.55
IVb	LOFA	0.055	0.055		280	1	15.40	0.055	1.3	1.01	0.99
	PA (0.9)	0.256	0.256		74.1	2	18.97	0.128			2.31
	TBPA (0.1)	0.028	0.028		231.86	2	6.49	0.014			0.25
	G	0.439		0.439	30.7	3	13.48	0.146			
	Total	0.778	0.339	0.439			54.34	0.343			3.55
IVc	LOFA	0.055	0.055		280	1	15.40	0.055	1.3	1.01	0.99
	PA (0.7)	0.199	0.199		74.1	2	14.75	0.099			1.79
	TBPA (0.3)	0.085	0.085		231.86	2	19.71	0.043			0.77
	G	0.439		0.439	30.7	3	13.48	0.146			
	Total	0.778	0.339	0.439			63.34	0.343			3.55
IVd	LOFA	0.055	0.055		280	1	15.40	0.055	1.3	1.01	0.99
	PA (0.5)	0.142	0.142		74.1	2	10.52	0.071			1.28
	TBPA (0.5)	0.142	0.142		231.86	2	32.92	0.071			1.28
	G	0.439		0.439	30.7	3	13.48	0.146			
	Total	0.778	0.339	0.439			72.32	0.343			3.55

 e_o , total equivalents present at the start of the reaction; e_A , number of acid equivalents; e_B , number of hydroxyl equivalents; E, equivalent weight; F, functionality; W, weight; m_o , total moles present at the start of the reaction; R, ratio of total –OH groups to total –COOH group; and K, alkyd constant (m_o/e_A)

scopy recorded in polymer/KBr pellets using Shimadzu FTIR 400 spectrometer, in conjunction with ¹H NMR spectra were recorded on a Varian Gemini 300 MHz spectrometer using DMSO solvent. The color (ASTM D 1544-04) of the prepared alkyd, and the new modified (TBPA) reactive FR alkyd resins, was undertaken using the Gardner 1953 standard color. Viscosity of the resin samples (ASTM D 1545-07) was established by using Gardner-Holt viscosity tubes.

Film casting and testing

A range of chemical, physical, and mechanical evaluations of the painted films were undertaken according to appropriate ASTM standard test methods. The chemical resistance (ASTM D 870-02, D 2792-04) of the dried resin coatings, to different solvent media, was carried out on glass panels (25×75 mm). Glass plates (100×150 mm) coated with the individual resins were utilized to measure the drying schedule, in terms of time of set-to-touch and dry-through times (ASTM D 1640-03). In addition, the same coated glass plates were used to measure the degree of gloss at an angle of 60° for the individual resin coatings (ASTM D 523-08), and to measure film hardness by means of the pencil test (ASTM D 3363-00). The prepared steel panels (ASTM D 609-95) were used to measure the film coating thickness (ASTM D 1005-07), the adhesion 'cross hatch' test (ASTM D 3359-02), the flexibility 'bend' test (ASTM D 522-93a), and the resistance to mechanical damage 'impact resistance' (ASTM D2794-04).

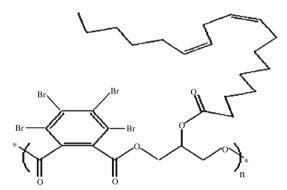
Flame-retardant testing method

The performance of the new modified (TBPA) reactive FR alkyd resins, was evaluated in a limited oxygen index (LOI) chamber. LOI values were determined using the standardized test methods ISO 4589-1:1996 and ASTM D 2863-06. The test panels were prepared using a combustible material (wood specimen). It was important that the panels were free from any surface contamination, or imperfections, prior to the coating application. Hand tool cleaning (sand paper) was used to treat the faces and edges of the individual panels. The final dry film thickness (DFT) of the panels was $50 \pm 5 \ \mu\text{m}$. In all cases, the film application was undertaken by means of conventional brushing. Following air drying for a period of 10 days, the panels under study were heated at a temperature range of 50-60°C for 2 h to eliminate any residual solvent.

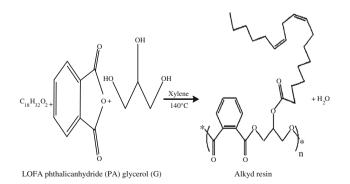
Results and discussion

Synthesis and spectral analysis of alkyd resin

In the present investigation, new modified reactive FR alkyd resins were prepared in the hope that they might demonstrate enhanced flame retardancy properties. For this series of resins, all were prepared and purified as outlined in "Experimental" section. The chemical structures of alkyd and new modified FR alkyd resin are represented in Schemes 1 and 2. Structural features of the alkyd resin were obtained using FTIR spectroscopy and ¹H NMR spectroscopy. This combination of techniques was utilized for structure confirmation (see Figs. 1, 2, respectively). The key FTIR functional groups observed [OH group at 3456 cm⁻¹ (CH) CH₂ asymmetric stretching group at 2958 cm⁻¹, symmetric stretching group at 2856 cm⁻¹, the CH₂ bending group at 1452 cm⁻¹, and the strong C=O carbonyl stretching group around 1734 cm⁻¹] are all typical for an alkyd resin. Also present on the resin spectrum are C-H bending, C-O and C-C stretching fingerprint peaks, specifically the peak at 1260 cm⁻¹ (likely to be due to the esters) and the peak at 1122 cm⁻¹. The peaks at 1600 and 1580 cm⁻¹ (doublet—due to stretching of the aromatic ring at 1070 cm^{-1}) correspond to unsaturated



Scheme 2: Chemical structure of reactive flame-retardant alkyd resin containing LOFA, TBPA, and G



Scheme 1: Chemical structure of an ideal alkyd resin containing LOFA, PA, and G

aromatics in plane deformation. Finally, the peaks at 748 and 705 cm⁻¹ corresponding to aromatic out of plane bending can be attributed to the polyester portion of an alkyd. The ¹H NMR spectrum was measured in DMSO- d_6 solvent and showed clear peaks at $\delta = 1.2$ –1.6 ppm (CH₂ of fatty acid chain), 4.1–4.2 ppm (glycerol CH₂, –CH), 5.4 ppm (hump, 2H, 2OH), and 7–8 ppm (m, 3H, Ar–h).

Evaluation of film properties

Evaluation the flame retardancy of the prepared reactive flame-retardant alkyd resins

The FR properties of the prepared reactive FR alkyd resins were evaluated by means of the limiting oxygen index (LOI) test. This test method was first proposed in 1966 by Fenimore and Martin²⁷ and is used to indicate the relative flammability of materials.²⁸ Standardized in France (NF T 51-071) and in the United States (ASTM D 2863), the LOI test is now subject to an international standard (ISO 4589). The LOI is defined as the minimum concentration of oxygen, expressed as a percentage that will support combustion of a polymer. It is measured by passing a mixture of oxygen and nitrogen over a burning specimen, and reducing the oxygen level until a critical level is reached.²⁹ The LOI is expressed as

$$\text{LOI} = 100 \frac{[\text{O}_2]}{[\text{O}_2] + [\text{N}_2]}.$$

This method proved suitable as a semiguantitative indicator of the effectiveness of the FR during the research and development phase of the work. This was due to the fact that the equipment is relatively inexpensive and the size of the test sample that is required is reasonably small. It is clear that the incorporation of TBPA into the backbone of short, medium, and long oil alkyd resins, in the ratio's mentioned in "Experimental" section, results in enhanced flame retardancy when compared with an alkyd control sample. The results obtained from the LOI test are shown in Table 2 and Figs. 3 and 4. It can be observed that the LOI value of the control alkyd resin (minus TBPA) is 18 and the maximum LOI with TBPA present is 31 (for formula IVd). Normal atmospheric air (i.e., the air that we inhale) contains approximately 21% oxygen, so a material with an LOI of less than 21% would burn easily in air. In comparison, a material with an LOI value greater than 21%, but less than 27%, would be considered to be 'slowburning.' However, a selfextinguishing material is one that has a LOI greater than 27% and this would stop burning after the removal of the fire, or the ignition ³⁰ The increased content of TBPA from source. formula 1b to formula IVd, increases the LOI value from 20 to 31. This is due to the incorporation of the

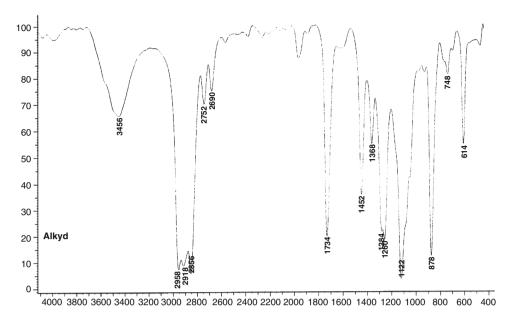


Fig. 1: IR spectra of the prepared alkyd resin

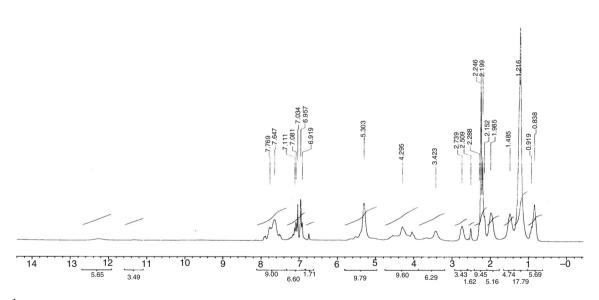


Fig. 2: ¹H NMR spectra of the prepared alkyd resin

TBPA into the backbone of the alkyd resin, resulting in: (i) a more stable chemical structure with a higher molecular weight, leading to a higher flame retardancy; (ii) the bond strength between the chains is a key factor for controlling flame retardancy of polymers, and the binding of bromine to the polymeric backbone by covalent bonds improves the flame retardancy of the polymers; (iii) the flame retardancy is increased by minimizing the presence of hydrogen because of its low bond enthalpy with carbon; and (iv) the bromine radical is much less active than the hydrogen radical, which slows the combustion process.

Evaluation of the physical and mechanical properties of the prepared reactive FR alkyd resins

The effects of incorporating TBPA into the alkyd resin backbone structure were evaluated using a variety of standard test methods for gloss, viscosity, hardness, adhesion, flexibility, and impact strength. These test methods were utilized to determine any negative aspects that might occur due to the incorporation of the TBPA into the alkyd resin structure. The physical and the mechanical characteristics of the reactive FR alkyd resins are presented in Tables 3 and 4 respec-

tively. First, all short, medium, and long oil alkyd resin compositions gave a very clear transparent and homogeneous appearance. The results show that the alkyd resins' time of reaction and viscosity increase with the higher concentrations of TBPA, due to the increase in molecular weight of the alkyd resin. However, the alkyd resin color also increases with the higher concentrations of the TBPA due to the presence of the bromine atoms within the alkyd resin backbone structure. Gloss result shows that the gloss increased with the higher concentrations of TBPA content. Thus, this is a positive result and may be attributed to the introduction of the lone pair of electrons on the bromine atom on the aromatic ring. The scratch hardness is observed to vary between H and 8H and it is clear from the data that the alkyd resistance toward scratch hardness increased in proportion with

Table 2: Flame retardant characteristics of reactive flame-retardant alkyd resin

Resin no.	0% excess-OH	Replacement of TBPA	Limiting oxygen index (LOI)		
la	0	0	18		
lb		10	20		
lc		30	23		
ld		50	25		
lla	10	0	18		
llb		10	21		
llc		30	26		
lld		50	28		
Illa	20	0	18		
IIIb		10	23		
IIIc		30	27		
IIId		50	29		
IVa	30	0	18		
IVb		10	24		
IVc		30	28		
IVd		50	31		

the level of TBPA content. Adhesion was improved with the increase of TBPA content; this may be attributed to the binding of bromine atoms to the polymeric backbone, by means of covalent bonding. The flexibility (bending) test shows that, the film for all the coating compositions passed the Mandrel bend test, with no obvious significant change being observed for the modified resins. Thus, it can be stated that all the films produced showed reasonably good flexibility. Resistance to mechanical damage (impact resistance) was shown to improve impact strength with the increased TBPA content. Again, this positive result can be attributed to the binding of the bromine atoms to the polymeric backbone by means of covalent bonding, which improves the polymer-substrate adhesion.

Evaluation of the chemical properties of the prepared reactive flame-retardant alkyd resins

The chemical resistance of the prepared reactive FR resins was undertaken using glass panels (25×75 mm). The coated glass panels were sealed,

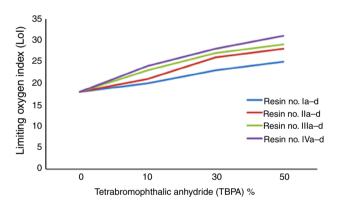


Fig. 4: Limiting oxygen index (LOI) characteristics of reactive flame-retardant alkyd resin



Fig. 3: Representative samples of coated wood specimens before ignition

Resin no.	0% excess- OH	Replacement % of TBPA	Time of reaction (min)	Color (Gardner)	Viscosity (Gardner)	Gloss at 60°
la	0	0	300	11	А	76
lb		10	315	12	В	81
lc		30	320	14	В	83
ld		50	330	15	D	91
lla	10	0	270	12	С	77
llb		10	285	13	D	79
llc		30	300	15	E	84
lld		50	320	16	G	87
Illa	20	0	250	12	D	82
IIIb		10	265	15	F	85
IIIc		30	290	17	Н	88
IIId		50	310	18	J	89
IVa	30	0	220	13	E	86
IVb		10	245	15	G	91
IVc		30	260	18	I	93
IVd		50	285	>18	Ν	93

Table 3: Physical properties of reactive flame-retardant alkyd resins

Table 4: Mechanical properties of reactive flame-retardant alkyd resins

Resin no.	0% excess- OH	Hardness	Adhesion	Flexibility	Impact (J)
la	0	Н	2B	Pass	1.82
lb		Н	3B	Pass	1.89
lc		2H	3B	Pass	1.95
ld		4H	4B	Pass	2.05
lla	10	Н	2B	Pass	1.82
llb		2H	3B	Pass	1.92
llc		ЗH	4B	Pass	1.98
lld		5H	4B	Pass	2.10
Illa	20	2H	3B	Pass	1.85
IIIb		4H	3B	Pass	1.97
IIIc		5H	4B	Pass	2.05
IIId		6H	4B	Pass	2.12
IVa	30	ЗH	3B	Pass	1.90
IVb		5H	4B	Pass	2.05
IVc		7H	4B	Pass	2.15
IVd		8H	5B	Pass	2.20

using paraffin wax at the edges, and immersed to half their length in each individual test solution: (i) 5 wt% anhydrous sodium carbonate in water, (ii) 10 wt% sulfuric acid, and (iii) 1:3 by volume benzene/mineral turpentine solvent mixture. The panels were then removed from the individual test solution, wiped carefully and then allowed to dry at room temperature, prior to assessment of any change. The obtained data indicates that the chemical resistance was not significantly changed by the resin modification.

Conclusion

TBPA is well known as a powerful reactive FR and this study focused on the incorporation of this component into the construction of a reactive FR alkyd resin as the dibasic acid source. The nonflammability of the prepared reactive FR alkyd resins was characterized by means of the LOI test method. The physical, mechanical, and chemical properties were examined to assess any negative aspects associated with the addition of the TBPA. The incorporation of TBPA into short, medium, and long alkyd resins results in significantly enhanced flame retardancy properties, when compared to a control alkyd containing no TBPA. This improvement may be attributed to a number of key factors. First, the introduction of high molecular weight TBPA, containing 68% bromine, enables it to impart a high degree of flame retardancy to the resulting resins. Second, the bond strength between the chains is an important factor controlling the flame retardancy of the polymer band. It is also known that the binding of bromine to the polymeric backbone by covalent bonding improves the flame retardancy of the polymer (also referred to as the reactive route). This makes the polymer itself fire retardant. Third, the flame retardancy is increased by minimizing the presence of hydrogen because of its low bond enthalpy with carbon. In addition, the bromine radical is much less active than the hydrogen radical which slows the combustion process. The results obtained indicate that the FR capability and mechanical properties improve with an increase in the TBPA content within the alkyd resin backbone structure. However, chemical resistance was not significantly changed by this modification. Finally, the prepared reactive FR resins overcome a variety of problems that weaken the attraction for using additive FRs, namely poor compatibility, leaching, and a reduction in mechanical properties.

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