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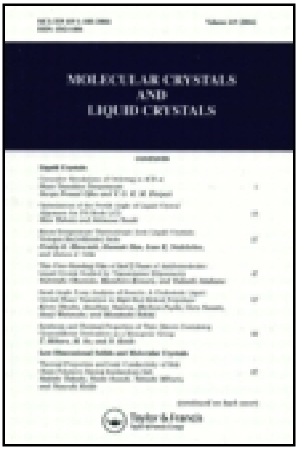

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Combustion Characteristics of Wood Protected by Intumescent Coatings and the Influence of Different Additives on Fire Retardant Effectiveness of the Coatings

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Combustion Characteristics of Wood Protected by Intumescent Coatings and the Influence of Different Additives on Fire Retardant Effectiveness of the Coatings

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Intumescent coatings make the most efficient way of fire retarding of flammable materials. The coatings swell under the influence of heat and form a thick porous charred layer. The latter perfectly insulates a substrate against excessive increase of temperature and oxygen access. In order to make intumescent coatings more effective, a proper selection of essential components, i.e. carbonising, foam-producing and dehydrating materials, is necessary. The choice of components for an intumescent fire retardant composition has an essential effect on the rate of charred mass formation and its structure.

In the paper, the influence of different modifiers, added to amino resins during the process of condensation, on fire retardant and heat-insulating efficiency of intumescent coatings for wood, is presented. The fireproofing efficiency was studied by cone calorimeter. This paper also presents the results of thermal analysis of samples dynamically heated from 298K to 823K in the presence of oxygen. The results are presented in the form of DTA, TG and DTA curves.

Keywords: fire retardant; intumescent coatings; cone calorimeter

INTRODUCTION

Intumescent coatings are gaining more and more importance to fire retardancy of combustible materials. When exposed to heat, they form a carbonaceous foam of cellular structure with simultaneous release of incombustible gases which effectively protect wood against the access of oxygen and flame spread[3,4,5].

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Our study was aimed at determining the effect of different compounds, introduced to amino resins in the process of their condensation, on fire retardant and heat insulation efficiency of intumescent coatings for wood. Cone calorimeter and analysers for thermogravimetry and differential thermal analysis were used for this purpose.

EXPERIMENTAL

Synthesis of coat-forming material

Successful attempts at preparing new amino resins of increased resistance to fire were made in this study. This aim was achieved by performing the condensation in the presence of monoammonium phosphate. The resins obtained in this way can play the role of a coat-forming material which at the same time is a subject to carbonisation - the source of carbon. It was assumed that the presence of urea and dicyandiamide in the product of condensation can result in the appearance of intumescent properties. Moreover, it was supposed that ammonium salt used, which is known for its fire retardant properties, can also function as a dehydrating agent. In order to strengthen the structure of foam, char-forming compounds, such as dextrin or pentaerythritol, were also introduced to the resins[*)].

The following components, most frequently present in the intumescent systems, were chosen for the study[1,2,6]

- dextrin, pentaerythritol, - as polyhydric carbonizing materials
- urea, dicyandiamide - as foam producing materials
- monoammonium phosphate - as acidic dehydrating agent and, at the same time, esterification catalyst.

[*)] *The resin synthesis is protected by Polish Patent P 306 789/1995 „Instumescent Fire Retardant and the Way of Its Manufacture".*

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Methods of investigation

To determine the effect of selected components on fire retardant efficiency and heat-insulating ability of intumescent coatings for wood, the following studies were carried out:

- Fire retardant efficiency of coatings as a function of modifiers used. The method for evaluation of this property was
  - *Cone Calorimeter*: The samples were exposed to heat flax of 35 kW/m^2 in the vertical position. A spark igniter was used to start gas combustion.
- Thermal transformations of resins synthesised by the authors. Methods for characterising the above transformations were:
  - *Thermogravimetric analysis* [TGA, DTGA] and differential thermal analysis [DTA].

    The analyses were carried out in two unrelated furnaces: in air under atmospheric pressure in an open platinum crucible. Heating rate was 5°C/min until reaching the final temperature of 823K.

RESULTS

Results of studies of fire retardant efficiency of coatings by using cone calorimeter

Wood covered with amino resins

Figure 1 shows heat release rate (HRR) curves for uncoated pine wood and that coated with amino resins: urea-formaldehyde [D1], dicyandiamide-formaldehyde [D5] and urea-dicyandiamide-formaldehyde [D2] ones. All curves recorded during the combustion of samples studied have similar shapes. A comparison of HRR curves recorded during the investigation of amino resin-coated wood with the respective curve for uncoated pine wood shows that the course of the mentioned curves is similar.

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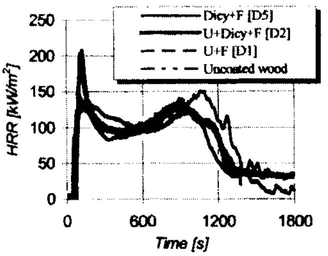

FIGURE 1. Heat release rate curves for wood protected with amino resins

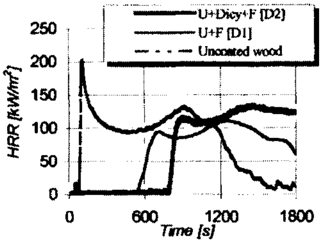

FIGURE 2. Heat release rate curves for wood protected with amino-phosphate-formaldehyde resins

Wood coated with amino resins with monoammonium phosphate added

In Figure 2 HRR curves are presented for wood coated with amino-phosphate resins, namely dicyandiamide-phosphate-formaldehyde resin [D6] and urea-dicyandiamide-phosphate-formaldehyde resin [D3]. The course of the above curves is different from those discussed earlier. They distinguish by a long segment of initial heating of samples with no increase in heat release rate. It is caused, on the one hand, by dilution of combustible gases, evolved from wood, with non-flammable gaseous products of the decomposition of amino-phosphate coatings. On the other hand, an insulating carbonaceous foam is formed on the surface of wood. After 575 and 788 seconds for dicyandiamide-phosphate-formaldehyde coating [D6] and urea-dicyandiamide-phosphate-formaldehyde coating [D3], respectively, released gases burst into flames and specific extinction area decreases rapidly. Right away after the flash point, heat release rate increases to reach extreme value of 123 kW/m^2 for D6 coating and 146 kW/m^2 for D3 coating.

Wood coated with amino resins supplemented by monoammonium phosphate and dextrin

Analysis of HRR (Fig. 3) as well as of parameters presented in Fig. 4 brings into conclusion that the introduction of dextrin together with monoammonium

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phosphate into amino resins results in the formation of a very efficient fire retardant coating. Wood protected by urea-dicyandiamide-phosphate-formaldehyde resin with dextrin added to the latter [D4] did not ignite during the entire measurement period, i.e. for 30 minutes. The carbonaceous foam formed on the surface results in a good heat insulation of the substrate and protects it against thermal decomposition. HRR shows linear behaviour and almost does not increase for all the time of investigation. Average heat release rate amounts to 0.83 kW/m^2 and average mass loss rate to 1.54 g/m^2×s. Total heat released and effective heat of combustion are also relatively low, namely 4.43 MJ/m^2 and 0.59 MJ/kg, respectively. These values indicate that the discussed coating absorbs large amount of heat and its heat conduction is low.

Wood protected by dicyandiamide-phosphate-formaldehyde resin supplemented by dextrin [D7] is characterised by very good fire retardant properties for 15 minutes. After the mentioned period of time the gases burst into flames and this is followed by a rise in heat release rate and in mass loss rate (MLR). Although the carbonaceous foam created on wood surface was comparable to that made by D4 resin, the discussed coating characterises by a lower fire retardant efficiency.

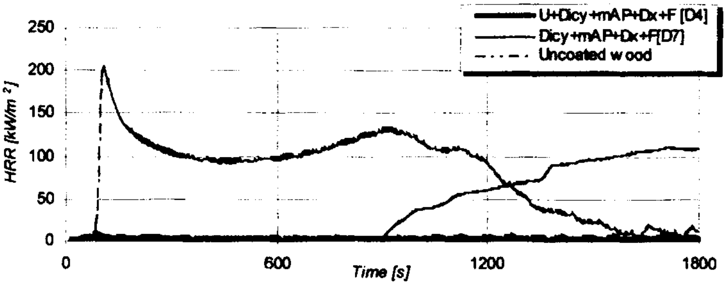

FIGURE 3. Heat release rate curves for wood protected with amino-phosphate-formaldehyde resins supplemented by dextrin.

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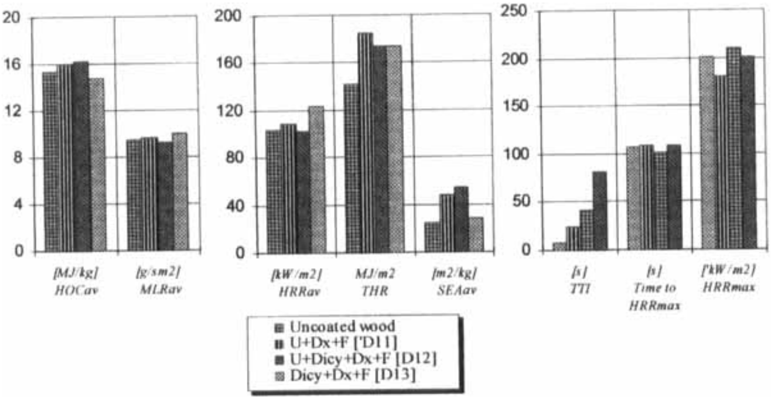

FIGURE 4. Combustion characteristic of wood protected with amino-phosphate-formaldehyde resins supplemend by dextrin

Determination of the effect of particular compounds on the course of thermal decomposition of resins

While analysing the course of thermal decomposition of amino resins investigated, it was found that decomposition courses of dicyandiamide resin (D5) and urea-dicyandiamide resin (D2) are similar. An increased dynamics of the decomposition at the first stage points to the presence of unsaturated systems. It is confirmed by the exothermic character of DTA curves, which suggests the occurrence of reactions unrelated to the thermal decomposition. The urea condensation resin (D1) contains small amounts of low-molecular mass products as indicated by slow rate of mass loss at the first stage of thermolysis and the absence of markedly pronounced exothermic and endothermic effects on DTA curve.

The addition of dicyandiamide increases thermal stability of the condensation product because it shifts the range of active thermolysis towards higher temperatures with simultaneous reduction in the extent of thermal destruction in this range.

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The course of thermal decomposition of the amino resins undergoes a significant change after the addition of monoammonium phosphate (D3, D6). The presence of the latter compound shifts given stages of decomposition by about 40K towards lower temperatures. The rate of decomposition decreases considerably to about 0.2-0.1 mg/min, i.e. over 2.5 times. The amount of incombustible residue after thermal analysis increases from several to 34%.

The decomposition of amino-phosphate resins occurs in a very similar way, irrespectively of the kind of polyhydric compound used. The similarity between courses of the curves suggests that similar processes proceed during the combustion, no matter whether amino-phosphate-formaldehyde resin contains dextrin or pentaerythritol. The course of TGA and DTGA curves indicates that the decomposition of the resins in the range of low temperatures (up to 415K), which is accompanied by mass loss of 8%, corresponds to the release of free formaldehyde as evidenced by an endothermic loop on DTA curve. In the range of 415-550K, active thermolysis with maximum rate of 0.09 mg/min at 513K, results in mass loss of 49%. Further course of the decomposition has the form of a plateau, i.e. no thermal effects are observed on DTA curves.

CONCLUSIONS

1. Coating prepared on the basis of urea-dicyandiamide resin, additionally containing monoammonium phosphate and dextrin, appeared to be the most efficient one both from the point of view of fire retardancy and heat-insulating properties.
    - Urea and dicyandiamide when reacted with formaldehyde give a coat-forming material which, on the one hand makes the source of carbon, and on the other, swells. This increases heat-insulating properties of coatings and reduces heat release rate (HRR) and mass loss rate (MLR).
    - Monoammonium phosphate causes the elimination of exothermic effects, decreases the amount of incombustible residue, reduces HRR

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and MLR and diminishes total heat released (THR) and effective heat of combustion (HOC).

- Dextrin, by itself, does not contribute to intumescent properties of coatings, but when combined with phosphate, it strongly increases the above properties and, in particular, strengthens the structure of foam created.

2. Wood covered with coating prepared on the basis of urea-dicyandiamide-phosphate-formaldehyde resin supplemented by dextrin does not ignite when exposed to heat flux, does not show increased HRR, MLR and HOC.
3. Coatings formed from amino-phosphate-formaldehyde resins with the addition of polyhydric compound intumesce evenly making a fine cellular carbonaceous foam which is characterised by low thermal conduction and very good heat-insulating properties. Moreover, the decomposition of coating is accompanied by the release of incombustible gases which cut off oxygen access.

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