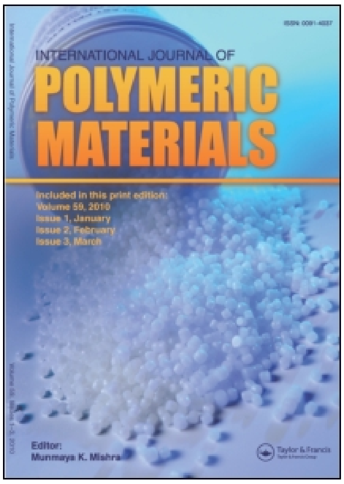

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Thermo-Protecting Properties of Intumescent Compositions

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Intumescent fire retardants have wide applications due to thermo-protective properties, which decrease heat flow on the polymer surface. In this work we present the experimental investigation of thermal-protection properties of a number of polymer mixtures (polycarbonate + ABS plastic, epoxy resin, *etc.*) with different fire retardant additions.

Keywords: Fire retardants; intumescent additives; polymer mixtures; polycarbonate; ABS; epoxy resins

Usually, an intumescent composition in a multi-components composition, creating under the influence of an external thermal source the volume carbonized residue, *i.e.*, a foamed coke. Thickness of the foamed cokes multiply exceeds thickness of the initial composition. Schematically the process is represented in Figure 1.

The following stages of char foam formation process are consequently represented: system heating until the temperature of beginning of destruction of the coating material components, formation of the foam layer and coke frame.

There exists two basic types of the foaming system:

1. Systems in which the polymer binding participates in the coke formation. Polycarbonate and epoxy resin belong to such systems.

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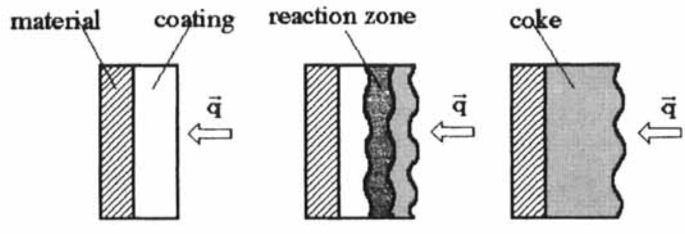

FIGURE 1 Consequent stages of foam formation.

2. Systems in which the polymer does not participate in the coke formation. In these compositions foam formation is provided by the special addition, *i.e.*, by foam formation system.

This foaming process goes through two stages. At the first stage the system transforms from solid into high elastic state. Simultaneously, there is a distraction of composition substances with arise of large amount of gaseous and volatile products, which foam the melted mass. At the second stage the system transforms into solid stage again, but this is related with foam hardening during carbonization reactions and exactly at this time the final formation of coke char takes place [1].

The mechanism of intumescent system action under influence of external heat flow possess specific features. The most important of them are the following:

- Change of polymer thermal-destruction in the direction of foam coke formation.
- Thermal-isolation of polymer surface by coke char from inverse heat flow.
- Obstacle for fuel diffusion into the gas-phase reactions zone.

After analysis of large number of data in literature one can propose the following ways of increasing efficiency of fire retardant properties of intumescent compositions.

- Rising of yield of the carbonized residue.
- Increasing of coke high-melting.
- Deterioration of heat conductivity of foamed cokes.
- Increasing of thickness of carbonized layer.

An intumescent system consists of:

- foamed formations – substances for forming of carbonized residue;
- gas-formations – compounds, which are decomposed with yielding gaseous and volatile substances, which foam the material;
- additional components – plasticity agents, stabilizers, dyes, inhibitors and *etc.*

EXPERIMENTAL METHODS

Thermal-protective properties of the material were estimated by the following method: on the back side of a sample in the form of a plate of definite thickness, a thermocouple is placed and the material are treated by laser radiation.

Thermocouple indications in independence on time were fixed by a computer system (Fig. 2).

Measurements of density and porosity of foamed cokes were carried out by dilatometer. Beforehand weighed sample were coated by silicone glue covering. The volume were determined by immersion of the sample in water. Density of foamed cokes were determined by equation $\rho = m/V$, when m and V are measured mass and volume, respectively. To find porosity, the coke char were pressed into a tablet, so that its volume could be determined. The porosity was calculated by the formula $P = V_{coke}/V_{char}$, where V_{coke} and V_{char}-correspond to coke volume and pressed coke char volume. Density of coke material were determined after preliminary pressing of material into a tablet.

RESULTS AND DISCUSSION

As a polymer basis for studied samples the following materials were used: polycarbonate (PC) trade-mark ≪Diphlon≫; produced by polycondensation di-phenyl-propane and phosgene; the mixture of PC with acrylo-nitrile-butadiene-styrolene plastic (ABS) trade-mark ABS – 2020 with mass ration 3 : 1 and with polyethylentherephthalate (PET) with mass ration 2 : 1; epoxy resin ED-20, hardened by polyethylene-polyamine; epoxy resin, hardened by polyamide trade-mark PO – 300. As fire retardants were used: bromine-contained fire retardant

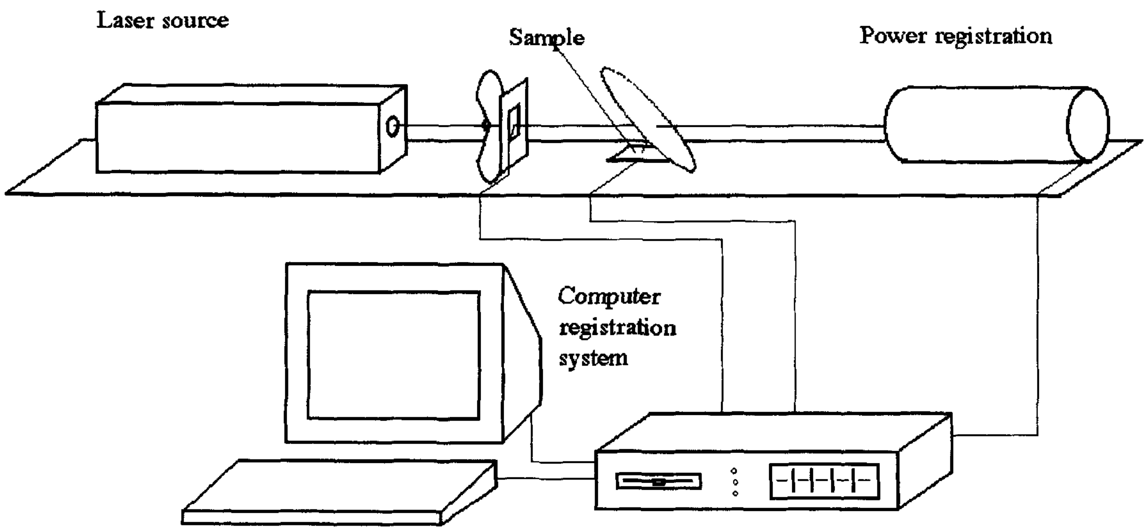

FIGURE 2 Plant for investigation of the thermo-protection properties.

on the basis of tetra-bromine-biphenol and phosgene. In fact, we used as a fire retardants: polycarbonate with contents of bromine ~50% (BFR), phosphorous containing fire retardant 3-phenilphosphate (PhFR) (content of phosphorous is equal 5%), phosphorous-bromine containing fire retardant tris-(polybrom-2-naphthyl) phosphite with bromine content 63,5% and phosphorous content 4% (PhBFR).

Foam formation systems consisted of pentaerythritol, ammonium polyphosphate, urea and boric acid at different rations.

For determination of efficiency of different fire retardants the element analysis of the cokes was carried out. The results are presented in the Table I.

From the presented data one can see that the chemistry compositions of cokes differ from each other slightly, through the percentage of coke residue yields are changed essentially. This proves different structure of foamed coke. Data on foamed cokes structure are presented in the Table II.

From the presented data it is seen, that the addition of fire retardants increases effective density of cokes and this, in turn, reduces its porosity. The coke structure becomes more fine and dense.

TABLE I The element content of the cokes

Composition	*Quantity of fire retardant*, %	*Yield of coke*, %	*Element content of coke*, %			
			C	*H*	*Br*	*P*
PC/ABS + BFR	13	11	71	5	0.5	–
PC/ABS + PhFR	12	17	81	4	–	0.35
PC/ABS + BFR	6	32	81	4	0.2	0.31
PC/ABS	–	8	79	6	–	–
PC/PET + BFR	12	14	61	5	0.7	–
PC/PET + PhFR	11	22	77	5	–	0.93
PC/PET + PhBFR	6	35	81	4	0.3	0.36
PC/PET	–	11	75	5	–	–

TABLE II Density and porosity of cokes chars

Composition	*Density of coke char*, g/sm^3	*Porosity*, %
PC/ABS + BFR	0.16	88
PC/ABS + PhFR	0.18	85
PC/ABS + PhBFR	0.17	86
PC/ABS	0.11	92
PC/PET + BFR	0.21	83
PC/PET + PhFR	0.18	85
PC/PET + PhBFR	0.16	88
PC/PET	0.10	92

Influence of fire retardants on thermo-protective properties of studied compositions are shown in the Figure 3.

Comparison of retardant properties shows, that addition of fire retardant at the beginning stage improves thermo-protective properties.

In the case of epoxy connectors (Figs. 4), addition of HDHCl to an intumescent system essentially changes the structure of foamed coke skeleton. Appearing during the process of formation of foamed coke layer the vapor–gas cavern has significantly smaller size at the presence of some quantity (a few percents) of HDHCl. Perhaps, this is

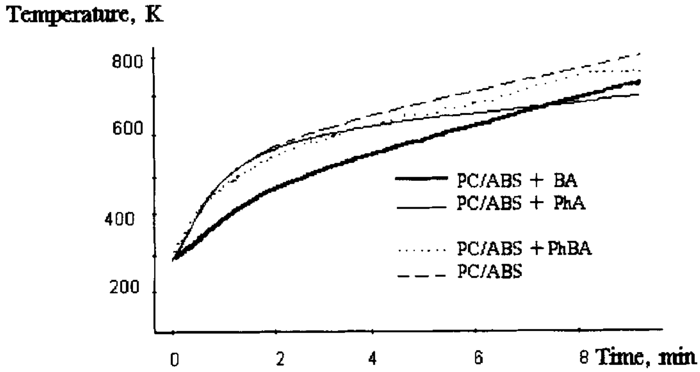

FIGURE 3 Thermo-protection properties of compositions PC/ABS.

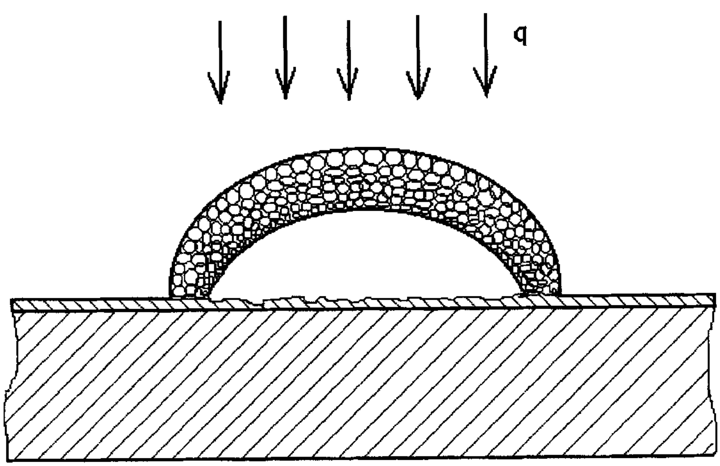

FIGURE 4a Thermo-protection coating. ED-20 + Intumescent addition (20%).

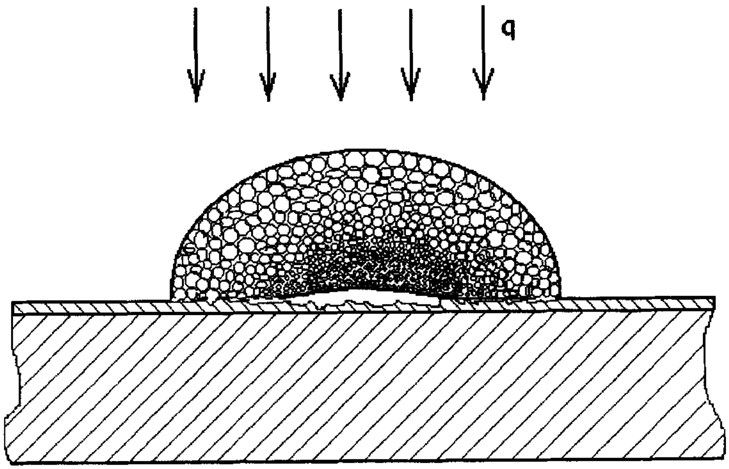

FIGURE 4b Thermo-protection coating. ED-20 + Intumescent addition (20%) + (2%).

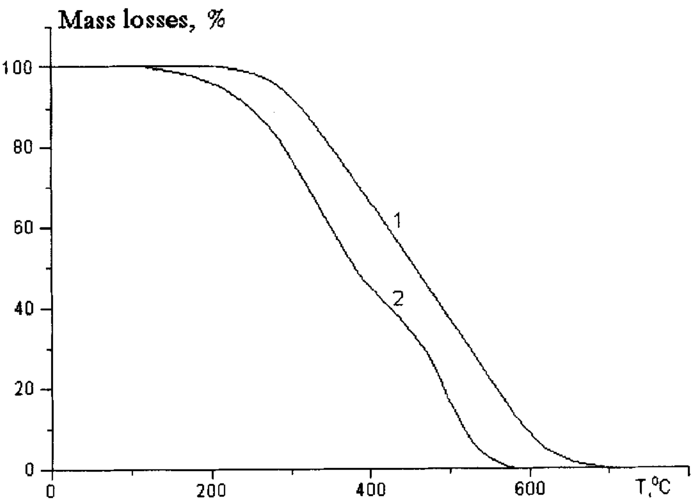

FIGURE 5 The curves of mass loss of following samples: 1. ED-20; 2. ED-20 + HDHCl.

explained, by the fact, that hydrazine-dihydro-chloride initiates destruction of the resin, shifting the temperature of beginning of destruction into the region of lower temperatures (Fig. 5).

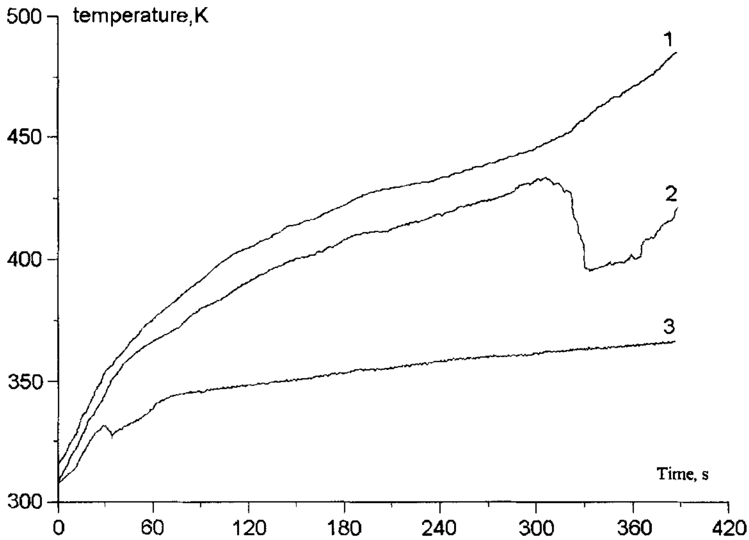

FIGURE 6 Thermo-protection properties of coating, contained ED-20: 1. ED-20 + PEPA; 2. (ED-20 + PEPA) + 40% Intumescent addition (IA); 3. (ED-20 + PEPA) +

Thermo-protective properties of coatings on the ED-20 basis are presented in the Figure 6. It can be seen from Figure 6, that insignificant addition of HDHCl essentially improves thermo-protective properties of the coating.

Reference

[1] Gibov, K. M. (1987). *Doc. Thesis*, Kazanskii Chemical-technology Institute, Kazan.