

Fire-safe polyurethanes modified with new antipyrène

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Introduction

The combustion phenomenon is inherent to the people in everyday life. External manifestation of spontaneous combustion process involving all the exothermic chemical reactions in the environment is fire. The mechanism of the phenomenon of burning plastic is a complex process. It is a multi-step process in which numerous physical and chemical phenomena occur [1 ÷ 4]. The phenomenon of polymer combustion depends on composition, chemical structure, volume, shape of material, density, surface porosity of the internal structure and other. Polyurethanes without flame retardants are flammable materials. Combustion is facilitated by very developed surface of pores. This is accompanied by the release of toxic fumes and gases. In the case when polyurethane foam with closed cells is subjected to combustion, the burning process proceeds more difficult than in the case of foams with open cells enabling so-called chimney effect. Polyurethanes decompose at temperatures above 200°C. Among the products of PU degradation, besides hydrogen cyanide and carbon monoxide, also nitrogen oxides, nitriles, hydrogen chloride and carbon dioxide are present. Isocyanate fumes hovering over the surface of burning polymer are condensed and liquid polyols are subjected to further decomposition [5 ÷ 8]. Fire resistance of polymers can be increased by adding certain chemicals in order to reduce or retard combustion (known as flame retardants) and/or mineral fillers, nanofillers or some plasticizers [9 ÷ 10]. These compounds, introduced into the polymer by chemical and/or physical modification, interfere in the mechanism of combustion process. In order to obtain foams which would have a good price and meet criteria connected with safe use and environment protection, it is required to develop a system of flame retardants free from halogens and based on easily available and raw materials. In the presented studies, an attempt was undertaken to develop new technology for producing polyols containing boron and nitrogen, which, when introduced into polyurethane premix as polyol, would effectively reduce flammability of foam material and would not disturb the production process [11 ÷ 12].

From a several years, authors of the paper carry out research works aiming at developing recipes for preparation of fire resistant rigid polyurethane foams. Due to requirements of the European Union countries to apply halogen-free methods, problem of flammability of polyurethane foams become very actual. Authors developed new polyol for production of rigid polyurethane-polyisocyanurate foams, which played the role of an effective flame retardant. Taking advantage of the synergistic effect occurring in combustion process of polymers, they produced boron-nitrogen flame retardant, in which flame retarding properties of boron and nitrogen were mutually complemented and intensified. Boron participates in endothermic reactions, whose final effect is release of water and formation of glassy protective layer. This layer protects the surface against oxygen and heat coming from the flame. Whereas, nitrogen compounds are decomposed to gaseous products, which in the condensed phase are involved in formation of expanded carbon layers, and after getting to gaseous combustion zone, they become scavengers of radicals.

Boric acid and N,N'-di(methyleneoxy-5-hydroxypentyl) urea were used to produce new compound reducing flammability and not described in literature.

Experimental

Raw materials

The polyether with trade name Rokopol RF-55 (product of oxypropylation of sorbitol, produced by Chemical Plants "Rokita", Brzeg Dolny, Poland) and Ongromat CR 30-20 (technical polyisocyanate whose main component is diphenylmethane 4,4'-diisocyanate, made in Hungary) were used to prepare the rigid PUR-PIR foams. The density of Ongromat CR 30-20 at temperature of 25°C was 1.23 g/cm³, its viscosity was 200 mPas. Polyols and polyisocyanate were characterized according to the following standards: ASTM D 2849-69 and ASTM D 1638 – 70.

Anhydrous potassium acetate in the form of 33% solution in diethylene glycol (Catalyst-12, POCh Gliwice, Poland) and „DABCO 33LV” (triethyleneamine produced by Hüls, Germany) in the form of 33% solution in dipropylene glycol were applied as catalysts in foam composition. The polysiloxanepolyoxyallyleneoxydimethylene surface-active agent “Sillicone L-6900” (Witco Corp., Sweden) was used as a stabilizer of the foam structure.

Carbon dioxide, formed in reaction of water with isocyanate groups, was a blowing agent. Moreover, tri(2-chloro-1-methylethyl) phosphate with trade name „Antiblaze TMCP” (by Albright and Wilson, Great Britain) was introduced into foam composition.

Analytical methods for the determination of raw materials

Raw materials used for production of polyurethane plastics must characterize by suitable properties of processing. They are essential both during composing the recipe of foam premix and during determining parameters of production of polyurethane plastics. Therefore, studies on polyol raw materials, first of all comprised the determination of hydroxyl number as well as percentage of boron, nitrogen and water. Hydroxyl number affects the amount of isocyanate required to produce urethane bonds, while boron and nitrogen will be introduced into polyurethane material together with boron-nitrogen polyol.

An important feature of polyisocyanate used in production of polyurethane foams is content of isocyanate groups. Since the amount of NCO groups can be slightly different in supplied lots of isocyanate, therefore, their percentage was determined in each new consignment of raw materials.

Determination of hydroxyl number

Hydroxyl number L_{OH} is defined by amount of mg of KOH, equivalent to amount of acetic anhydride reacting with free hydroxyl groups contained in 1 gram of substance tested. L_{OH} is determined by acylation with acetic anhydride in xylene medium. Excess of acetic anhydride after hydrolysis together with acid formed as a result of reaction is titrated with KOH solution in presence of phenolphthalein. Determination of hydroxyl number was performed according to standard PN-93/C-89052.03.

Determination of water in polyol

Content of water was determined by Karl Fisher's method by using pyridine-free reagent with trade name Titraqual according to standard PN-81/C-04959. Determination consists in dissolution of adequate weighed sample of the tested product in Titraqual (titrant) and potentiometric titration of this solution to equivalence point of titration.

Determination of boron content

Content of boron was determined by HACH Lange tests. Principle of determination consists in the fact that boron ions react with azomethine-H to form yellow compound that can be determined photometrically. The pH value of the sample must be within a range from 4 to 9. Colour reaction is strongly dependent on temperature, so the sample and cuvette for analysis should be at a temperature of 20°C. Fluctuations of temperature upwards lead to undervaluation of results but deviations downwards - to overestimation of results.

Determination of nitrogen content

Content of nitrogen was determined by use of Kjeltac 2200 apparatus (FOSS, USA).

Determination of percentage of isocyanate groups

The percentage content of isocyanate groups in polyisocyanate was determined by volumetric method. Polyisocyanate in amount of 0.1 – 0.2g was weighted to flask containing 10 cm³ of 0.5 molar solution of di-n-butylamine in chlorobenzene and 10cm³ of diethylformamide as a solvent was added. After 30 minutes, excess of unreacted di-n-butylamine was titrated back by 0.2 molar hydrochloric acid in presence of bromophenyl blue as an indicator. Blank test was also performed. Percentage content of isocyanate groups was calculated according to equation (1):

$$\%NCO = \frac{4.2 \cdot (V_1 - V_2) \cdot N}{C} \quad (1)$$

where:

V_1 – volume of 0.2molar HCl used for blank sample [cm³]

V_2 – volume of 0.2molar HCl used for tested sample [cm³]

C – weight of polyisocyanate

N – molar concentration of hydrochloric acid solution.

Equivalent of polyisocyanate was calculated according to equation (2):

$$R_{PI} = \frac{4200}{\%NCO} \quad (2)$$

Results of studies on basic raw materials for production of rigid polyurethane-polyisocyanurate foams

Results of studies will allow to assess suitability of selected raw materials for production of rigid polyurethane-polyisocyanurate foams. Results of studies on basic properties of raw materials used in recipe for preparation of rigid polyurethane-polyisocyanurate foams were presented in Table 1.

Table 1

Basic properties of the selected raw materials

Raw materials	Properties				
	Hydroxyl number mgKOH/g	Content of isocyanurate groups %	Concentration of nitrogen % w/w.	Concentration of boron % w/w.	Content of water %
Rokopol RF 55	495	-	-	-	0.1
Ongromat CR-30-20	-	31	Not tested	-	-
Tri[N,N'-di(methyleneoxy-5-hydroxypropyl)urea] borate	430	-	9.7	1.3	0.2

Structure of the new flame retardant indicates presence of boron and nitrogen atoms which have significant effect on flammability of

plastics. Percentage of these atoms in the new polyols is about 10%, thus, positive result of studies on reducing flammability of new foams can be expected.

The fundamental parameter of polyol, essential during the determination of recipe of polyurethane composition, is its hydroxyl number. Therefore, it was determined for polyol raw materials used. Hydroxyl number is equal to 430 mgKOH/g for tri[N,N'-di(methyleneoxy-5-hydroxypropyl)urea] borate and 495 mgKOH/g for Rokopol RF 55 and it is close to its theoretical values calculated. Hydroxyl number of polyol compounds tested (above 200 mgKOH/g) confirms that they are an interesting raw material for preparation of rigid PUR-PIR foams. Water, which reacts with polyisocyanate, exerts a significant effect on process of foaming. Therefore, knowledge of water quantity supplied in raw materials in foaming reaction is very important. Studies showed that water content in the studied raw materials is less than 1%. Such a small water content does not disturb the production of polyurethane foams and need not be taken into account in recipe.

Synthesis of rigid PUR – PIR foams

The rigid PUR - PIR foams were produced according to the recipes (Table 2) in such a manner that the weighted amount of polyisocyanate was introduced into one polypropylene vessel of 1 dcm³ and polyol with auxiliary substances were placed into another one. Polyols were thoroughly mixed with other components using electric stirrer (1800 rpm).

Table 2

Recipes for preparation of rigid polyurethane-polyisocyanurate foams

	Unit	F1	F2	F3	F4	F5	Standard foam [W1]
Rokopol RF-55	ch.eq. g	0.9 50.99	0.8 45.32	0.7 39.66	0.6 33.99	0.5 28.28	1 55.65
Tri[N,N'-di(methyleneoxy-5-hydroxypropyl)urea] borate	ch.eq. g	0.1 6.52	0.2 13.05	0.3 19.57	0.4 26.10	0.5 32.62	0 0
Silikon L6900	g	4.6	4.6	4.6	4.6	4.6	4.6
DABCO	g	2.8	2.8	2.8	2.8	2.8	2.8
Catalyst 12	g	6.5	6.5	6.5	6.5	6.5	6.5
AntiblazeTMCP	g	46.1	46.1	46.1	46.1	46.1	46.07
Water	ch.eq. g	0.7 3.15	0.7 3.15	0.7 3.15	0.7 3.15	0.7 3.15	0.7 3.15
Ongromat 30-20	ch.eq. g	3.7 250.1	3.7 250.1	3.7 250.1	3.7 250.1	3.7 250.1	3.7 250.1

Polyisocyanate was merged with polyol component and precisely mixed with mechanical stirrer for about 15s, then, it was poured into a mould and expansion of foam was observed. An open mould was applied in our studies in order to enable so called free expansion of foam. The open mould was made from steel (thickness of 3 mm, internal dimensions 25x25x30 cm).

In the first stage, the standard foam (reference) without addition in the form of new polyol was prepared and then, foams containing from 0.1 to 0.5 of chemical equivalent of each borate were synthesized.

During synthesis of polyurethane-polyisocyanurate foams, the course of foaming of the reaction mixture was monitored by measuring the respective technological times by the use of stop-watch i.e. start time, time of expansion and time of gelation.

Start time – it is time measured by stopwatch from the moment when all components were mixed to the moment when so-called “creamy state” was achieved. It is demonstrated as the beginning of foam expansion.

Time of expansion – it is time measured by stopwatch from the moment when the components were mixed to the moment when foam achieved maximum volume.

Time of gelation - it is time measured by stopwatch from the moment when the components were mixed to the moment when the free surface of foam ceased to glue to a clean glass rod.

Determination of flammability

In order to verify behaviour of foams in flame, the various tests were carried out:

According to ASTM D3014-73 standard – simplified chimney test - (Butler’s vertical test).

Apparatus used to determine the flammability according to the vertical test consisted of a vertical column (300x57x54mm) whose three walls were made from metal plate and the fourth one was a movable pane. Six foam samples (150x19x19mm) were tested. Before burning, the samples were weighted accurate to 0.0001mm and then they were placed inside a chimney. A pane was installed and a flame from the burner, supplied with a gas: propane – butane, was brought into contact with the sample for 10 seconds. Then, the burner was removed and the time of the sample free burning and retention (residue after burning) were measured by stopwatch in vertical test. Retention was calculated from the equation (3):

$$R_e = \frac{m}{m_0} \cdot 100\% \tag{3}$$

where:

R_e – retention

m_0 - mass of sample before combustion [g]

m – mass of sample after combustion [g].

Using the test method described in the standard ISO 5660-1:2001

The basic research tool in this method is a cone calorimeter. During the test, the standardized test samples with dimensions of 100 mm x 100 mm were subjected to the influence of thermal radiation. The following parameters were recorded: time required to initiate the combustion reaction, thermokinetic parameters i.e. rate of heat emission and total amount of heat released as well as the selected toxic and smoke-generating properties. Thermokinetic properties were determined on the basis of the calorimetry theory of oxygen consumption, which says that for each 1g of oxygen consumed, 13.1 kJ ± 5% of heat is released. The testing was conducted for samples of materials placed horizontally, reaction of combustion was initiated by ignition. Samples of the materials were subjected to influence of thermal radiation at intensity of 30 kW/m². End of testing was after decay of flame combustion.

Oxygen Index (IO) was determined according to ASTM D 2863-1970 standard

The method consists in the determination of the limiting percentage concentration of oxygen in the mixture of oxygen and nitrogen, sufficient to sustain burning of the sample in the form of small beam with dimensions of 150x13x13. Oxygen index was calculated in percentage according to equation [4]:

$$OI = \frac{O_2}{O_2 + N_2} \cdot 100\% \tag{4}$$

where:

- O_2 – volumetric flow of oxygen at limiting concentration [m³/h]

- N_2 – volumetric flow of nitrogen at limiting concentration of oxygen [m³/h].

Results and discussion

Method for reducing flammability of polymeric materials with application of boron compounds is used since recently. Preliminary studies are promising. It is believed that boron compounds can help to solve the problem of flammability of foams. Flame retarding properties of boron can be aided by additional antipyrène i.e. nitrogen, and then, effect of flammability reduction will be more intense.

Method that allows to define flammability of materials is the method of cone calorimetry. It also allows characterization of phenomena accompanying combustion process. Parameters of pyrolysis process of selected rigid polyurethane-polyisocyanurate foams produced using the new boron polyols are presented in Table 3.

Table 3

Results of fire tests of selected rigid PUR-PIR foams

Symbol of foam	Mean time to sustained ignition T_{zap}	Total heat released THR	Mean mass loss rate HLR	Maximum heat release rate HRR	Time to reach HRR max.	Amount of emitted CO	Amount of emitted CO ₂	Oxygen index
	[s]	[MJ/m ²]	[gm ² /s]	[kW/m ²]	[s]	[g/g]	[g/g]	%
W1	1.48	14.3	10.22	278.90	10	1.352	11.2	19.6
F1	6.79	3.6	7.07	229.94	24	0.634	1.4	22.9
F5	14.07	2.9	4.91	198.79	30	0.333	1.0	23.8

The rigid polyurethane-polyisocyanurate foams containing the lowest (0.1 of chemical equivalent) and the highest (0.5 of chemical equivalent) amounts of boronitrogen compound and standard foam W1 (not modified with boronitrogen compound) were subjected to studies on combustion process by cone calorimetry method. In the case of foam W1, ignition time was very short i.e. 1.48 s. It is typical for plastics with porous structure, which are characterized by high flammability. It was observed that after modification of recipe, ignition time of these foams was considerably longer. The longest time of ignition was in the case of F5 foam, containing 0.5 of chemical equivalent of tri[N,N'-di(methyleneoxy-5-hydroxypentyl)urea] borate, i.e. 14.07s. This result is consistent with expectations due to the highest content of antipyrène in foam composition. During flammability test conducted by cone calorimetry method, THR value informing about total amount of heat released by burning foam was also measured. The highest amount of heat (14.3 MJ/m²) was released during testing the standard foam. Application of the new compound in composition contributed to significant reduction in THR value, not exceeding 4 MJ/m². The lowest amount of heat released was observed in the case of rigid PUR-PIR foams containing the highest amount of borates. Total amount of heat released can indicate that boronitrogen compound, by reducing this parameter by about 80%, shows a cooling effect.

Moreover, the course of HRR curves, illustrating maximum heat release rate, provides information concerning mechanism of flammability reduction. It is a very important parameter, which is determinant of the ability to self-extinguishing of material under fire conditions. HRR values presented in Table 3 were determined for data from the moment of ignition to the end of test. Curves characterizing the course of heat release rate (HRR) of the foams obtained are presented in Figure 1.

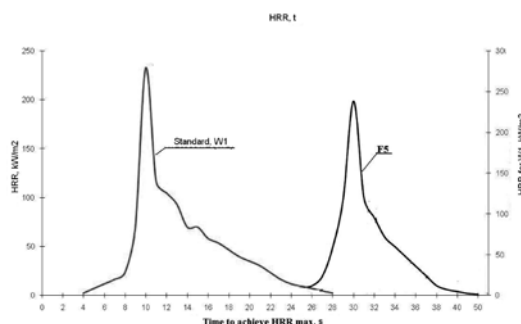


Fig. 1. Heat release rate (HRR) of rigid PUR-PIR foams

HRR curves for modified and unmodified rigid PUR-PIR foams show the successive stages of combustion process. At first heating of the samples can be observed followed by release of volatile matter and flammable gaseous products. Burning of the emitted gases is the cause of release of large amounts of heat. Course of curves in the figure indicates that in the case of standard PUR-PIR foam (W1), increase in heat release rate occurs vigorously. Under conditions of test, HRR value for standard foam was achieved in very short time and sustaining of flame was observed. However, foam modified with boron compound burns more gently, reaching lower value of heat release rate than unmodified foam. Extension of time to reach maximum HRR values (from 10 s for standard foam W1 to 40 s for foam F5 – containing 0.5 of chemical equivalent of tri[N,N'-di(methyleneoxy-5-hydroxypentyl)urea] borate) also indicates flame retarding properties of the compound studied. Similar conclusions can be drawn for the curves showing carbon monoxide release rate – Figure 2.

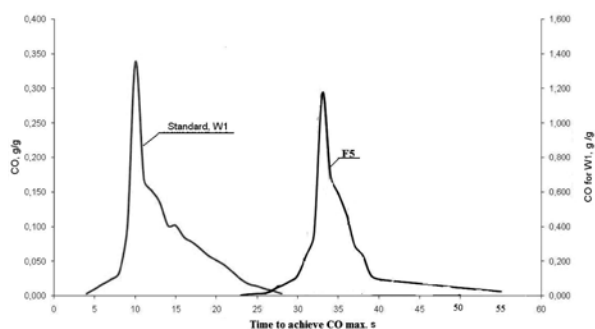


Fig. 2. Rate of carbon monoxide release from rigid PUR-PIR foams

The W1 curve is characteristic for standard foam without boronitrogen antipyrène. Amount of the released CO is considerably higher than in case of foams modified with the new compound. For standard foam, CO was emitted in amount of 1.352 g/g. After modifying the recipe, amount of emitted carbon monoxide was reduced almost five times, to value of 0.234 g/g for the foam F5, containing 0.5 of chemical equivalent of tri[N,N'-di(methyleneoxy-5-hydroxypentyl)urea] borate. Similar relations can be formulated for carbon dioxide evolved during the flammability test.

Rigid polyurethane-polyisocyanurate foams modified with boronitrogen compound show much longer time to sustained ignition than the standard foam, which is consistent with results obtained for the determination of oxygen index. The value of oxygen index (OI) i.e. percentage of oxygen in the mixture with nitrogen, which allows to sustain burning of sample was determined. It reflects primarily the degree of flammability of volatile decomposition products of plastic. Oxygen index for standard foam is equal to 19.6%. After modification of PUR-PIR foam, oxygen index increased to 22.9% for F1 foam containing 0.1 of chemical equivalent of boronitrogen compound and to 23.8% for F5 foam containing 0.5 of chemical equivalent of this compound, respectively.

It was confirmed by oxygen index method that presence of the new compound in composition of PUR-PIR foam reduced its flammability by about 21%. Under conditions of tests by cone calorimetry method, foams prepared with addition of the new polyol form charred residue to a lesser extend. During flammability test performed by cone calorimeter method, it was observed that the modified foams did not burn but only glowed. It is a causes of slight loss in mass of these foams during flammability tests. The lowest HLR value, which defines average rate of mass loss, was recorded for foams characterized by the longest times to durable ignition. Analysis of combustion process of rigid PUR-PIR foams in cone calorimeter showed that the new compound was an effective antipyrène. The compound contains both boron and nitrogen atoms in the molecule, therefore, synergistic

interaction of these elements can be confirmed. Under conditions of large amounts of heat supply (35 kW/m²), modification of foam causes delayed ignition. In subsequent stage of combustion process, it acts as diluting agent and demonstrates cooling effect. It confirms the conclusion about formation of boric acid at high temperature followed by formation of glassy protective layer, which task is to reduce or even interrupt process of burning. Presence of nitrogen as antipyrène was also noted in the new PUR-PIR foams. It participates in formation of foamed carbon layers and considerably reduces intensity of liberation of fumes.

Another method, used for determining flammability of polymeric material is Butler's test (so-called vertical test). In this test, sample of foam is subjected to combustion and then, its retention i.e. residue after combustion is calculated from formula given in the standard. Retention value, given in percentage, determines combustible properties of foam. The retention value is higher, the material is less flammable. Results of studies on flammability of foams by Butler's test are presented in Table 4.

Table 4

Results of studies on flammability of rigid PUR-PIR foams by Butler's method

Symbol of foam	W1	F1	F2	F3	F4	F5
Retention [%]	78.4	84.1	84.9	85.8	86.8	87.6

Results of studies on flammability of rigid PUR-PIR foams by Butler's method confirmed fire tests of these foams conducted by cone calorimeter method. Dependence between retention of foams and amount of boron compound in foam composition is presented in Figure 3.

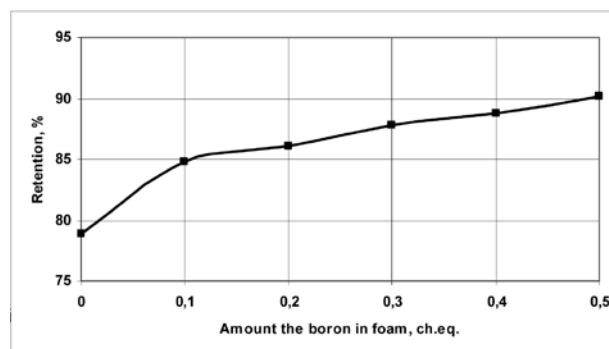


Fig. 3. Dependence between retention of PUR-PIR foams and amount of boron polyols in foams

It was found that residue after combustion (retention) increased with increase of boronitrogen compound content in the new foams. Its value was within the range from 78.4% for standard foam to 87.6% for F5 foam containing the highest amount of the new compound. In summary, it can be concluded that Butler' test confirmed the assumption that the new compounds contribute to reduction of flammability of rigid PUR-PIR foams.

Summary and conclusions

Due to the specific, porous structure of polyurethane materials, they are classified as combustible materials. Searching for the method to reduce flammability of PUR-PIR foams is a particularly important subject. Flammability tests confirmed that use of the new compounds containing boron and nitrogen atoms in molecule structure, effectively increased their fire resistance. The effectiveness of boronitrogen compounds in reducing the flammability was demonstrated by decrease in total amount of heat emitted, prolonged time to sustained ignition, smaller amount of carbon dioxide and monoxide released, increase in oxygen index and retention and lower maximum combustion temperature achieved in a shorter time than standard

foam. The results presented in this paper are the answer for search of fire-safe polyurethanes. Studies have proved that modifications of premixes for production of polyurethane foams allow to obtain the desirable properties. It was shown that extremely important is the suitable selection of polyol raw materials, isocyanate and auxiliaries already at the level of determination of recipe. Application of boron compound as a flame retardant for production of rigid polyurethane - polyisocyanurate foams is an innovative solution that can affect efficiency of applied technologies and provide favourable changes in characteristics of foams.

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