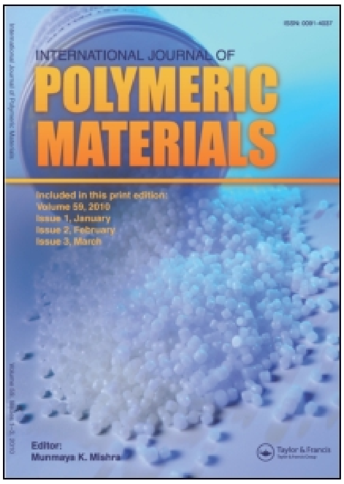

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Flame Retardant Polyolefinic Materials

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Flammability and thermal properties of industrial flame retardant polyolefines used for making parts in color televisors have been studied. The influence of thermal ageing on mechanical properties and fire behaviour of polypropylene material has been shown. Thermogravimetric data have been used to evaluate kinetic parameters for thermo-oxidative decomposition of some flame retardant polypropylene materials.

Keywords: Flammability; polyolefine materials; thermal ageing; the decomposition kinetics

INTRODUCTION

Polymers are widely used as constructional, electroinsulating or decorative materials in manufacturing domestic radio-electronic products. In Russia, the products are made in accordance with general State Standard (GOST): GOST 12.2 006-89 "Domestic radio-electronic apparatus. Safety needs and methods of testing", which is analogous to International Standard of IEC 65. This standard set forth minimum requirements for polymeric materials used in electronic apparatus and equipment to prevent injury or damage due not only to fire but also electric shock, energy, mechanical and heat hazards, radiation and chemical hazards. Some of the issues in this Standard are devoted to fire safety requirements and the description of basic test methods for evaluating flammability of polymeric materials used in TV devices.

Other important document regulating fire safety for plastics applied in making electrotechnical and electronic products is GOST 12.1.044-89 "Fire and explosive hazards of substances and materials.

The parameters and methods for the assessment". This Standard is used for classifying materials by combustibility and ignitability, flame propagation, smoke release and toxicity of pyrolysis and combustion products. The Standard includes also small-scale test of limiting oxygen index (LOI) to evaluate flammability of different organic materials. The LOI test corresponds to ASTM D 2863.

Polymeric materials for making domestic electrotechnical and electronic products have been usually ranked by flammability in accordance with well known Standards such as IEC 707; IEC 112; UL 746; UL 1470 and other. USA Standard of Underwriters Laboratory, Inc. of UL 94 is one of the basic methods for testing material flammability, which is included in above Standards [1]. In this test the specimen in horizontal or vertical orientation is exposed to small flame, and extent of burning and dripping are monitored. The most severe conditions are realized in vertical test. In this case, the bottom of vertically-mounted strip is exposed to a flame twice, each time for a 10 s duration. To achieve the top rating of V0 by UL 94 classification, burning specimen must self extinguish in less than 10 s after removal of burner flame. Glowing time should not be over 30 s. No flaming drips are permitted to fall from the specimen. The UL 94 V2 rating is less stringent. Some flaming drips are allowed and afterflame time for each individual specimen may be up to 30 s. After glowing time may achieve to 60 s.

The applications for polymeric materials in color televisors include covers, coils, barriers, winding details, enclosures and other appliances in deflexion systems, high-voltage block, line transformer or other functional parts. Only the materials with UL 94 V0 are permitted in making important parts for the electronic apparatus.

During the development of native Standard -analogous IEC 707 and UL 94 [2] we investigated different industrial flame retardant polymeric materials recommended for making parts in TV apparatus.

The objective of the present paper has been to evaluate the flammability of some flame retardant polyolefinic materials by methods of UL 94 V and LOI (ASTM D 2863, GOST 12.1.004-89). It was interesting to ascertain the correlation of the testing results and to determine the possibility of UL 94 V method for a more detailed selection of flame retardant materials with top rating of V0.

The solid phase decomposition of polymeric materials plays the important role in their fire behaviour. Therefore, the comparative investigation of thermooxidative decomposition of the polyolefinic materials has been carried out. The influence of the accelerated thermal ageing on the change in flame retardancy and physico-mechanical properties for one of the polyolefinic materials has also been considered.

EXPERIMENTAL

Materials

In the work, the commercial flame retardant polypropylene and polyethylene materials developed by domestic and foreign producers have been used. The samples were supplied by TV design burea (Moscow) without a detailed description of formulations. It is known that some compositions inlcude halogen-containing substances and antimony oxide as general flame retardants and synergist.

Flame retardant polypropylene compositions are:

Trade mark of PP 22-01-3C, Technical Conditions TC-6-05-1968-84, Russia
PP 22-01-5C, TC-6-05-1968-84, Russia
PPTG, Russia
PP 2654, Chisso, Italy
PP 2038, Chisso, Italy
FR PP, Moplen, Italy
Polyflam RPP 371, NDLP 733, Belgium.

Flame retardant polyethylene compositions are:

PETG based on PE 21008-75, Byelorussia
FR PE Funicon E-205, Japan.

The samples for flammability testing are in the form of plates of 100–130 mm length and of 1.3–3.5 mm thickness. Before testing the samples were conditioning at room temperature and 50% relative humidity.

The Investigation Methods

Flame retardancy level for PO samples was determined by LOI and Ul-94 test methods. The influence of PO sample sizes on LOI and UL-94 flammability indices is shown in Table I. Limiting oxygen

TABLE I Flammability Indices for Flame Retardant Polyolefine Materials

Material	*Section size, mm*	*LOI, v%*[a]	*LOI** %[a]	*UL-94V*[b]					*Ignition number of*
				t1, s	*t2, s*	*tgl, s*	*dripping*	*rating*	*dripping*[a]
PP 22-01-3C									
Russia	13 × 3	27.2	25.7	2	< 10	2–8	No	V0	5
PP 22-01-5C	13 × 3	27.5	23.8	0	< 10	3–10	No	V0	4
Russia	13 × 1.8	27.5	23.6	0	< 10	20–40	No	V1	–
PPTG, Russia	13 × 1.5	27.5	21.5	0	< 10	< 60	Yes	V2	2
PP Chisso	13 × 3	28.2	24.0	0	< 10	2.5–8	No	V0	3
2654, Italy	13 × 2	28.1	23.8	0	< 10	4.2	No	V0	3
PP Chisso	13 × 3	27.5	22.0	1	1	< 60	Yes	V2	2
2038, Italy	13 × 3.5	27.1	–	0	0	< 30	No	V0	3
FRPP									
Moplen, Italy	13 × 3	27.7	24.0	0	< 10	0.3	No	V0	3
Polyflam RPP,									
Belgium	13 × 3	28.0	24.3	0	< 10	2.5–9	No	V0	3
PETG,	13 × 3	32	–	0	< 10	5–10	No	V0	3
Russia	13 × 2.1	29.7	–	0.5	6.6	20	No	V0	3
	13 × 1.3	28.7	25.5	2	5.4	10	Yes	V2	2
PE Funicon,	10 × 2	30.8	27.7	0	0.2	< 60	No	V1	3
E 205	13 × 2	30.8	27.7	0	0	< 60	No	V1	3

[a] The average values are based on 3 sample's results.
[b] The average results are based on 5 run's ones.

index was evaluated using the testing conditions specified by GOST 12.1.044-89 but at different combustion direction of the samples: downward and upward flame spread.

Preliminary heating samples and air surroundings results in LOI decrease. "Temperature index" apparatus was developed at the Institute of Chemical Physics, Russian Academy of Sciences to estimate the effects of temperature and oxygen content on critical characteristics of the combustion for polymeric materials [3].

Testing PMMA samples with known LOI values by analogous apparatus of Stanton Redcroft, England, showed that the parameters of standard flame of gas burner are changed with the increase of temperature and oxygen content in oxidizer flux. Flame sizes and its temperature grow. As result, the ignition conditions of polymeric samples during testing LOI dependence on temperature are different. This circumstances affects the precision and the reproduction of testing results.

For "Temperature index" apparatus of IChPh system the device was developed to ignite the sample in regulated regime. The device provides automatic ignition of preliminary stoichiometrically mixed air and combustible gas by electrical spark, the transfer of pilot burner in neutral position after the ignition with simultaneous cessation of supplying the combustible gas. This device improves, the exactness and the stability of ignition parameters and LOI values. "Temperature index" apparatus of IChP system includes also the device for measuring flame spread rate at burning conditions given for the studied polymeric sample. The device consists of two ionizational probes connected with timer, which are placed at the sample surface at the distance of 50 mm one from another.

The comparative study of thermooxidative decomposition of polyolefin materials was performed by TGA method with using OD-102 derivatograph (Hungary). Sample weight was 50–100 mg. Heating rate was 7 degree/min.

TGA data were used to determine temperature characteristics of polymeric materials: temperatures of mass loss beginning and termination, Tbg and Tt, temperature corresponding to maximum mass loss rate, T max. Mass losses, W, for each stage of the decomposition were also determined. The kinetic parameters of the decomposition for some polypropylene samples were evaluated by method described in work [4].

Thermal ageing of PP-22-01-5C composition was carried out in accordance with the recommendation of Standard for former Community of States for Economical Mutual Aid (ST SEV 4127-83). Thermal ageing of PP samples was carried out at 100, 120, 140 and 150°C. The duration of the exposition was to 182 days. Air temperature supplied by ventilators was controlled with precision of 1C.

Studied samples of standard sizes in the form of slabs or spades for testing flammability or mechanical properties are placed in special holders. These holders are fastened in horizontally on trays in the thermostat to realize the circulation of air along sample surface.

RESULTS AND DISCUSSION

Table I data show a general tendency of LOI to decrease burning direction change from the candle-type to the opposite upward flame spread. The extent of LOI change depends on polymer nature and the composition formulation. It should be noted that the variation of sample width in limits of 10–13 mm and its length from 100 to 150 mm had practically no effect on LOI values. However, the thickness of samples affects LOI. Polymer nature, the composition and thermo-physical properties of materials stipulate for observed LOI dependence on sample thickness. For example, PP materials show similar LOI with thickness change in the above limits. LOI for PETG decreases at the thickness change from 3 to 1.3 mm. Such LOI decrease is regular: leading mechanism for heat transfer is changed with the transition from "thermal thick" to "thermal thin" burning behaviour of material [5].

From Table I one can see that at vertical orientation of sample and upward flame spread in LOI test all studied polyolefinic materials (excepting PPTG) do not ignite in air medium. PPTG becomes flammable in these testing conditions (LOI = 21.5%). LOI is reduced due to preheating of the material ahead of the pyrolysis zone by flame heat flux.

The influence of oxidizer flow temperature on LOI values for polyolefinic materials is shown in Table II. It should be noted that oxidizer flow rate grows with its temperature. As the result, heat losses from flame reaction zone increase and thus affect LOI values. Therefore, oxidizer flow rate has been calculated for given temperature to keep

TABLE II The Effect of Oxidizer Flux Temperature on LOI for Polyolefine Materials

Temperature, C	*U1, l/min*	*LOI, %*	
		PP 22-01-5C (130 × 13 × 1.5, mm)	*PETG (130 × 13 × 1.5, mm)*
20	10.6	27.2	29.7
50	9.0	26.3	29.5
80	8.4	25.8	27.5
100	7.8	25.3	25.6

constant level of 4 cm/s in the accordance with GOST 12.1.044-89. The dependence for volume oxidizer flow rate on temperature and pressure can be described by the following equation:

$$(P1 \times U1)/(P2 \times U2) = [(T1)/(T2)]^{1/2},$$

where P1, U1, T1 are pressure, volume rate and temperature of oxidizer flow at normal conditions; P2, U2, T2 are parameters of gas medium at changed conditions.

For constant pressure P1 = P2 and U2 corresponding to linear rate of 4cm/s, it is easy to calculate U1 values needed by above equation in testing flammability (Tab. II).

For candle burning type of polyolefin samples LOI decrease is observed over 50°C. The dependence of LOI on temperature for polypropylene material of PP 22-01-5C corresponds to the following linear equation: LOI = 27.59–0.0299 T, and for polyethylene material of PETG it is described by the equation: LOI = 31.3–0.0516 T. Here temperature is given in C. Above equations allow us to evaluate "Temperature Index" (TI) for polymeric materials. TI is the temperature at which the LOI-temperature dependence curve of a material intercepts an oxygen concentration of 21% present in normal air atmosphere. TI profile provides a more reliable and complete picture of the flammability behaviour of polymers than does the LOI at normal temperature. The results obtained were extrapolated to higher temperature. TI = 220°C for PP 22-01-5C and TI = 200°C for PETG were found.

If we assume that LOI is changed with temperature by exponential law, the extrapolation to 21% oxygen content in oxidizer flow gives a higher TI for the flame retardant polyolefins. For example, for PP

22-01-5C exponential dependence of LOI on temperature described by the equation: LOI = exp(3.32 – 0.000876 T), where T is in °C results in a less reliable temperature index: TI = 314°C.

The influence of polymeric sample thickness on flammability characteristics has been also observed in testing by UL-94 V method (Tab. I). Therefore, the thickness of samples at ranking flammability by UL-94 method should be indicated. One and the same polymeric material may pass from top rank of UL-94 V0 to a lower one of V1 or V2 with its thickness change. As example, PETG samples have rating UL-94 V0 for thickness of 3–2 mm and V2 for thickness of 1.3 mm.

There is no straight correlation between flammability characteristics of the polyolefinic materials, obtained by LOI and UL-94 V methods. The polymeric materials of the same chemical nature have similar LOI values found by standard testing method (candle-like test). Note, however, the interconnection of flammability rank change by UL-94 V with extent of reducing LOI at the transition from standard candle-type test to upward burning one: the smaller this change the higher a rank of material by UL-94 V method and greater number of repeated ignition needed to dripping. This conclusion holds for the samples with the same thickness.

The effect of twofold ignition in UL-94 V test is connected with additional heating of test sample. If repeated 10 s ignitions of the specimens with standard 10 s interval for pilot flame action are used in the test, the distinctions in their burning behaviour can be revealed. In this case, a self-burning time of the samples increases with the repeated number of ignitions and the flame may be spread upon full length of the testing sample. After several repeated ignitions of polyolefinic samples flaming drips are formed, which may be able to ignite a cotton in UL-94 V test. The repeated ignition number for the appearance of flaming drips depends on polymer grade and flame retardancy pathway of the material. Table I shows the distinction in flaming dripping behaviour of polyolefinic materials when repeated of ignition the samples is used in UL-94 test. The best results are obtained for PP 22-01-5C and PP 22-01-3C materials, which show a dripping only after 4-th and 5-th ignition of the samples by pilot flame of gas burner.

Other samples with thermal thick burning behaviour (3 mm thickness) and top rating UL-94 V0 show a dripping after third ignition by pilot flame. The results obtained show the potential of using UL-94 test for more detailed screening flame retardant thermoplastics.

The comparative study of the decomposition of the polyolefinic materials clearly showed similarities as well as differences between sample behaviour. It has been found that under programmed heating with constant rate the thermal decomposition of flame retardant polyolefinic materials proceeds in a few stages. Typical TG, DTG and DTA curves for flame retardant PP samples are presented in Figure 1. Analysis results are given in Table III. Endothermal peak stipulated

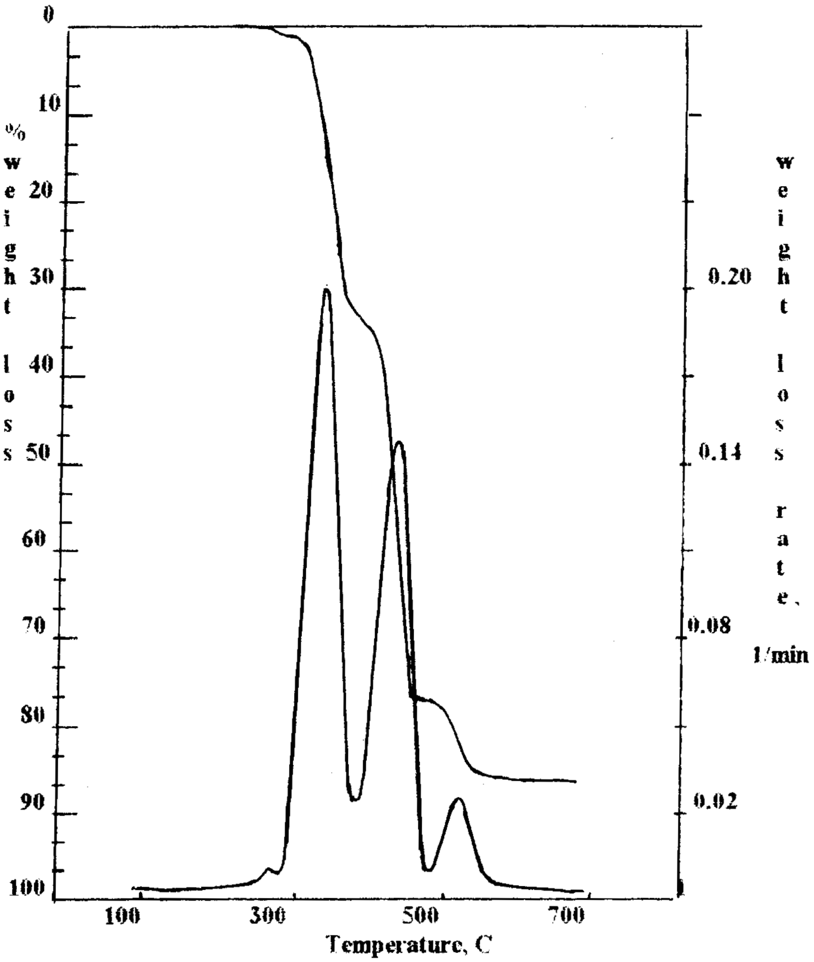

FIGURE 1 TG and DTG curves for the PP 22-01-5C material, obtained at heating rate of 7 degree/min in air.

TABLE III Thermal Characteristics of Flame Retardant Polyolefine Materials

Stage	*Index*	*PP Chisso 2654*	*FRPP Moplen*	*Polyflam Rpp371*	*PP 22-01-5C*	*PP 22-01-3C*	*PETG*	*PE Funikon E-205*
I	Tbg, C	292	290	285	285	280	295	288
	Tmax, C	345	340	335	338	340	343	333
	Tt, C	372	372	370	373	373	390	398
	W1, %	33	34.2	30	32	32.5	22	46
II	Tbg, C	372	372	370	373	373	390	398
	Tmax, C	428	422	425	427	440	483	463
	Tt, C	460	453	465	475	478	507	490
	W2, %	74.5	74	69.5	77	75.5	65	67.5
III	Tbg, C	460	453	465	475	478	507	490
	Tmax, C	512	510	510	512	512	528	530
	Tt, C	540	530	525	533	545	540	610
	W3, C	83.5	83	77	85	83	69	84

by PP melting is observed on DTA curves in temperature range of 140–170°C. Low weight losses at heating PP samples up to 280–290 C (1–1.5%) are accompanied by heat absorption. The decomposition of flame retardant PP materials is carried out at the first stage as endothermal process and then it is developed with essential heat release particularly at last stage.

The total yield of nonvolatile residue for PP samples is within the limits of 15–22.5%. Maximum yield is observed for Polyflam RPP 371, Belgium.

It can be noted that for PP-22-01-5C and PP-22-01-3C samples the decomposition stage associated with intensive polymer charring is propagated in a wider temperature range (up to 475–478°C) than in other PP materials.

The flame retardant polyethylene samples melt at 95–130°C. Endothermal maximum intensity on DTA curve associated with the polymer transformation in melting state is higher for PETG than for PE Funikon E-205. The second and third stages of the decomposition for PETG are less exothermal than those of PE Funikon E-205.

Kinetic parameters for the first and second stages of thermo-oxidative decomposition of PP-22-01-5C and PP 22-01-3C are calculated using TG and DTG curves in accordance with the method [4].

The process for maximum decomposition rate is described by the equation:

$$(dc/dT)\max = -(Z/b)\exp(-E/RT\max)f(c),$$

where c – the degree of conversion on the stage, Z – the pre-exponential factor, b – the heating rate, E – the activation energy, R – the gas constant and T – peak temperature in K.

For the decomposition of sample at each stage:

$$f(c) = c^n,$$

where n is the order of the reaction.

Table IV demonstrates the effective kinetic parameters obtained for the decomposition and carbonization stages at the heating of PP 22-01-5C and PP 22-01-3C in air medium.

TABLE IV The Effective Kinetic Parameters for Thermo-oxidative Decomposition of PP samples

State	*Parameter*	*PP 22-01-5C*	*PP 22-01-3C*
1	Activation energy, Eeff, kJ/mol	130 + 2	120 + 2
	ln Z (Z:1/s)	9.021	8.100
	Reaction order, n	1.5	1.47
11	eff, kJ/mol	138 + 2	108 + 2
	ln Z (Z:1/s)	8.278	5.690
	n	1.52	0.72

The decomposition process of above flame retardant PP materials at first stage follows the reaction of fractional order with $n = 3/2$. This order of reaction is retained in the second stage of PP 22-01-5C decomposition. However, for PP 22-01-3C sample the reaction order is drawing near 2/3. Fractional order of the decomposition indicates complex character of this process. The effective activation energies for the first stage of the process of thermooxidative decomposition are 130 and 120 kJ/mol accordingly for PP materials studied. These quantities are typical for the oxidation of polyolefins. The second stage in temperature range of 370–480°C is associated with the formation of a carbonaceous material, which is partially burnt out during the heating of samples up to 545°C or above. Data obtained show that the composition formulation affects the kinetic parameters of general processes of the material decomposition. It should be noted that the formation of PP 22-01-3C carbonaceous product and its following oxidation lead to the production of a somewhat lesser amount of evolved fuel than that for PP 22-01-5C sample. It can be assumed that a favorable course for carbonization and a higher resistance to the oxidation of carbonized product formed result in a higher level of flame retardancy for PP 22-01-3C than that for PP 22-01-5C.

Accelerated thermal ageing or a 3 year keeping the PP materials at normal conditions had no visible effects on their flame retardancy level. As example, Table V shows the influence of temperature and ageing duration on flammability of PP 22-01-5C material.

However, the physico-mechanical properties of the material are essentially changed, in particular, during ageing at 140–150°C. The initial characteristics of PP 22-01-5C and standard methods for their testing are given in Table VI. Figure 2 shows deformation diagrams

TABLE V PP 22-01-5C Flammability Dependence on Temperature and Duration of Accelerated Ageing

Temperature, C	*100*	*150*	*150*	*150*	*150*	*150*
Ageing duration, days	168	4	7	14	21	28
LOI, % vol.	26.5	27.5	27.6	27.6	27.6	27.6
UL-94 V Rating (3mm)	V0	V0	V0	V0	V0	V0

TABLE VI The Initial Properties of Flame Retardant PP Material

Indices	*Testing Method*	*PP 22-01-5C*
Density, kg/m^3	GOST 15139-69	1250
MFI, g/10 min (230/21.N)	GOST 11645-75	13
Tensile strength, MPa	GOST 11262-80	18–19
Elongation at break, %	GOST 11262-80	130
Fragile temperature, C	GOST 16783-73	minus 15
Impact strength, kJ/m^2	GOST 4643-71	48
LOI, %	GOST 12.1-044-89	27.5
UL-94 V Rating	GOST 28157-89	V0(3mm) V0(2mm)

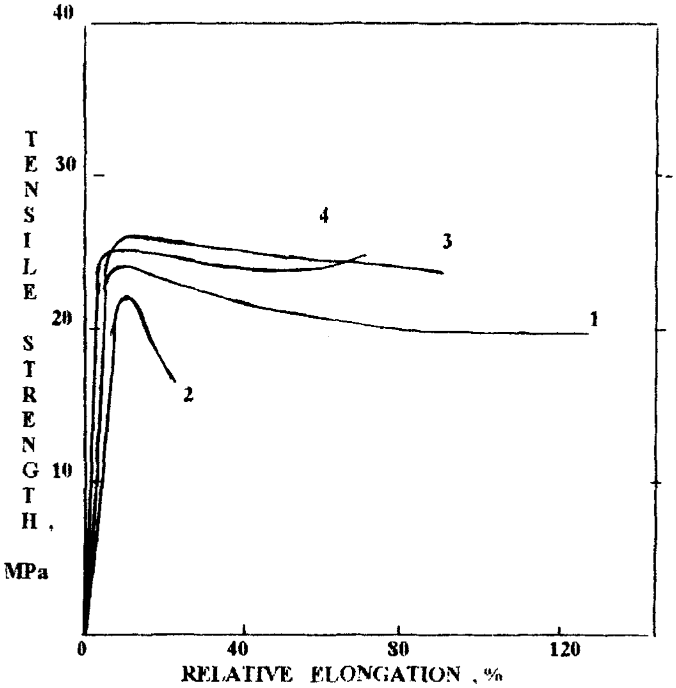

FIGURE 2 Deformation diagrams for flame retardant PP 22-01-5C material: 1-initial sample; 2-after 23 day's ageing at 150 C; 3-after 42 day's ageing at 120 C; 4-after 42 day's ageing at 100 C.

for the samples after some duration of thermal ageing. The shape of initial plots of the deformation diagrams for the PP 22-01-5C samples during their thermal ageing shows no considerable changes (Fig. 2). The exception are diagrams obtained for ageing PP 22-01-5C samples at 150°C and 21–23 day's duration of heat action. These diagrams are characterized by a strong decrease of strain after the fluidity point up to a full destruction of the samples (Fig. 2, curve 2). It should be noted that the values for fluidity limit and conventional modulus of elasticity are changed during thermal ageing of the above material in the range of experimental data scattering.

Tensile strength of the material remained almost constant during the exposure of samples up to 42 days at 100 and 120°C. At 140°C exposure the tensile strength of PP 22-01-5C samples increases over 25% after 2–4 ageing days and achieves 22–23 MPa. This level of tensile strength for the material remains during ageing up to 21 days. Subsequent thermal ageing increases the evolution of gaseous decomposition products and decreases the tensile strengh of PP samples.

The increase of temperature up to 150°C intensifies the thermo-oxidative ageing of PP materials. A 20–23 MPa level of tensile strength for PP 22-01-5C samples is observed during 21 day's exposure. By 23 days, the strength decreases to 16.4 MPa.

Maximum elongation at break for the flame retardant PP samples is decreased from 130% to 70–80% during first days of ageing at all temperatures. Afterwards, a monotonous decrease in elongation to 62% during 42 day ageing at 100°C is observed.

The essential decrease of maximum relative elongation takes place in time interval of 7–14 days, when the samples are exposed to heating at 140° and 150°C. A lowering of elongation follows up to a total destruction of the sample.

The dependence of the sample impact strength on temperature and ageing duration has a complicated form (Fig. 3). Impact strength is increased during 1–2 day's exposition at any temperature studied but, in particular, at 120°C (almost double comparing to the initial value). After 28 day's ageing at 120°C impact strength of the material is 85–88 kJ/m^2.

Increasing temperature and the duration of ageing result in the decrease of impact strength. After 14 day's ageing at 140°C the sample impact strength achieves its initial value and after 21 day's ageing at 150°C it is 60–68 kJ/m^2.

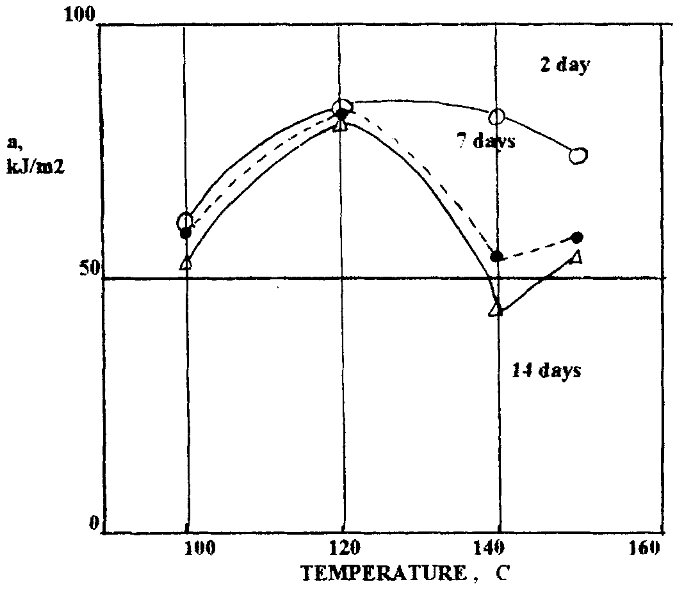

FIGURE 3 The influence of Temperature and ageing duration on impact strength of PP composition.

It should be noted that the reactions, which lead to ageing of PP material begin to develop at temperature corresponding to the increase of molecular mobility of the polymer. The shape of curves for the dependence of elongation and impact strength on ageing conditions indicates the competition of the degradation and chain cross-linking reactions. The growth of impact strength and the decrease of elongation in ageing initial period are typical for predominance of cross-linking reactions over the degradation ones in this time.

The change of physico-mechanical indices for flame retardant PP material during its ageing can be described by exponential equation:

$$I = I_0 \exp - \{A\, t \exp(B/RT)\},$$

where

I and I_0 are current and initial physico-mechanical index,
T is temperature in K,
t is ageing duration in days,
A and B are numeral coefficients.

Similar equation mode predicts the appearance of an extreme in functional dependence of the index on ageing time. As example, the appearance of maximum on plot for tensile strength versus ageing duration in initial period of PP 22-01-5C ageing at 140°C.

Flame retardancy level has no essential change even after accelerated PP 22-01-5C ageing at high temperature (Tab. V). The comparison between flame retardancy change and physico-mechanical properties during PP 22-01-5C ageing permits us to conclude that the flame retardant system used does not migrate from the material and does not lose its flame retardant performance under the investigated ageing conditions.

These investigations show that the flame retardant polyolefinic materials developed by domestic producers and used for making parts in TV devices are equivalent to foreign materials recommended for this application.

CONCLUSION

The study of flammability and thermal properties of a number of flame retardant polyolefinic materials used in Russia or recommended for making parts in color televisors and other electronic apparatus has been carried out. Bench-scale testing methods of UL-94V and LOI have been used for comparative evaluation of the polymeric material's flammability. The influence of sample size and testing conditions on flammability parameters has been noted.

The flame retardant materials based on the same polymer show similar LOI values and UL-94V ranking, when the samples are evaluated in the same standard conditions.

The distinction in burning behaviour of the polymeric materials studied is noted in LOI test with ignition of the vertical sample at the bottom and upward flame spread over its surface. The distinction between the flame retardant polymeric materials with top UL-94 VO ranking is also revealed on repeated ignitions of the samples.

The results obtained show the potential of using UL-94V test for more detailed screening of flame retardant thermoplastics with top V0 rating.

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