



Research article

UDC 699.812+691.175

DOI: 10.34910/MCE.116.7



Intumescent compounds for fireproofing of polymer pipelines

V.V. Bogdanova¹ , O.I. Kobets¹ , O.N. Buraya¹ , A.A. Ustinov² , O.A. Zybina² 

¹ Research Institute for Physical Chemical Problems of the Belarusian State University, Minsk, Republic of Belarus

² Peter the Great St. Petersburg Polytechnic University, St. Petersburg, Russia

✉ au.spbpu@gmail.com

Keywords: fire clutch, thermoformable composite, polymer binder, intumescent system, foaming ability, heat-insulating efficiency, thermal analysis

Abstract. Reducing the rate of fire spread in buildings through the intersections of enclosing building structures with polymer pipelines is achieved by using fire clutches equipped with liners made of expandable materials. In case of a fire, it is ensured that the spread of flame through polymer pipelines is hindered due to formation of a heat-insulating foamed layer. This layer does not allow the polymer low-melting pipelines to heat up to 120°C. Comparative assessment of heat-insulating and mechanical properties of two intumescent composites and their charred products were carried out to clarify the reasons for their heat-insulating efficiency. Composites included the same intumescent system (ammonium polyphosphate / pentaerythritol / dolomite / thermally expanded graphite) and different polymer binders. The research was conducted using the following methods: complex thermal and X-ray analyses, scanning electron microscopy, a number of standard and original techniques. It was established that heat-insulating ability of a charred layer is determined by temperature intervals in which interactions of initial components occur. The best mechanical, heat-insulating and morphological properties of investigated intumescent composites and their charred layers are achieved when temperature ranges referred to formation of organo-mineral framework and volatile thermolysis products are coinciding. For the composites studied in this paper, this temperature range was 350–400 °C. Thanks to this knowledge, it becomes possible to develop new fire-retardant composites with improved properties instead of selecting the components in empirical way. As a result, the general quality of fire-retardant materials may grow and their properties will be sufficient not only to meet the construction requirements, but to properly operate in case of fire as well, practically justifying the predicted effect.

Acknowledge: The research is partially funded by the Ministry of Science and Higher Education of the Russian Federation as part of World-class Research Center program: Advanced Digital Technologies (contract No. 075-15-2020-934 dated 17.11.2020)

Citation: Bogdanova, V.V., Kobets, O.I., Buraya, O.N., Ustinov, A.A., Zybina, O.A. Intumescent compounds for fireproofing of polymer pipelines. Magazine of Civil Engineering. 2022. 116(8). Article no. 11607. DOI: 10.34910/MCE.116.7

1. Introduction

Fire retardant coatings (or thermoformable coatings, TFC) have proven themselves effective as means of preventing the fire spread along building communications. They are also finding the niche as a part of constructive elements themselves, for example, in fire clutches (Fig. 1, a-d). These composites include such ingredients as polymer binders, pore-forming agents, dehydrating agents, combustion retardants, mineral fillers [1–9], and carbon materials (e.g., thermally expanded graphite, TEG, that has

recently found a lot of applications [10–18]). Combined together and exposed to heating, such composites tend to expand and provide thermal insulation to protected surface.

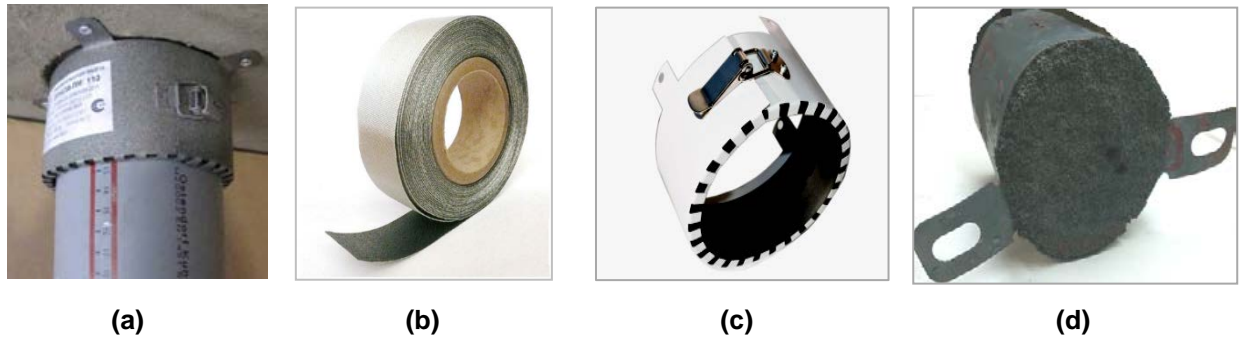


Figure 1. Fire sleeve – preventive fire protection of polymer pipelines crossing floors in a multi-storey building: (a) fireproofing sleeve fixed on a protected polymer pipeline; (b) a tape-insert made of a polymer expandable composite - the main working element of a fire clutch; (c), (d) fire clutch with liner before and after fire tests.

There are requirements for fire retardant composites used as filler in fire couplings: the integrity of polymer pipeline exposed to heating for 15-180 minutes must be ensured; and the temperature on unheated side of a pipeline, when thermal degradation already begins, must not exceed 120 °C. In addition, TFCs themselves must be durable and elastic, as well as the foamed layer must not only provide thermal insulation, but also be able to withstand the convective air currents in case of fire.

Applying fire retardant composites in order to protect the pipelines is not a new technique; in [19] authors suggest a fire retardant material with thixotropic properties and high viscosity, which at high temperatures becomes a foam-silicate with low conductivity and low mechanical strength; such properties ensure both the heat insulation of a pipeline or a cable connection, and an easy removal of foamed substance from the cavity between pipeline's surface and clutch.

Intumescent coatings may also be applied to surfaces of polymer cables directly; however, the dry coating must possess high values of flexural strength, which is a problem. It's being solved by using an intumescent compound as a filler in roll materials; such material, for example, was developed in [20], and it passed certification tests showing R15, R45 and R60 fire resistance limits when applied to steel beam. In [21] authors propose a set of fire retardant bendable roll materials describing their advantages over intumescent paints: adoption of the form of constructive element (waste minimization), the speed of installation, installation at any time of the year and under different climatic conditions; such materials are also finding good application in oil and gas complex where hydrocarbon fires may take place. So, there is no doubt that intumescent compounds in different forms are finding a good niche in different areas of construction.

However, development of new TFCs is mostly based on empirical ideas and hindered because of many requirements applied to them. The lack of theoretical knowledge about the processes occurring in these materials under heating conditions doesn't allow to affect their properties directly at the development stage. The abovementioned factors mostly refer to TFCs containing TEG as a foaming agent. Thermally expanded graphite, after decomposition of its layered structure at the initial stages of heating, forms bulk folded heat insulating structures [22]. In general, the heat insulating effect of TEG is based on chemical (interaction of graphite with an acid in its interlayer space resulting in formation of water vapor, CO₂, SO₂) and physical mechanisms [22, 23]. However, TEG being an ingredient of fire retardant compositions deteriorates physical and mechanical properties of initial coatings and products of their thermolysis [14, 23, 24].

The studies were carried out to optimize the formulation of a fire retardant composition based on melamine-formaldehyde resin [25]; the main goals were to reduce the time and cost of new TFCs development, and to obtain information about the factors that make a decisive contribution to their fireproofing ability. At the same time, a correlation was found between the heat insulating ability and the quantitative content of carbon-containing (pentaerythritol) and foaming (TiO₂) agents in the TFC. Also, the contribution of a polymer binder to heat insulating ability of TFC must also be taken into account and studied properly.

Such studies focused on assessing the correlation between the nature of polymer binder and the properties of TFCs were carried out in [4, 26, 27]; TFCs contained ammonium polyphosphate, pentaerythritol, melamine and / or titanium dioxide. The authors of [4, 26] used water-dispersed copolymers of vinyl acetate, acryl, styrene and vinyl toluene acrylate as polymer binders. In [27], organosilicon polymer and acrylic copolymer were used as binders. However, there is a lack of information about the reasons for

the different efficiency of TFCs containing various polymer binders, thus direct development of expandable materials with required heat insulating properties is hindered.

Comparative studies of TFCs containing identical char-forming system and various polymer binders were carried out in order to obtain information on the reasons for the different heat insulating efficiency. The abovementioned is the main goal of the current study.

2. Materials and Methods

Two waterborne compositions (TFC1 and TFC2) were selected for the study, made on the basis of the following polymer dispersions: a vinyl acetate-ethylene emulsion of Mowilith LDM 1865 WP and a styrene-acrylic emulsion of Osacril OSA H with the same content (40 %) in the investigated compositions. A mixture of ammonium polyphosphate (APP, NH_4PO_3), pentaerythritol (PET, $\text{C}_5\text{H}_{12}\text{O}_4$), and dolomite (DI, $\text{CaMg}(\text{CO}_3)_2$) was used as a char-forming system (CFS) in both TFCs. Each of the compositions additionally contained the same amount of TEG. The fractional mass ratio of the CFS components in both compositions was: AFP: PET: DI: TEG = 1.7: 0.8: 1: 1.

The foaming capacity of TFC was estimated by the coefficient of foaming, calculated from the relative change in the thickness of the samples during heating in the dynamic mode in the temperature range from 20 to 200–800 °C every 100 °C: $K = \delta_l / \delta_0$, where K is a coefficient of foaming; δ_l is the thickness of foamed layer mm; δ_0 is the initial thickness of the TFC sample, mm [28].

The value of heating rate was 10 °C / min. The coating was considered satisfactorily foamed if the value of K was at least 10.

The mechanical strength (relative compression deformation) of the foamed products formed as a result of TFCs heating was determined according to the laboratory method: the products of heat treatment of a TFC sample at 500 °C for 10 min were subjected to tests. The relative compression deformation (ε , %) was determined by the formula: $\varepsilon = \frac{h_0 - h_{res}}{h_0} \cdot 100\%$, where h_0 and h_{res} are the height (mm) of the heated foamed sample before and after applying the load ($\sim 8 \text{ kg} / \text{m}^2$) [29].

X-ray phase analysis (XPA) of initial and charred composites was carried out on a DRON-2 diffractometer (Cu $K\alpha$ – radiation). Crystalline phases were identified using the ICDD software [30].

The microstructure of the surface of sections of foamed char residues after testing TFC samples was investigated using a LEO1420 scanning electron microscope (K. Zeiss, Germany).

The data of complex thermal analysis (differential scanning calorimetry – DSC, thermogravimetric analysis – TG, differential thermogravimetric analysis - DTG) was obtained in temperature range 20–600 °C (heating rate 10 °C / min in an oxygen / nitrogen atmosphere) using Netzsch STA 449 C setup.

Fire tests on heat insulating ability of a TFC sample (50×350 mm) embedded in two turns inside the body of a standard fire sleeve fixed to a fragment of a polypropylene (PP) pipe were carried out according to the laboratory method described in [31]. The method of conducting the fire tests, simulating the conditions stated in GOST R 53306–2009, was somewhat different from the standard method: in the laboratory furnace, the temperature regime was set with a lower final temperature inside the furnace (850 °C instead of 1000 °C), while taking into account the smaller size of the test chamber (17×22×32 cm) respectively scaled the amount of combustible load (the length of the PP pipe with a diameter of 40 mm with a wall thickness of 4 mm was 250 mm). During the tests, the time was measured in minutes until the limit state was reached: temperature of 120 °C on the unheated surface of a polymer pipe fragment and/or deformation, destruction of a polymer pipe fragment behind the enclosing structure from the unheated side. The tests were considered completed when one of the limit states was reached. The layout of the sample, the arrangement of thermocouples and the graph of the temperature regime in the laboratory test setup are shown in Fig. 2 a, b.

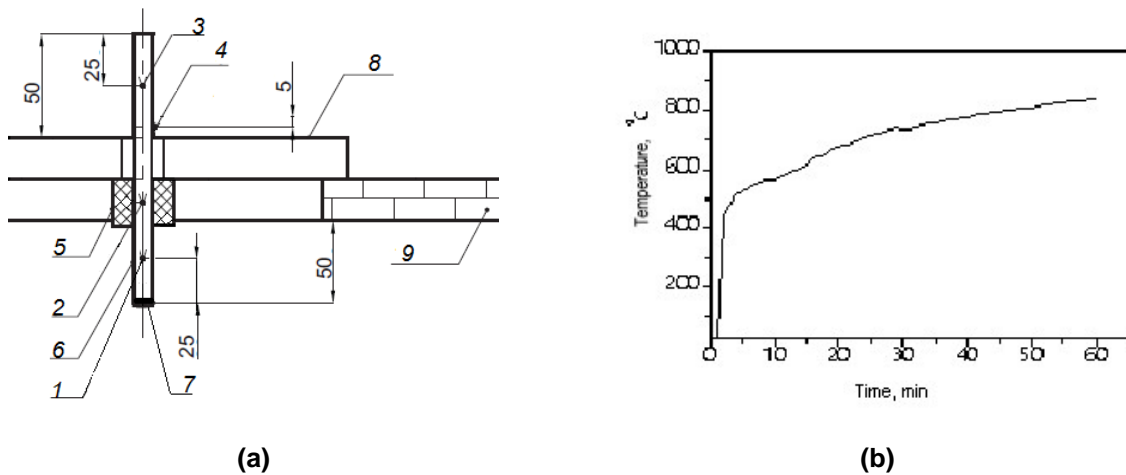


Figure 2. Schematic layout of the sample and thermocouples in the laboratory setup (a), graph of the rate of temperature rise in the chamber of the laboratory test furnace (b).

1–3 – thermocouples located in the internal cavity of the pipeline fragment;
 4 – thermocouples located on the unheated surface of the pipelines fragment;
 5 – fire coupling (cut-off protective device); 6 – a fragment of a polymer pipeline;
 7 – plug; 8 – furnace cover; 9 – furnace; all dimensions in fig. (a) are in mm

3. Results and discussion

To obtain more complete information on heat insulating ability of TFC1 and TFC2, not only the limiting factor was measured - the time of reaching 120 °C on the unheated side of the pipeline, but also the change in temperature inside the cavity of the pipeline from heated (thermocouple 1) and unheated (thermocouple 3) sides, as well as in the middle of the sleeve (thermocouple 2) with a sample of the thermoformable composition (Fig. 3, a).

It follows from the results of fire tests that the fire resistance limit of TFC2 is 1.8 times higher compared to TFC1. At the same time, thermocouples 1 – 3 (Fig. 3, a, b) located in the fire clutch (2), inside the polymer pipe from the heated side (1) and from the unheated side above the foamed test sample (3), show that the rate of temperatures rising during 10- and 40-minute heating are higher for TFC1 compared to TFC2 by 1.3; 1.8; 3.1 and 1.1; 1.4; 1.8 times respectively.

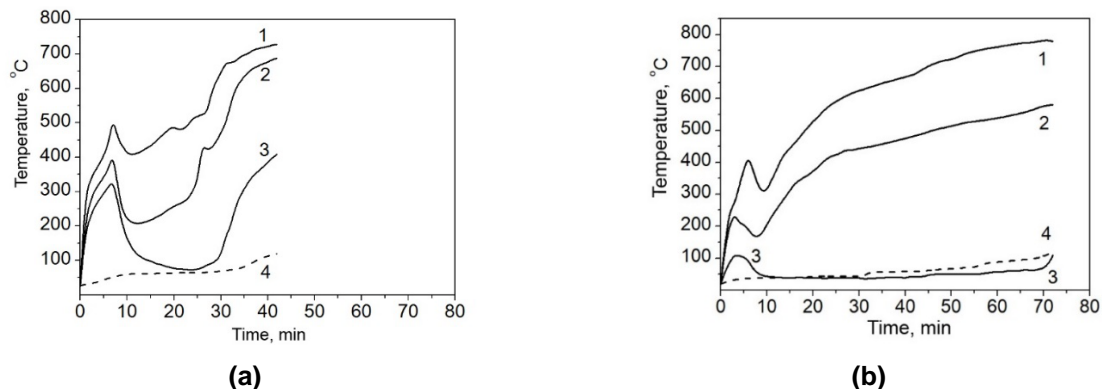


Figure 3. Graphs of temperature change depending on time while heating a fragment of a polymer pipeline with a fire sleeve containing: (a) TFC1; (b) TFC2.

For composition TFC1, as compared to TFC2, on all thermocouples (1–3) located inside the polymer pipe, at the initial stage of testing (the first 10 min), higher maxima of temperature rise were recorded. This fact indicates a lower thermal insulating ability of the solid products of TFC1 thermolysis. It should be noted that during the first 10 minutes from the start of the tests, when in both cases a rapid short-term rise in temperature was observed inside the polymer pipe, no change in its external state was observed. Fire tests were stopped when one of the limiting states was reached: for TFC1 after registering the beginning of deformation of the polymer pipe (Fig. 4, a), and for TFC2 – upon reaching a temperature of 120 °C on the unheated side of the polymer pipe while maintaining its original integrity (Fig. 4, b).

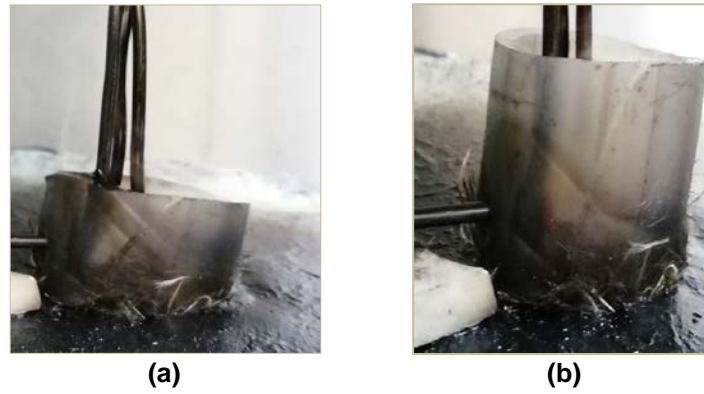


Figure 4. Photo fixation of the state of polymer pipe from the unheated side at the end of fire tests of TFC1 (a) and TFC2 (b).

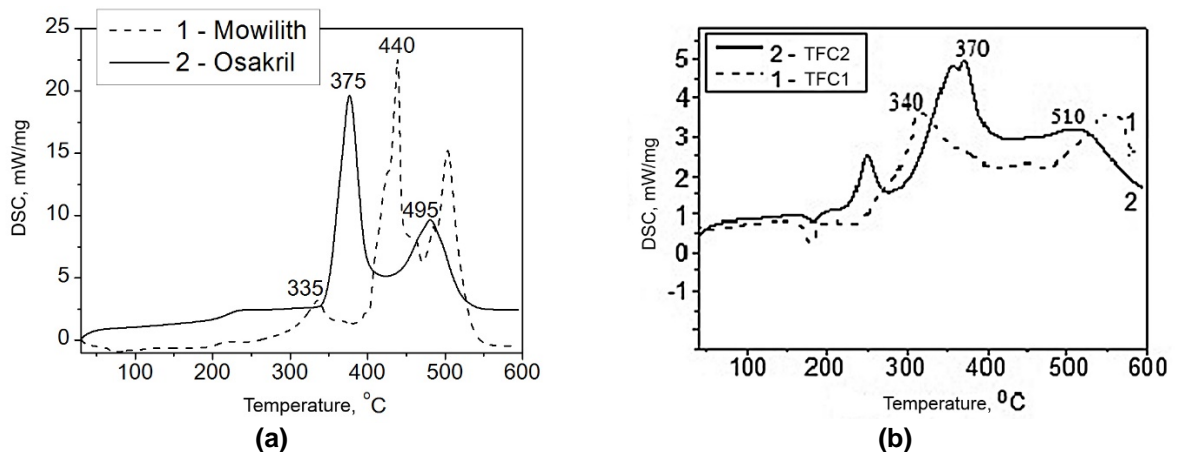
To determine the reasons for the different thermal insulating properties of the investigated compositions, comparative studies of the foaming ability and physico-mechanical properties of solid products of TFC1 and TFC2 at temperatures of 20–800 °C with an interval of 100 °C were carried out (Table 1) [32]. When comparing mechanical characteristics of the investigated compositions with their thermal protective efficiency, it should be noted that the rate of foaming is higher in the entire investigated temperature range for sample TFC2 with best fire resistance. The relative compression strain (ε , %) for the thermolysis products of TFC1 (20 %) is 5.7 times bigger than that of TFC2 (3.5 %), which is an additional factor that determines the low thermal insulating ability of TFC1.

Table 1. Physical and mechanical properties of TFCs charred products.

Sample	Parameter	Temperatures range, °C					
		20–300	20–400	20–500	20–600	20–700	20–800
TFC1	K	5.3	7.8	8.1	7.2	5.3	7.3
	ρ , g/cm ³	0.139	0.062	0.067	0.047	0.057	0.034
	Δm , %	12.5	36.4	46.2	57.1	64.7	70.6
TFC2	K	8.9	19	14.9	10.5	9.2	9.2
	ρ , g/cm ³	0.136	0.034	0.037	0.038	0.041	0.039
	Δm , %	9.4	44.8	52.4	64.3	70	72.9

Making a conclusion on the factors that determine effectiveness of TFCs is incorrect if only the values of mechanical parameters of charred layers are assessed. Comparative studies on behavior of both TFCs themselves and their components under heating conditions were carried out at the next stage.

Fig. 5 (a-c) shows the differential scanning calorimetry data for individual polymer binders, TFCs based on them, and a charring system. Thermal transformations of individual polymer binders occur in different temperature ranges (Fig. 5, a): temperatures of the onset of weight loss and exo-effects accompanied by almost complete weight loss are 216 °C, 335, 440, 503 °C for Mowilith; and 231, 375, 495 °C for Osakril.



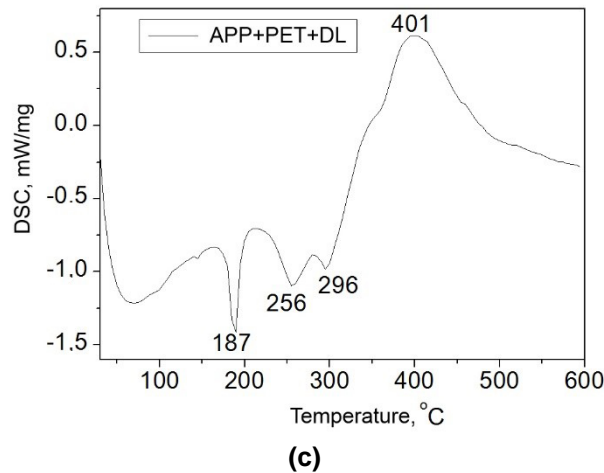


Figure 5. DSC data for: a – binders; b – TFCs; c – charring system; curve numbers correspond to: 1 – Mowilith binder or TFC1 based on Mowilith; 2 – Osakril binder or TFC2 based on Osakril.

According to DSC data (Fig. 5, b), changes in the maxima of thermal effects are observed when comparing data for polymer binders and TFCs. This is explicitly for TFC1, where the exo-effects of individual Mowilith in the range of 440 and 503 °C (Fig. 5, a) were transformed into exo-peaks with maxima at 340 and 540 °C (Fig. 5, b). A slightly different effects occur in TFC2, where the shift of the main exo-effect of the individual Osakril (375 °C) is practically not observed, while the temperature of the second exo-effect (495 °C) shifts to higher point (510 °C).

Endo-effects in charring system are recorded in the range of 200–320 °C which can be observed in DSC curves (Fig. 5, c); such effects are typical for components of intumescent compound. With a further increase in temperature, a broad exo-effect is recorded with a maxima of 401 °C. Proceeding from the fact that this effect is accompanied by insignificant weight loss, it is assumed to be due to structural transformations of thermolysis products of CFS components.

To verify this assumption, an X-ray study of the charred products and their physical state in the temperature range of 200–600 °C was conducted (Table 2). No visible changes in the state of the initial intumescent compound are observed in the range from 200 °C to 250 °C. The onset of sintering without changing the color of the original compound becomes noticeable at about 300 °C. The processes of melting and formation of a foamed layer occur in the temperature range of 300–400 °C, and complete foaming of the heated compound occurs at 400–500 °C [33].

Table 2. X-ray phase analysis (XRD) data and the physical state of charred products obtained by heating the intumescent compound.

T, °C	Physical state	XRD data
200	P	CaMg(CO ₃) ₂ , NH ₄ PO ₃ , C ₅ H ₁₂ O ₄
250	P	CaMg(CO ₃) ₂ , NH ₄ PO ₃ , C ₅ H ₁₂ O ₄
300	S	CaMg(CO ₃) ₂ , NH ₄ PO ₃ , C ₅ H ₁₂ O ₄ , UP
400	F	CaMg(CO ₃) ₂ , CaCO ₃ , UP
500	F	CaMg(CO ₃) ₂ , CaCO ₃ , UP
600	F	CaMg(CO ₃) ₂ , CaCO ₃ , UP

Note 1 – Indication of physical state: P – powder; S – sintering; F – foaming.

Note 2 – UP – unidentified phase.

According to X-ray phase analysis, the change in intensity of reflections of individual components of the CFS on the X-ray diffraction patterns appears at about ~ 300 °C. At this temperature, in addition to the initial components (APP, PET, DL), an unidentified phase was detected by X-ray diffraction, the appearance of which can be explained by APP thermolysis resulting in formation of a mixture of polyphosphoric acids that initiate PET esterification reactions [34], as well as formation of products of interaction with carbonate component. At the same time, in temperature range from 300 °C to 400 °C, according to XRD, the main crystalline phases are CaMg(CO₃)₂ and CaCO₃ with a decrease in intensity of their reflections followed by an increase in heating temperature. Presumably, as the temperature rises, the carbonate Ca-Mg-containing salt dissociates into its constituent CaCO₃ and MgCO₃ with their simultaneous interaction with condensed phosphoric acids formed during APP thermolysis [35]. At 400 °C, the peaks referred to crystalline phases of PET and APP disappear from X-ray diffraction patterns of CFS, which indicates the intensification of

interaction between products of thermal decomposition of CFS components. Thus, as a result of X-ray examination of solid products of TFC thermolysis, it was found that the exothermic effect on DSC curve in range of 300–400 °C (Fig. 5, c) is due to formation of an unidentified organo-mineral product.

An important aspect affecting the qualitative and quantitative characteristics of thermoformable composites is the formation of gaseous products while TFCs heating; those products take part in formation of heat insulating porous structures [35, 36]. In accordance with this, it is of interest to consider the data of DTG study of TFCs and their components. According to DTG data for film-forming agents (Mowilith, Osakril) and polymer compositions based on them (TFC1, TFC2), it can be seen (Fig. 6 a, b) that weight loss occurs at a maximum rate in the temperature range of 300–400 °C. At the same time, for Osakril and TFC2, the maximum temperatures are 30–40 °C higher than those for Mowilith and TFC1. Intensive thermolysis of the charring system is recorded in the temperature range 220–350 °C with a maximum at 244 °C (Fig. 6, c).

Comparison of thermal transformations of individual binders, TFCs and CFSs with the fire resistance limit of the composites suggests the existence of a dependence of heat insulating efficiency of TFCs both on the nature of polymer binder and on the coincidence of temperature ranges of thermal transformations of all components of charring composites.

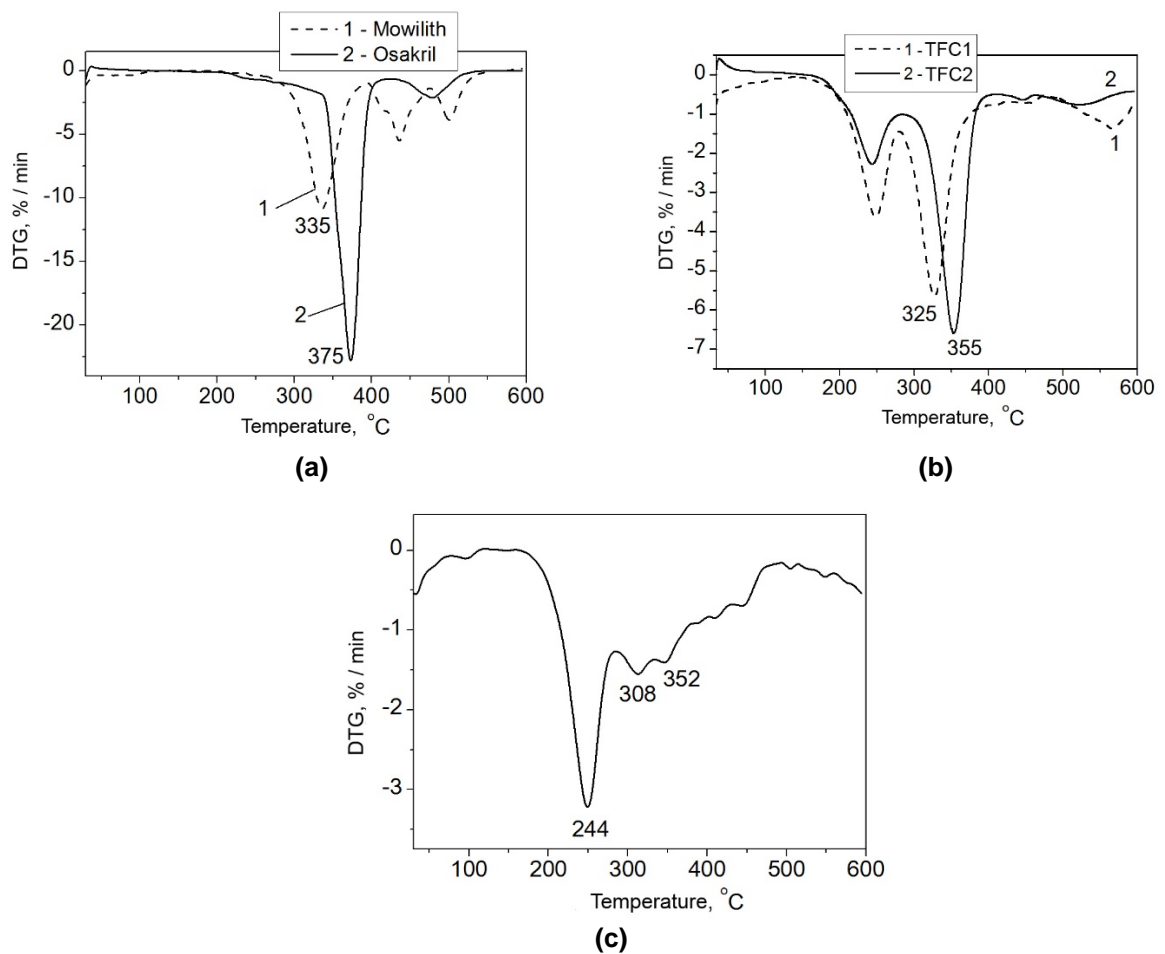


Figure 6. DTG study data for: a – binders; b – TFCs; c – gas-char-forming system; curve numbers correspond to: 1 – Mowilith binder or TFC1 based on Mowilith; 2 – Osakril binder or TFC2 based on Osakril.

To understand the reasons for the different heat-insulating performances of TFC1 and TFC2 containing the same charring compositions and different polymer binders, it is of interest to compare the temperature ranges of thermal transformations of CFS, Mowilith and Osakril. Thus, individual binders differ significantly in temperature maxima of the rate of weight loss caused by the formation of gaseous products (Fig. 6, a): for Mowilith – 335 °C, for Osakril – 375 °C. The same trend persists for compositions based on these binders (Fig. 4, b): TFC1 – 325 °C, TFC2 – 355 °C. DTG curves show a peak in the rate of weight loss with a maxima of 244 °C, due to gasification of CFS (Fig. 6, c). X-ray studies show that an intense interaction between products of CFSs decomposition occur in the temperature range of 300–400 °C leading to formation of amorphous organo-mineral products. Due to this, we assumed that high heat-insulating efficiency of TFC2 may be explained by coincidence of temperature intervals in which the formation of organo-mineral framework and its foaming occurs. Coincidence of the temperature ranges (350–400 °C)

referred to intense formation of gaseous products and, at the same time, formation of an organo-mineral framework was established for TFCs based on Osakril binder; this composite has the highest heat-insulating ability.

This assumption is confirmed by the data of thermogravimetric analysis: the total mass loss at 500 °C for TFC1 is 68 %, and for TFC2 – 50 %.

An additional fact in favor of the formation of a frame structure are values of physical and mechanical properties of charred products: the foaming coefficient of TFC1 and TFC2 in temperature range of 20–400 °C is 19.0 and 7.8 (Table 1). In addition, charred layers formed as a result of fire tests were investigated by scanning electron microscopy (SEM). It was found that in case of TFC2 a more uniform porous charred structure was formed in comparison to TFC1 (Fig. 7, a, b). The morphological structure of charred layer is known to affect its mechanical strength and heat insulating properties [3, 36].

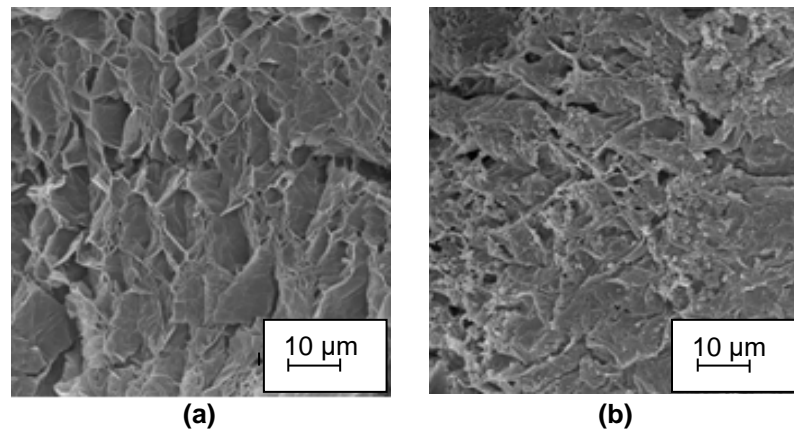


Figure 7. Microstructure of charred layers according to scanning electron microscopy: a – TFC1; b – TFC2.

The assessment of data on thermal and phase transformations of TFCs and their components was conducted. It shows the importance of knowing the temperature ranges in which transformation of components occur. This knowledge allows to consciously choose the components of TFC on development stage, thus creating new composites with better properties. Such composites may find wider niche in modern construction because of their better compliance to building requirements.

4. Conclusions

1. A study of two intumescent composites based on two different polymer binders (vinyl-acetate ethylene (Mowilith) and styrene-acrylic (Osakril) emulsions) and the same char-forming system (APP: PET: DL: TEG = 1.7: 0.8: 1: 1) was conducted in order to clarify their possible application as a part of fire clutches. Such properties of intumescent composites and charred layers as foaming ability, mechanical strength, structural morphology and heat-insulating ability were investigated. Test methods included thermal analysis (DSC, TG, DTG), X-ray phase analysis and scanning electron microscopy.

2. It was established that heat-insulating ability of a charred layer is determined by temperature intervals in which interactions of TFC components occur. The best mechanical, heat-insulating and morphological properties of investigated intumescent composition with higher heat-insulating ability and its charred layer are achieved when temperature ranges referred to formation of organo-mineral framework and volatile thermolysis products are coinciding. Such temperature range for this composition that was established in the current work is 350–400 °C.

3. Results of this research are opening the way to more conscious approaches to developing of fire retardant composites based on intumescent systems. As many components of such composites are chosen empirically, the properties of final materials may not be sufficient; and if polymer binder and intumescent system are selected according to their thermolysis ranges, a more effective composite may be developed. Such statement may result in a wider use of fire retardant composites in civil engineering.

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Information about authors:

Valentina Bogdanova, Doctor of Chemical Sciences

ORCID: <https://orcid.org/0000-0002-8557-9925>

E-mail: bogdanova@bsu.by

Olga Kobets, PhD of Chemical Sciences

ORCID: <https://orcid.org/0000-0002-6702-7430>

E-mail: kobets@bsu.by

Oksana Buraya,

ORCID: <https://orcid.org/0000-0002-6241-1281>

E-mail: fhp@bsu.by

Andrei Ustinov,

ORCID: <https://orcid.org/0000-0001-6350-5338>

E-mail: au.spbpu@gmail.com

Olga Zybina, Doctor of Technical Sciences

ORCID: <https://orcid.org/0000-0001-9401-7206>

E-mail: zybina.oa@spbstu.ru

Received 27.09.2021. Approved after reviewing 08.07.2022. Accepted 19.07.2022.