

Improvement of Methodology for Assessing Fire-Protective Efficiency of Intumescent Coatings Applied on Metal Constructions

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Abstract. The article observes the possibility and feasibility of introducing new and improved methodology for assessing the performance of fire retardant intumescent coatings for steel structures. The results of a comparative assessment of coatings based on a set of parameters obtained in laboratory conditions are presented: swelling coefficient, adhesion coefficient, adhesion of the coating system under high temperature conditions, kinetic parameters of thermolysis, thermal insulation. The small-scale laboratory test was developed to evaluate thermal insulating ability of 4 intumescent coatings applied to small steel plates. An improvement of the control method for assessing the fire retardant efficiency of intumescent coatings is proposed – instead of one test plate with dimensions of 600x600x5 mm with intumescent coating applied, a design of a metal frameholder is proposed, in which 4 plates with dimensions of 300x300x5 mm can be fixed, and a set of thermocouples can be installed on each of them; all 4 plates are heated in the same conditions, thus the contradictions which may occur between the results of separate experiments are avoided. Such improvements made in the methodology of investigating the properties of intumescent coatings appeared to be fruitful, as correlation can be established between the laboratory tests and large-scale tests.

1 Introduction

The method of fire protection of building structures with thin-layer intumescent compositions is one of the most demanded at the present time, and the growth rate of the market for intumescent compositions is increasing annually according to market reports, e.g. the one made by Paints and Coatings Industry [1]. It is mostly referred to the increased growth of building, development of gas and oil industry, and to the restrictions applied by governments in the scope of health care (intumescent coatings are halogen free which makes them a preferable choice both in design and fireproofing). The application of intumescent

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compositions (hereinafter referred to as IC) significantly increases the fire resistance of building structures: under heat exposure, the coating formed by the IC expands as a result of thermolytic synthesis of a foamed polymer layer (charred layer). The charred layer prevents the effects of heating and convective flame flows on the building structure for a certain time, thereby increasing the time during which the structure will retain its load-bearing capacity and other functions in the event of a fire. Thermolytic synthesis of charred foam occurs due to the superposition of IC components, capable of forming crosslinked polymer structures under thermal action (which, as shown in previous studies, are based on melamine-aldehyde resins [2]).

The main parameter of IC which determines the possibility of their application and is determined by the regulatory documents of the Russian Federation, is fire retardant efficiency, which, in the case of IC application on metal structures, is characterized by the time (in minutes) from the start of the fire test until the critical temperature (500 °C) is reached by a standard steel structure with a fire retardant coating, and is determined by the method described in part 5 of Russian National Standard GOST R 53295-2009 [3]. According to this method, a fire retardant is applied to steel columns of I-section of profile N20 or profile N 20B1 (hereinafter - samples); samples with thermocouples (pre-installed on them) are placed in a test furnace, where they are exposed to heating; the test is stopped when the sample reaches the critical temperature of 500 °C. The time from the beginning of the heating process until the moment the sample reaches the critical temperature characterizes the fire-retardant efficiency of the IC, and the IC is assigned the fire-retardant efficiency group from the 1st (not less than 150 minutes) to the 7th (not less than 15 minutes). Also, GOST R 53295-2009 provides for a control method for testing fire retardant coatings, within which the sample is a steel plate with a size of 600x600x5 mm with a fire retardant coating applied to it; in the process of heating the sample, the temperature on the unheated side of it is recorded, and the test is also stopped when the critical temperature of 500 °C is reached.

A similar, yet with a number of differences, method for determining the fire-retardant efficiency of ICs is described in the European standard EN 13381-8 [4], which provides rod structures of various types and sizes as test samples, thermal insulation with concrete blocks at both ends of the sample, as well as the critical temperature at which the test is stopped, at 1000 ° F (corresponding to ~ 537 °C).

The described approaches to conducting fire tests of intumescent coatings have a number of shortcomings and are criticized by some authors. [5,6]. Basically, this is due to the fact that ICs belong to reactive fire protection means, i.e. their fire-retardant efficiency is critically influenced by the amount of heat flux absorbed by the intumescent layer. The described methods do not take into account heat transfer in the expanding layer of charring foam, as well as the effect of physical and chemical transformations proceeding with heat absorption on the fire retardant efficiency of intumescent coatings. The conditions in the test furnaces are also quite far from those of a real fire. Also, these methods do not take into account the kinetics of chemical reactions underlying the thermolytic synthesis of charred foam, and this circumstance is extremely important, since the thermolytic synthesis proceeds differently depending on the temperature regime [7-10]. Thus, the conditions of the described fire tests take into account only external factors, such as temperature in the furnace, the temperature of the gas phase, etc., but do not take into account such factors as the kinetics of chemical reactions in the process of thermolytic synthesis of charred foam, its thermal conductivity, etc.

It is also important that large-scale fire tests are costly and time consuming. Also, there are no verified laboratory methods that would allow assessing the fire-retardant performance of an IC directly in the process of its development and adjusting its formulation without the need for large-scale fire tests. This circumstance affects the market of fire retardant materials

- the availability of express methods for assessing the fire retardant properties of ICs would significantly reduce the volume of counterfeit products on the market.

The current work presents the results obtained by the authors in the course of the development and research of a number of intumescent compositions, and in the course of further work it is planned to make proposals to a number of national regulatory documents (in particular, GOST R 53295-2009) based on these results. The authors have developed a model laboratory setup for determining the fire retardant efficiency of intumescent coatings. Such approaches were described in some previous works of other researchers, for example in [5] two installations were proposed for determining the fire-retardant efficiency of ICs, based on the use of emitting panels, which are able to be driven by a computer system and move in different directions relative to the test sample (plates with applied IC), thereby changing the amount of heat flux incident on the sample (system H-TRIS - Heat-Transfer Rate Inducing System). This method is quite costly and may be hardly performed in other laboratory.

2 Materials and methods

In accordance with the regulatory document of the Russian Federation, which establishes general requirements for fire protection for steel structures [3], to determine the fire retardant efficiency of intumescent fire retardant compositions, standardized complex thermophysical large-scale tests are used that are as close as possible to the conditions of a real fire; these methods are laborious and costly. Their use is advisable at the stage of material certification. Therefore, at the initial stage, to obtain data, we used laboratory methods for assessing individual parameters of an intumescent coating, which, of course, can characterize a change in its fire-retardant properties. As such parameters, we chose the volume of the intumescent layer (swelling coefficient), adhesion coefficient, kinetic parameters of IC thermolysis and thermal insulation capacity.

For the tests, the basic formulation of the fire retardant intumescent composition was used as an initial material (Table 1).

Table 1. Basic intumescent composition

No.	Component	Amount, %
1	Polymer binder	15
2	Pentaerythritol (PE)	10
3	Melamine (MA)	10
4	Ammonium polyphosphate (APF)	30
5	Additives	1
6	Solvent	Up to 100

On the basis of this composition, samples of fire retardants (table 2, samples IC 1 – IC4) were made in the laboratory of VDM "Pigment" LLC, differing in the type of polymer binder and the origin of intumescent components – melamine (MA), pentaerythritol (PE), ammonium polyphosphate (APP).

Table 2. Samples of intumescent coatings

Difference factors	IC 1	IC 2	IC 3	IC 4
Polymer binder	Acrylic copolymer	Acrylic copolymer	Vynil copolymer	Vynil copolymer
Country of component's origin	China	Europe	China	Europe

Samples of intumescent compositions were tested by various methods, some of which are well-known, and the other developed in the framework of this study. The volume of charred layer (swelling coefficient) when applying IC samples to steel plates and their further heating was measured [11,12]; the coefficient of adhesion of the charred layer to the substrate was determined by the method of reverse strike, steel plates were also used as the substrate [13,14]; the kinetic parameters of ICs thermolysis were calculated by the methods of non-isothermal kinetics [10]. Also, two types of fire tests were carried out – in laboratory (small-scale fire tests) using the methodology developed by the authors; and large-scale fire tests, but also taking into account the innovations proposed by the authors.

To assess the swelling coefficient and the adhesion coefficient, the samples of ICs were applied to plates $40 \times 120 \times 3$ mm (Figure 1); in this experiment, plates were used without a primer. The compositions were applied with an applicator in 4 layers with layer-by-layer drying for 24 hours, trying to achieve a layer thickness of 1 mm. After applying the last layer, the samples were kept for at least 7 days at a temperature of 20 ± 2 C°.



Fig. 1. Samples for laboratory tests of fire retardant performance

The swelling coefficient (K) was determined as the ratio of the thickness of the layer formed after heating exposure (h_1) to the thickness of the initial coating layer (h_0).

The adhesion of charred layer to the surface of the structures and materials is one of the determining factors in the fire-retardant efficiency of intumescent char-forming coatings, especially if the protected substrate is metal.

Due to the fact that one of the criteria for the fire-retardant efficiency of an intumescent coating is its safety on the protected surface under fire conditions, it is advisable to assess the adhesion and strength characteristics of charred layers [15,16]. When analyzing the literature, it was not possible to find references to indicative methods for assessing the characteristics of the charred layers, which are quite easily implemented in laboratory conditions, therefore, a previously developed installation that implements the method of reverse impact (Fig. 2) was used [13]. Columns (2) are fixed in the basis (1); sample (4) fixed in frame (3) can move up and down the columns. A charred layer (5) is turned downside. A sample (4) is a steel plate ($80 \times 140 \times 4$ mm). A frame (6) with a peen (7) can also move and fall down to the sample. So, the device has two applications: sample, fixed in a frame, may fall down itself, or a peen may fall down and strike the sample.

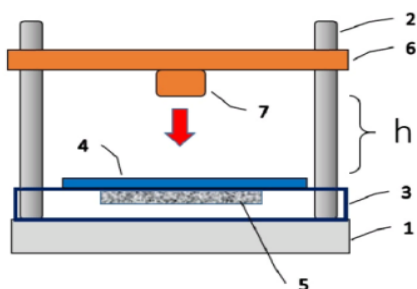


Fig. 2. Scheme of a device for back strike method

The essence of the method is to determine the quantitative indicator (in units of mass) of the safety of char on the substrate after three strikes on the reverse side of the plate.

An alternative to the chemical description of charring of ICs can be the presentation of the kinetics of the charring mechanism based on the results of thermal analysis (TA), which is currently widely used to study various fire retardant materials [17-20]. In particular, in the Russian Federation, thermal analysis is actively used to identify materials, substances and means of fire protection, according to the methodology declared in Russian Standard GOST R 53293-2009 «Fire hazard of substances and materials. Materials, substances and means of fire protection. Identification by Thermal Analysis Methods». The development of modern TA methods has led to the emergence of the so-called nonisothermal kinetics [10], within which the solution of the inverse kinetic problem is based on the processing of experimental data within the framework of the general kinetic model.

Using the results of the TA experiment, it is possible to determine the mechanism of IC thermolysis. Of course, the conditions of the TA experiment and the conditions of real fire are significantly different, however, the results of the application of non-isothermal kinetics will help to clarify thermolysis phenomena under fire conditions. In turn, from the kinetic data, important information can be obtained about the change in the mechanism of the fire-retardant action of intumescent compositions when they are modified by additives. In particular, this approach can be used in the study of aging of intumescent coatings [21].

Thermoanalytical studies were carried out using a synchronous complex (Netzsch; Germany). The obtained experimental data were processed using software packages based on Research Center for Expertise of Fire.

To carry out the small-scale fire tests, special samples were made: a hollow steel cylinder with a height of 20 mm and a wall thickness of 2 mm was welded to one side of a steel plate with dimensions of $40 \times 120 \times 3$ mm; the side of the plate on which the cylinder was not welded, was covered with a flame retardant, and heated by a gas burner; a thermocouple was applied to the plate from the unheated side and fixed with a heat-insulating material in the cavity of the welded cylinder (Fig. 3). Thus, due to the design of the sample, it was possible to achieve fixation of the thermocouple from the unheated side and its simultaneous thermal insulation in order to reduce the influence of the heat flux on the test result. A thermocouple recorded the temperature of the unheated side of the plate during the test, and the data was written to a flash drive using a thermoelectric converter. As a result, an array of time-temperature data was obtained, which was processed in MS Excel, and the dependence of the temperature on the unheated side of the sample on the time from the beginning of the test was plotted.

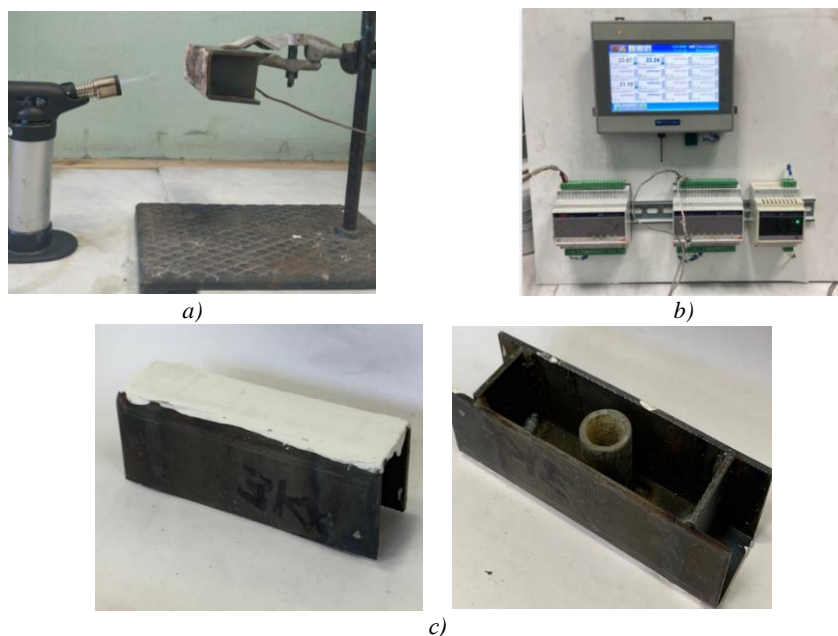


Fig. 3. Laboratory installation for testing fire retardant efficiency: *a)* plate with fixed thermocouple; *b)* thermoelectric converter; *c)* test plate

The disadvantage of the described installation at the current working stage is a significant amount of heat loss, which did not allow bringing the temperature on the unheated side of the plate to 500 °C. Therefore, the graphical equations were used to determine the time the graph reached a "plateau", the temperature difference between the control sample (without a flame retardant) and samples with a flame retardant at the 15th minute of the test, as well as the limiting temperature that the sample reached by the 15th minute of the test. Also, at the end of the test, the state of the charred layer itself was assessed – e.g. its thickness, burnout pattern under the influence of the burner flame.

Two series of tests were carried out: in each series, a control sample (without a fire retardant coating) and samples coated with compositions IC1-IC4 were tested.

To assess the fire retardant efficiency according to the control method [3], it was proposed to make changes to the test procedure, namely, to implement the possibility of simultaneous testing of several samples.

The existing IC control test method [3] regulates the use of one test sample (steel plate with dimensions 600x600x5 mm with IC applied), on the unheated side of which a heat-insulating layer is mounted and 3 thermocouples are applied. The essence of the method lies in the thermal effect on the plate and determining the time from the beginning of the thermal exposure to the onset of the limiting state of the prototype (for a steel plate - reaching a temperature of 500 °C on its unheated side). It is obvious that testing only one sample at a time can affect the overall reproducibility of the results; this is relevant both for repeated tests of the same IC, and for tests of different ICs, and the divergence in the test results can be observed even within the same test setup.

As part of the improvement of this technique, 4 samples were prepared – plates with dimensions of 300x300x5 mm with IC1-IC4 flame retardants applied to them. Two thermocouples were applied on each plate (Fig. 4). Each plate was placed in a special frame - a holder with 4 sections (Fig. 4).

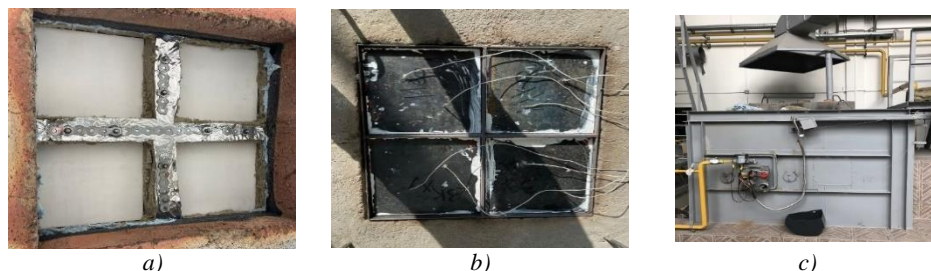


Fig. 4. Large-scale fire tests: *a)* steel plates in a holder; *b)* position of thermocouples on the test plates; *c)* one of the testing furnaces

The frame was placed in a furnace, covered with a heat-insulating material, and the time of reaching a temperature of 500 °C on the unheated side of the plate was recorded according to the data of 2 thermocouples.

At the end of the test, the frame with the test plates was removed from the furnace and a visual assessment of the state of charred layers was made - the degree of its burnout, the degree and nature (adhesive / cohesive) of its possible collapse from the plate.

3 Results and discussion

The results of determining the swelling coefficient and the adhesion coefficient of the IC1-IC4 samples are presented in Table 3. Fig. 5 shows a view of the samples after annealing in a furnace.

Table 3. Swelling coefficients and the adhesion coefficients of the IC1-IC4 samples

No. of the sample	IC 1	IC 2	IC 3	IC 4
Swelling coefficient	28,3	35,2	39,6	36,4
Adhesion coefficient	77,1	80,1	97,2	99,4



Fig. 5. View of the samples after annealing in a furnace

Sample IC 3 showed the highest values of swelling and adhesion coefficients. In general, the swelling coefficient values for all samples are relatively close, but both vinyl binder coatings (IC3 and IC4) have a swelling coefficient higher than that of acrylic binder coatings (IC1 and IC2). This confirms earlier data that acrylic copolymers as binders in intumescent systems perform worse than vinyl copolymers, probably due to the peculiarities of the structure of the polymer network formed during thermolysis, which, in a certain extent, inhibits the release of the charred layer.

Sample IC3 also showed the highest value of the adhesion coefficient, and this value is ~ 2 times higher than that of the IC4 composition; in this case, the significant influence of the quality of intumescent components is obvious - moreover, for the preparation of the IC3

composition, Chinese components were used, which are cheaper; and taking into account that the IC3 sample showed the best results in this experiment, it becomes possible to significantly reduce the cost of producing intumescent compositions, increasing their availability to users, and, accordingly, popularity in the market. Samples IC1 and IC2 showed close and rather high values of adhesion coefficient, as well as a rather high cohesive strength in comparison with the adhesive one.

Results of thermal analysis performed for samples IC1-IC4 are presented in figure 6 and table 4.

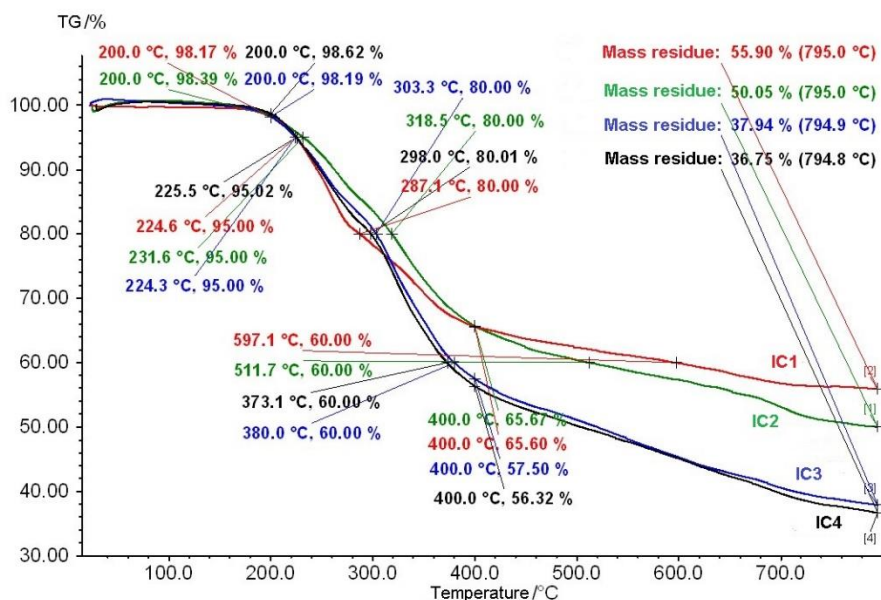


Fig. 6. TG-curves for samples IC1-IC4

Table 4. Values of mass loss of IC1-IC4 samples at temperatures 200, 400 and 795 °C

No. of sample	Δm_{200}	Δm_{400}	Δm_{795}
IC1	1,83	34,40	44,1
IC2	1,61	34,33	49,5
IC3	1,81	42,50	62,06
IC4	1,38	43,68	63,25

Observing the results of thermal analysis makes it clear that samples IC1 and IC2 (based on acrylic binder) show higher thermal stability than samples IC3 and IC4 (based on vinyl binder) as the values of mass residue for samples IC1 and IC2 are higher. A contradiction between the results of small-scale tests (and our expectations) and results of thermal analysis occurs, as samples IC3 and IC4 are expected to show higher thermal stability. Further tests are already planned to be conducted in order to clarify the results.

Results of determining the activation energy are presented below. The experiments were carried out in a dynamic air atmosphere, the flow rate was 50–70 ml / min, four series of experiments were performed, for each sample, at a heating rate of 20 °C / min, the sample weight was 5 mg. Sample holders - platinum crucibles. Samples for testing were prepared from coatings based on IC1 – IC4.

Table 6 shows the numerical values of the kinetic parameters of the first stage of thermolysis of the investigated compositions.

Table 5. Values of activation energy of IC1-IC4 samples

No. of sample	E_a , kJ / mol
IC 1	29,09
IC 2	18,29
IC 3	65,6
IC 4	71,47

Both acrylic based samples (IC1 and IC2) have significantly lower activation energies than vinyl based samples. This may indicate a greater additivity of the components of the intumescent composition distributed in the acrylic binder, and an earlier onset of thermolysis. In general, this means that such coating will begin to expand and perform thermal insulation earlier, but this does not provide information about its further state during heating.

The results of large-scale fire tests are presented in table 6.

Table 6. Results of large-scale fire tests according to modified control method

Test No.1		Test No.2	
No. of sample	Time, min	No. of sample	Time, min
IC1	40	IC1	36
IC2	31	IC2	33
IC3	61	IC3	40
IC4	65	IC4	60

Photographs of charred layers formed by samples IC1-IC4 during the fire test are presented below (fig. 7)



Fig. 7. Charred layers formed during fire tests; on the left – test No.1, on the right – test No.2; samples are located clockwise, starting from top left segment – IC3, IC4, IC2, IC1

Results of large-scale fire tests, in common, correspond to the results of small-scale tests; however, in the second test here the sample IC4 (vinyl binder, European components) showed better fire-proofing efficiency, than sample IC3 (vinyl binder, Chinese components). In the first test the results are mostly similar.

However, if comparing two acrylic-based samples and two vinyl-based samples, ICs based on vinyl polymer binders show much more greater results in fire-proofing efficiency; charred layer which formed from acrylic coatings had worse adhesion to steel surface and fallen off during the test, which could be the reason why the testing plate was more intensively exposed by heating. Also, the charred layer itself had worse quality (it had many hollowed areas) when speaking about acrylic coating; on the contrary, the charred layer formed by both vinyl coatings had no hollows and had high enough density to protect the

steel plate from heat exposure for more than 60 minutes. In general, these results prove our (and not only our, but also other researchers) thoughts that acrylic copolymers don't operate well as binders in intumescent systems.

As for the raw materials from different manufacturers which were used when making the samples – it can be seen that there is a little difference between the parameters of European and Chinese-based compositions; in some cases, the ICs based on Chinese components (which are much cheaper) showed better results than European-based. This statement may prove useful for the manufacturers of intumescent compositions, as their final price could be lower when using the cheaper raw materials, which are not inferior in quality. It also may lead to the development of the intumescent compositions market, because the lower price may allow more and more people to try these materials thus making them more reliable for those who have not utilized them.

4 Conclusion

The current work presents initial results which were obtained by the authors using new and improved methodologies of investigating the parameters of intumescent fireproofing materials and charred layers which they form as a result of heating. The main achievement is a correspondance of results obtained by laboratory tests and modified large-scale tests: it reveals a possibility for specialists who work on developing the new fire retardant materials to test them 'right here, right now' and estimate their parameters without the need to conduct large-scale fire tests for all developed samples. And the modified methodology of conducting the large-scale fire tests could increase their reliability and let to avoid the dependence on external factors.

Also, such instrumental methods like thermal analysis, microscale calorimetry and etc., which investigate the properties of materials in the process of heating, should be considered not only as identification methods in case of intumescent materials, but also as the methods of quality control. This opinion may be found in the works by other researchers, however the process of approving such methods depends on the government decisions and may vary from country to country.

As an initial part of this process, the results described in the current work can be a basis for further improvement of methodologies that are used to investigate intumescent materials, thus it should positively affect the approaches to development of these materials which can result in increase of their final quality.

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