Intumescent coatings with improved properties for fireproofing of wooden building constructions

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Abstract. The paper overviews the mechanism of fire-retardant action of intumescent coatings in relation to wooden structures. A comparative assessment of fire-retardant performance of various intumescent compositions, their effectiveness in relation to wood and materials based on it, has been carried out. For this, tests were carried out using the «ceramic tube» method and tests in the cellulosic fire mode. As a result of tests, it was shown that glass microspheres, kaolin, and water-soluble sulfonated graphene are significantly effective functional additives in the intumescent composition. The mechanism of their influence on fire retardant efficiency is considered. It has been shown that compositions containing sulfonated graphene provide a delay in heating in the surface layer of wooden sample, whereas classical intumescent compositions more effectively provide a delay in heating in the depth of the sample. Almost all of the considered compositions demonstrated satisfactory performance when tested by the «ceramic tube» method, thus these compositions allow to make the wood hardly flammable and hardly combustible.

1. Introduction

The fireproofing of wooden constructions is an effective preventive procedure due to the aim to reduce the damage from possible fires. Fire protection of wood is considered to be a mandatory stage when putting a building into operation.

First of all, fire protection of wood is needed to solve the problem of minimizing the percentage of fires as a result of the occurrence of accidental fire sources, which are usually called low-calorie. They include a cigarette that has not been extinguished, short circuit, molten metal dripping during welding, etc. These are the cause of fire in 80% of cases based on the total number of fires. Wood is one of the most widely used materials in construction; at the same time, according to statistics, 75% of fires in the field of residential construction in the territory of the Russian Federation occur in the private sector – these are both wooden buildings themselves and a large number of structures inside buildings (beams, roofs, floors). The increased flammability of wood is the main reason why its fireproofing is one of the most pressing problems in the construction industry.

There are studies dedicated to the problem of wood fireproofing. For example, [1] describes the studies on intumescent compositions based on vinyl acetate copolymer (VAC) (29.15 wt.%) as a binder; ammonium polyphosphate (APP) (18.50 wt.%) as an acid source; pentaerythritol (PER) (9.25 wt.%) as a carbon source; melamine (9.25 wt.%) as a blowing agent; and water (23.85 wt.%). The varied amounts of industrial fillers, i.e., titanium dioxide (TiO₂) and aluminium trihydroxide (Al(OH)₃), and biological fillers, i.e., Rice Husk Ash (RHA) and Eggshell (ES) were incorporated into these formulations. Cone calorimetry was used to establish combustivity of wooden samples covered by intumescent compounds, and Raman spectroscopy was used to observe the char residues after the fire tests. The authors achieved the increase in back-surface temperature for the wood sample coated
with the formulation F (combining two industrial and two bio-fillers) to be delayed to 990 s with the highest temperature reaching around 250 °C, compared to 530 °C maximum for the uncoated sample. The Raman spectroscopy showed that char residue obtained from the wood substrate protected with the coating A, containing titanium dioxide and RHA fillers, was characterized by the most ordered char structure, very similar to the one of unprotected wood, whilst the char from the sample coated with the formulation B, based on the industrial fillers only, was the most disordered.

In [2] the alder and spruce samples were covered by commercial intumescent paint containing titanium dioxide (TiO$_2$) and antimony trioxide (Sb$_2$O$_3$) as fireproofing additives. The fire performance of these chemicals combined with the fire retardant paint was evaluated in an oxygen index instrument (Dynisco Limiting Oxygen Index Chamber), which is generally applied to evaluate and classify the fire performance of a material, under nitrogen flowing upward in a test column according to ASTM-D 2863. Limiting oxygen index (LOI) values give information about the flammability of the material. For fire performance test by the in-house method, the blowtorch was kept at a distance of 5 cm from the coated and uncoated wood surfaces. The flaming angle was set to approximately 90°. After the flame was applied onto wood surface for 60 s, the observations were performed. The flame extinguishing time was recorded. The best LOI result was obtained from KA2 (42.5; alder, 2 wt.% of Sb$_2$O$_3$) for the alder solid wood surface whilst the maximum LOI value was achieved from LA2 (spruce, 2 wt.% of Sb$_2$O$_3$) and LT2 (spruce, 2 wt.% of TiO$_2$) (41) for the spruce wood surface. These findings showed the antimony trioxide and titanium dioxide enhanced physical properties as well as flame retardancy.

Most other researches [3-10] concentrate on fireproofing of metal (in particular steel) constructions; there is not much information particularly on the intumescent coatings for wood surfaces. Though the materials and approaches in development of fireproofing compositions for metal and wood are quite similar; in both cases, the researchers aim to apply different carbonaceous additives in intumescent compositions in order to improve their fireproofing and mechanical properties.

Our research is specifically devoted to fire retardant intumescent coatings for wood based on the melamine-pentaerythritol-ammonium polyphosphate composition, and the purpose of the work is to select the optimal composition in terms of fire retardant properties; a number of experiments were carried out to reach the aim of the research.

2. Materials and methods

2.1. Preparation of intumescent compositions

The original intumescent composition was a mixture of melamine (MA), pentaerythritol (PE) and ammonium polyphosphate (APP) in an aqueous dispersion of vinyl acetate copolymer [11,12]; titanium dioxide was used as a pigment [13,14]. Five compositions were prepared on the basis of composition 1; their formulations are presented in Table 1

| Components, mas.% | Comp. No.1 | Comp. No.2 | Comp. No.3 | Comp. No.4 | Comp. No.5 |
|-------------------|------------|------------|------------|------------|------------|
| Aqueous dispersion of vinyl acetate copolymer | 20.00      | 20.00      | 20.00      | 20.00      | 20.00      |
| APP               | 25.00      | 25.00      | 25.00      | 25.00      | 25.00      |
| Melamine          | 9.00       | 9.00       | 9.00       | 9.00       | 9.00       |
| Pentaerythritol   | 9.00       | 9.00       | 9.00       | 9.00       | 9.00       |
| Titanium dioxide  | 6.00       | 6.00       | 6.00       | 6.00       | 6.00       |
| Talc              | 2.00       | 2.00       | 2.00       | –          | 2.00       |
| Kaolin            | –          | –          | 4.00       | –          | –          |
| Sulfonated graphene | –        | –          | –          | –          | 0.05       |
| Glass microspheres | –         | –          | –          | –          | –          |
| Other functional additives | 0.20 | 0.20 | 0.20 | 0.20 | 0.20 |
| Water             | 25.80      | 24.80      | 21.80      | 27.80      | 25.75      |
The compositions were prepared by mixing the components in a metal beaker using a mechanical stirrer. At the stage of dispersion, the stirring speed was 1000-1200 rpm, then, with the addition of PFA, it decreased to 600 rpm. Composition 2 contains hollow glass microspheres (Fig. 1), composition 5 contains water-soluble sulfonated graphene [15] (manufactured by Laboratory of composite materials in Building, RUDN University, Russia).

**Figure 1.** SEM images of microspheres used as an additive in composition No.2; 
*a)* x200; *b)* x500

### 2.2. Swelling coefficient

The swelling coefficient is a parameter which means the ability of intumescent coating to expand in volume when exposed to heating. To determine the swelling coefficient, the test the compositions were applied to clean and degreased metal plates with a size of 150 mm x 75 mm x 2 mm. The coating was dried at room temperature for 72 hours. The thickness of the dry layer of 1 mm was achieved on all samples.

The thickness of the initial coating layer was measured on the plate with the ETARI ET-11P magnetic thickness gauge in five points (results were averaged).

The samples with applied flame retardant compositions were placed in a furnace heated to 600°C and exposed for 5 minutes so that the charred layer could form.

Using a Zipower PM 4265 caliper, the thickness of charred layer was measured at five points. The final value was averaged. The swelling coefficient was defined as the ratio of the charred layer thickness to the thickness of the initial coating.

### 2.3. Thermal analysis

To assess the behavior of intumescent compositions in the process of thermolysis, the thermal analysis was used. Thermal analysis of the compositions was performed using SETSYS Evolution TG-DSC/DTA 1750 by SETARAM (France).

The following data was recorded during thermal analysis:
- values of weight loss at determined temperatures (in the range of 300-550 °C);
- ash or char residue, %, at the temperature of destruction completion;
- temperatures of maximum DTA peaks in the range of 150-550 °C.

The average of the three measurements for each flame retardant composition was analyzed.

### 2.4. Tests by «ceramic tube» method

After evaluating the fire-retardant properties of the compositions themselves, the further step is fire tests, in which the compositions are applied to wooden samples and their fire-retardant effectiveness in relation to wood is being assessed. One of the methods for assessing the effectiveness of fire retardants for wood is the «ceramic tube» method described in [16] (Fig. 2). The essence of the method for testing wood samples with a fire retardant coating in the «ceramic tube» installation is the following: a standard wood sample treated with an intumescent composition is weighed, then placed in a ceramic box, the walls of which are lined with aluminum foil; the sample is exposed to the flame of a gas
burner from below (200 ± 5 °C, 2 min); the change in mass of the sample after the test is registered. The result of test is a difference in sample mass before and after testing, i.e. weight loss (in g and %): the lower the weight loss, the more effective fire protection is provided by the intumescent composition. The fire retardant composition belongs to the I group of fire retardant efficiency (hardly combustible) if the value of the weight loss is not more than 9%. If the weight loss of the sample is not more than 25%, then the composition is assigned the II group of fire retardant efficiency (hardly flammable) [16]. Preliminary comparative tests were carried out on three samples, expanded tests – on 10 samples. After the tests, the samples were re-weighed. The final test result was the arithmetic mean of three tests.

2.5. Tests in cellulosic fire mode

However, the most complete picture emerges after conducting the real fire tests, which make it possible to evaluate the behavior of the sample under conditions close to those of a real fire. The tests were carried out in the Academy of State Fire Service of the Ministry of Emergencies, Moscow. The authors of the methodology are E. Kruglov, R. Aseeva.

The testing device was a furnace (Fig. 3) in which the conditions of a cellulosic fire were created in accordance with [17]. Samples of wood were placed in the furnace in such a way that heating was carried out only on one side. The unheated side was protected with intumescent coatings. The spots for thermocouples on a sample are shown in Figure 4.

![Figure 2. a) Drying of wood samples with applied fire retardants; b) General view of the «ceramic tube» installation](image)

![Figure 3. Furnace design 1, 2, 3, 4 - sequence of layers of the structure; 5 - frame beam; 6 - tongue; 7 - external isolating structure made of wood; 8 - gypsum; 9 - mineral wool (heat-insulating layer); → direction of heat transfer](image)
During the tests, the main recorded indicator was considered the time of heating the sample to a temperature of 270 °C on the surface, and at a depth of 5, 10, 15 mm, respectively.

![Figure 4. Placement of thermocouples on a wood sample: T₁, T₂, Tₙ - temperature measurement points: 1 – radiation panel, 2 – wood sample, 3 – heat-insulating material](image)

3. Results and discussion

3.1. Results of determining the swelling coefficient of intumescent coatings
The test results showed that the swelling coefficient increases for all compositions, except for No. 2. For composition No. 5 with the addition of sulfonated graphene, an increase in the expansion of intumescent char was detected, which is corresponding with the data of previous studies [18,19]. A decrease in the value of the swelling coefficient for composition No. 2 may be due to the fact that the hollow microspheres act as a heat shield on the coating surface, delaying the heating of the underlying layers [20,21]. Values of swelling coefficient are presented in Table 2 (Results of thermal analysis subsection).

3.2. Results of thermal analysis
Results of thermal analysis are presented in Figures 5-9. Based on the thermal analysis data, the temperature values of the main stages of thermolysis and the characteristics of thermal stability of intumescent coatings (Table 2) were determined.

![Figure 5. TA curves of composition No. 1](image)

![Figure 6. TA curves of composition No. 2](image)
Based on the data presented in Table 2, the endothermic peaks corresponding to the thermal decay of the main components of intumescent composition (pentaerythritol and ammonium polyphosphate) are shifted along the temperature axis when various additives are included into the composition. So, for example, the synthesis of char-forming resin in compositions No. 4 and 5 begins earlier in comparison with others. Composition No.2 has the highest thermal stability, which is confirmed by the values of temperature of total weight loss and ash residue. The temperature of total weight loss for compositions No.4 and 5 is the lowest, which indicates an earlier formation of intumescent char.
Presented data confirms the fact that functional additives directly affect the process of thermolytic synthesis of the charred layer: in modified compositions No. 4 and 5 it occurs faster, which ultimately has a positive effect on their fire retardant efficiency.

### 3.3. Results of tests by «ceramic tube» method

First of all, comparative tests were carried out for each composition on three wooden samples; the results are shown in table 3.

**Table 3.** Comparative parameters of fire tests of 5 compositions by «ceramic tube» method

| № of sample | Weight of a wooden sample before treatment, g | Weight of a treated wooden sample before the test, g | Approximate composition consumption, g/m² | Weight of sample after the test, g | Mass loss, g | Mass loss, % |
|-------------|---------------------------------------------|-------------------------------------------------|-----------------------------------------|----------------------------------|-------------|-------------|
| 1           | 142.34                                      | 147.42                                          |                                         | 137                              | 10.42       | 7.1         |
| 2           | 143.6                                       | 149.56                                          |                                         | 140.5                            | 9.06        | 6.1         |
| 3           | 139.16                                      | 144.23                                          | 280-290                                 | 132.4                            | 11.83       | 8.2         |
| 4           | 139.71                                      | 145.23                                          |                                         | 116                              | 29.23       | 20.1        |
| 5           | 133.39                                      | 139.81                                          |                                         | 132.2                            | 7.61        | 5.4         |

According to experimental data, all wooden bars protected by test compositions, except for composition No. 4, demonstrated a weight loss which corresponds to group I of fire retardant efficiency (less than 9% weight loss).

Bars protected by compositions No. 2 and No. 5 showed the most significant results in all tests, therefore extensive tests were carried out for them using the «ceramic tube» method (on 10 samples). Results of these tests are presented in Tables 4 and 5.

**Table 4.** Results of extensive tests according to the «ceramic tube» method for composition No. 2

| №  | W, % | Weight of a wooden sample before treatment, g | Length of sample, mm | Δm | Weight of a treated wooden sample before the test, g | Approximate composition consumption, g/m² | Weight of sample after the test, g | Mass loss, g | Mass loss, % |
|----|------|---------------------------------------------|----------------------|----|-------------------------------------------------|-----------------------------------------|----------------------------------|-------------|-------------|
| 1  | 7    | 143.36                                      | 151.60               | 8.24 | 147.03                                          | 271.0                                    | 130.08                           | 16.95       | 11.5        |
| 2  | 7.5  | 143.50                                      | 151.80               | 8.30 | 145.50                                          | 268.1                                    | 129.70                           | 15.80       | 10.8        |
| 3  | 7    | 145.50                                      | 153.85               | 8.35 | 147.30                                          | 271.2                                    | 133.52                           | 13.78       | 9.4         |
| 4  | 7.5  | 145.01                                      | 153.41               | 8.40 | 147.86                                          | 275.0                                    | 132.78                           | 15.08       | 10.2        |
| 5  | 6    | 146.15                                      | 154.80               | 8.65 | 148.45                                          | 274.0                                    | 137.30                           | 11.15       | 7.5         |
| 6  | 7.5  | 149.16                                      | 157.78               | 8.70 | 153.31                                          | 282.0                                    | 141.20                           | 12.07       | 7.9         |
| 7  | 7    | 147.30                                      | 155.92               | 8.62 | 149.65                                          | 276.4                                    | 136.05                           | 13.60       | 9.0         |
| 8  | 6    | 144.36                                      | 153.05               | 8.69 | 148.42                                          | 284.0                                    | 137.10                           | 11.32       | 7.6         |
| 9  | 7.5  | 144.70                                      | 153.38               | 8.68 | 145.45                                          | 268.0                                    | 128.40                           | 17.05       | 11.7        |
| 10 | 6    | 147.64                                      | 156.24               | 8.60 | 151.10                                          | 281.0                                    | 137.48                           | 13.62       | 9.0         |

Average value: 275.1 14.04 9.46
Table 5. Results of extensive tests according to the «ceramic tube» method for composition No. 5

| №  | W, % | Weight of a wooden sample before treatment, g | Length of sample, mm | Δm | Weight of a treated wooden sample before the test, g | Approximate composition consumption, g/m² | Weight of sample after the test, g | Mass loss, g | Mass loss, % |
|----|------|--------------------------------------------|----------------------|----|-----------------------------------------------|------------------------------------------|-------------------------------|---------------|--------------|
| 1  | 7.5  | 150.96                                     | 159.70               | 8.70| 155.45                                        | 284                                      | 146.25                        | 9.20           | 5.9          |
| 2  | 6.6  | 148.58                                     | 157.23               | 8.65| 152.30                                        | 283                                      | 143.35                        | 8.95           | 5.8          |
| 3  | 7.5  | 147.58                                     | 156.18               | 8.60| 152.52                                        | 281                                      | 143.40                        | 9.12           | 6.0          |
| 4  | 7.7  | 143.30                                     | 151.90               | 8.60| 148.40                                        | 283                                      | 139.25                        | 9.15           | 6.1          |
| 5  | 6.6  | 147.23                                     | 155.87               | 8.64| 151.43                                        | 282                                      | 139.40                        | 12.03          | 7.9          |
| 6  | 6.5  | 145.30                                     | 154.00               | 8.70| 149.00                                        | 284                                      | 140.02                        | 9.08           | 6.1          |
| 7  | 7.7  | 144.59                                     | 153.19               | 8.60| 149.04                                        | 281                                      | 139.92                        | 9.12           | 6.1          |
| 8  | 6.5  | 146.20                                     | 154.85               | 8.65| 151.20                                        | 283                                      | 141.45                        | 9.75           | 6.5          |
| 9  | 7.7  | 144.08                                     | 152.76               | 8.68| 149.88                                        | 284                                      | 139.93                        | 9.95           | 6.6          |
| 10 | 7.5  | 145.20                                     | 153.50               | 8.30| 149.00                                        | 284                                      | 140.02                        | 9.08           | 6.1          |

Average value: 282.9 9.54 6.31

According to the data in Tables 4 and 5, the weight loss did not exceed 9% only for wooden samples treated with composition No. 5. It was decided to conduct further tests in a cellulosic fire mode with a composition No.5. Composition No. 1 was chosen as a control sample.

3.4. Test results under cellulosic fire conditions [17]

Three samples were tested: W1 – an untreated wooden block, W2 – a wooden block treated with composition No.1 and W3 – a wooden block treated with composition No.5. The results of tests under cellulosic fire conditions are presented on Figures 10-12 and in Table 6. Thermocouples were measuring the following temperatures (see Figures 10-12): T₁ – standard fire mode in the furnace (°C); T₂ is the temperature on the heated side of the sample; T₃ is the temperature in the wooden samples section at a depth of 5 mm; T₄ – temperature in the wooden samples section at a depth of 10 mm; T₅ – temperature in the section at a depth of 15 mm.

Figure 10. Heating of an untreated wooden sample W1 under the influence of cellulosic fire mode
Figure 11. Heating of a wooden sample W2 covered by composition No.1 under the influence of cellulosic fire mode

Figure 12. Heating of a wooden sample W3 covered by composition No.5 under the influence of cellulosic fire mode

Table 6 shows the generalized characteristics of the time of reaching a temperature of 270 °C on all samples surface, and at a depth of 5 mm, 10 mm and 15 mm, respectively.

Table 6. Comparison of data on the time to reach a temperature of 270 °C for all samples

| Depth of a section | δ = 0 mm | δ = 5 mm | δ = 10 mm | δ = 15 mm |
|--------------------|----------|----------|-----------|-----------|
| Sample W1          | 1.66     | 7.33     | 13.66     | 14.17     |
| Sample W2          | 1.83     | 13.33    | 17.83     | 20        |
| Sample W3          | 6.30     | 12.5     | 17.33     | 19.33     |

According to the data in Table 6, the maximum time to reach a temperature of 270 °C on the wood surface is for sample W3.

At a depth of 5 mm, the results for samples W2 and W3 are mostly similar, but significantly higher than those for untreated sample W1. This means that the samples treated with intumescent compositions are heating up more slowly.
At a depth of 10 mm, samples W2 and W3 are heated to a temperature of 270 °C in 17.83 and 17.33 minutes respectively. Untreated wooden sample W1 is heated in 13.66 minutes. The time difference is stable around 4 minutes.

At a final depth of 15 mm, samples W2 and W3 have heating times of 20 and 19.33 minutes, respectively. For sample W1, this time was 14.17 minutes. The final difference in time between samples W2 and W1 is 5.83 minutes.

The results obtained show that the compositions belonging to the I group of fire retardant efficiency with a thickness of the applied layer of 1 mm provide a heating delay on average of 4-6 minutes. At the same time, the composition containing sulfonated graphene (sample W3) as an additive slows down heating on the wooden samples surface for 5 minutes longer in comparison with untreated wood (sample W1). Composition No.1 (Sample W2), which is less effective in the surface layer, turns out to be slightly more effective when a sample is heated at depths of 5, 10 and 15 mm. These results allow a more conscious approach to the choice of additives when solving specific problems of fire protection of wood and materials based on it.

4. Conclusions

In this work the behavior and fire-retardant characteristics of intumescent compositions for wood were investigated by various methods (including thermal analysis and tests in the «ceramic tube» installation). Comparative tests were carried out on five intumescent compositions based on the classical intumescent triad, some of them were modified with functional additives such as glass microspheres and water-soluble sulfonated graphene. Composition 1, 2, 3 and 5 demonstrated a satisfactory result in establishing the fire retardant efficiency according to weight loss parameter. Composition 5, modified with sulfonated graphene, demonstrated the best performance, providing the ability to transfer wood to the category of hardly combustible. The effectiveness of this additive from the point of view of improving the properties of the final charred layer, in our opinion, lies in its active catalytic action in the process of thermolytic synthesis of intumescent char. It was also shown that the process of thermolytic synthesis begins earlier, and this leads to the formation charred layer with bigger thickness.

This composition was tested under cellulosic fire conditions when applied to a wood sample. As a result of the tests, it was found that the fire-retardant efficiency of intumescent coatings under the conditions of a cellulosic fire for wood samples consists in a delay in the time of the beginning of wood heating, its charring and decomposition under the action of high temperatures and in charred layers ability to be an obstacle to the spread of flame along the wood surface. In comparison with untreated wood, the delay in heating is 5.8 minutes. In case of a real fire, this time can play a significant role. However, under the same conditions, composition No.1, which showed less efficiency in preliminary tests on determining the group of fire retardant efficiency, provides bigger values of the heating time under conditions of a cellulosic fire. The results of this work will be useful in solving problems of fireproofing of wood and materials based on it.

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