

Investigation of the Processes of Formation of a Fire Retardant Coating

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Abstract. The processes of phase formation in a fire retardant composition based on liquid glass and potassium carbonate have been investigated. The mechanism of the process has been studied. It was found that the use of potassium carbonate provides controlled gas evolution during heat treatment of the composition, as a result of which the integrity of the fire retardant coating is preserved.

Introduction

Fire protection is the primary task in the creation of building materials and structures [1,2]. Wood is actively used in construction as load-bearing structures and finishing materials. Fire retardants for wood are known. Impregnating compounds require a large number of applications, coatings make it difficult to control the required thickness to create the desired degree of fire retardant efficiency, plasters are difficult to apply. Each type of coating has its own advantages and disadvantages; therefore it is effective to create a coating with a combined effect.

Unresolved Issues

Recently, the development of effective methods for increasing the fire safety of industrial and residential buildings has been actively pursued [3,4]. To study combustion in rooms, innovative methods are used [5,6]. In this regard, research related to the development of effective fire retardants [7,8], coatings [9,10], and impregnating agents [11] is especially relevant.

Known compositions of fire retardant coatings based on organic substances [12], but organic substances are capable of thermal decomposition with the release of combustible products of thermal destruction [13,14]. Production from organic raw materials [15,16] is technologically complex and not environmentally friendly [17,18]. However, the use of organic substances is due to the ability to swell [19,20] when exposed to temperature. at ignition temperatures. The disadvantage of such coatings is [21] the uncontrolled size and coalescence of pores formed during the swelling of liquid glass.

Main Part

The aim of the work was to develop a fire retardant coating based on liquid glass with controlled porosity. According to the information, aqueous solutions of sodium silicate are strongly alkaline and can undergo hydrolysis. At $\text{pH} < 10.9$ they lose their stability and can partially release silicic acid. This fact leads to a detailed study of the processes of polycondensation of silicic acid and their influence on the formation of a fire retardant coating. Mixing a concentrated solution of potassium carbonate with a solution of sodium metasilicate (liquid glass) leads to a fairly rapid gelation throughout the entire volume of the mixture under study [22]. It is known that when using highly concentrated solutions of polymers, converted into a gelatinous state, no separation of a synergistic liquid is observed during aging. This is explained by the fact that it can persist for an infinitely long time in the gel in the form of micro- and submicron regions, since the formation of open capillary

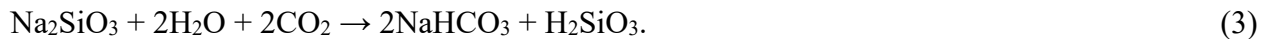
porosity is necessary for the release of this liquid into the external environment. Submicroscopic liquid separation can be judged by the cloudiness of the gel system.

When the obtained gel is examined under a microscope in reflected light, a dense, colorless-cloudy gel matrix with evenly spaced inclusions of small, white prismatic crystals is observed. Considering that during the partial hydrolysis of sodium silicate, silicic acid is released, and also taking into account the information given on the processes of its polycondensation, we will consider the processes occurring in the studied gel based on sodium silicate and potassium carbonate.

When mixing a solution of water glass with a solution of potassium carbonate with the latter, silicic acid enters into a chemical reaction, which is an intermediate product of the partial hydrolysis of sodium silicate, with the formation of potassium silicate:



This reaction proceeds until the potassium carbonate is completely consumed. The second reaction product (1), carbonic acid [$\text{H}_2\text{O} + \text{CO}_2$], can react with sodium silicate, also in solution, to form various carbonates:



The presence of sodium carbonates in the mixture compensates the charge on the silicic acid particles and causes its polycondensation followed (2) coagulation of the mixture and gelation (3). The assumptions put forward are confirmed by X-ray: the diffraction pattern of the studied mixture contains clear diffraction maxima of weak intensity of aqueous sodium carbonate ($\text{Na}_2\text{CO}_3 \cdot \text{H}_2\text{O}$) and trone – double salt of carbonate and dihydrate sodium bicarbonate (Fig. 1). Diffraction maxima corresponding to aqueous sodium carbonate are less intense and more diffuse, which suggests an imperfect crystalline structure of this salt and its lower content in comparison with trone.

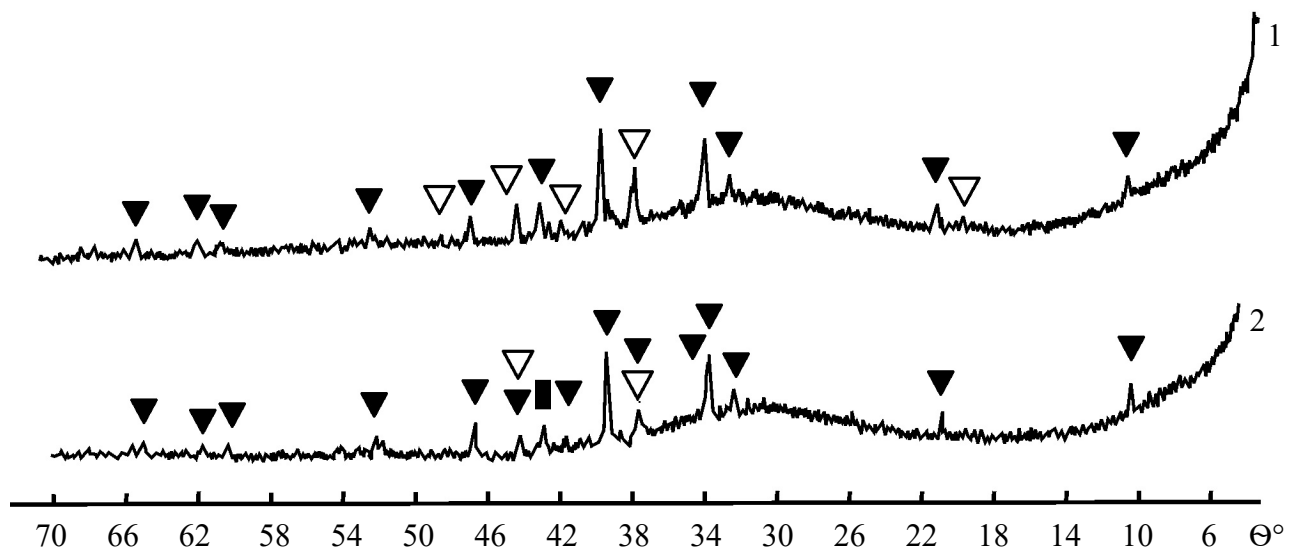


Fig. 1. X-ray phase analysis of a mixture of potassium carbonate and sodium silicate:
1 – without heat treatment; 2 – heat treatment 500 °C; ▽ – $\text{Na}_2\text{CO}_3 \cdot \text{H}_2\text{O}$; ■ – Na_2CO_3 ;
▼ – $\text{Na}_2\text{CO}_3 \cdot \text{NaHCO}_3 \cdot \text{H}_2\text{O}$

The presence of a halo in the diffractogram indicates the content of the X-ray amorphous component in the mixture under study. Taking into account the presence of a halo and the above reactions it can be assumed that the X-ray amorphous component of the obtained gel contains potassium silicate in a semi-amorphous state, as well as various intermediates of the metasilicic acid polycondensation process.

Table 1. Weight loss in a mixture of sodium and potassium salts during heat treatment

Temperature, [°C]	Weight loss		Increase in losses, [%]
	[mg]	[%]	
100	5.1	5.05	0
150	45.9	45.4	40.4
200	61.2	60.6	15.2
250	71.4	70.7	10.1
300	78.2	77.4	6.4
350	86.7	85.8	8.4
400	88.4	87.5	1.7
450	95.2	94.3	6.8
500	100	100	5.4

In Fig. 2 showed the heating curves for a mixture of sodium and potassium salts. The DTA curve shows a deep endothermic effect at 150 °C, accompanied by a significant (up to 45 %) weight loss (Table 1). According to the information and results of X-ray phase analysis, it can be assumed that this effect corresponds to the removal of crystalline hydrate water from sodium carbonate, and also accompanies the decomposition of trone.

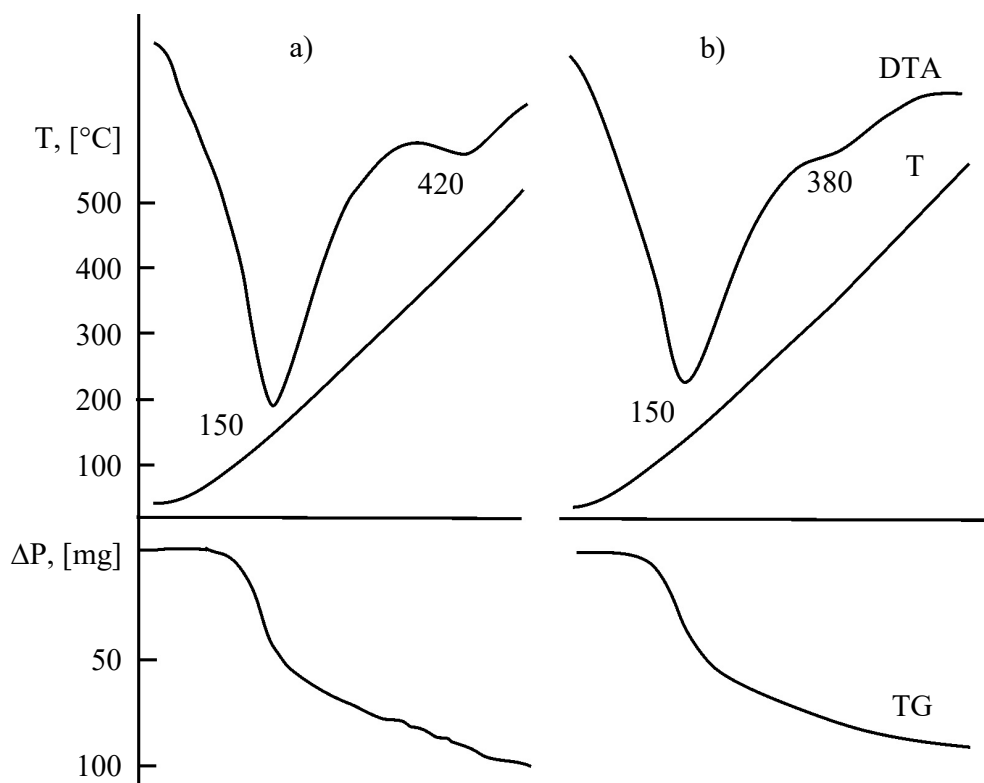


Fig. 2. Differential thermal analysis: a) mixtures of potassium carbonate and sodium silicate; b) fire retardant mixture

A weakly pronounced endothermic effect at 420 °C is accompanied by additional weight losses of up to 3 % and corresponds to the residual thermal decomposition of trone to anhydrous sodium carbonate.

The DTA results are consistent with the conclusions of X-ray phase analysis: when the mixture is heat-treated to 500 °C, a water molecule is split off in aqueous sodium carbonate, which is accompanied in the diffractogram by the appearance of a set of diffraction maxima corresponding to anhydrous sodium carbonate and the disappearance of characteristic maxima $\text{Na}_2\text{CO}_3 \cdot \text{H}_2\text{O}$. The intensity of the main diffraction maxima of trone slightly decreases, which can presumably be attributed to the partial loss of crystalline hydrate water by the salt. The halo intensity does not change (Fig. 3).

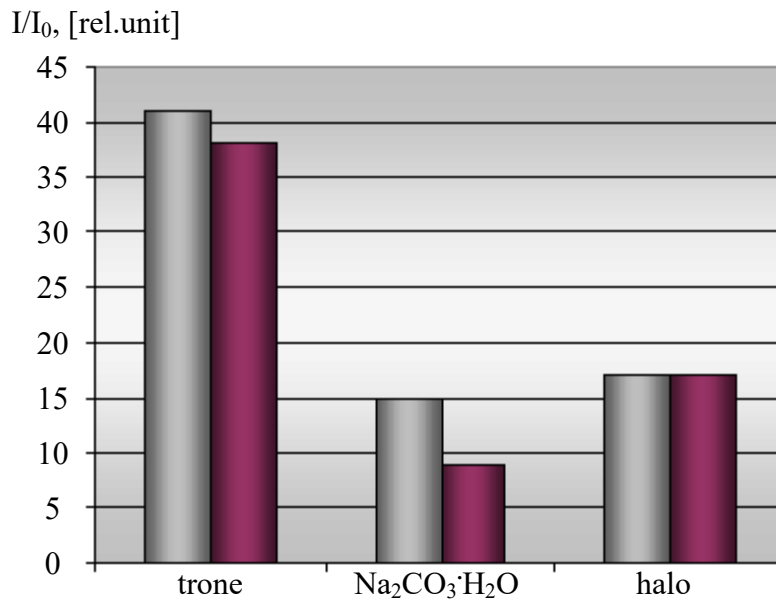


Fig. 3. Changes in the intensity of characteristic diffraction maxima depending on the heat treatment temperature of the fire retardant coating: ■ – thermally untreated composition; ■ – processing temperature 500 °C. Diffraction maxima d , Å: trone – 2.647, Na₂CO₃·H₂O – 2.37, halo – 3.40

The introduction of asbestos and expanded vermiculite into the composition of the gel mixture does not change the mechanism of phase formation in the untreated coating, as evidenced by the shape of the DTA curve of the coating shown in Fig. 2. Microscopic examination of the flame retardant coating showed the presence of a vitreous substance consisting of small vitrified particles 3–5 μm in size combined into porous aggregates up to 40–50 μm in size (Fig. 4).



Fig. 4. Cut type of fire retardant coating

The pore diameter in the aggregates is 5–10 μm. The pores between aggregates are 60–80 μm in size. Rarer are individual large pores 150–300 μm in size. Apparently, the formation of the smallest pores is associated with the release of carbon dioxide during the reaction of silicic acid with potassium carbonate. This reaction proceeds rather slowly due to the limited amount of silicic acid released during the hydrolysis of sodium silicate; therefore, a pronounced coalescence of gas bubbles in the gel mixture is not observed. The average pore size is formed, apparently, when physical water is removed from the mixture, which is located in the voids between large gel globules. Large pores are formed due to the coalescence of medium-sized pores during heat treatment. Swelling of the flame retardant coating does not lead to its destruction (Fig. 4). Vitrified gel particles form a dense bonding layer with a wooden surface without chips. The introduction of vermiculite and asbestos into the gel does not violate the integrity of the coating.

Conclusion

The use of a two-component gel composition based on water glass and potassium carbonate provides, due to its chemical characteristics, a consistent course of reactions for the formation of sodium carbonates. The sequential passage of these reactions ensures controlled gas evolution during heat treatment of the mixture and thereby ensures the integrity of the fire retardant coating.

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