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DEVELOPMENT OF A FIRE-PROOF COATING CONTAINING SILICA FOR POLYSTYRENE

The possibility of applying a silica-containing coating to the surface of XPS extruded polystyrene foam, which is characterized by a high degree of flammability, was evaluated. The effect of the content and concentration (11, 22, 44 and 85 %) of orthophosphate acid on the optical properties of silicic acid sols obtained by the exchange reaction between aqueous solutions of liquid glass and acetic acid was studied. The fact of incorporation of orthophosphate acid into the gel structure was confirmed by the results of acid-base titration with a sodium hydroxide solution of the intermicellar liquid isolated as a result of gel syneresis. Using an optical microscope, the structure of the polystyrene film coating after treatment with orthophosphate and sulfuric acid solutions was investigated. In both cases, the effect of an increase in the pore area and a general increase in the looseness of the surface was noted, which can help reduce its hydrophobicity and improve adhesion to the coating. The increase in hydrophilicity of the surfaces of polystyrene films after treatment with acids was also confirmed by the flatter, non-spherical shape of the drops of the composition on them. The structure of the obtained coatings on polystyrene films was analyzed. The similarity of the directions of the cracks in the case of treatment of the films with solutions of both acids was noted, and an assumption was made about the presence of uniform deformation stresses during gel shrinkage. A microscopic study of coatings on the surface of extruded polystyrene foam was conducted, and a positive effect of orthophosphate acid on the density of their structure was established. It was determined that the optimal solution for obtaining a uniform coating is the modification of the sol with the help of a 22 % solution of orthophosphate acid. Schemes of the interaction of the silica coating and the polystyrene base in cases of electrostatic interaction and in the case of the formation of covalent bonds between the coating and the polystyrene surface are proposed.

Keywords: liquid glass, silica-containing coatings, fire protection of building materials, extruded polystyrene foam

1. Introduction

One of the most urgent problems today is energy saving, among the key aspects of which should be highlighted the preservation of heat in houses and apartments. To insulate facades, roofs, plinths and foundations of buildings, heat-insulating materials are used, which usually have a porous structure and are characterized by a low coefficient of thermal conductivity.

Among such construction materials, polystyrene foam has become widely used, which is waterproof, easy to process and cheap.

According to the state standards of Ukraine, harmonized with European standards, there are two types of expanded polystyrene: expanded (or foamed) and extruded, labeled as EPS (DSTU B EN 13163:2012) and XPS (DSTU B EN 13164:2019), respectively. Since EPS polystyrene belongs to building materials of low and moderate flammability (groups G₁–G₂), it is more often used for facade insulation. XPS polystyrene has a denser monolithic structure, is stronger compared to EPS polystyrene and frost-resistant, which is why it is called a new generation heat-insulating material.

Extruded polystyrene foam is used to insulate surfaces where there are high requirements for mechanical stability and adhesion of heat-insulating boards to the base on which they are fixed.

Despite high energy efficiency, one of the main disadvantages of polystyrene XPS is a high degree of flammability – it is classified as materials with medium and high

flammability (groups G₃-G₄). Of course, certain types of polystyrene contain flame retardant additives, but such brands have a higher price, so they are not always in demand among consumers. In addition, flame retardants often contain halogens and emit toxic substances during combustion.

In view of the above, the study of ways to increase the fire safety of highly flammable heat-insulating materials is relevant.

2. Analysis of literary data and formulation of the problem

The analysis of recent publications shows that effective polystyrene fire-retardant coatings are compositions based on SiO₂ gels obtained from organosilicon precursors by sol-gel technology, with the addition of phosphorus- and nitrogen-containing flame retardant agents [1–3], but the release of toxic substances into the atmosphere is a big problem during their disposal or fire. In addition, it should be noted that the coatings on an organic basis, proposed by the authors of the works [1–3], have a high cost and a high smoke-generating capacity, compared to the coatings formed on the basis of inorganic substances.

Adhesion of the coating to the base material is of great importance [4]. It is known that polystyrene has adsorption properties, which, in particular, is used during the manufacture of synthetic ion exchange resins. This is stated in the paper [5], where the use of such adsorbents for the purification of wastewater from dyes is considered, but other fields of application are not described. The results of numerous studies on the absorptive capacity of polystyrene show that the adsorption of the polymer can be caused by van der Waals interactions, capillary condensation, the formation of a Langmuir monolayer, and the filling of micropores [5–7]. The works [5–7] are of great theoretical and practical importance, at the same time we should not forget that it will not be possible to achieve the adhesive strength determined only by the surface forces described by the authors of these publications. Works [8–9] proposed a method of modifying the surface of polystyrene by introducing compounds that give the surface a positive or negative charge. In work [8], for example, vinylpyridine and cetyltrimethylammonium bromide were used as surfactants, in work [9] polyvinylpyrrolidone and polyacrylic acid, and then a silicic acid sol obtained by hydrolysis of tetraethylorthosilicate was applied. But it should be taken into account that under the influence of meteorological conditions, partial compensation of charges on the surface of the coating and polystyrene foam may occur, which leads to a significant decrease in the adhesion of the coating to the base. Therefore, publications devoted to the issue of fixing coatings on the surface of the base by chemical transformation are of great interest.

The results of work [10] deserve attention, where the structural and chemical modification of the polystyrene surface was investigated during treatment with a solution containing a phosphate buffer solution (Na₂HPO₄ and KH₂PO₄, pH 7,39) and sodium chloride. The authors assumed that it is the phosphate ions adsorbed on the polymer surface and the corresponding value of the ionic strength that contribute to the photooxidation process, the scheme of which is shown in Fig. 1. Adsorption of ionic groups can lead to the extension of polymer chains due to electrostatic repulsion and subsequent hydration.

The appearance of charged particles on the surface of polystyrene can contribute to the strengthening of the electrostatic interaction between the organic matrix and sols of silica precursors, which also carry a positive charge in an acidic medium, and a negative charge in an alkaline one [11]. However, adsorption and electrostatic attraction cannot ensure reliable and strong adhesion between the coating and the substrate material.

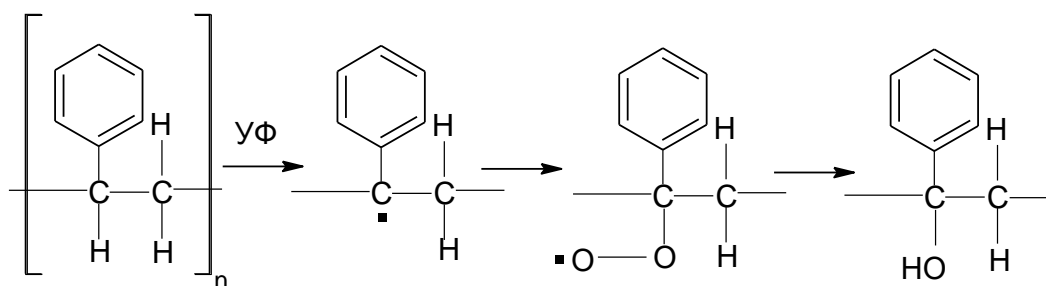


Fig. 1. Scheme of photooxidation of polystyrene after treatment with a phosphate buffer solution

It is known that the silica-containing coating is much better retained on the surface if covalent bonds with functional groups (hydroxyl, carboxyl) are formed. Structurally, polystyrene is a non-polar vinyl polymer, the main chain of which contains phenyl groups (C_6H_5-) as substituents. To improve adhesion, chemical modification of polystyrene with the formation of functional groups or adsorption immobilization of certain substances on the surface of polystyrene is necessary.

Among the methods of formation of functional groups on the surface of polystyrene, it is possible to consider the hydration of the vinyl fragment, the catalysts of the process should be strong acids. The results of the work of the author of the publication [12], where the hydration of polystyrene was carried out in the medium of sulfuric acid and perchloric acid at 25 °C, deserve attention (Fig. 2).

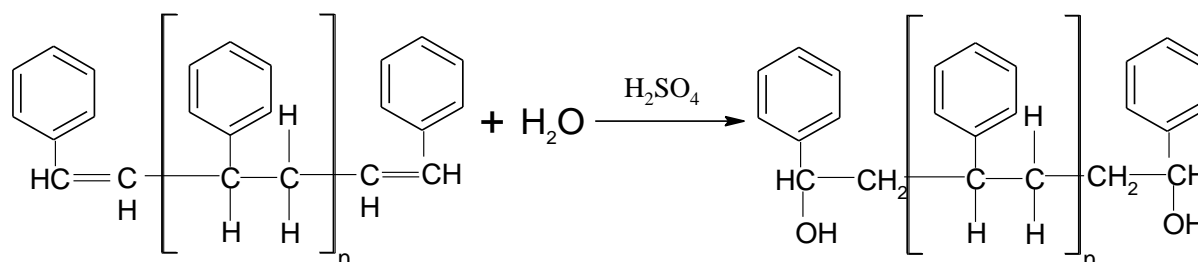


Fig. 2. Scheme of hydration of final fragments of polystyrene under the action of sulfuric acid

Under the action of these acids, the surface of polystyrene is somewhat activated, but the reverse process is likely during the action of fire. In addition, the percentage of release of toxic gases increases. On the other hand, the authors of the work [13] investigated that an acidic medium is favorable for the development of pathogenic microorganisms, viruses, fungi, and, taking this into account, the preliminary treatment of polystyrene with an acid solution is questioned.

It is known that orthophosphate acid is able to polymerize, especially in the presence of an alkaline component. Therefore, it is often used as a binding component in refractory concrete compositions, as well as during the pressing of refractory products for various purposes. Under the influence of temperature, the phosphate anion is able to integrate into the structure of the siloxane framework and form a heat-resistant silicophosphate chain.

Orthophosphate acid can also be used to hydrate the double bond, but stricter conditions are required: high pressure and heat, which can cause destruction of the material [14]. Therefore, it seems appropriate to study in more detail the influence of the content of orthophosphate acid on the changes in the properties of the silica fire-resistant coating and its fixation on the polystyrene base.

3. The purpose and tasks of the research

The purpose of the work is to substantiate the composition of the silica composition as a basis for the fire-resistant coating of heat-insulating polystyrene building materials.

To achieve the set goal, the following scientific tasks must be solved:

- to investigate the effect of the content and concentration of orthophosphate acid on the physical and chemical properties of silicic acid sol;
- to investigate the possibility and determine the ways of fixing the protective coating on the surface of polystyrene.

4. Research materials and methods

The object of research is silica sols based on liquid glass. The subject of research is the processes that occur during gelation in sols based on liquid glass and thermal destruction in case of contact with flames.

The main hypothesis of the research is the assumption that the effect of strong mineral acids can activate the surface of polystyrene to increase the probability of covalent bonds formation with the silicate coating.

Silicic acid sol was obtained as a result of the exchange reaction between sodium silicate (liquid glass) and acetic acid. A solution of orthophosphate acid with a concentration of 11, 22, 44 and 85 % was used as a modifying additive. The sol was applied to the surface of XPS extruded polystyrene foam samples measuring 5x5x3 cm using a bath method. Application of 2–3 layers of coating was carried out after drying the previous layer in the thermal shock mode at 80 °C in a drying cabinet. At the same time, changes in the optical density of the sols over time were determined using a KFK-2 photocolormeter. Distilled water was used as a comparison solution.

To determine the mass fraction of orthophosphate acid embedded in the gel structure, an acid-base titration of the intermicellar fluid released as a result of gel syneresis was performed with a 0.001 M sodium hydroxide solution using phenolphthalein and methyl orange as indicators.

The influence of surface treatment of polystyrene with acids was determined on samples of polystyrene film with a smooth, dense surface. The samples were kept for a day in solutions of concentrated orthophosphate and sulfuric acids, after which a silicic acid sol was applied to the surface of the film by the bath method.

The structure and characteristics of the coating on the polystyrene foam surfaces were evaluated using a Digital Microscope S10 1000x.

5. Study of the influence of orthophosphate acid on the physical and chemical properties of experimental sols

The dependence of the change in the optical density of the experimental silicic acid sols over time on the concentration of the orthophosphate acid used is shown in Fig. 3. With an increase in the concentration of orthophosphate acid, an increase in the rate of change in the optical density of sols over time is observed: in the case of using 85 % H_3PO_4 , the optical density of the sols is maximum.

The dependence of the viability of experimental sols on the concentration of orthophosphate acid is shown in Fig. 4. The figure shows that an increase in concentration has an almost linear effect on the increase in survival time. The sol without the addition of orthophosphate has a slightly different structure of the siloxane framework and is therefore not shown in the graph.

The use of concentrated acid (85 %) leads to a sharp increase in the acidity of the sol, therefore, an acid variant of polycondensation of silicic acid with the formation of clathrates is realized, which proceeds slowly enough within 3 days.

It was determined how completely orthophosphate acid is incorporated into the siloxane framework, for which the pH and mass fraction of the acid in the intermicellar liquids above the experimental gels solidified in closed boxes were determined (Table 1). The mass fraction of the acid that remained in the intermicellar liquid, which was released as a result of the syneresis of the silicic acid gel with the addition of 85 % orthophosphate acid, was determined by titration with an alkali solution in the presence of phenolphthalein and methyl orange indicators.

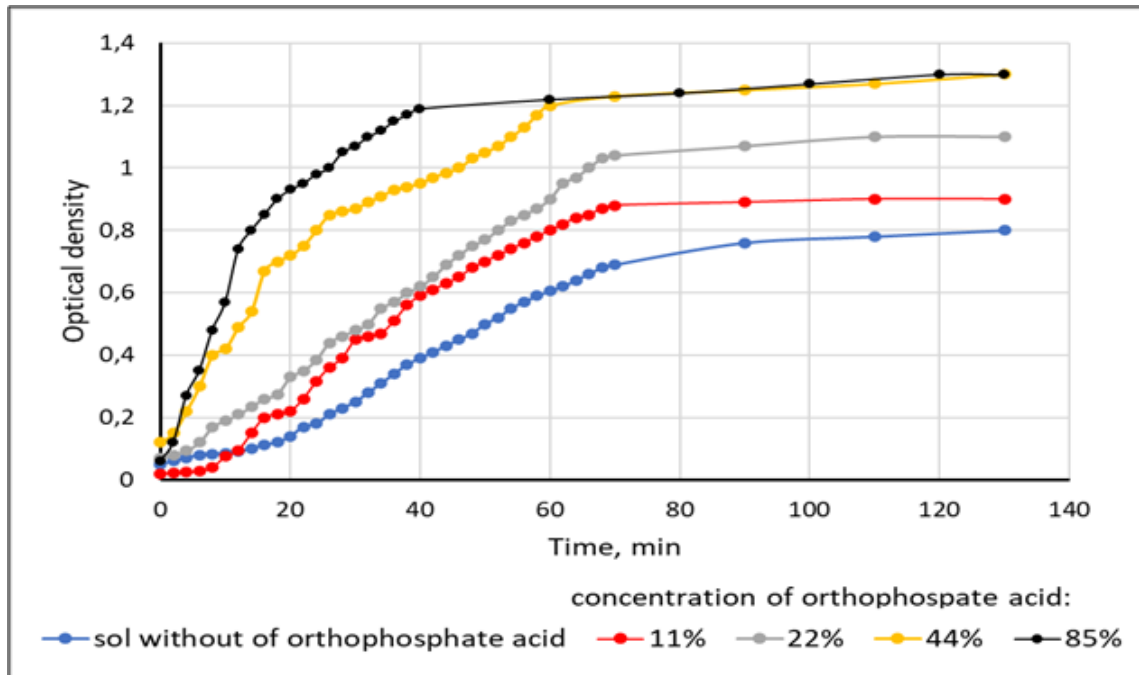


Fig. 3. Dependence of the change in the optical density of the experimental silicic acid sols over time on the concentration of the orthophosphate acid used

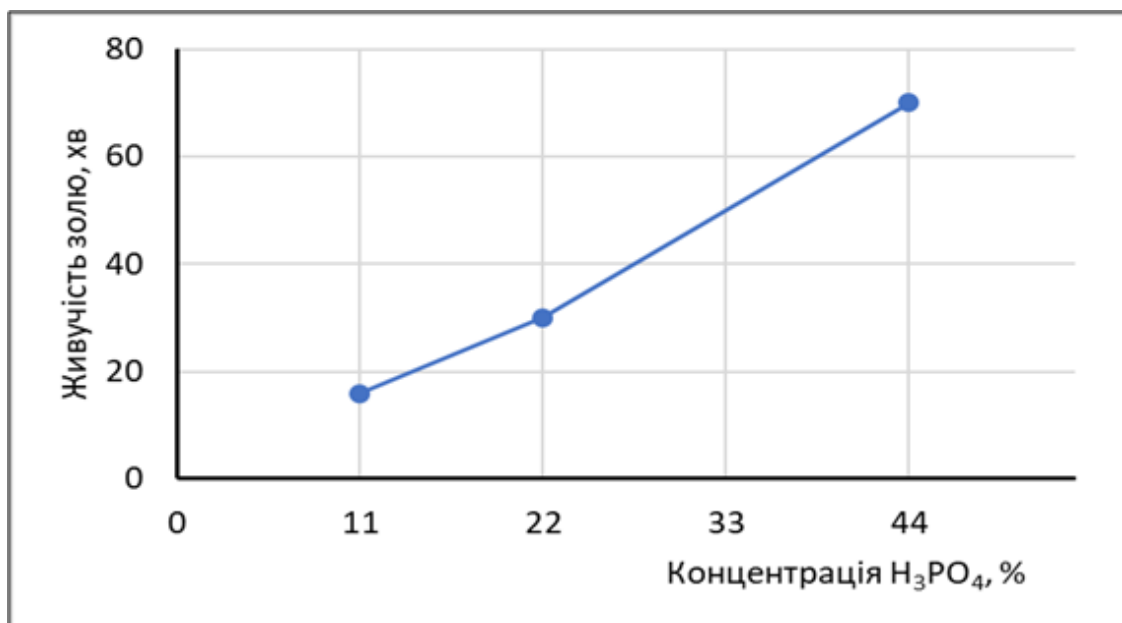


Fig. 4. Dependence of the survivability of silicic acid sols on the concentration of orthophosphate acid

Tab. 1. Results of determining the pH of intermicellar liquids over gels with different content of orthophosphate acid

№	H ₃ PO ₄ concentration, %	pH
1	0	5
2	11	5,5
3	22	5
4	44	4
5	85	3

It was established that the mass fraction of orthophosphate acid incorporated into the siloxane framework is 97,1 %.

6. Study of the possibility of coating the surface of polystyrene

The results of surface treatment of polystyrene with orthophosphate and sulfuric acids and subsequent application of the composition are shown in fig. 5.

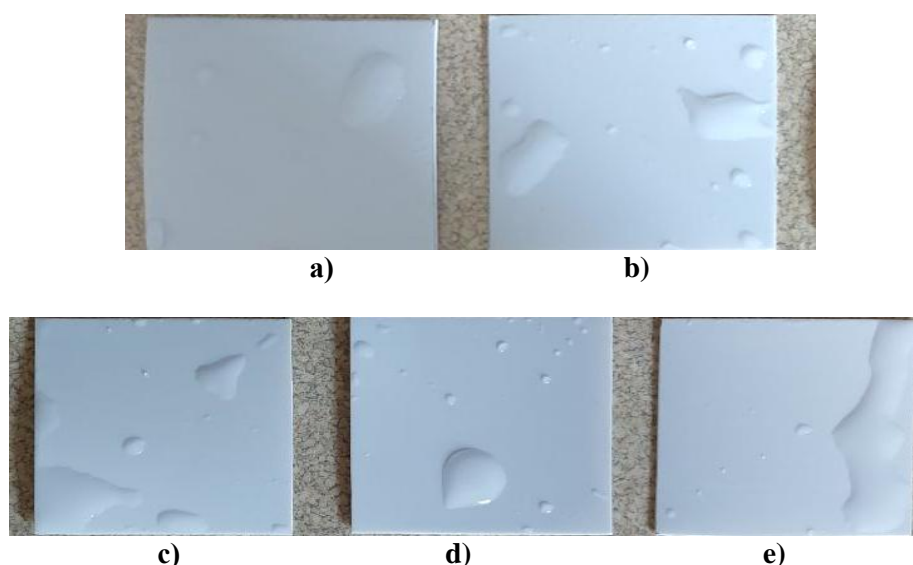


Fig. 5. Photo of drops of the composition on the surface of polystyrene films: a – untreated film; b–e – films kept for a day in solutions of orthophosphate (b, d) and sulfate (c, e) acids. After etching: b, c – washed with running water; d, e – unwashed

On the untreated polystyrene film, the drop has a shape closer to spherical, which indicates the hydrophobicity of the surface. After treating the surface with acids, the drops acquire a flatter shape, that is, the wettability of the surface increases. It is possible to clearly observe the spreading of the composition on the surface of the polystyrene film treated with sulfuric acid without further washing (Fig. 5, e), which demonstrates the maximum positive effect of H₂SO₄ on increasing the hydrophilicity of the polystyrene surface.

The microstructure of the surfaces of polystyrene films after treatment with acids is shown in Fig. 6.

Analysis of the microstructure of the films shows that treatment of polystyrene films with acids makes their surface more voluminous and porous. Despite the fact that sulfuric acid is stronger compared to orthophosphate, treatment with them gave similar effects. The images clearly show an increase in the total area of indentations on the surface of acid-treated polystyrene films compared to the untreated sample. It should also be noted that washing the surface of the film with running water after treatment with acids did not have a decisive effect on modifying the structure of the polystyrene surface.

The coating obtained on the acid-treated polystyrene films held slightly better than on the untreated sample, although the purpose of this experimental study on the control

samples was not to achieve a sufficient level of adhesion. The coatings obtained on the processed samples had cracks, and attention should be paid to their uniform branching, which suggests the presence of uniform deformation stresses during gel shrinkage.

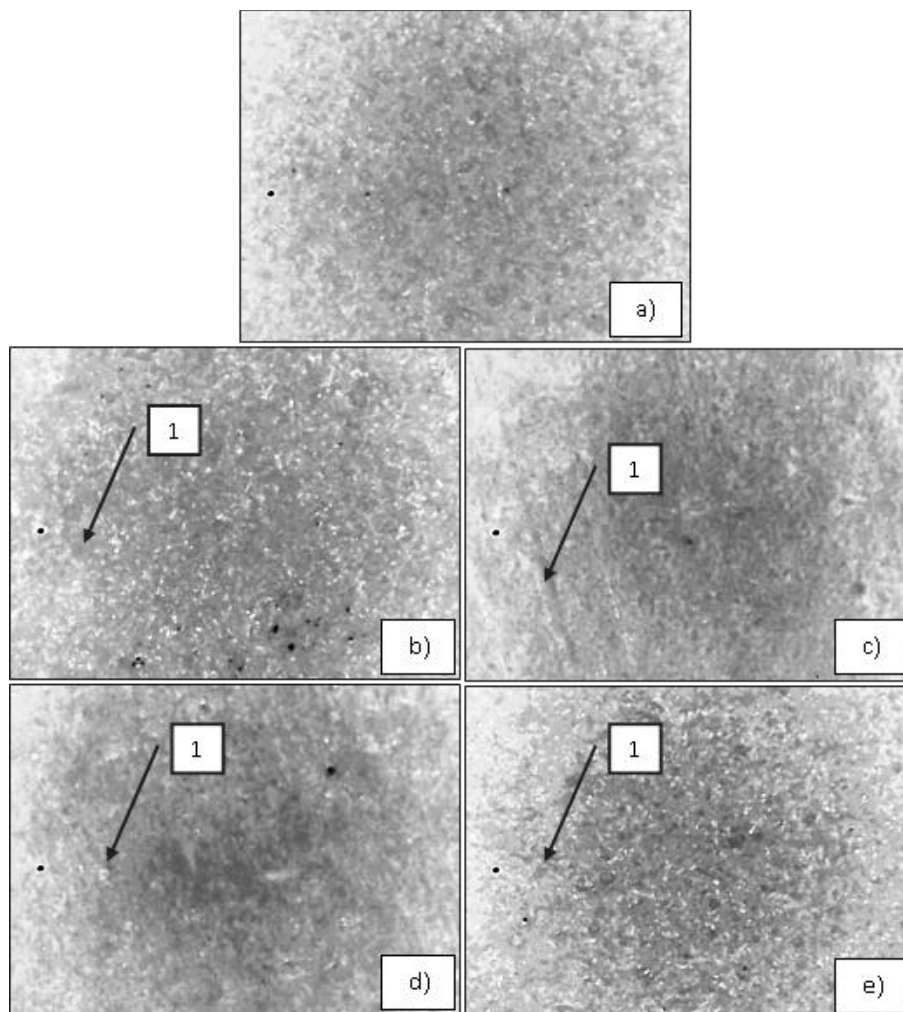


Fig. 6. Microstructure of the surfaces of polystyrene films treated with solutions of sulfuric and orthophosphate acids: a – untreated film; b–e – films kept for a day in solutions of orthophosphate (b, d) and sulfate (c, e) acids. After etching: b, c – washed with running water; d, e – unwashed, 1 – indentations formed after treatment with acids

The microstructure of coatings obtained on the surfaces of extruded polystyrene foam based on compositions with different concentrations of orthophosphate acid is shown in Fig. 7.

We observe that the coatings obtained on the basis of compositions with the addition of orthophosphate acid (Fig. 7, c–f) are denser compared to those obtained without the use of additives (Fig. 7, b). The most homogeneous in thickness are coatings based on compositions with the addition of orthophosphate acid with a concentration of 11 and 22 %.

Polystyrene adsorbs phosphate ions, and its surface is negatively charged. Due to the electrostatic attraction of the positively charged silicic acid sol to the surface, better adhesive characteristics and fixing of the protective coating on the surface of the material are ensured, so it is possible to assume the scheme of the electrostatic interaction of the coating and the polystyrene surface shown in Fig. 9.

Based on the conclusions made, the following chemical mechanism of the formation of a silica-containing coating on the surface of polystyrene can be assumed, as shown in Fig. 10.

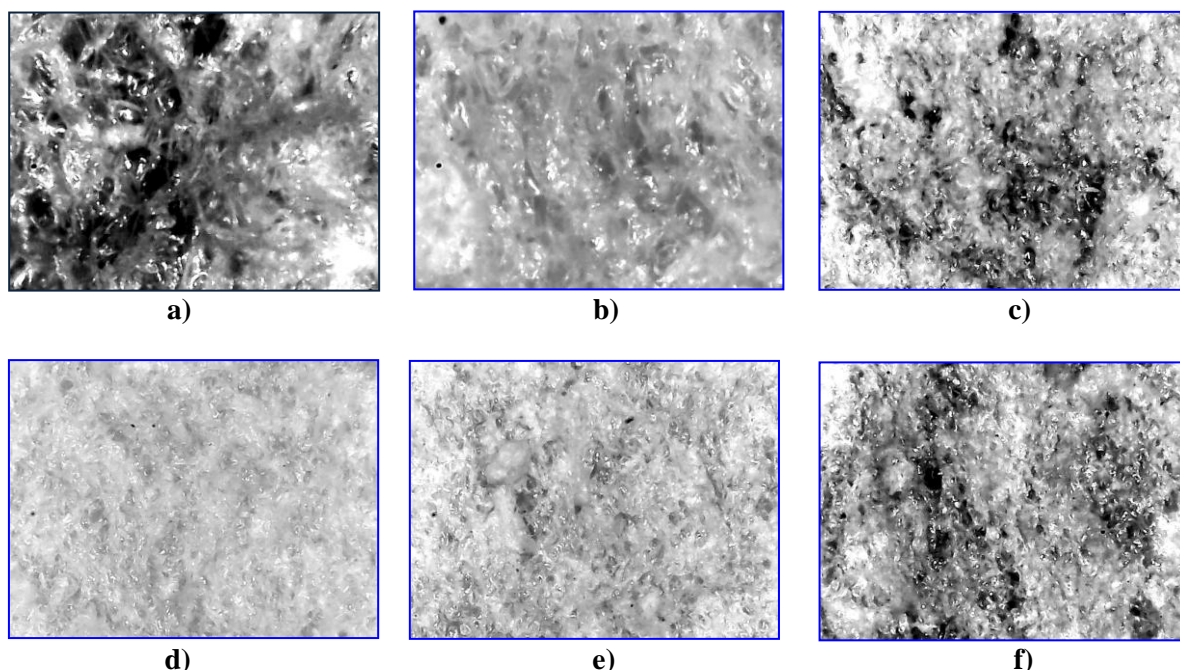


Fig. 7. Microstructure of the surface of extruded polystyrene: a – without coating; coatings based on compositions: b – without addition of orthophosphate acid and with addition of orthophosphate acid of the appropriate concentration; c – 11 %, d – 22 %, e – 44%, f – 85 %

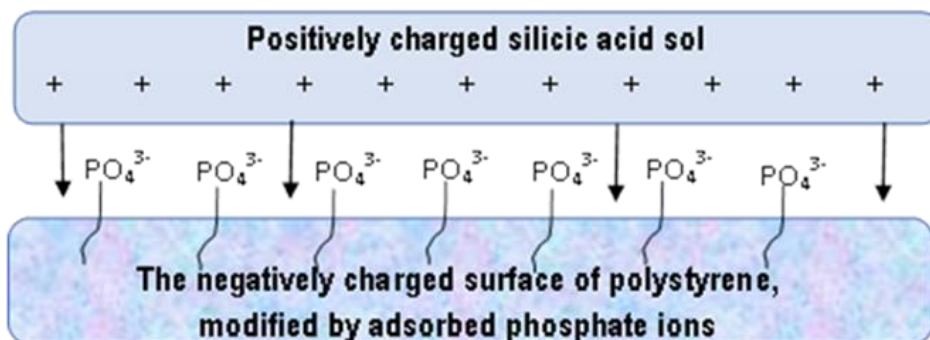


Fig. 9. Scheme of the electrostatic principle of adhesion of a silica-containing coating on the surface of polystyrene

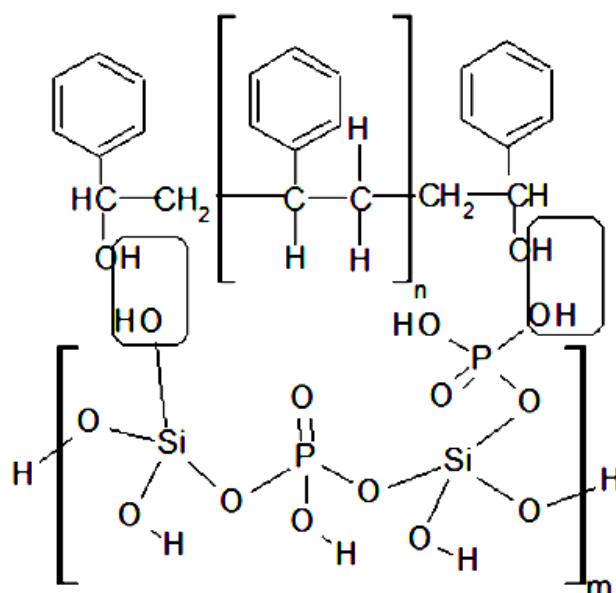


Fig. 10. Scheme of the formation of a silica-containing coating on the surface of polystyrene

It can also be assumed that the orthophosphate will covalently bind the $\equiv\text{Si-OH}$ groups of the gel and the $-\text{CH}_2\text{OH}-$ group of polystyrene and be incorporated into the siloxane structure.

7. Discussion of the results of the study of the impact of orthophosphate acid on the physicochemical properties of experimental sols

Analysis of the dependence of the optical density of silicic acid sols with H_3PO_4 additives showed that with an increase in the concentration of orthophosphate acid, the time of solidification of the sol increases, but the time of latent coagulation decreases. Just before the beginning of hidden coagulation, when colloidal particles begin to thicken, it is advisable to apply sol to the surface of the building material in order to obtain a uniform coating. An interesting observation is that the solidification time of the sol with H_3PO_4 of 11 % concentration is the shortest among those studied. On the other hand, it is known that it is precisely at a pH of $\sim 5,5$ that the rate of silica gel formation is maximal (the pH value in the intermicellar liquid above the solidified gel is exactly 5,5). Taking into account the time of onset of hidden coagulation and the durability of the composition, which will be acceptable and satisfactory for applying a coating to the surface of the building material, it is possible to give preference to the use of sol with the addition of a 44 % solution of orthophosphate acid.

However, the study of the microstructure of coatings on the surface of extruded polystyrene shows that greater homogeneity in thickness can be achieved by adding a solution of orthophosphate acid with a concentration of 11 or 22 % to the sol. Taking into account the fact that the survivability of sols with 11 % H_3PO_4 solution is the lowest among those studied, it is optimal to modify the sols using a solution of orthophosphate acid with a concentration of 22 %.

Calculations of the mass fraction of orthophosphate acid in the gel structure suggest that orthophosphate acid forms covalent bonds with silanol groups. However, it should be noted that not all orthophosphate acid is incorporated into the structure, a certain amount of it is neutralized by the action of the alkali formed as a result of the hydrolysis of sodium silicate.

Treatment of the polystyrene surface with acids has a positive effect – the hydrophilicity and wettability of the surface increases. Considering the higher heat resistance of silicophosphate compositions compared to silicosulfate compositions, it is more expedient to use orthophosphate acid for surface treatment.

However, under the influence of certain conditions, partial compensation of charges on the surface of the coating and polystyrene foam may occur, which will lead to a significant decrease in the adhesion of the coating to the base (Fig. 9), so the option of more reliable fixation of the coating on the surface of polystyrene should be considered, namely due to the formation of covalent bonds connections (Fig. 10). Then it will be more effective to treat the surface with sulfuric acid, which contributes to the formation of hydroxyl groups that will covalently bind the applied coating and improve adhesion. It can be asserted that in the case of application of silicic acid sol on the surface of polystyrene and subsequent heating, water molecules condense between the $\equiv\text{Si-OH}$ groups of the gel and $-\text{CH}_2\text{OH}$ of polystyrene, resulting in the formation of covalent bonds.

It should be noted that the key to conducting the chemical polycondensation reactions shown in Scheme 10 is the preservation of the conditions for the electrostatic fixation of the coating on the surface of polystyrene.

It remains unclear how stable the bond between the coating and the polystyrene

base will be under conditions of high temperatures or open fire, so further studies are expected to study the influence of the composition of silicate sol on the stability of fire-resistant coatings and changes in the fire resistance of extruded polystyrene.

8. Conclusions

1. The effect of the content and concentration of orthophosphate acid on the properties of silicic acid sol was investigated spectrophotometrically. The density and structure of coatings on the surface of extruded polystyrene were evaluated using a microscopic study. It was established that the addition of a 22 % H_3PO_4 solution is optimal for obtaining a uniform coating on the surface of polystyrene foam.

2. The positive effect of treating the surface of polystyrene with solutions of sulfuric and orthophosphate acids to reduce hydrophobicity and improve the adhesive characteristics of the silica-containing coating was noted. Schemes of the interaction of the silica coating and the polystyrene base in cases of electrostatic interaction and in the case of the formation of covalent bonds between the coating and the polystyrene surface in places of partial destruction of the double bonds of the polymer are proposed.

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РОЗРОБКА ВОГНЕЗАХИСНОГО КРЕМНЕЗЕМВМІСНОГО ПОКРИТТЯ ДЛЯ ПОЛІСТИРОЛУ

Оцінено можливість нанесення кремнеземвмісного покриття на поверхню екструдованого пінополістиролу марки XPS, що характеризується високим ступенем легкозаймистості. Досліджено вплив вмісту та концентрації (11, 22, 44 та 85 %) ортофосфатної кислоти на оптичні властивості золів кремнієвої кислоти, добутих реакцією обміну між водними розчинами рідкого скла та оцтової кислоти. Факт вбудовування ортофосфатної кислоти в структуру геля підтверджено результатами кислотно-основного титрування розчином натрій гідроксиду інтерміцелярної рідини, виділеної внаслідок синерезису гелю. За допомогою оптичного мікроскопа досліджено структуру покриття полістирольної плівки після обробки розчинами ортофосфатної та сульфатної кислот. В обох випадках відзначено ефект зростання площі пор та загальне збільшення рихлості поверхні, що може сприяти зменшенню її гідрофобності та покращенню адгезії до покриття. Підвищення гідрофільності поверхонь полістирольних плівок після обробки кислотами також підтверджувалося більш плоскою, несферичною формою крапель композиції на них. Проаналізовано структуру отриманих покриттів на полістирольних плівках. Зауважено схожість направленості тріщин у випадку обробки плівок розчинами обох кислот та висунуто припущення про наявність рівномірних деформаційних напружень під час усадки гелю. Проведено мікроскопічне дослідження покриттів на поверхні екструдованого пінополістиролу, встановлено позитивний вплив ортофосфатної кислоти на щільність їхньої структури. Визначено, що оптимальною для отримання однорідного покриття є модифікація золю за допомогою 22 %-го розчину ортофосфатної кислоти. Запропоновано схеми взаємодії кремнеземистого покриття та полістирольної основи у випадках електростатичної взаємодії та у разі утворення ковалентних зв'язків між покриттям та полістирольною поверхнею.

Ключові слова: рідке скло, кремнеземвмісні покриття, вогнезахист будівельних матеріалів, екструдований пінополістирол

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