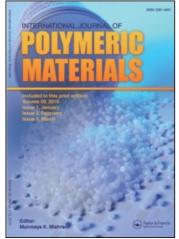
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Intumescent Fireproof Coating Based on Water Glass

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The effectiveness of fireproof intumescent coatings based on water glass can be considerably increased if the coagulated sodium water glass is used as an intumescent component. When heated, the coagulated sodium water glass creates a steam-gas cloud above the protected surface which together with foam formation decreases the temperature of the protected surface. The mixture of sodium water glass, portland cement and dehydrated clay which, besides adhesion, maintain the durability of the composition are used as binders due to their cohesive properties.

Keywords: Intumescent coatings; water glass; fireproof dehydrated clay; sodium

INTRODUCTION

Fireproof intumescent coatings based on sodium water glass are complex compositions which usually contain: binder intumescent additives and a hardening agent [1]. They are applied to cover the surface of constructive structures by a thin layer (up to 4-5 mm). At temperatures $350-500^{\circ}$ C a coating intumescents and a porous thermoinsulating layer (up to 3-4 cm thick) is formed. Due to the low heat conductivity of the porous layer fast heating and destruction of protected constructive elements are prevented.

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EXPERIMENTAL

An intumescent component is previously prepared due to the coagulation of sodium water glass in calcium chloride solution medium. When adding sodium water glass being sprayed by compressed air into CaCl₂ concentrated solution, hydrolysis reaction prevails on two liquid phase boundary due to the solutions pH difference. The neutralization anions charge of leads to their immediate coagulation at the interface boundaries. If silicates concentration is high enough a membrane with negative charge from the silicate side and with positive charge from calcium chloride solution side is formed. When the silicate solution viscosity is high the membrane transforms gradually into a gellike shell from coagulated silica with a small concentration gradient by calcium from CaCl₂ solution side and by sodium from silicate side [2]. After drying from outer damp grains from water glass drops, which obtain water-resistance of outer, partly calcined layer but are not (due to shrinkage microcracks formation (Fig. 1a)) steam-and moisture-proof, are formed.

Thermogravimetric analysis of the granules obtained allow to determine the level of dehydration temperatures and water mass losses during heating. As it is shown in the thermogram (Fig. 2) up to 14,5% of the water adsorbed by mass are evolved at temperature up to 120° C but the water does not participate in the intumescence process as at this temperature level the liquid (molten) phase, which can hold the water vapours being formed, is absent. The intumescence process occur (according to the thermogram) at 320° C and higher. In this temperature interval the removal of water from ligand shells takes place. This process is the most intensive (up to 13,3% of mass) up to 560° C. The overall losses of the sample with mass 195,4 g are 28,7% at 970° C.

It is necessary to note that during the real fire due to the considerable gradient of the temperatures on the coating surface, the process of the liquid phase formation leads or is simultaneous to the evolution of the water adsorbed. So besides chemically bound (coordinated) water the considerable part of adsorbed water participates in the intumescence process.

The grains obtained are used fire-proof compound preparation. Sodium water glass with density $\rho = 1,39 \text{ kg/m}^3$ is used as a binder. Portland cement is used as a sodium water glass hardening agent.

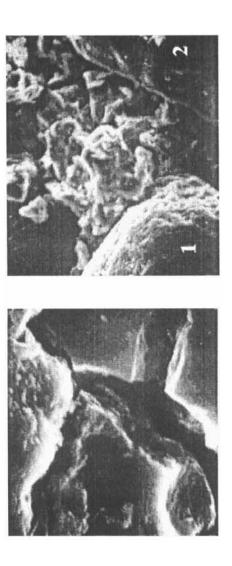


FIGURE 1 Microstructure of: a - shell after drying the calcined layer (*3000 magnification); b - composition (*300 magnification); (1 - portland cement particle, 2 - dehydrated clay particle).

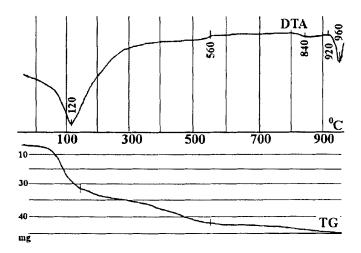


FIGURE 2 Thermogram of the sodium silicate coagulated by calcium chloride.

The analysis of samples chosen for spectroscopic investigations are carried out by means of spectrometer IRS-29 in frequency interval $450-1400 \text{ cm}^{-1}$. The samples are fine powders mixed with vaseline. They are placed between KBr plates.

RESULTS AND DISCUSSION

Due to the high rate of calcium ions transition into solution, which are formed during the hydrolysis and hydratation of portland cement minerals, practically immediate coagulation of sodium water glass with its subsequent hardening proceeds. In this case, the hydration processes of cement do not have considerable influence on the strength of coating applied.

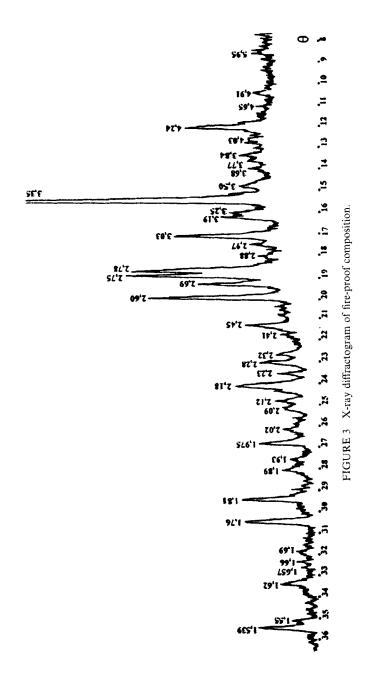
Taking into account the significance of the conditions control of coating structure formation during the initial period the influence of retardants is studied. These retardants (immediately after mixing with water) neutralize or bind free or evoluting calcium hydroxide obtained as a result of Portland cement hydration.

The immediate hardening of this systems is prevented due to the blocking of a portland cement clinker surface by phosphate-ions which are introduced into the composition as the 15% solution of sodium phosphate Na₃PO₄ while making cement slip. Depending on the concentration of the retardant introduced, the process flows till its complete neutralization. This allows to regulate the composition vitality in the limits given by applying technology. When a retardant used in the composition is exhausted, the coagulation and hardening of sodium water glass occur. In this case the intumescent component surface has a gradient of calcium atoms content, which favour the hardening. There is the fast hardening of composition by means of silicates which are formed from sodium water glass during the transition of calcium cations into solution. This can be concluded from the X-ray diffractogram (Fig. 3) where the reflections corresponding (due to [3]) to tobermorite gel ($d_{\alpha} = 0,278 \text{ nm}$) are consistently indicated. Afterwards the water from the colloids formed during the water glass coagulation is actively removed, and pore space formed is filled with new formations of hydrating portland cement of tobermorite - like calcium hydrosilicates type CaO · SiO₂ · H₂O ($d_{\alpha} = 0,303$ nm). The presence of bound water in calcium hydrosilicates increases the intumescence effectiveness of fire-proof composition during fire action. The crystalline hydrates evolute water vapours in temperatures intervals up to 500-700°C.

Thus, during a period of time the strength of a coating increases due to cement minerals hydration. This is followed by the growth of contacts number between all composition components and, consequently, of the fire-proof coating density.

As it is seen from IR spectra (Fig. 4) the spectrum of powder obtained form intumescented grains differs mainly by the appearance of more intensive and right-biased wide stripe $760-790 \text{ cm}^{-1}$ and stripes in the frequency area 835 and 834 cm^{-1} (also more intensive but left-biased). It is known [4] that anion vibrations in the majority of cases determine the spectrum character only, the influence of cation has generally an effect on the displacement of absorption stripes in the area of higher or lower frequencies depending on the radius of the given cation. So, according to the noted differences in spectra grains before and after fire effect the changes of calcium and sodium cations states in the composition can be supposed.

The photographs of the composites after fire action (Fig. 5) show the effective protection of paper honeycombed plastic by an intumescent coating. Above plastic is made of pressboard cladded by polyethylene.





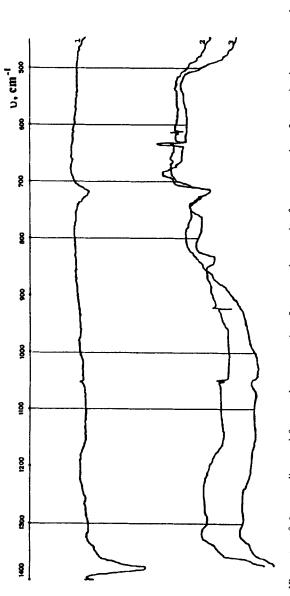


FIGURE 4 IR-spectra of: 1 - vaseline used for samples preparation, 2 - coagulant grains from water glass, 3 - grains intumescented at $450^{\circ}C$ temperature.

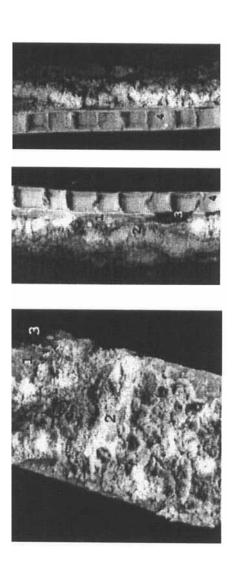


FIGURE 5 The external appearance of the composition applied to the surface of paper honeycombed plastic after fire action: 1 - the surface protected by the intumescent coating removed; 2 - intumescent fire-proof coating; 3 - the charry surface of paper honeycombed plastic not covered with fire-proof composition; 4 - the section of paper honeycombed plastic.

In this case only control surface areas (number 3 Fig. 5) are subjected to destruction due to charring. At the same time the honeycombed plastic surface, protected by the coating, saves its properties completely as it is seen from Figure 4 where number 1 marks the surface areas of honeycombed plastic, from the surface of which intumescent (after fire action) fire-proof coating is removed.

CONCLUSION

Due to the dehydration of initial hydrosilicates during the fire action on the composition their sintering with amorphous (glassy) phase formation takes ace. The presence of a glassy phase predetermines the formation of a dense melt (impenetrable for steam-gas mixture) on the surface of the material protected, with the help of which the intumescented coating is formed during the fire.

It is necessary to add that the system including together with a crystal phase the considerable volume of compressed dehydrated gel of silicic acid (having the structure of polymer compound) has a high strength not only during compression but also during tension and breaking off. The above mechanic characteristics exclude coating peeling during operation which is connected with sharp temperature vibrations of the environment.

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