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CHEMICAL SOLUTIONS FOR FIRE PROTECTION PROBLEMS



The modern approaches to the creation of fire protective coatings through modifying intumescent systems by nanomaterials have been considered; chemical reaction mechanisms at high temperature have been studied. Interactions among the components of polyphosphate type intumescent blend have been researched, the correlations between the directions of chemical processes and fire retardant properties of intumescent coatings have been found. Efficient methods for raising fire-protective efficiency and improving performance characteristics of intumescent coatings (operation life, resistance to environmental factors and bio-contamination) have been proposed. The results of fundamental research have enabled developing new flame retardant compositions whose properties are confirmed by tests in accordance with existing standardized methods; these results have been commercialized.

Keywords: *intumescent system, fire-protective efficiency, operation life, nanomaterials, montmorillonite, and biocides.*

In Ukraine, advanced fire-retardant technologies have been developed, at least, thirty years, starting with R&D works of the L.M. Litvinenko Institute of Physical, Organic and Coal Chemistry of the NAS of Ukraine (IPOCC), in cooperation with R&D Fire Protection Institute that was the leading agency of the USSR in the field of fire safety. This cooperation has resulted in the creation of technique for obtaining an intumescent graphite-based fireproof coating of new generation and several unique polymeric fire-retardant agents [1–3] produced at the IPOCC pilot plant and used for fire protection of strategic facilities such as military complexes, ammunition depots, and NPP generator halls [4, 5]. The R&D works concerning the design of effective fire-retardant formulas based on fundamental research of reaction ability and chemical transformation mechanisms continue and are funded by the NAS of Ukraine [6–10].

This research is of paramount importance in the view of Euro-integration aspiration of Ukraine and respective harmonization of the national fire safety regulations and standards with the European stand-

ards containing requirements that cannot be met unless advanced chemical methods are used [11–13]:

- ✦ Prohibition of the use of halogen-containing materials and flame retardants in the construction industry;
- ✦ Ensuring of stable composition of fireproof coating, prevention of its adverse effect on the human health during operation, and minimization of toxic effect of combustion products on the human being during the fire.

Proceeding from the above, this research is aimed at searching and designing effective and feasible fireproof coatings for construction objects having good operational, sanitary and hygienic, and environment friendly properties.

INTUMESCENT COATINGS

On the basis of the IPOCC researches and the world fire-fighting experience, an intumescent system of conventional composition is chosen as subject of study [9, 14–18]:

- ✦ Acid donor: phosphates, ammonium polyphosphate (APP);
- ✦ Carbonizing agent: pentafluoride (PF) and its analogues;

✦ Gasifier, foaming agent: melamine derivatives (MD), derivatives of dicyandiamide, carbamide.

The IS-containing coatings are referred to thin fireproof intumescent coatings. At high temperature, the coatings expand 20–80 times and transform into incombustible coke layer preventing increasing temperature of material for a certain time (Fig. 1).

Fire-retardant effect of these systems is connected with two main factors, *the chemical* and *the physical* ones. The chemical factor is determined by carefully selected (in terms of properties and quantity) components that, at high temperature, undergo endothermic reactions with the formation of incombustible coke layer or decay releasing incombustible substances thereby inhibiting the combustion processes. The physical factor is associated with thermal insulation properties of the layer that prevents the protected object from heating during the fire and with strength, density, and thermal conductivity of the coke layer.

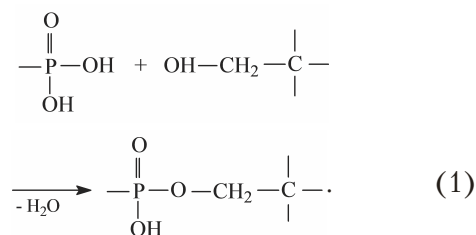
The con-calorimetric method is widespread and practically the only technique for studying fireproof effectiveness of IS [19–21], which enables getting information on the rate of heat release, which is, basically, the key factor for assessing the material flammability with indirect data on probable chemical transformations. However, these data do not give a comprehensive understanding of physical and mechanical parameters (density, adhesion to substrate, temperature of destruction) of the coke layer, which makes impossible to ensure long-term fire resistance of construction objects during the fire.

The authors of this paper have proposed a gradual study of chemical and physical transformations in IS with identifying the structure of chemical reaction products and the physical parameters of the coke layer [22, 23]. The method foresees keeping the samples of intumescent coatings at specified temperature (within the range of 200–700 °C) with further measurement of volume intumescence factor (K , cm³/g), mass loss Δm (%), density of intumesced material ρ (g/cm³), and adhesion of intumesced layer to substrate. These studies enable predicting fireproof effectiveness

of IS-based coatings [23]. At the same time, the chemical composition of coke layer is analyzed using IR and NMR spectroscopy.

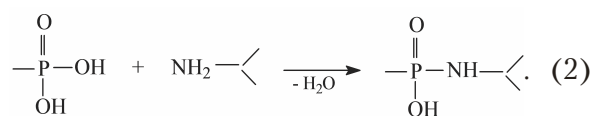
CONTROL OF CHEMICAL TRANSFORMATION MECHANISM IN INTUMESCENT SYSTEM

The fireproof mechanism in intumescent technology is conventionally described by the scheme of chemical reactions of blend components, namely [25, 26]: thermal decomposition of phosphates or poly phosphates with the formation of phosphoric acids and ammonia; decomposition of foaming agent (amine) with the formation of incombustible gases (NH₃, CO₂, N₂ etc.); dehydration and esterification of polyol by phosphoric acid with the formation of spatial structures of the coke layer – the base of heat insulating envelope.



The proposed scheme of transformations gives a very simplified and formalized understanding of transformations taking place in the system of phosphoric acid donor/polyol/foaming agent within the temperature range of basic chemical processes (100–400 °C). In this interpretation, the functional role of organic amine reduces to the formation of incombustible gases that inhibit combustion and ensure intumescence of envelope formed by polyol and phosphate.

The systematic study of chemical composition of the coke obtained within the range of temperature from 100 to 400 °C using IR and NMR spectroscopy [8] has showed that a nucleophilic replacement reaction (2) occurs near the 5th coordinating atom of phosphorus between phosphoric acid (or its derivatives) and amine, in the system:



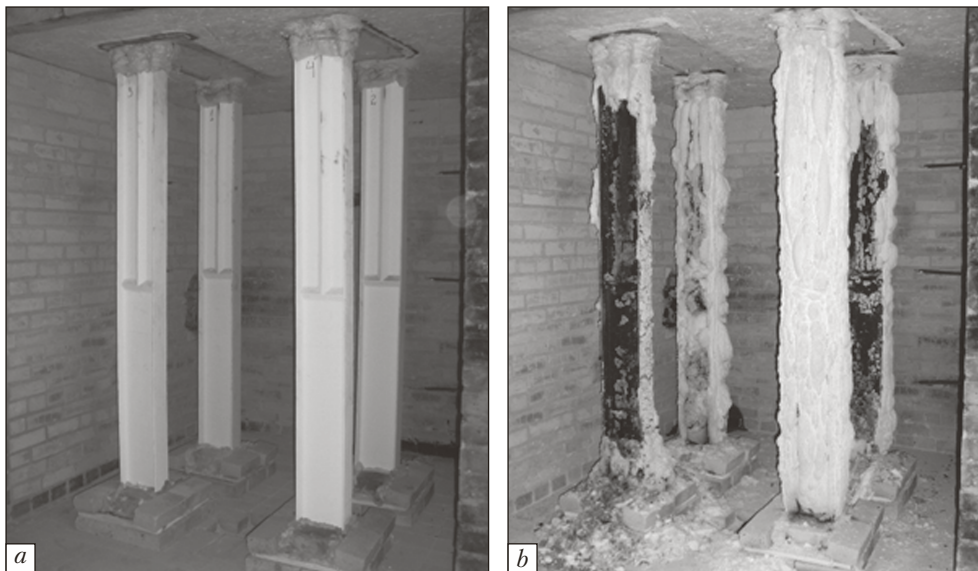


Fig. 1. Steel piers with intumescent coating before (a) and after (b) fire tests according to DSTU B. V.1.1-14:2007. Intumescent paints for metallic structures have been tested by *Donstroitest*

The strength and heat resistance of the coke layer increase as the rate of formation of phosphamide bond $-P-N=$ by aminolysis raises (2). This conclusion stimulated the search of optimal conditions for the realization of scheme (2) in IS and enabled designing a procedure for the production of new complex fire-retardant additives for intumescent paints containing amine and phosphate components, which are not manufactured in Ukraine. A fire-retardant additive playing roles of acid donor and foaming agent simultaneously has been used for developing a new efficient fire-retardant composition for the timber with improved fireproof and operational properties [6, 7], which is manufactured in Ukraine and branded *Endoterm 250103* (*Spetsmaterialy* RPC, Kyiv).

The hypothesis that the foaming agent is an active component of IS for building a heat insulating envelope [24] has been confirmed by studying the mechanisms of chemical reactions and the parameters of intumesced layer of APP/PF/amine system. It has been established that amines which are the most widespread in the production of intumescent coatings (melamine, dicyandiamide, carbamide) react with other system components in different di-

rections, which enables harmonizing the processes of expansion, formation and time of service life of the coke layer by combining foaming agents within one IS in order to raise fireproof effectiveness of coating as a whole. These studies have enabled establishing a clear dependence between the amine structure and the limit of fire resistance of metal structure ensured by fireproof coating as showed in Fig. 2.

The shares of melamine, dicyandiamide, and carbamide in IS for ensuring respective limits of fire resistance (R , min) are important data for preparing cost-effective formulas of fire resistant agents, which makes possible to decrease the costs of fire protection of structures of the 2nd and the 3rd classes of fire resistance, at least, by 10–15%.

WAYS OF MODIFICATION OF INTUMESCENT SYSTEM Fireproof effectiveness

Numerous researches of IS on searching optimal components of process have demonstrated that the range of these substances is very limited: APP, PF and analogues, and MA derivatives. The inert fillers (metallic oxides, fire-retardant agents, ceramic materials, wollastonites, etc.) do not improve radically

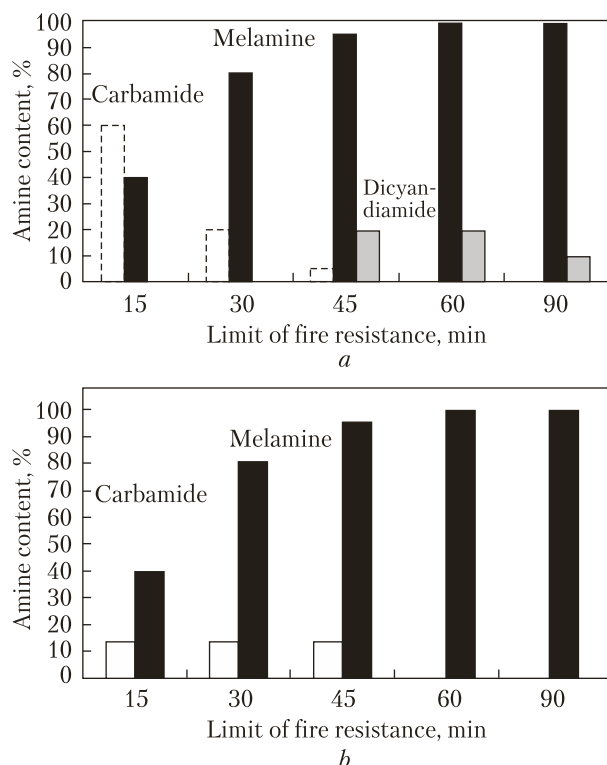


Fig. 2. Approximate proportions of amines (%) in intumescent coating for ensuring respective limits of fire resistance (min) of steel structures tested according to DSTU B. V.1.1-14:2007: *a* – intumescent composition with styrene acrylate copolymer in organic solvent; *b* – emulsion intumescent composite with ethylene vinyl acetate copolymer

the fireproof properties of IS [22, 25–28]. Therefore, the scientific approach to the design of new effective intumescent composites in terms of varying both the chemical nature of the components and their proportion seems to be almost exhausted.

Therefore, optimization of IS with nanomaterials as admixtures improving fireproof, operational,

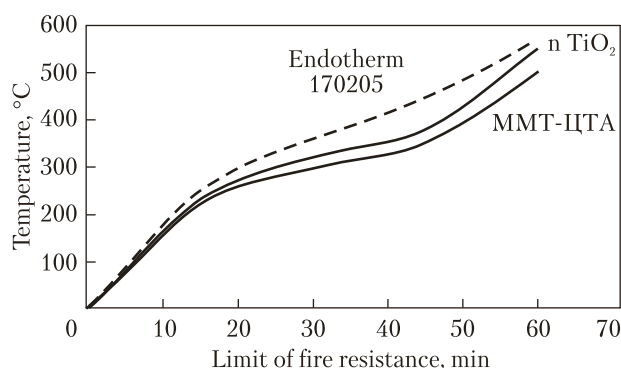


Fig. 3. Measurement of fire resistance of steel pier according to DSTU B. V.1.1-14:2007 (coefficient of profile section 192 m⁻¹) protected by *Endotherm 170205* intumescent coating with nanocomposite additives: *n*-TiO₂ – titan nanooxide, MMT-CTA – cetyl-trimethyl-ammonium montmorillonite (from records of *Donstroitest*)

and environment properties of the coatings is a good solution [29–30]. The study of effect of nanooxide and nanoclay admixtures (montmorillonite and bentonite clays) has enabled establishing the fact that positive action of nanoparticles manifests itself in four basic directions:

- ✦ Catalysis of chemical processes between the IS components with modification of mechanisms of reactions, their rates and composition of the products at all stages of transformations under the action of high temperature;
- ✦ Creation of barriers for oxygen penetration to the combustion area and for migration of off-gases;
- ✦ Involvement into carbonization with the formation of heat insulating coke layer having a high strength and a strong thermal resistance;
- ✦ Extension of service life of the coating during operation and reducing its toxicity during the fire.

Effectiveness of Decontamination of Materials Treated with EVAFARB in the Case of Artificial Contamination by Test Strains

Material	Effectiveness of decontamination in 24 hours/30 days				
	<i>S. aureus</i>	<i>E. coli</i>	<i>C. albicans</i>	<i>A. niger</i>	<i>M. tuberculosis</i> B ₃
Wood	90.4/97.0	92.2/99.2	94.5/99.5	91.2/91.2	100/100
Gypsum wallboard	93.1/91.0	90.5/99.4	90.5/95.0	91.5/93.5	100/100
Concrete	90.0/93.9	91.5/97.0	89.9/91.0	90.5/95.5	98.7/100

The mechanisms of chemical effect of metallic nanooxides and nanoclays on the reactions running in IS at a temperature of 100–500 °C have been considered in [29–32]. These studies have allowed the researchers to identify montmorillonite modified by quaternary ammonium salts as optimal admixture for IS, which increases the fire resistance limit of protected steel structures by 10–15% (Fig. 3).

The application of modified *Endoterm 250103* fire-retardant composition for timber has enabled to significantly reduce costs of fire protection and to raise its resistance to moisture and atmospheric effects.

Service life

Pursuant to ETAG 018-2 [33], the fireproof effectiveness of coatings for steel structures has been estimated before and after climatic tests for working conditions Z2. The service life of intumescent coating (APP/PF/melamine/EBA copolymer/titan oxide with MMT admixture) tested by ETAG 018-2 method has been established to reach 15 years that is 3 years longer as compared with the basic coating.

Antimicrobial properties

The nanoclays used for fireproof coatings also enhance antimicrobial properties of fireproof means by adding MMT nanocomposites with biocide substances [34]. The application of this product has led to raising bioresistance of *EVAFARB* antimicrobial paint having a low flammability (*Spets-materialy* RPC, Kyiv). MMT nanocomposite containing nanosilver ions and polyhexamethyleneguanidine cations imparts to *EVAFARB* a wide range of biocide action: bactericide, virulicide, fungicide, sporicide, and algicide effects and meets the requirements of European Directive on biocide products [35]. Some data on antimicrobial properties of *EVAFARB* coating obtained at the Gromashevskiy Institute for Epidemiology and Infection Diseases of the AMS of Ukraine pursuant to national standards and methods [36, 37] are given in Table below

Due to its good antimicrobial properties, especially, with respect to tuberculosis bacillus, *EVAFARB*

paint is used as a disinfectant for emergency exits in public places, medical and child care establishments, enterprises of food industry and agriculture, in prisons and barracks.

We publish information on several R&D accomplishments of IPOCC in the sphere of reactive fire protection that have been commercialized. The experience of IPOCC researchers in the field of fire protection has been used for drafting national fire safety standards [38, 39]. They are among the members of working groups of technical boards of the Ministry of Regions and Standardization (TK25 Fire Safety and Fire Fighting Facilities, TK304 Protection of Buildings and Structures, and TK-315 Fire and Anthropogenic Safety Systems of Buildings and Structures) and founders of international NGO, *Fire Safety and Audit* Association. The R&D results have been published in monograph studies and guidelines [10, 40] on fire protection of structures.

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ХІМІЧНІ РІШЕННЯ ПРОБЛЕМ ВОГНЕЗАХИСТУ

Розглянуто сучасні підходи до створення вогнезахисних покриттів шляхом модифікації інтумесцентних систем наноматеріалами з вивченням механізму хімічних

перетворень в умовах впливу високих температур. Проведено систематичне дослідження взаємодій компонентів інтумесцентної суміші поліфосфатного типу, знайдено чіткі кореляції між напрямками хімічних процесів та вогнезахисними властивостями інтумесцентного покриття. Запропоновано дієві способи одночасного підвищення вогнезахисної ефективності та експлуатаційних характеристик інтумесцентних покриттів у частині терміну служби, стійкості до впливу факторів навколишнього середовища та біоуражень.

Результати фундаментальних досліджень дозволили розробити нові рецептури вогнезахисних сумішей, властивості яких були підтверджені випробуваннями згідно з діючими стандартизованими методиками та впроваджені у виробництво.

Ключові слова: інтумесцентна система, вогнезахисна ефективність, термін служби, наноматеріали, монтморилоніт, біоциди.

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ХИМИЧЕСКИЕ РЕШЕНИЯ ПРОБЛЕМ ОГНЕЗАЩИТЫ

Рассмотрены современные подходы к созданию огнезащитных покрытий путем модификации интумесцентных систем наноматериалами с изучением механизма химических превращений в условиях воздействия высоких температур. Проведено систематическое исследование взаимодействий компонентов интумесцентной смеси полифосфатного типа, найдены четкие корреляции между направлениями химических процессов и огнезащитными свойствами интумесцентного покрытия. Предложены действенные способы одновременного повышения огнезащитной эффективности и эксплуатационных характеристик интумесцентных покрытий в части срока службы, устойчивости к воздействию факторов окружающей среды и биопоражения. Результаты фундаментальных исследований позволили разработать новые рецептуры огнезащитных составов, свойства которых были подтверждены испытаниями в соответствии с действующими стандартизованными методиками, и внедрить их в производство.

Ключевые слова: интумесцентная система, огнезащитная эффективность, срок службы, наноматериалы, монтмориллонит, биоциды.

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