

## COATINGS WITH INTUMESCENT FILLER BASED ON ALKALI ACTIVATED GLASS

Adrian Ionut NICOARA<sup>1</sup>, Nicoleta CIRSTEA<sup>2</sup>, Cristian BOSCORNEA<sup>3</sup>,  
Alina BADANOIU<sup>4\*</sup>

*Intumescent coatings are used on steel structures in order to prevent the overheating during a fire event, which could lead to structural damage of construction.*

*This paper presents some preliminary results regarding the fire behavior of coatings based on acrylic binder or commercial waterborne paint with intumescent filler i.e. alkali activated glass waste (AABSIP). The paints with intumescent filler were applied to a steel substrate. The coating based on acrylic binder and higher dosages of AABSIP filler has a good behavior when exposed to direct flame i.e. reduces the temperature of substrate.*

**Keywords:** coatings, intumescent filler, waste glass, alkali activated materials

### 1. Introduction

Fire represents a serious problem which can cause human deaths or injuries or important property damage. There are many fire protection methods (active and passive) and materials which can be used to reduce the effect of fire in buildings [1–4].

Steel is widely used worldwide to build modern, attractive and durable structures. Fires can cause considerable damage to metal structures due to loss of strength and rigidity of steel at high temperatures (over 500°C). Fire protection is therefore often required to protect metal structures [5].

There are many types of materials used for the protection of steel structures against fires, for example, plating plates, sprayable coatings and intumescent coatings [1,5,6]. Intumescent coatings swell when exposed to high temperature or in contact with the flame, thereby causing a volume increase and a decrease in density [7].

The regular intumescent polymer-based coatings contain an inorganic acid, a carbon source (char former) and a blowing agent [8,9]. In this case the intumescence process is determined by the char formation (at the surface of substrate) and its foaming. The char layer represents a physical barrier reducing the heat transfer, therefore preventing the ignition and burning of substrate [8,9].

---

<sup>1</sup> Assistant, Dept. SIMONA, University POLITEHNICA of Bucharest, Romania

<sup>2</sup> MSc student., Dept. SIMONA, University POLITEHNICA of Bucharest, Romania

<sup>3</sup> Associated Professor, Dept. BSP, University POLITEHNICA of Bucharest, Romania

<sup>4</sup> Professor, Dept. SIMONA, University POLITEHNICA of Bucharest, Romania, \*Corresponding author: e-mail: alina.badanoiu@upb.ro

Previous research performed in our group pointed out the possibility to obtain inorganic intumescent materials based on alkali activated glass waste with various additions [7,10].

This paper explores the possibility to obtain intumescent coatings, by mixing of a polymeric binder/commercial waterborne paint with an intumescent filler resulted by the milling of alkaline activated glass waste.

## 2. Materials and methods

### 2.1. Materials

Waste glass powder (WGP) was obtained by fine grinding of soda-lime glass cullet (from glass bottles with various colors). The Blaine specific surface area of the waste glass powder was 3000 cm<sup>2</sup>/g. Borax decahydrate (Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·10H<sub>2</sub>O), sodium hydroxide, used as alkali activators, were chemical reagent grade.

The intumescent material (further referred as AABSIP) was obtained by dry mixing the solid component (WGP) with the alkaline activator solution (obtained by the solubilization of sodium hydroxide and borax decahydrate in water). The mixture was homogenized for 5 minutes using a mechanical stirrer (800 rpm). After hardening, the AABSIP was stored in air at 20°C for 4 weeks and then milled in order to obtain a fine powder.

AABSIP powder was dispersed in two acrylic dispersions/binders (Acronal LR 9014 and Acronal S562) - paint 1 and 2 and a commercial waterborne paint – paint 3 - see table 1.

*Table 1.*

**The composition of the studied paints**

Paint	AABSIP %	Water %	Acrylic binder %	Additives* %	CaCO <sub>3</sub> %
Paint 1	8.26	33.05	51.25	3.31	4.13
Paint 2	15.45	30.88	47.87	5.8	-
Paint 3	5.26	47.37	47.37 (commercial waterborne paint)		

\*) Antifoaming, film forming, dispersing, coalescing, thickening agents.

These paints were applied on steel plates (50x50x2 mm) in several layers (2, 3 and 4 – layers) with similar thickness (~0.1 mm). After the application the coated substrates were stored in air for 21 days until the fire test was performed.

## 2.2. Methods

The new prepared paints were characterized by specific tests [11].

The density was determined by the pycnometer method, according to the standard DIN EN ISO 2811-1:2011 [12].

The solids content (amount of non-volatile materials) was calculated according to the standard ISO 3251:2008 [13]:

$$\text{Solids content} = (m_2/m_1) * 100 (\%) \quad (1)$$

where:

$m_1$  = paint mass before thermal treatment (g);

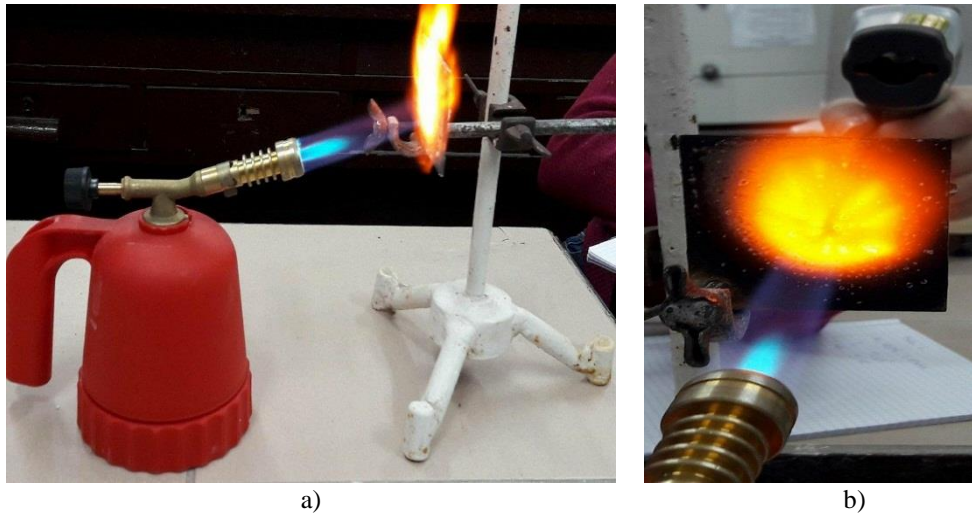
$m_2$  = paint mass after thermal treatment at 135°C for 30 min (g).

The coating gloss, opacity and adhesion to the substrate were determined by applying layers of paint to the Leneta type cards and matching with similar standards. The gloss was measured at 20°, 60° and 85° by using a Micro-TRI-gloss from BYK according to the standard ISO 2813:2014 [14]. At least 4 locations on a coating surface were measured to obtain an average value. The opacity was measured with portable spectrophotometer Spectro-guide 45/0 (BYK) by standard test method established by ASTM D2805 - 11(2018) [15]. The adhesion was measured by the cross-cut test, according to ISO 2409:2013 [16].

The pH of paints was determined with portable pH-meter AL10pH (Aqualitic).

The micrographs of coatings surfaces were obtained with a digital pocket microscope DPM 300 (BYK).

The fire behavior was determined by contacting the steel plate with a propane flame (average temperature of 1000°C). The steel plates were mounted in a stand (Fig.1) in this way that the propane flame is in contact with the coated face of the steel plate. The distance between the burner and the plate was kept constant for all determinations. The "cold" face (opposite to the coated face on which the flame was applied) temperature was measured with a pyrometer.



a) b)  
Fig.1. Fire test panel (a), detail (b)

### 3. Results and discussion

According to the literature data, the exposure of steel to temperatures above  $500^{\circ}\text{C}$  causes a significant decrease in mechanical strength, thus affecting the strengths of building structures in the event of a fire [5,9].

The temperature of the “cold face” of steel plate increases rapidly and the critical temperature ( $500^{\circ}\text{C}$ ) is achieved after 1 minute.

The presence of coatings on the surface of steel plate causes, as expected, a decrease of “cold” face temperature (Figs. 2 and 3).

The increase of coating thickness (by successive deposition of multiple layers) causes a reduction of “cold” face temperature in the case of coatings based on acrylic binders (paints 1 and 2); however, the temperature rises rapidly within the first minutes from the moment of contact with flame and exceeds the critical value of  $500^{\circ}\text{C}$ .

From the two graph presented in Figs.2 and 3, it can be seen that the steel plate coated with acrylic resin with a higher amount of intumescent material (paint 2) reaches lower temperatures of “cold” face than those of the steel plate with paint 1 coating (with lower amount of intumescent filler). The increase of the coating thickens (3 layers) in the case of paint 2 (with a higher amount of intumescent filler), improves the fire behavior i.e. the critical temperature of “cold face” is achieved after a longer time compared with the specimens coated with 2 layers.

In the case of paint 3 (Fig. 4), from the first minutes of contact with the propane flame, the “cold” face temperature abruptly rises above  $500^{\circ}\text{C}$ , which shows that this coating is not suitable for protecting the steel elements. This fire

behavior could be due to the lower amount of intumescent filler dispersed in this paint as compared with the paints based on acrylic dispersions.

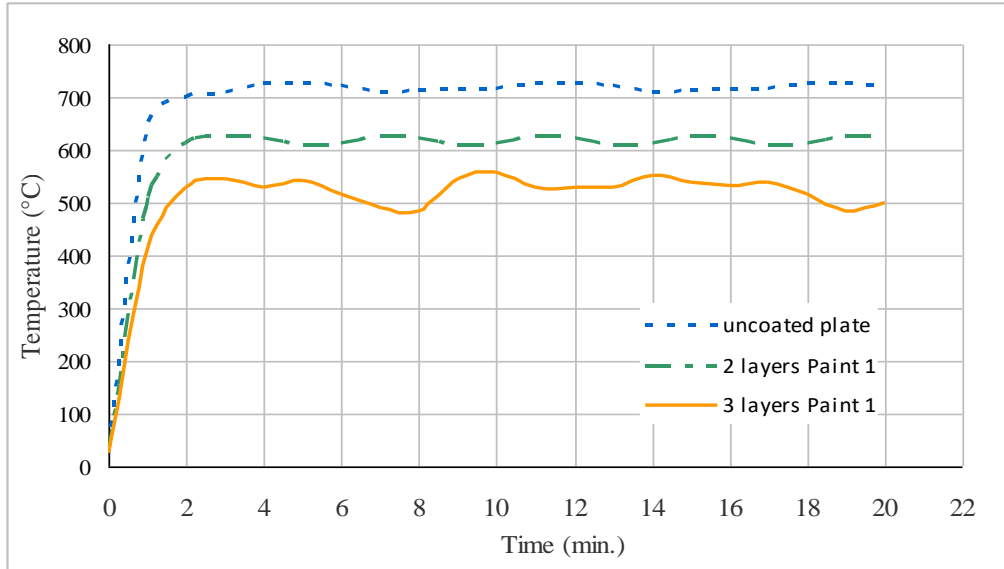


Fig. 2. "Cold" face temperature vs. time for the steel plate without / with paint 1

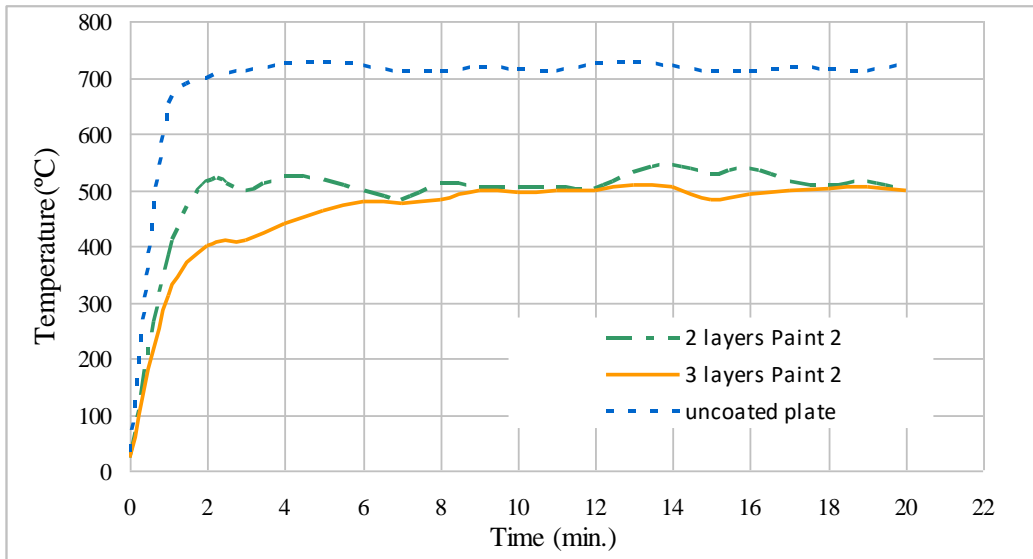


Fig. 3. "Cold" face temperature vs. time for the steel plate without / with paint 2

Figs. 5 and 6 show the visual aspect of the metal plates coated with paint 1 and paint 2 before and after the contact with propane flame.

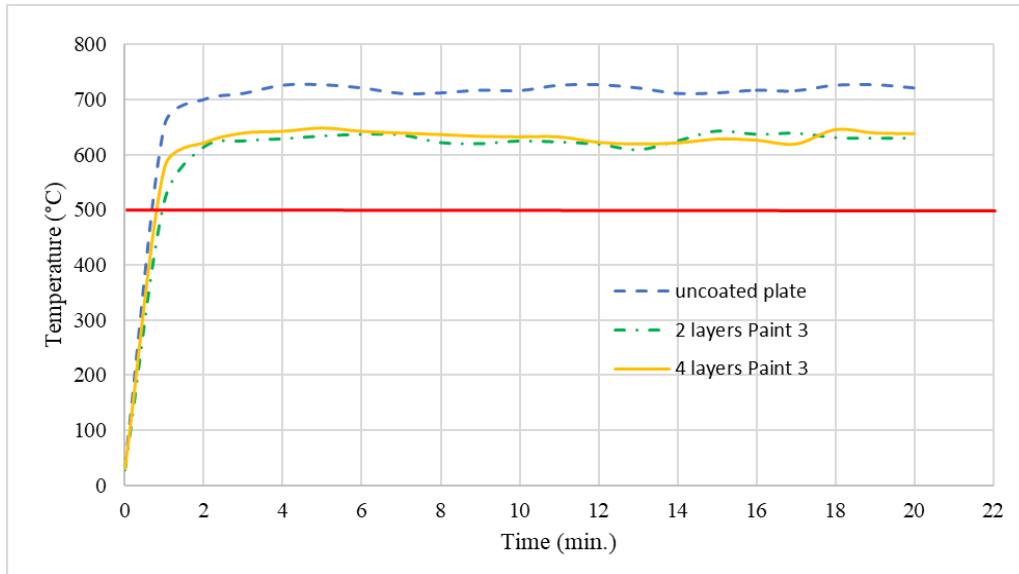


Fig. 4. "Cold" face temperature vs. time for the metal plate without / with paint 3

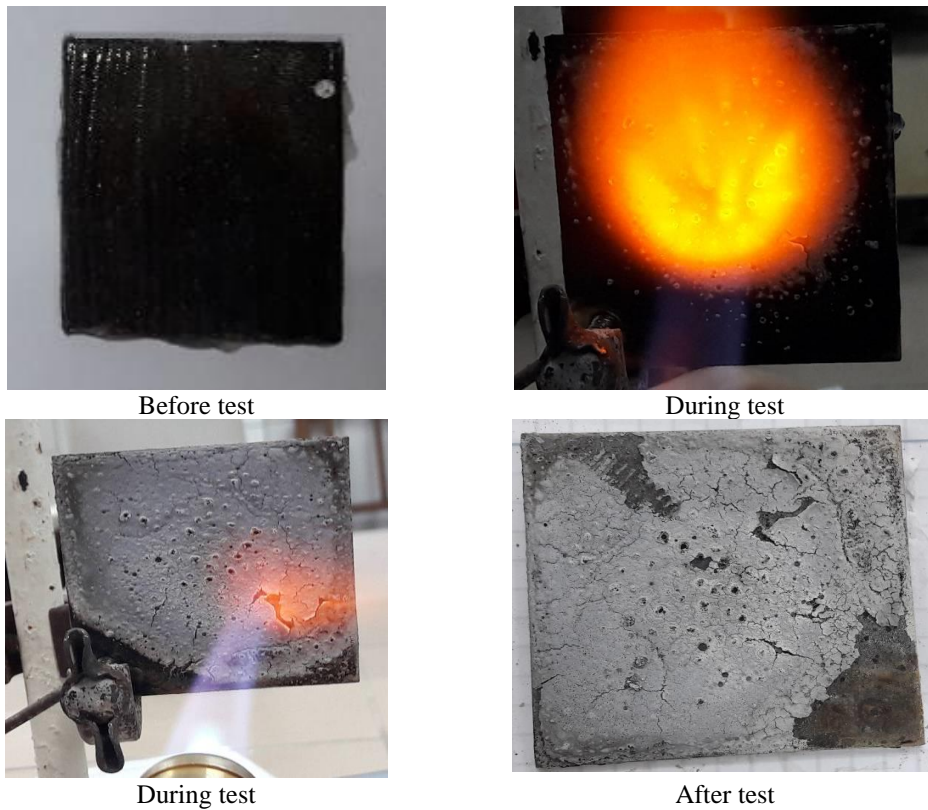
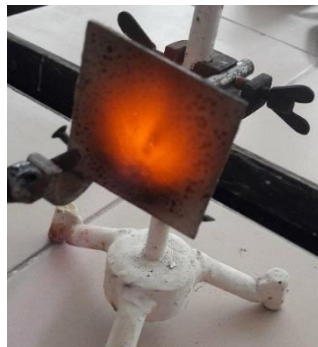


Fig. 5. Visual aspect of the steel plate coated with paint 1 (3 layers) before, during and after contact with propane flame

In the case of the steel plate coated with paint 1 (Fig.5), the contact with the propane flame produces the swelling of the coating (intumescent process), after which the partial cracking and exfoliation occurs. The intumescent process is due to the water loss from the hydrates formed by the alkaline activation of WGP [7].

A similar fire behavior is observed in the case of metal plate coated with paint 2 (Fig. 6). At the surface of the steel plate one can assess some zones (with various sizes) covered with a glassy like layer.

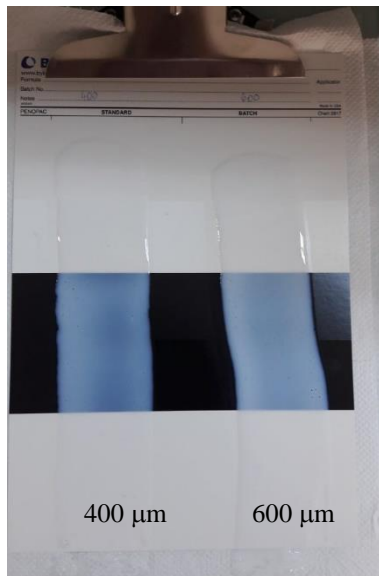


During test



After test

Fig. 6. Visual aspect of the steel plate coated with paint 2 (3 layers) during and after contact with propane flame



a)



b)

Fig. 7. Films with various thicknesses: a) coating with lower amount of AABSIP (paint 1); b) coating with a higher amount of AABSIP (paint 2)

For the paints 1 and 2, which showed a promising fire behavior, were assessed some specific properties; therefore, films with two thicknesses, i.e. of 400  $\mu\text{m}$  and 600  $\mu\text{m}$ , were applied on the Laneta cards using a film casting knife with width of 5 cm. One can see that more coverage is achieved with paint 1, for both thicknesses (fig.7).

In table 2, there are presented some of the specific properties determined for the studied paints/coatings. The paints have an alkaline pH due to the inclusion of the AABSIP filler, which was obtained by the alkaline activation of the waste glass powder [7].

Table 2.

**Specific properties of paints/coatings**

Paint	pH	Density (g/cm <sup>3</sup> ) DIN EN ISO 2811- 1:2011	Solids content (%) ISO 3251:2008	Adhesion (ISO 2409:2013)	Gloss (GU)			Opacity (%) ASTM D2805 - 11(2018)
					20°	60°	85°	
Paint 1	10.28	1.13	40	Class 0 - completely smooth cuts without delamination	3.6	19.6	21.9	12.14
Paint 2	10.16	n.d.	44	Class 1 - there are no fragments at intersection points; the delaminated area is about 5%	1.4	3.1	0.9	10.09

The solids content is higher for paint 2 compared to paint 1, consistent with the higher AABSIP dosage.

The micrographs of the surface of dry coatings (Figs. 8 and 9) show an adequate dispersion of AABSIP filler in the polymeric binder. Paint 2 coating contains smaller grains well dispersed in the film (Fig.8). Paint 1 coating contains bigger grains (possible agglomeration) – Fig. 9; this could be due to the presence of limestone filler in this formulation.

The adhesion of the studied paints assessed according to ISO 2409:2013 [16], is good i.e. Class 0 - for Paint 1 and Class 1 - for Paint 2, of the 5 grades of adhesion (where Class 0 is the best and Class 5 - the weakest).

The opacity of the two coatings is comparable. According to the data presented in Table 2, it can be seen that the paint formulation with a lower amount of AABSIP filler (paint 1) is glossier and the paint with a larger quantity of AABSIP filler (paint 2) is opaquer.



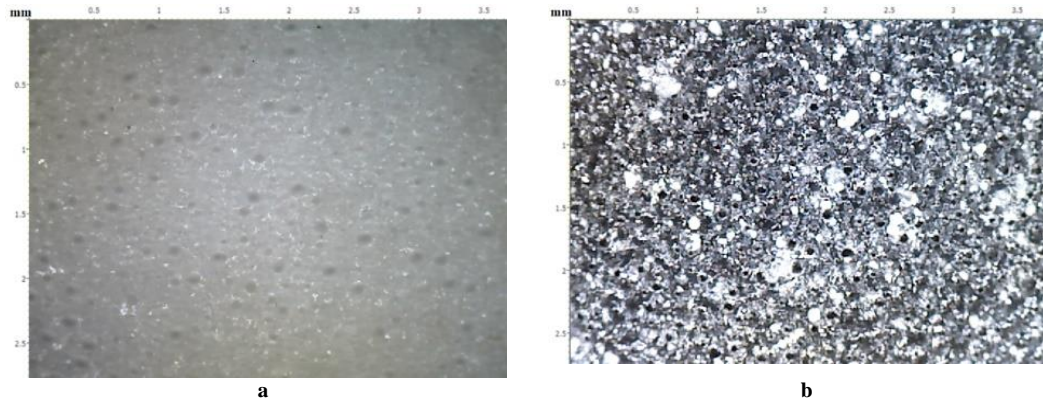


Fig. 8. Micrographs of the surface of paint 2 coating applied on Leneta card: a) white area; b) black area

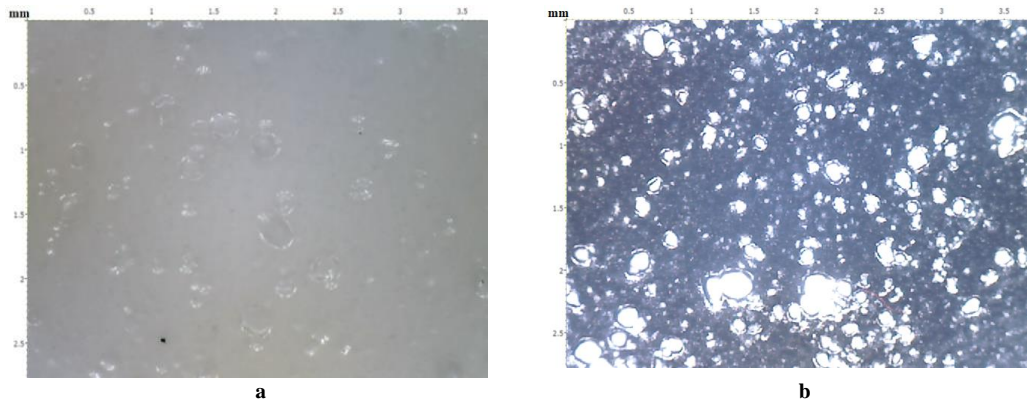


Fig. 9. Micrographs of the surface of paint 1 coating applied on Leneta card: a) white area; b) black area

#### 4. Conclusions

The results obtained in this paper can be summarized as follows:

- The coatings based on acrylic binders with intumescent filler (AABSIP), decreases the temperature of substrate (steel plates), when exposed to fire; as expected, the increase of film thickness (by applying multiple layers) increases the intumescence process (specific for AABSIP materials) and prevent the increase of substrate temperature.

- The steel plates coated with commercial waterborne paint containing AABSIP addition, did not prevent the increase of the temperature of steel substrate over the critical value ( $500^{\circ}\text{C}$ ). For the protection of metal structures, the coatings with a higher AABSIP content have a better behavior to fire. It is expected that the increase of intumescent filler (AABSIP) content in the paint formulation to improve the thermal insulation ability of this coating; therefore, more studies should be performed.

- Preliminary tests on the properties of films obtained with prepared paints have shown adequate results in adhesion and compatibility binder – filler (AABSIP), but improvements in hiding power are required.

- The results presented in this paper are promising, but specific tests regarding the fire behavior of this type of coatings should be also performed.

## REFERENCES

- [1] *Han Z, Fina A, Malucelli G, Camino G.* Testing fire protective properties of intumescent coatings by in-line temperature measurements on a cone calorimeter. *Prog Org Coatings* 2010;**69**:475–80.
- [2] *Zhang HY, Kodur V, Qi SL, Cao L, Wu B.* Development of metakaolin-fly ash based geopolymers for fire resistance applications. *Constr Build Mater* 2014;**55**.
- [3] *Villaquiran-Caicedo MA, de Gutierrez RM, Sulekar S, Davis C, Nino JC.* Thermal properties of novel binary geopolymers based on metakaolin and alternative silica sources. *Appl Clay Sci* 2015;118:276–82.
- [4] *Lyon R. E., Balaguru P. N., Foden A., Sorathia U. DJ and DM.* Fire-resistant aluminosilicate composites. *Fire Mater* 1997;**21**:67–73.
- [5] *Cirpici BK, Wang YC, Rogers B.* Assessment of the thermal conductivity of intumescent coatings in fire. *Fire Saf J* 2016;81:74–84. doi:10.1016/j.firesaf.2016.01.011.
- [6] *Erdoğan ST.* Inexpensive intumescent alkali-activated natural pozzolan pastes. *J Eur Ceram Soc* 2015;**35**:2663–70..
- [7] *Al Saadi THA, Badanoiu AI, Nicoara AI, Stoleriu S, Voicu G.* Synthesis and properties of alkali activated borosilicate inorganic polymers based on waste glass. *Constr Build Mater* 2017;**136**:298–306. doi:10.1016/j.conbuildmat.2017.01.026.
- [8] *Alongi J, Han Z, Bourbigot S,* Intumescence: Tradition versus novelty. A comprehensive review, *Progress in Polymer Science* 2015; **51**; 28–73.
- [9] *Puri RG, Khanna AS,* Effect of cenospheres on the char formation and fire protective performance of water-based intumescent coatings on structural steel, *Progress in Organic Coatings* 2016; **92**; 8–15
- [10] *Badanoiu A, Al Saadi THA, Stoleriu S, Voicu G ,* Preparation and characterization of foamed geopolymers from waste glass and red mud, *Construction and Building Materials* 2015; **84**: 284-293
- [11] *Koleske J.V.* (Editor), *Paint and Coating Testing Manual - Fifteenth Edition of the Gardner-Sward Handbook*, ASTM International, 2012.
- [12] ISO 2811-1:2011 - Paints and varnishes -- Determination of density - Part 1: Pyknometer method
- [13] ISO 3251:2008 - Paints, varnishes and plastics -- Determination of non-volatile-matter content
- [14] ISO 2813:2014 - Paints and varnishes -- Determination of gloss value at 20 degrees, 60 degrees and 85 degrees
- [15] ASTM D2805 - 11(2018) - Standard Test Method for Hiding Power of Paints by Reflectometry
- [16] ISO 2409:2013 - Paints and varnishes— Cross-cut test