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Research on the fire retardant coating for steel structure protection based on Epikote 240 epoxy with the presence of environmentally friendly additive: nanoclayI30.E

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Abstract- The research content is to develop nanocomposite coatings to provide fire resistance to structural steel substrate.This study presents the results of the different formulations developed to study the effects of nanoclay on coating expansion and thermal shielding during the fire test. The anti-fire effect of nanocomposite coating is improved by 5 wt.% dispersed nanoclay particles. The coatings are characterized by using scanningelectron microscope (SEM), Flame-retardant evaluation methods: LOI, UL 94 rating. Effect of additional content of nanoclay on combustion efficiency is studied by flame retardant test and measurement of limited oxygen index and effective thermal conductivity.

Keywords: Flame retardant, nanoclay, nanocomposites, limiting oxygen index.

I. INTRODUCTION

The devastating effects of fires lead to disasters causing loss of life and property, and scientists have researched to develop new materials and new methods to protect people and property from the destructive effect of fire. Previous studies have shown that steel structures can maintain strength below or up to a maximum temperature of 500^0C; structure breaks down at higher temperature. Therefore, to ensure the safety of steel structures, the temperature of the steel structure must be kept below 500°C in the event of a fire incident [1-3]. To address this need, a variety of flame retardants have been developed and marketed; These include phosphorus and halogen based flame retardants ... Flame retardants used as additives to make composite coatings to protect steel structures have been studied such as: Ammonium polyphosphate (APP), kaolin clay, Boric. acid ...The outer coating is designed to perform under extreme conditions to maintain steel integrity for between 1 and 3 hours when the ambient temperature exceeds 1100^0C [4]. This time period is considered sufficient for staff evacuation and fire control. The active coating effectively protects the surface against the rapid rise in temperature, thus maintaining the structural integrity of the steel surface. The components used in the coating to protect steel structures: Ammonium polyphosphate (APP), kaolin clay, Boric acid, Pentaerythritol (PER) and Melamine [5-7]. Additive nanoclay has been studied as one of the environmentally friendly flame retardant additives, with only 2% by weight of nanoclay, the fire resistance is significantly improved [8-10].
In this work, research on the effects of nanoclay additive on the fire resistance of the coating on the system: epoxy resin Epiokte 240, polyamide amine, Ammonium polyphosphate (APP), Boric acid, Melamine. The objective of this study is to investigate the synergistic effect of nanoclayI.30E ith other flame retardant ingredients in improving the flame resistance of the paint film on Epikote 240 epoxy film forming agent.

II. MATERIALS AND METHODS

2.1. Materials

- Curing agent: LUCKAMIDE Polyamine N-153-IM-65 (China).
- Nanoclay I.30E (Nanocor USA) is a surface-changing montmorillonite (MMT) mineral that will disperse into nano-particles in epoxy resin systems.
- Boric acid (BA), Ammonium polyphosphate (APP), Melamine were purchased from Sigma.
- Epoxy Epikote 240 (EP) resin of Shell Chemicals(USA):

Property	Test method	Unit	Value
Epoxy group content	SMS 2026	mmol/kg	5100 - 5400
Epoxy molar mass*		g	185 - 196
Viscosity at 25°C	ASTM D445	Pa.s**	0.7 - 1.1
Colour	ASTM D1209	Pt-Co	200 max
Density at 25°C	ASTM D4052	kg/L	1.12

* no. of grams of resin containing 1g-equivalent of epoxide. (Weight per equivalent, WPE, is an alternative term).
** 1 Pa.s = 10 poise

2.2. Methods
2.2.1. Sample preparation
All intumescent ingredients were mixed with their respective weight percentage as stated in the Table 1. Technological condition is stirring speed 1500 rpm, stirring time is 90 minutes. A structural steel plate measuring 100 cm^2 was used as the substrate.

Table 1. Weight Percentage of the Coating

No.	APP	Melamine (Mel)	PEG	Boric acid	Epoxy	Hardener	Nanoclay
S1	12.39	5.87	5.95	12.25	42.76	20.29	1
S2	12.39	5.87	5.95	12.25	41.20	19.34	3
S3	*12.39*	*5.87*	*5.95*	*12.25*	*40.15*	*18.39*	*5*

2.2.2. Characterization and testing
Limiting Oxygen Index (LOI) according to JIS K720 standard (Japan): the sample bars used for the test were 150 x 6.5 x 3 mm^3.The Horizontal Burning tests (UL-94HB): Standard bar specimens are to be 125 ±5 mm long by 13.0 ±0.5 mm wide, and provided in the minimum thickness and 3.0 (-0.0 +0.2) mm thick. (ASTM D635-12).The UL 94 flame retardant and oxygen limit tests are conducted at the Polymer Materials Research Center - Hanoi University of Technology, Vietnam.The morphology of the samples was carried out by scanningelectron microscope (SEM, S4800, Japan).

III. Results and Discussion

3.1. Mechanical properties

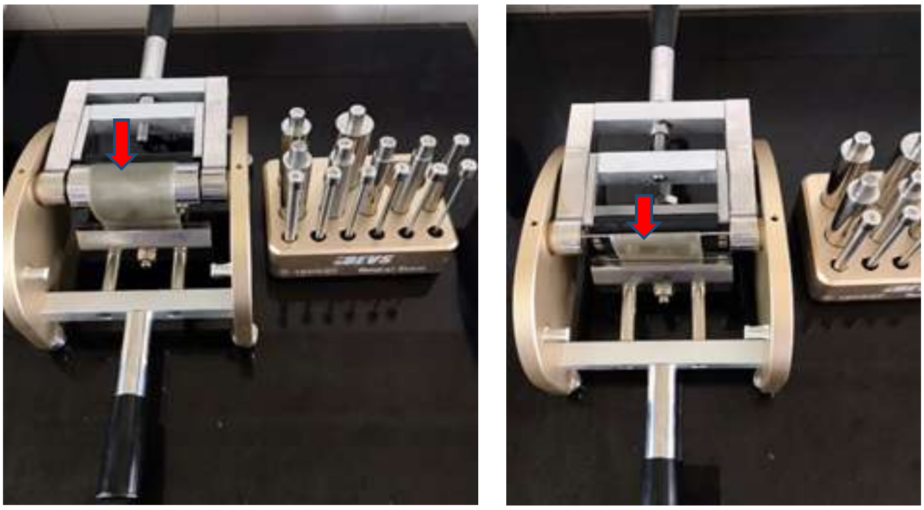


Figure 1. Measure the desired durability of the paint film

From Figure 1, the flexural strength of the paint film is passed by a measuring device (BEVS 1603) with a diameter of 32 mm - 2 mm. After testing through all measuring axes of 3 coating films (S1, S2 and S3) at the nanoclay ratios, the paint film achieves a tensile strength of min (2mm). The results of flexural strength measurement show that the paint film has high plasticity without breaking or cracking at many measurement results and is suitable for the construction of the project.

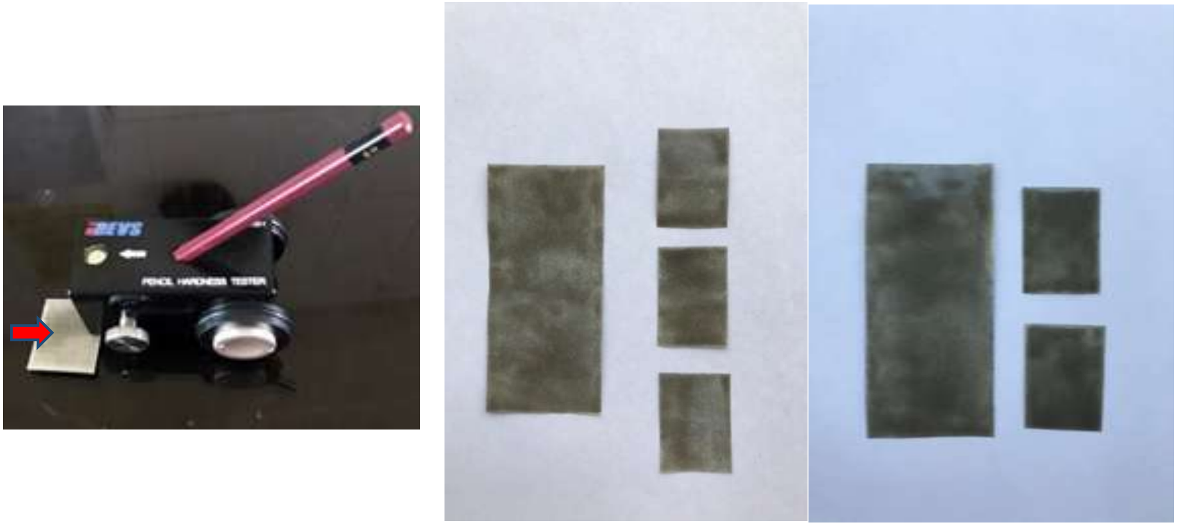


Figure 2. Measure the scratch of the paint film on the BEVS 1301 device

Table 1. Measurement of adhesion with a pencil

No.	6B	5B	4B	3B	2B	B	HB	F	H	2H	3H	4H	5H	6H
S1	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓		
S2	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	
S3	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	

The table 1, figure 2 scratching results of the nanoclay ratios in the paint film show that: at a high nanoclay ratio for good hardness because nanoclay is a compatible support, creating uniformity and cohesion of substances in the paint film.

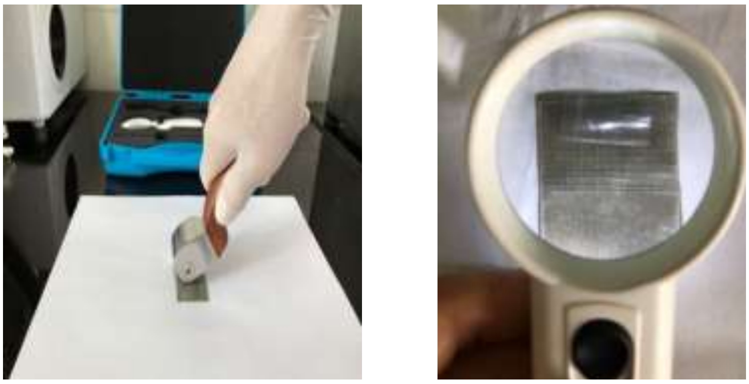


Figure 3. Measurement of adhesion on the BEVS 2202 device

See Fig 3, through the results of measuring the standard plates of the different nanoclay ratios according to the criteria for evaluating the adhesion of the paint film, the cut is completely smooth, with no inflection point (point 1). S3 coating membrane, gives the cut completely smooth, with no inflection point.

3.2. Structural morphology of the coating material

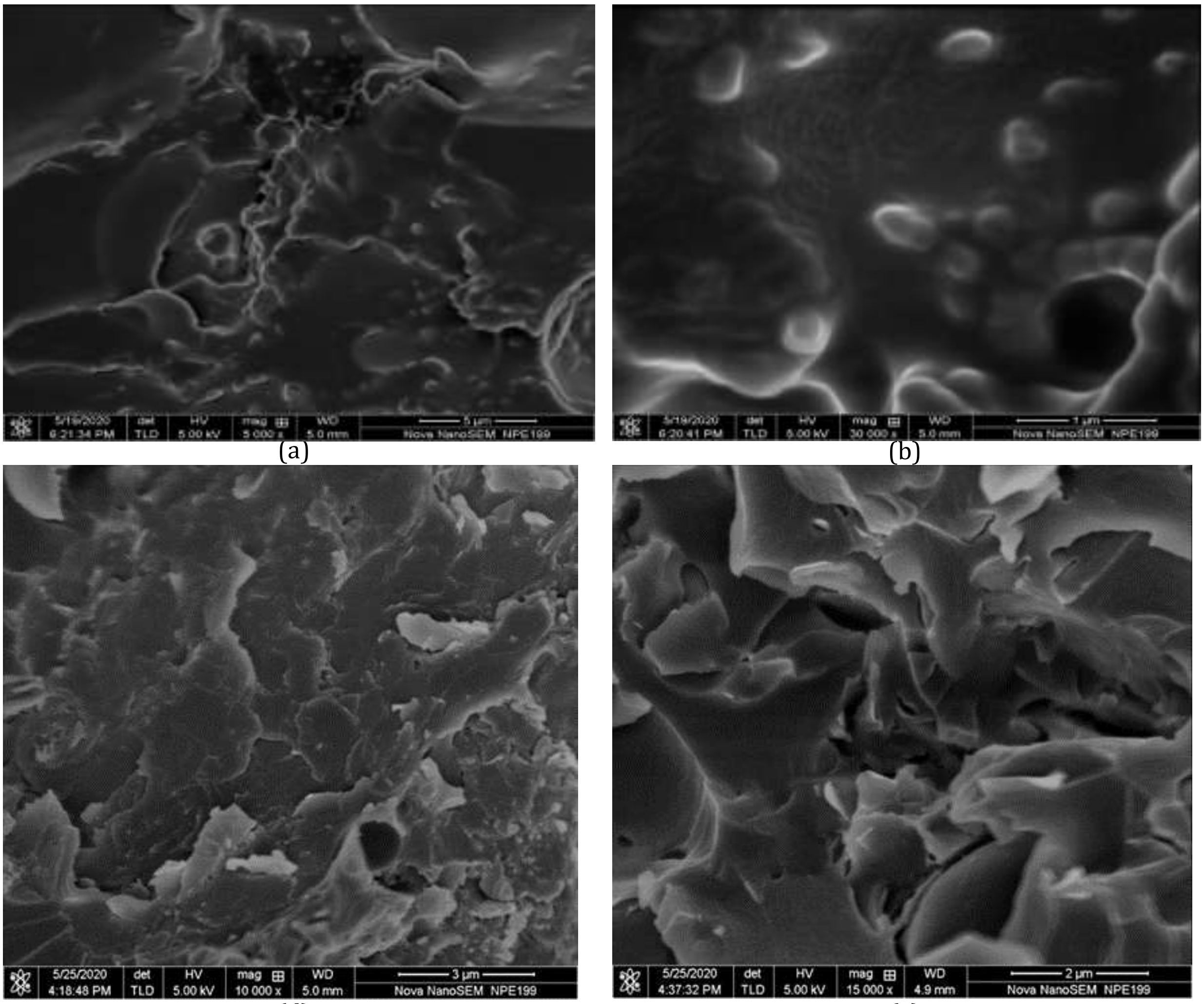


Figure 4. SEM image of coating material:*(a) – Epoxy/APP/MEL/PEG/Boric acid; (b) – 1 wt.% nanoclay/ Epoxy/APP/MEL/PEG/Boric acid; (c)- 3 wt.% nanoclay/ Epoxy/APP/MEL/PEG/Boric acid; (d)- 5 wt.% nanoclay/ Epoxy/APP/MEL/PEG/Boric acid*

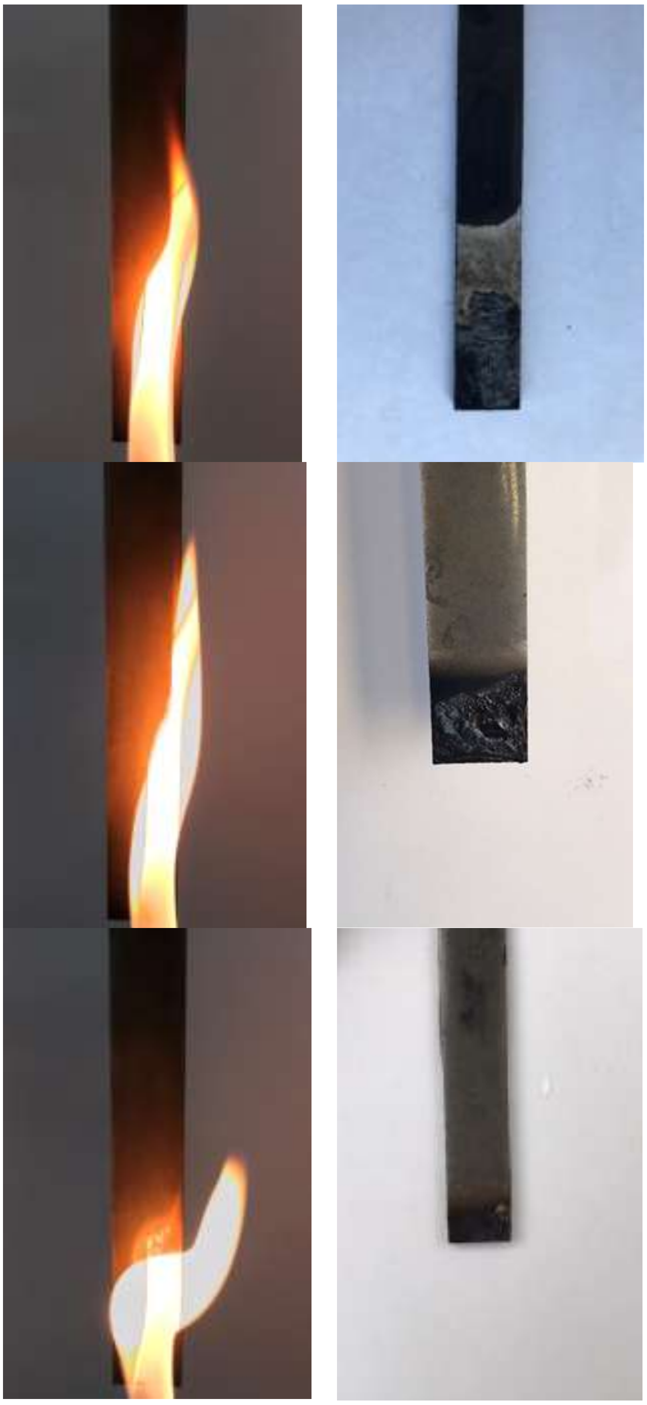
Figure 4 shows the uniformity of nanoclay particles in the flame retardant coating system. The uniformity of the nanoclay particles helps to increase the stability of the paint film, in addition it also prevents oxygen from contacting the substrate so the material does not produce a flame, reducing the ability to spread the fire. significantly when a fire occurs.

The addition of organic nanoclay can significantly aid in fire resistance by facilitating the formation of a carbon carbon layer. The nanoscale dispersed in the clay mineral thin layer, or interwoven in the polymer, all enhances the formation of coal upon burning. After pyrolysis the nanocomposites form coal with a multi-layered structural carbon silicate. The clay mineral layers first form an alternating structure and then convert into a multi-layered carbon silicate structure. Carbon carbon layers accumulate on the surface during combustion and segregate materials. The advantages of organic clay in retarding fire are very diverse: it does not generate harmful gases (the flame has a green flame retardant) and no discoloration.

By using a small amount of clay mineral, it is possible to significantly reduce the number of conventional flame retardant additives, giving the material optimal flame retardant properties. Conventional flame retardants always have some negative effect on the mechanical properties of the polymer substrate, so by using nanoclay, such a bad impact can be minimized and correcte.

3.3. Flame-Retardant Properties of of the coating

After burning the material, the results obtained in Fig 5 show that the ignition of the paint film is quite poor in sample S2 and sample S3. The paint film protects the steel surface when it burns quite well, after the fire test. Under these conditions, we remove the protective paint film that has burned to check the steel surface for expected results. The steel surface remains shiny and does not appear deformed or charred. At three test pieces, sample 3 gave the best results and met the V-0 requirements of UL- 94V standards suitable for application for flame retardant coating.

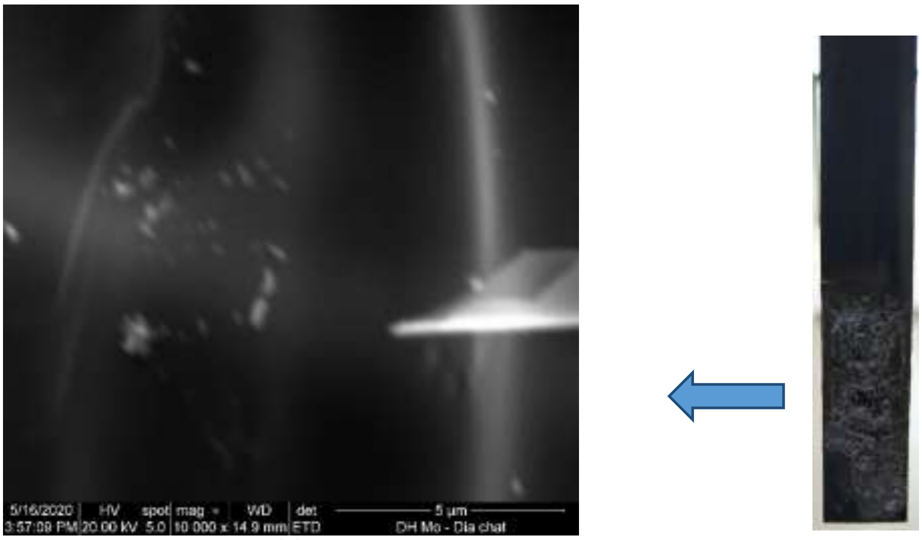


(a). Coating material with 1 wt.%
Burning stops within 60 seconds after two applications of ten seconds each of a flame to a test bar.

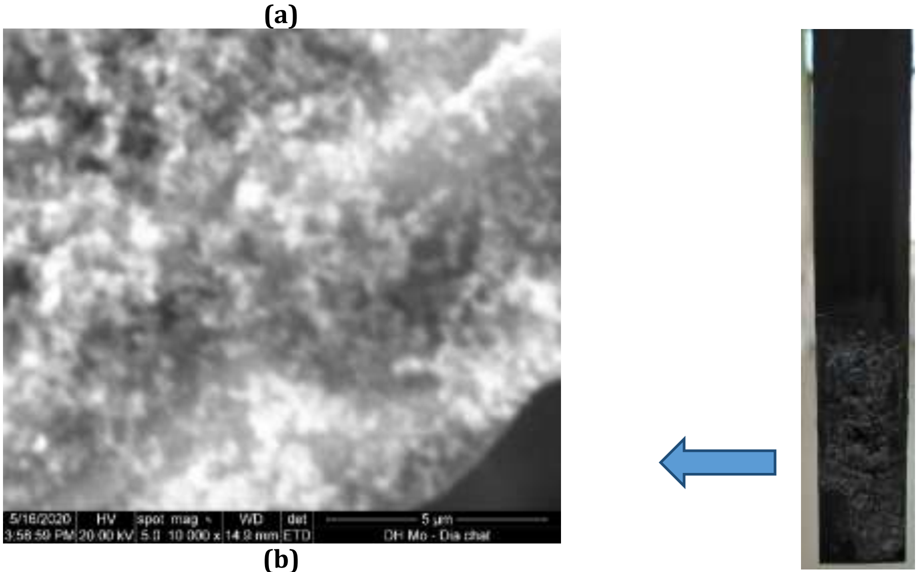
(b). Coating material with 3 wt.%
Burning stops within 60 seconds after two applications of ten seconds each of a flame to a test bar.

(c). Coating material with 5 wt.%
Burning stops within 10 seconds after two applications of ten seconds each of a flame to a test bar. t_1 = 2.5 s and t_2 = 3.7 s. (belongs to the group of highest level of fire-proof material of 94V-0).

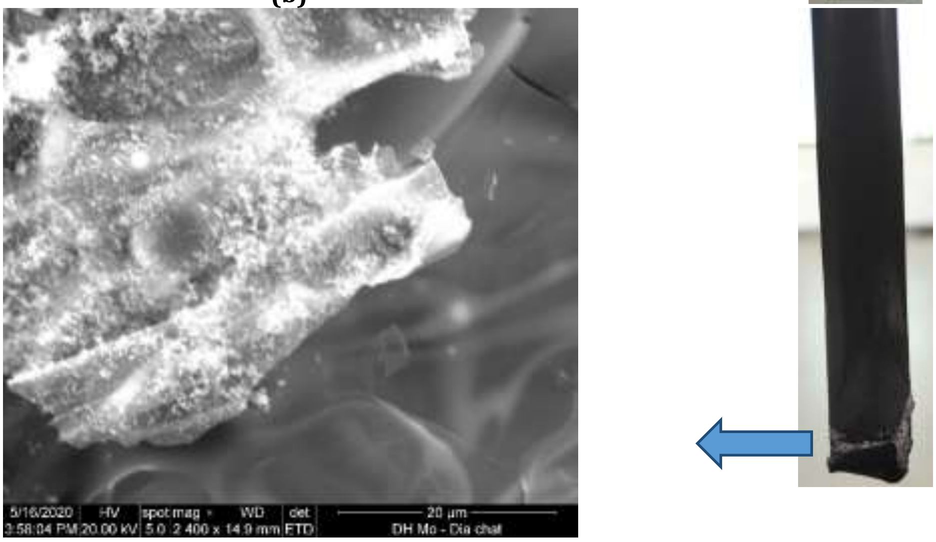
Figure 5. Image of fire retardation according to UL-94V method



(a)



(b)



(c)

Figure 6. Char morphology of intumescent coating formulations:
(a): 1 wt.% nanoclay; (b): 3 wt.% nanoclay; (c): 5 wt.% nanoclay

See Figure 6, thick coal layers reduce heat penetration in the steel substrate. Figure 2 (b, c), The inner surfaces of the coal layer are thick and porous because of air bubbles and aggregates, formed by CO_2 and

NH_3 emissions during fire testing. When the membrane was burned, N_2, NH_3 and CO_2 were produced, which bubbled through the viscous liquid and expanded the membrane, increasing the ability to protect against the burning agent.
This explains the synergies of APP, boric acid, PEG and melamine with epoxy resin in the bulging field film formation. The effect of the swelling coating is to form a porous coal layer on the steel surface. This coal layer is a physical barrier against heat exchange to the surface of the material, acting as a flame retardant layer providing insulation and thus, protecting the steel structure.

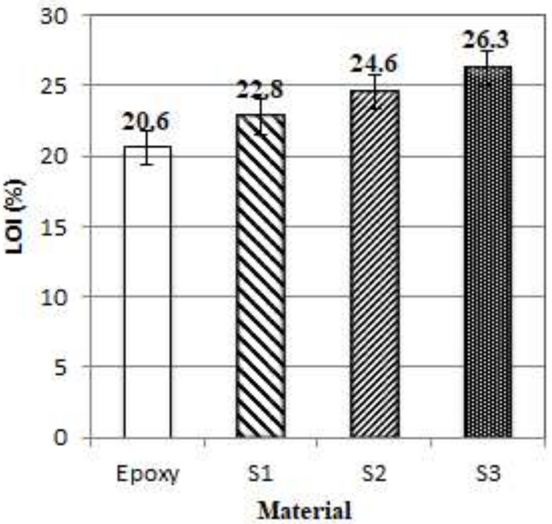


Figure 7. Results for flammability tests (reaction to small flame) oxygen index (OI)

The flame retardant properties of the coating are checked by the method of measuring the oxygen limit, the results are shown in the figure 7. The addition of different nanoclay materials leads to the change in the limited oxygen index. At% nanoclay mass, the oxygen index was limited to 26.3%, belonging to the flame retardant material.

IV. CONCLUSION

Coating material on epoxy resin Epikote 240 and additives: Ammonium polyphosphate (APP), Boric acid, Pentaerythritol (PER) and Melamine are combined with 1, 3, 5% by weight of nanoclay respectively. The results of the study show that reinforcement of the nanoclay creates a protective barrier on the surface of the insulator that minimizes heat flow to the substrate. As a result, it provides a better absorption effect on coating formulations on steel substrates. Additive nanoclay with 5% mass fraction is a reinforcing material that produces a ceramic like barrier protection on the surface of the insulation that reduces heat transfer to the substrate. Coating materials manufactured in the group of flame retardant materials at the highest level UL-94 V0.

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