



Investigation of the Oxygen Index of Intumescent Fire-Protective Polymer Composite Coatings

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ABSTRACT: This article investigates the fire-resistance properties of epoxy resin-based intumescent fire-protective polymer composite coatings using the limiting oxygen index (LOI) method. During the study, various amounts of flame retardants and intumescent-forming chemical additives (pentaerythritol and hexamethylenetetramine) were incorporated into the polymer composite formulation, and their effects on the oxygen index were analyzed. The experimental results showed that increasing the flame retardant content led to a rise in the oxygen index value. When 25% oligomeric flame retardant was added, the oxygen index reached 32%, while the physical and mechanical properties remained at the required level. Based on the obtained experimental data, statistical and mathematical models were developed, and their absolute maximum error values were determined. The research results make it possible to scientifically optimize the composition of intumescent fire-protective polymer composite coatings.

KEYWORDS: fire-resistance, limiting oxygen index, epoxy resin, pentaerythritol, polymer composites, mathematical modeling.

I. INTRODUCTION

In recent years, due to the rapid development of industrial sectors worldwide and the sharp increase in production volumes, significant attention has been paid to the use of various effective means for protecting building structures and materials from fire. In particular, this includes the treatment of metal structures and materials with polymer composites, the development of thermal insulation materials, fire-resistant paints and coatings, and the study of their properties.

Determining the structural behavior of metal constructions in buildings and сооружения under the effects of fire and high temperatures, as well as the scientifically justified selection of fire-resistant polymer composite coatings, is of great importance in organizing emergency rescue operations.

Experimental studies on determining the limiting oxygen index (LOI) of intumescent fire-protective polymer composite coatings were carried out by igniting specially prepared polymer composite samples and calculating the oxygen concentration in an oxygen–nitrogen gas mixture. The oxygen index value was determined based on the measured minimum oxygen concentration required to sustain combustion.

Bitumen, a viscoelastic hydrocarbon material derived from crude oil distillation, is a fundamental component in the construction industry, particularly for road pavements, roofing, and waterproofing applications. Its widespread use stems from its desirable properties, including flexibility, water resistance, and cost-effectiveness. The 60/90 penetration-grade bitumen, commonly employed in road construction, is valued for its moderate viscosity and durability under varying climatic conditions [1-2]. However, a significant limitation of bitumen, including the 60/90 grade, is its inadequate adhesion to aggregates such as sand, gravel, or crushed stone, especially in the presence of moisture or under thermal and mechanical stresses. Poor adhesion results in stripping, where bitumen detaches from aggregates, leading to pavement degradation, such as cracking, raveling, and pothole formation, which compromises the structural integrity and longevity of road surfaces [3].

To mitigate these challenges, researchers have explored various additives to enhance bitumen's adhesion properties. Traditional modifiers, such as styrene-butadiene-styrene (SBS), ethylene-vinyl acetate (EVA), and amine-based adhesion promoters, have been effective in improving adhesion and mechanical performance. However, these additives often involve high costs, complex processing, and environmental concerns due to their synthetic nature and energy-intensive production processes [4]. As the construction industry increasingly



prioritizes sustainability and cost-efficiency, there is a growing demand for eco-friendly, locally sourced, and economically viable alternatives to conventional bitumen modifiers [5].

MDEA (methyldiethanolamine) waste, a byproduct of natural gas sweetening processes in the oil and gas industry, presents a novel and sustainable option for bitumen modification. MDEA is commonly used to remove acidic gases (e.g., CO₂ and H₂S) from natural gas streams, and the resulting waste stream contains a mixture of amines, water, and organic compounds. The chemical composition of MDEA waste, particularly its amine functional groups, suggests potential for improving bitumen's adhesion to aggregates by enhancing chemical interactions at the bitumen-aggregate interface. The polar nature of amines can facilitate stronger bonding with siliceous or calcareous aggregate surfaces, while their compatibility with bitumen's hydrocarbon matrix ensures effective integration [6-7]. Moreover, repurposing MDEA waste aligns with circular economy principles, transforming an industrial byproduct into a value-added material, thereby reducing waste disposal challenges and environmental impacts [8].

Previous studies on bitumen modification have primarily focused on synthetic polymers and chemical additives, with limited attention to industrial waste materials as modifiers. For instance, SBS-modified bitumen is widely used for its ability to enhance elasticity and adhesion, but its high cost and environmental footprint limit its applicability in resource-constrained regions [9]. Recent research has explored bio-based and waste-derived modifiers, such as crumb rubber, waste cooking oil, and lignin, with varying degrees of success in improving bitumen performance [10]. However, the use of MDEA waste as a bitumen modifier remains largely unexplored, representing a significant research gap. Preliminary studies on amine-based additives suggest that their polar functional groups can improve bitumen's wetting ability and adhesion to aggregates, particularly in wet conditions (Curtis et al., 1993). Given its availability in regions with active oil and gas industries, MDEA waste offers a promising opportunity to develop cost-effective and sustainable bitumen formulations [11].

The primary objective of this study is to investigate the effect of MDEA waste as an additive on the adhesion properties of 60/90 penetration-grade bitumen, with a focus on its performance under wet conditions [12]. The research also evaluates the impact of MDEA waste on bitumen's viscosity, softening point, and thermal stability to ensure its suitability for road construction applications. By testing various concentrations of MDEA waste, the study aims to identify the optimal formulation that maximizes adhesion while maintaining desirable mechanical and thermal properties. The findings are expected to contribute to the development of high-performance, sustainable bitumen for road pavements, particularly in regions with significant oil and gas processing activities. Additionally, this research promotes the valorization of industrial byproducts, offering economic and environmental benefits for the construction industry [13].

The significance of this study lies in its potential to address critical challenges in road construction while advancing sustainable material development. Improved adhesion properties can enhance pavement durability, reduce maintenance costs, and improve safety by minimizing pavement failures [14]. By utilizing MDEA waste, a low-cost and abundant byproduct, this study supports the adoption of environmentally friendly practices in infrastructure development. Furthermore, the research provides a foundation for future investigations into waste-derived bitumen modifiers, fostering innovation in the use of industrial byproducts for sustainable construction solutions [15].

II. DEVICE DESCRIPTION

The determination of the limiting oxygen index (LOI) of epoxy-based polymer composites was carried out as follows. According to the requirements of GOST 12.1.044-2018 [1, 2] and GOST 21793-76, [3, 4, 5, 6, 7] polymer composite samples were prepared. Testing of the prepared samples was initiated with a gas mixture containing 18% oxygen for the oxygen index measurement.

The oxygen index (OI) was calculated as a percentage.

$$KI = \frac{V_k}{V_k + V_a} \cdot 100 \quad (1)$$

Here: V_k – total volume of oxygen, in dm³/min or cm³/s; V_a – total volume of nitrogen, in dm³/min or cm³/s. Based on this formula, the average values of the oxygen index were determined.



Figure 1. Laboratory procedures for investigating the limiting oxygen index of intumescent fire-protective polymer composite coatings.

III. TESTING OF SYSTEM

The determination of the limiting oxygen index of epoxy resin-based polymer composites was carried out as follows.

Nowadays, the limiting oxygen index (LOI) is widely used to analyze the properties of fire-resistant intumescent coatings, which are extensively applied to protect metal structures from high temperatures and fire. According to literature data, the LOI of the proposed polymer composite materials before forming the composite is reported to be 17–18%. Taking this into account, newly prepared polymer composites were studied in oxygen–nitrogen mixtures with LOI values ranging from 18% to 42%. In Table 1 below, the effects of varying the ratios of the polymer binder and other chemical additives (flame retardants, intumescent-forming agents, surfactants, fillers, etc.) on the oxygen index were investigated for the preparation of E-44-1 epoxy resin-based intumescent fire-protective coatings [8, 9, 10].

Table 1

Experimental results of studying the effects of the ratios of chemical components forming intumescent fire-protective polymer composites on the oxygen index.

Name of the Polymer Composite	Amounts of Intumescent-Forming Components and Flame Retardant, wt %	Oxygen Index, %																														
Epoxy Resin	0	17-19,0																														
E-44-1	<table style="width: 100%; border-collapse: collapse;"> <tr> <td colspan="2">Polymer Binder:</td> <td></td> </tr> <tr> <td>Epoxy Resin.</td> <td style="text-align: right;">28</td> <td></td> </tr> <tr> <td>Polysulfide Rubber</td> <td style="text-align: right;">10</td> <td></td> </tr> <tr> <td>Zinc Oxide</td> <td style="text-align: right;">1</td> <td style="text-align: center;">32</td> </tr> <tr> <td>Hardener.</td> <td style="text-align: right;">3</td> <td></td> </tr> <tr> <td colspan="2">Intumescent Additive:</td> <td></td> </tr> <tr> <td>Pentaerythritol</td> <td style="text-align: right;">20</td> <td></td> </tr> <tr> <td>Hexamethylenetetramine</td> <td style="text-align: right;">8</td> <td></td> </tr> <tr> <td colspan="2">Flame Retardant: ARM-5</td> <td></td> </tr> <tr> <td>Metal-Containing Adduct</td> <td style="text-align: right;">30</td> <td></td> </tr> </table>	Polymer Binder:			Epoxy Resin.	28		Polysulfide Rubber	10		Zinc Oxide	1	32	Hardener.	3		Intumescent Additive:			Pentaerythritol	20		Hexamethylenetetramine	8		Flame Retardant: ARM-5			Metal-Containing Adduct	30		
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In epoxy resin-based intumescent fire-protective polymer coatings, the composition includes polymer binders, flame retardants, intumescent chemical additives that release gas at 200–300 °C, and fillers. The effects of varying the ratios of these components, ranging from 5% to 40%, on the oxygen index were investigated. Additionally, in this type of polymer coating, hardeners are used at 10% relative to the mass of the polymer binder. These hardeners consist of nitrogen-containing organic compounds.

IV. DIAGRAMMATIC REPRESENTATION

The effects of varying the percentages of nitrogen-, phosphorus-, and metal-containing flame retardants on the oxygen index were studied in the composition of the E-44-1 epoxy resin-based intumescent fire-protective

polymer coating. During the research, the proposed formulation included oligomeric ARM-5 flame retardants, obtained from epichlorohydrin and containing nitrogen- and phosphorus-based compounds, as well as imported Pylax flame retardants and the proposed ARM-1 and ARM-3 flame retardants. Their various ratios were incorporated into the polymer composite coating and analyzed [11, 12, 13, 14].

The results can be analyzed from Figure 1. According to the data, in the proposed E-44-1 epoxy resin-based intumescent fire-protective polymer coating, oligomeric flame retardants obtained from dichlorohydrin and containing phosphorus- and nitrogen-based compounds were studied in ratios ranging from 5% to 35%. It was found that at a 25% flame retardant content, the oxygen index reached 32%. Although the oxygen index increased to 34% or higher when the flame retardant content exceeded 35%, experimental analysis showed that such high amounts negatively affected the physical and mechanical properties of the polymer composite, making it non-compliant with required standards. Therefore, a 25% flame retardant content was determined to be the most effective [14].

It was found that the imported analogue flame retardant (Pylax), as well as the newly proposed polymer composite formulations containing ARM-1 and ARM-3 flame retardants, were less effective compared to the proposed oligomeric ARM-5 flame retardant.

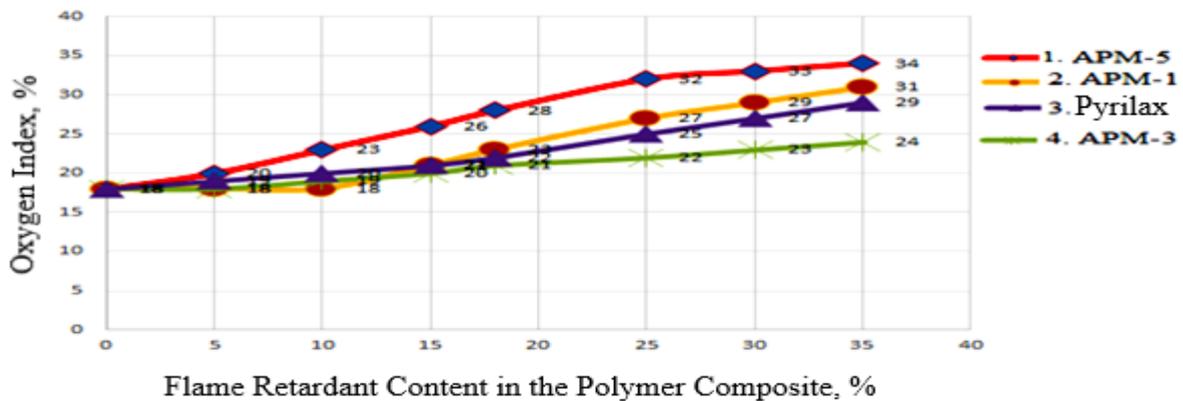


Figure 2. Effect of varying concentrations of flame retardants on the oxygen index of E-44-1 epoxy resin-based intumescent fire-protective polymer coating.

RESULTS

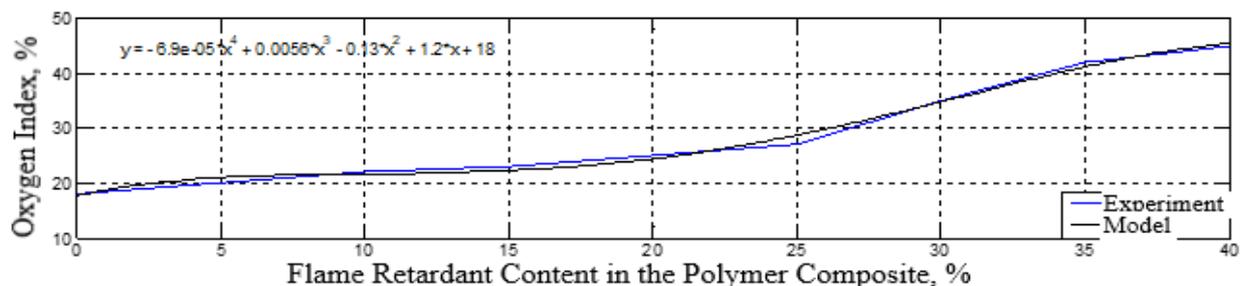
A statistical–mathematical model of the effect of varying concentrations of flame retardants on the oxygen index of E-44-1 epoxy resin-based intumescent fire-protective polymer composite coating [14].

Given the polymer composite composition with ARM-5 flame retardant content, %:

$Q = [0, 5, 10, 15, 20, 25, 30, 35, 40]$ and oxygen index, %: $q = [18, 20, 22, 23, 25, 27, 35, 42, 45]$ the statistical–mathematical model is:

$$y = -6.9 \times 10^{-5} x^4 + 0.0056 x^3 - 0.13 x^2 + 1.2 x + 18$$

This model describes the relationship between the ARM-5 flame retardant content (xxx) and the oxygen index (yyy) of the polymer composite.



The absolute maximum error of the statistical–mathematical model is 2.4555.

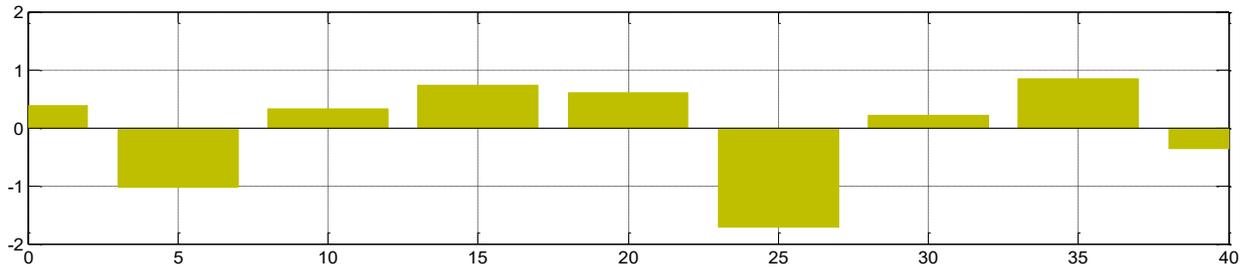


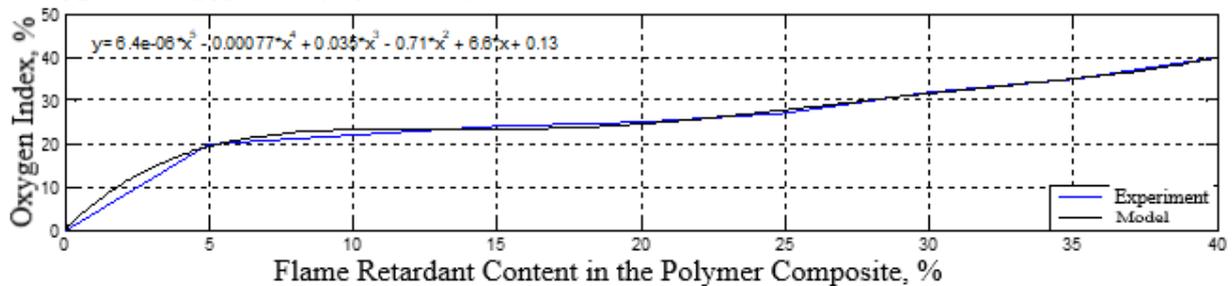
Figure 3. Statistical–mathematical model of the effect of ARM-5 flame retardant content on the oxygen index of E-44-1 epoxy resin-based intumescent fire-protective polymer composite coating.

Given the polymer composite composition with ARM-1 flame retardant content, %:

$Q = [0, 5, 10, 15, 20, 25, 30, 35, 40]$ and oxygen index, %: $q = [0, 20, 22, 24, 25, 27, 32, 35, 40]$ the statistical–mathematical model is:

$$y = 6.4 \times 10^{-6} x^5 - 0.00077 x^4 + 0.035 x^3 - 0.71 x^2 + 6.6 x + 0.13$$

This fifth-degree polynomial describes the relationship between the ARM-1 flame retardant content (xxx) and the oxygen index (yyy) of the polymer composite.



The absolute maximum error of the statistical–mathematical model is 1.8283.

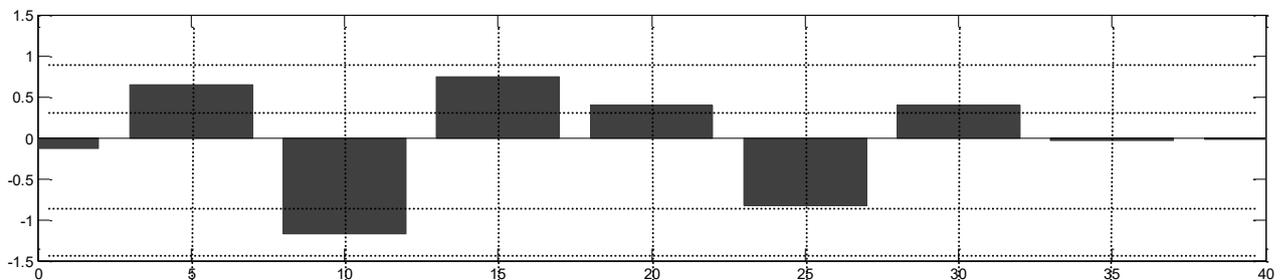


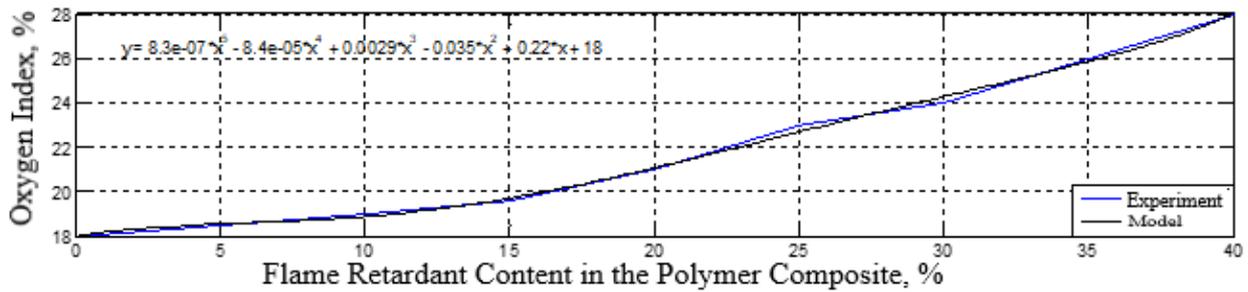
Figure 4. Statistical–mathematical model of the effect of ARM-1 flame retardant content on the oxygen index of E-44-1 epoxy resin-based intumescent fire-protective polymer composite coating.

Given the polymer composite composition with Pyrilax flame retardant content, %:

$Q = [0, 5, 10, 15, 20, 25, 30, 35, 40]$ and oxygen index, %: $q = [18, 18.5, 19, 19.6, 21, 23, 24, 26, 28]$ the statistical–mathematical model is:

$$y = 8.3 \times 10^{-7} x^5 - 8.4 \times 10^{-5} x^4 + 0.0029 x^3 - 0.035 x^2 + 0.22 x + 18$$

This fifth-degree polynomial describes the relationship between the Pyrilax flame retardant content (xxx) and the oxygen index (yyy) of the polymer composite.



The absolute maximum error of the statistical–mathematical model is 1.9353.

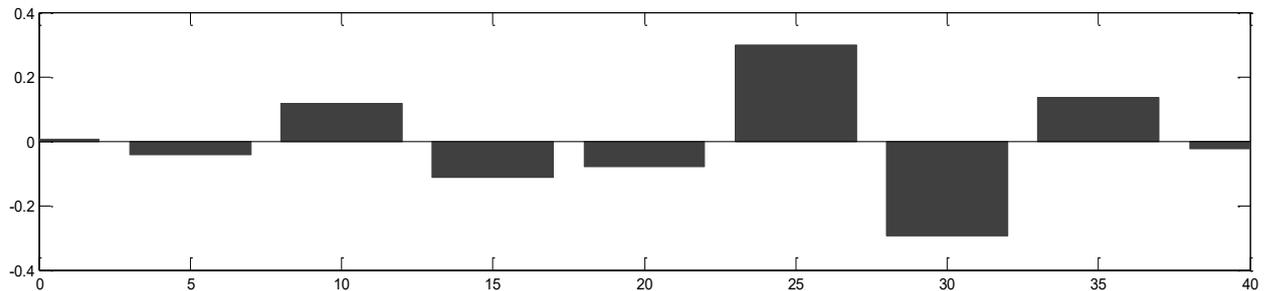


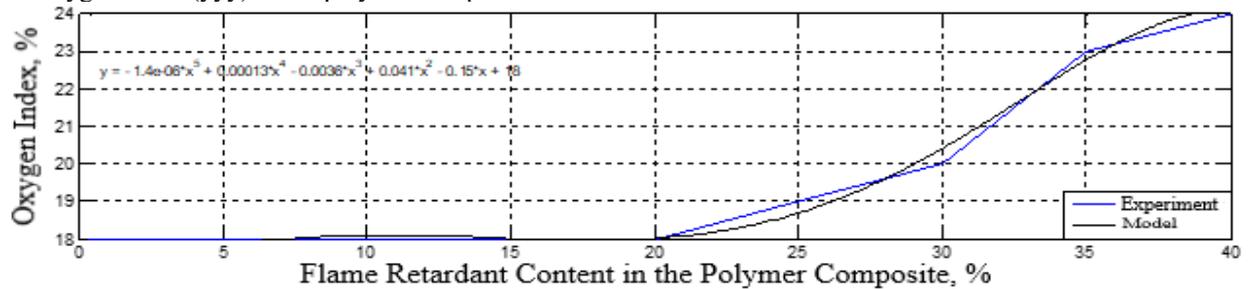
Figure 5. Statistical–mathematical model of the effect of Pyrilax flame retardant content on the oxygen index of E-44-1 epoxy resin-based intumescent fire-protective polymer composite coating.

Given the polymer composite composition with ARM-3 flame retardant content, %:

$Q = [0, 5, 10, 15, 20, 25, 30, 35, 40]$ and oxygen index, %: $q = [18, 18, 18, 18, 18, 19, 20, 23, 24]$ the statistical–mathematical model is:

$$y = -1.4 \times 10^{-6} x^5 + 1.3 \times 10^{-4} x^4 - 0.0036 x^3 + 0.041 x^2 - 0.15 x + 18$$

This fifth-degree polynomial represents the relationship between the ARM-3 flame retardant content (xxx) and the oxygen index (yyy) of the polymer composite.



The absolute maximum error of the statistical–mathematical model is 0.57937.

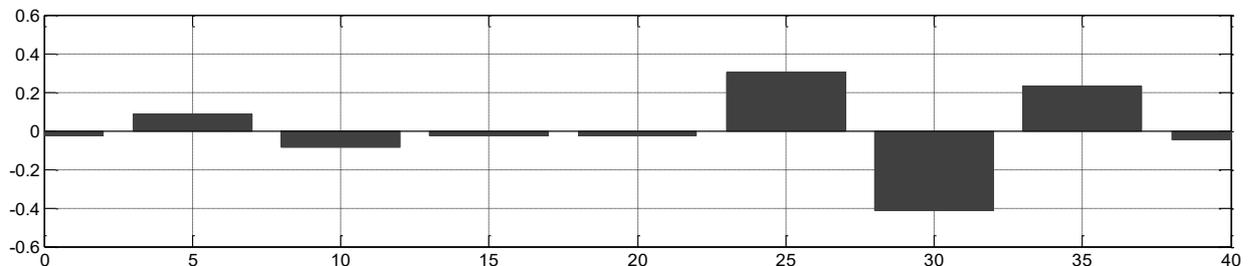


Figure 6. Statistical–mathematical model of the effect of ARM-3 flame retardant content on the oxygen index of E-44-1 epoxy resin-based intumescent fire-protective polymer composite coating.

During the production of E-44-1 epoxy resin-based intumescent fire-protective polymer coatings, the effect of intumescent chemical additives—pentaerythritol and hexamethylenetetramine—on the oxygen index was studied

in various ratios: 1:1.5, 1:1, and 1.5:1. In the study, mixtures of pentaerythritol and hexamethylenetetramine in a 1:1.5 ratio were added to the polymer composite in amounts ranging from 5% to 30%, and their effect on the intumescence properties was analyzed. Additionally, mixtures in the 1:1 and 1.5:1 ratios were also investigated. [14].

The results of this experiment are shown in Figure 6, indicating that mixtures of pentaerythritol and hexamethylenetetramine in a 1:1.5 ratio, when added to the polymer composite at a 25% proportion, achieved an oxygen index of 32% or higher. This demonstrates that this ratio is more effective for intumescence compared to the other proposed mixtures.

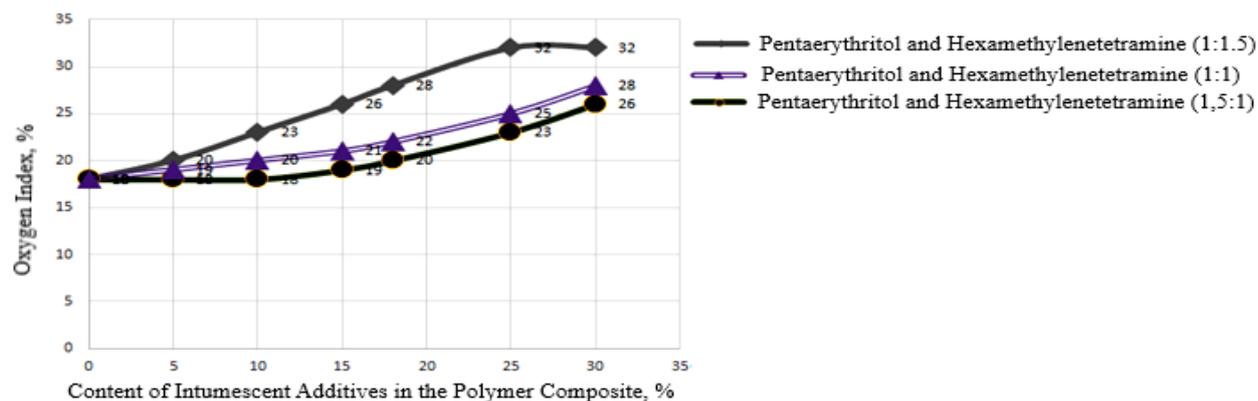


Figure 7. Effect of different ratios of intumescent chemical additive mixtures on the oxygen index of E-44-1 epoxy resin-based intumescent fire-protective polymer coating.

V. CONCLUSION

Thus, the fire resistance and intumescence properties of fire-protective polymer composites based on acrylic copolymers and epoxy resin are most effectively enhanced when flame retardants and intumescent chemical additives are incorporated in total amounts ranging from 10–15% up to 30–35% of the composite [14].

The conducted studies revealed that the fire-resistance properties of E-44-1 epoxy resin-based intumescent fire-protective polymer composite coatings are directly dependent on the ratios of their constituent components.

Among the flame retardants, the oligomeric ARM-5 was proven to be the most effective. When used at a 25% proportion, it achieved an oxygen index of 32%, while the physical and mechanical properties of the composite remained at the required level. Although adding more than 35% of the flame retardant further increased the oxygen index, it was found to negatively affect the material's strength characteristics.

Among the intumescent additives, the mixture of pentaerythritol and hexamethylenetetramine in a 1:1.5 ratio achieved the highest efficiency when applied at a 25% proportion.

The developed statistical–mathematical models accurately represented the effect of flame retardant content on the oxygen index and were confirmed to be consistent with the experimental results.

In general, incorporating flame retardants and intumescent additives into fire-protective polymer composites in an optimal range of 10–35% enhances the material's fire resistance and ensures reliable protection of structures.

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