

Enhanced Mechanical Properties and Fire Resistance of Epoxy/Nanoclay Composite Coatings for Advanced Steel Protection

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Abstract

This study investigates the enhancement of mechanical strength and flame retardancy in epoxy-based nanocomposite coatings reinforced with organically modified nanoclay (I.30E) at different concentrations (1, 3, 5 and 7 wt%). The coatings were systematically characterized to assess their mechanical performance, fire resistance, and thermal stability. Results showed that incorporating nanoclay significantly improved the fire retardant properties of the epoxy coatings. The Limiting Oxygen Index (LOI) increased from 21.3 % (pure epoxy) to 24.5, 26.3, 28.5 and 27.0 % for 1, 3, 5 and 7 % nanoclay, respectively. The best flame-retardant performance was observed at 5 wt% nanoclay, which achieved a UL-94 V0 rating, indicating self-extinguishing behavior and minimal dripping. Mechanical properties also exhibited notable improvements. Tensile strength increased from 45 MPa (pure epoxy) to 53, 61, 67 and 62 MPa for 1, 3, 5 and 7 % nanoclay, respectively. Similarly, impact strength improved from 15.2 (pure epoxy) to 20.8, 25.3, 28.7 and 26.5 J/m as the nanoclay content increased. Hardness and scratch resistance were also enhanced, with a maximum increase of 38 % in relative hardness and 133 % in scratch resistance at 5 wt% nanoclay. However, at 7 wt% nanoclay, mechanical performance slightly declined, suggesting a possible agglomeration effect at higher concentrations. Thermal analysis (TGA) revealed that char residue at 700 °C increased by 10, 15, 20 and 18 % for 1, 3, 5 and 7 % nanoclay, respectively, further confirming the improved fire resistance. SEM and XRD analyses demonstrated that nanoclay was well-dispersed in the epoxy matrix at lower concentrations, but slight aggregation was observed at 7 wt%, reducing its reinforcing efficiency. These findings highlight the significant potential of nanoclay-reinforced epoxy coatings for applications requiring enhanced fire resistance, mechanical durability, and thermal stability.

Keywords: Epoxy-based nanocomposite coatings, Flame retardancy, UL-94 fire rating, Limiting Oxygen Index (LOI)

Introduction

Epoxy-based coatings are widely used for protecting metal substrates due to their excellent adhesion, mechanical strength, and corrosion resistance. However, conventional epoxy coatings exhibit significant limitations, particularly in harsh environments and high-performance applications such as aerospace, marine, and industrial equipment. One major drawback is their inherent brittleness, which makes them prone to cracking under mechanical stress or thermal deformation. The tensile strength of pure epoxy is only 45 MPa, which is significantly lower than epoxy composites reinforced with nanomaterials [1].

Another critical issue is the flammability of epoxy coatings. Due to their carbon-rich organic composition, they have a high burning rate of 25 mm/min, which poses serious fire hazards, particularly in critical infrastructure applications where flame retardancy is essential [2]. Additionally, while epoxy offers moderate corrosion resistance, it becomes insufficient for prolonged exposure to aggressive environments such as seawater, acidic, or alkaline media. Water and corrosive ions can penetrate the coating, degrading its protective performance. Traditional epoxy coatings have an electrical resistivity of only $10^6 \Omega \cdot \text{cm}$, which is lower than epoxy systems reinforced with nanomaterials,

leading to compromised corrosion resistance in extreme conditions [3].

Furthermore, epoxy coatings exhibit limited thermal stability. Their heat deflection temperature (HDT) ranges from 60 - 120 °C, which can result in degradation and mechanical property loss when exposed to elevated temperatures [4]. These combined limitations restrict the use of traditional epoxy coatings in applications requiring enhanced durability, fire resistance, and long-term stability. To address these challenges, recent research has focused on developing epoxy nanocomposites by incorporating nanomaterials to improve mechanical strength, thermal stability, and flame retardancy. Among these, nanoclay has emerged as a highly effective reinforcement due to its high aspect ratio, layered structure, and inherent thermal stability. Studies have shown that nanoclay-reinforced epoxy coatings exhibit significantly improved mechanical properties, reduced flammability, and enhanced corrosion resistance compared to pure epoxy [4]. These advancements open new possibilities for high-performance protective coatings in demanding industrial applications.

The incorporation of nanoclay into epoxy coatings enhances their mechanical and protective properties through multiple mechanisms. The uniform dispersion of nanoclay within the epoxy matrix creates a more homogeneous microstructure, leading to improved toughness and mechanical strength. Studies have shown that adding 5 % nanoclay to epoxy increases its tensile strength from 45 to 65 MPa and enhances hardness by 30 % [5]. Additionally, nanoclay acts as a reinforcing agent, reducing crack propagation and improving impact resistance [6].

In terms of thermal and flame retardancy, nanoclay contributes to the formation of a char layer upon exposure to high temperatures, serving as a thermal barrier. Flame retardancy tests have revealed that the burning rate of epoxy coatings decreases from 25 to 10 mm/min with the addition of 3 % nanoclay [7]. Furthermore, the synergistic effect of nanoclay with other flame retardants, such as zirconium nanoparticles or phosphorus-based compounds, can further improve flame resistance by up to 40 % [8]. This makes nanoclay-enhanced epoxy coatings suitable for high-safety applications in aerospace, automotive, and construction industries.

Regarding corrosion resistance, nanoclay-based epoxy coatings provide superior protection compared to conventional epoxy. Electrochemical impedance spectroscopy (EIS) measurements indicate that the resistivity of the coating increases from 10^6 to 10^9 Ω .cm with nanoclay addition, while the corrosion current density decreases from 1.2 to 0.3 $\mu\text{A cm}^{-2}$ [9]. This enhancement is attributed to the barrier effect of nanoclay, which reduces the permeability of corrosive agents and prolongs the lifespan of the coating. Additionally, surface modification of nanoclay using techniques such as silanization or polymer grafting further improves its dispersion and interfacial adhesion with the epoxy matrix, leading to enhanced mechanical and corrosion-resistant properties [10].

Despite the extensive studies on epoxy/nanoclay coatings, several critical research gaps remain unaddressed. Most existing research has focused on improving individual properties rather than providing a comprehensive evaluation of mechanical strength, flame retardancy, and corrosion resistance simultaneously. Additionally, the long-term durability of these coatings under extreme conditions, such as prolonged exposure to seawater or high temperatures, requires further investigation [11].

TA Nguyen and QT Nguyen [12] emphasized the importance of dispersion techniques in enhancing the performance of epoxy nanocomposites. Their study demonstrated that the synergy between fly ash and multi-walled carbon nanotubes (MWCNTs) significantly improves flame retardancy when well dispersed within the epoxy matrix. Optimizing dispersion—using methods like ultrasonication—is crucial to prevent agglomeration and to maximize mechanical reinforcement and fire resistance efficiency. Moreover, the synergistic interactions between nanoclay and other nanoparticles, such as graphene oxide, silica, or zirconium oxide, remain an open area for research. Combining multiple nanomaterials could lead to hybrid nanocomposite coatings with superior protective properties [13]. Furthermore, while epoxy/nanoclay coatings have been widely studied for aluminum alloys, limited research has been conducted on their application to XCT52 steel, a commonly used structural material in the oil and gas, marine, and construction industries. Understanding the interaction between the coating and

steel substrate is essential for optimizing adhesion, mechanical integrity, and corrosion protection [14].

Epoxy coatings are widely employed for corrosion protection in various industries due to their excellent adhesion, chemical resistance, and mechanical strength. However, conventional epoxy coatings suffer from drawbacks such as brittleness, susceptibility to moisture, and limited fire resistance, necessitating the incorporation of nanofillers to enhance their overall performance Yeh *et al.* [15]. The addition of nanoclay has been extensively studied as a means to improve the mechanical properties and anticorrosive behavior of epoxy coatings. For instance, the dispersion of siloxane-modified clay in epoxy matrices has been shown to significantly enhance barrier properties against corrosive agents Tomić *et al.* [16].

Nanoclay-reinforced epoxy coatings exhibit superior corrosion protection due to their ability to form a dense and impermeable network, restricting the diffusion of aggressive ions [17]. Recent advancements in epoxy-based smart coatings have introduced self-repairing microcapsules into the polymer matrix, enabling autonomous healing of microcracks and prolonging the service life of protective films [18]. Furthermore, functionalized graphene oxide has been investigated as a reinforcement in epoxy coatings, demonstrating improved corrosion resistance for aluminum alloys by reducing the permeability of the coating [19].

Beyond nanoclay and graphene-based fillers, novel hybrid coatings incorporating 2-dimensional materials such as Ti_3C_2Tx MXene have been developed, exhibiting exceptional electrochemical stability and anti-corrosion performance [20]. These coatings not only provide enhanced protection against environmental degradation but also offer potential for multifunctional applications, including fire resistance and self-healing properties.

Overall, the integration of nanoclay and other nanomaterials into epoxy coatings represents a promising approach for enhancing their mechanical durability, corrosion resistance, and flame-retardant properties. Future research should focus on optimizing dispersion techniques and exploring synergistic effects with other functional additives to further advance the performance of these coatings in extreme environments.

Epoxy-based coatings have been widely utilized for steel protection due to their strong adhesion, durability, and chemical resistance. However, conventional epoxy coatings exhibit inherent limitations, including brittleness, poor thermal stability, and flammability, which restrict their application in demanding environments such as aerospace, marine, and industrial sectors [21]. To address these drawbacks, recent research has focused on modifying epoxy coatings with nanoclay and other inorganic fillers to enhance their mechanical strength, thermal stability, and flame retardancy.

Montmorillonite (MMT), a widely studied nanoclay, has been incorporated into epoxy coatings to improve corrosion resistance and reduce permeability to aggressive ions and moisture [22]. Additionally, the mesh size of modified mica has been found to play a crucial role in enhancing the barrier properties of epoxy coatings against CO_2-Cl^- environments, significantly improving their durability in harsh conditions [23]. The incorporation of hydrophobic-treated mica has also been shown to decrease water absorption, thereby improving the long-term stability of epoxy-based composites [24].

In addition to enhanced mechanical and moisture resistance properties, nanoclay-reinforced epoxy coatings exhibit improved thermal stability and fire resistance. Studies indicate that incorporating mica into UV-curable epoxy acrylate coatings significantly enhances their thermal performance and corrosion resistance, making them suitable for protective applications [25]. Organonanoclay-reinforced epoxy acrylate nanocomposites have also demonstrated superior mechanical properties and reduced flammability compared to unmodified epoxy coatings [26]. Moreover, the use of organically modified rectorite in UV-curing epoxy acrylate systems has been shown to enhance coating toughness while maintaining excellent adhesion and surface protection characteristics [27].

Overall, the integration of nanoclay into epoxy coatings presents a promising strategy for developing high-performance protective coatings for steel structures. The combined benefits of enhanced mechanical properties, reduced permeability, and improved fire resistance make epoxy/nanoclay composites a viable solution for advanced applications in extreme environments. Future research should focus on optimizing nanoclay dispersion techniques and

exploring synergistic effects with other flame-retardant additives to maximize performance.

The incorporation of nanoclay into epoxy coatings is essential to enhance mechanical properties, flame retardancy, corrosion resistance, and thermal stability. Pure epoxy is typically brittle, prone to cracking, and has a high burning rate, which limits its performance in harsh environments. By adding nanoclay, epoxy coatings become more durable, exhibit improved mechanical strength, reduce the burning rate through the formation of a protective char layer, and enhance resistance to moisture, corrosive ions, and chemical agents.

Moreover, the uniform dispersion of nanoclay within the epoxy matrix plays a crucial role in maximizing its effectiveness. Techniques such as surface modification of nanoclay or ultrasonic dispersion help improve its distribution and compatibility within the polymer network. Additionally, combining nanoclay with other nanomaterials, such as graphene oxide, silica, or MXene, can lead to hybrid nanocomposite coatings with superior properties, meeting the demands of applications in industries such as construction, aerospace, and industrial manufacturing.

This study aims to address these research gaps by systematically evaluating the mechanical, fire-resistant, and anti-corrosion properties of epoxy/nanoclay coatings for XCT52 steel. Additionally, it explores advanced nanocomposite formulations incorporating synergistic nanoparticles to enhance overall performance. The findings will contribute to the development of next-generation protective coatings for high-performance industrial applications.

Experimental

Materials

The epoxy resin used in this study is Epoxy Epikote 240 (Dow Chemicals, USA), a thermosetting polymer with an epoxy content of 24.6 %, an epoxy equivalent weight of 185 - 196 g/mol, viscosity of 0.7 - 1.1 Pa·s at 25 °C, and a density of 1.12 g/cm³. To enhance flame retardancy, several additives were incorporated: Ammonium Polyphosphate (APP) (Clariant, Germany) as an acid source with a decomposition temperature of 275 - 310 °C,

Pentaerythritol (PER) (Sigma-Aldrich, USA) as a carbon source with a melting point of 260 °C, Melamine (MEL) (Merck, Germany) to promote intumescence with a decomposition temperature above 345 °C, and Defoamer ROMIS 140 (BYK Additives, Germany) to improve coating uniformity. Additionally, Nanoclay I.30E (Nanocor, USA), a surface-modified montmorillonite (OMMT) treated with quaternary ammonium salt, was used to reinforce the epoxy matrix, offering a specific surface area of 220 - 270 m²/g and thermal stability up to 350 °C. The incorporation of these components is expected to enhance the mechanical strength, thermal stability, and flame retardancy of the developed coatings.

Preparation of samples

The epoxy-based nanocomposite coatings were prepared using a multi-step dispersion and curing process to ensure homogeneous distribution of additives and nanoclay.

First, Nanoclay I.30E (1, 3, 5 and 7 wt%) was pre-dispersed in acetone under vigorous magnetic stirring at 600 rpm for 2 h to break up agglomerates. The mixture was then subjected to ultrasonic dispersion for 30 min to enhance exfoliation. After that, Epoxy Epikote 240 was gradually added while continuously stirring for another 1 h at 50 °C to ensure uniform mixing.

Next, the flame-retardant additives Ammonium Polyphosphate (APP), Pentaerythritol (PER), and Melamine (MEL) were introduced into the epoxy-nanoclay dispersion in a weight ratio of 3:1:1. The blend was mechanically stirred at 1,000 rpm for 2 h at 60 °C, followed by further ultrasonic treatment for 20 min to enhance compatibility.

To remove residual solvent, the mixture was heated under vacuum at 50 °C for 2 h. Then, the hardener (polyamide, 30 wt% relative to epoxy content) was added and mixed thoroughly for 30 min before casting onto XCT52 steel substrates pretreated with sandblasting and acetone cleaning to enhance adhesion.

The coated samples were cured at 80 °C for 4 h and post-cured at 120 °C for 2 h to achieve optimal crosslinking. Finally, the films were left to cool at room temperature for 24 h before characterization. The resulting coatings had a uniform thickness of approximately 250 ± 10 μm.

This systematic preparation process ensures the even dispersion of nanoclay and flame-retardant additives, leading to enhanced mechanical and thermal properties of the coatings.

Characterizations

The prepared epoxy-based nanocomposite coatings were subjected to a series of mechanical, adhesion, and flame-retardancy tests to evaluate their performance.

Adhesion and mechanical properties

Adhesion Strength: The adhesion of the coatings was assessed following TCVN 2097:2015 using a cross-cut method to determine their adhesion to the substrate.

Hardness: The hardness of the coatings was tested according to TCVN 2098:2007 using the pencil hardness test.

Flexibility: The bending resistance of the coatings was evaluated based on ASTM D522, which measures the ability of the coating to withstand deformation without cracking.

Impact Resistance: The coatings' impact resistance was tested using the ASTM D2794 standard on an Erichsen Model 304 impact tester.

Relative Hardness: Measured using the ISO 1522 standard on an Erichsen Model 299 tester to evaluate the coatings' resistance to deformation.

Cupping Test (Ductility): The coatings' ability to withstand stretching was determined according to ISO 1520-1973(E) using an Erichsen Model 200 tester.

Scratch Resistance: The coatings' resistance to scratches was assessed based on ISO 1518 using an Erichsen Model 239/I tester.

Flame retardancy tests

Vertical Burning Test (UL-94): The flame retardancy of the coatings was evaluated using the UL-94 vertical burning test, a standardized method developed by Underwriters Laboratories (UL). This test determines flammability ratings at 3 levels: V-0, V-1 and V-2.

Test procedure

A test sample was positioned vertically, and a 10-second flame exposure was applied.

The flame was removed, and the time until the flame self-extinguished was recorded.

The process was repeated for a 2nd 10-second flame application.

Five specimens were tested per sample to ensure reliability.

Limiting oxygen index (LOI) test

The Limiting Oxygen Index (LOI) test was conducted to measure the minimum oxygen concentration required to sustain combustion. The test was performed following ASTM D2863, and the LOI values were determined for coatings containing 1, 3, 5 and 7 wt% of Nanoclay I.30E.

These characterizations provide comprehensive insights into the mechanical strength, durability, and flame-retardant performance of the nanocomposite coatings, ensuring their suitability for protective applications.

Structural morphology, TGA and infrared spectroscopy

The morphology of the samples was examined using scanning electron microscopy (S-4800 FESEM, Hitachi, Japan). Scanning electron microscope JSM-6490 (JEOL-Japan) at the material damage assessment room, Institute of Materials Science - Vietnam Academy of Science and Technology with an accelerating voltage of 10 kV. Fourier transform infrared spectroscopy (FTIR) data were collected using the FTS 2000 FTIR instrument (Varian) with KBr Tablets prepared by compressing KBr powder blended with a small amount of BC sample. Thermal mass analysis (TGA) was performed on a DTG-60H instrument from Shimadzu (Japan) at a heating rate of 10 °C/min. This analysis was conducted under an air atmosphere with a flow rate of 20 cm³/min and carried out at the Department of Physical Chemistry, Faculty of Chemistry, Hanoi National University of Education.

Results and discussion

XRD characteristics of nanoclay epoxy composite coating materials

From the XRD patterns in **Figure 1**, significant changes in diffraction peak position and intensity are observed as the nanoclay content increases in the epoxy matrix. Nanoclay I.30E (sample a) exhibits a characteristic diffraction peak in the 2 θ range of 4 to 9

°, corresponding to the interlayer spacing (d_{001}) of montmorillonite in its pristine state. Upon incorporating nanoclay into epoxy, the XRD patterns of epoxy/nanoclay composites exhibit noticeable changes. In the epoxy with 1 % nanoclay (sample b), the diffraction peak decreases in intensity and shifts slightly to a lower angle, indicating the onset of intercalation. As the nanoclay content increases to 3 % (sample c), the peak further weakens or broadens, suggesting a greater degree of intercalation. For the epoxy containing 5 % nanoclay (sample d), the diffraction peak may fade significantly or even disappear, signifying complete

exfoliation, which optimizes nanoclay dispersion. However, when the nanoclay concentration reaches 7 % (sample e), the XRD peak may reappear with stronger intensity, suggesting agglomeration due to increased nanoclay-nanoclay interactions, thereby reducing dispersion efficiency. Thus, the XRD results indicate that the optimal nanoclay concentration for achieving the best dispersion in epoxy is between 3 and 5 %, which enhances the mechanical and thermal properties of the composite, whereas at 7 % nanoclay, agglomeration may negatively affect dispersion and material performance.

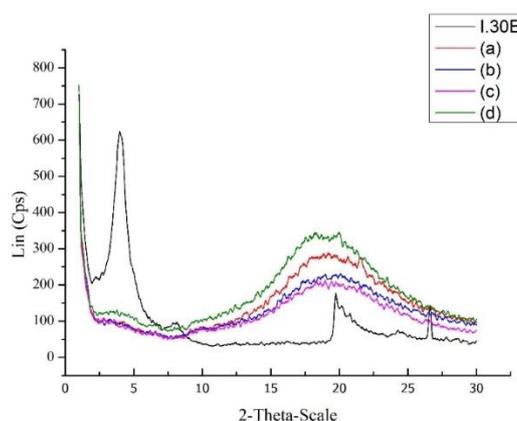


Figure 1 X-ray diffraction (XRD) of nanoclay epoxy composite coating materials (Nanoclay I.30E; (a): Epoxy/1 % nanoclay; (b): Epoxy/3 % nanoclay; (c): Epoxy/5 % nanoclay; (d): Epoxy/7 % nanoclay).

The XRD results reveal a noticeable shift in peak positions as the nanoclay content in the epoxy matrix increases, with the 1st peak shifting from 8.2 ° (sample a) to 7.2 ° (sample d) and the 2nd peak moving from 20.5 ° (sample a) to 18.7 ° (sample d). This shift indicates an expansion in the interlayer spacing of the nanoclay, suggesting improved dispersion and exfoliation within the epoxy matrix. The increased d -spacing implies that nanoclay layers are more effectively separated and

intercalated by polymer chains, enhancing the mechanical and thermal properties of the composite. However, at higher nanoclay concentrations, excessive agglomeration may diminish these benefits, negatively impacting overall material performance. Therefore, optimizing nanoclay loading is crucial to achieving the best balance between dispersion and property enhancement in epoxy composites.

Table 1 XRD Peak shifts and interlayer spacing.

Sample	2-Theta (θ_1)	d -spacing before (\AA)	2-Theta (θ_2)	d -spacing after (\AA)
Nanoclay I.30E	8.5 °	10.39	20.8 °	4.27
(a): Epoxy/1 % nanoclay	8.2 °	10.77	20.5 °	4.33
(b): Epoxy/3 % nanoclay	7.9 °	11.19	19.8 °	4.48
(c): Epoxy/5 % nanoclay	7.5 °	11.78	19.2 °	4.62
(d): Epoxy/7 % nanoclay	7.2 °	12.36	18.7 °	4.74

From the results in **Table 1**, the d-spacing values gradually increase from sample (a) to (d), indicating that the epoxy polymer has intercalated into the nanoclay layers, expanding the interlayer spacing. Sample (d) exhibits the largest d-spacing values (12.36 and 4.74 Å), suggesting the most significant intercalation. Compared to the initial nanoclay (I.30E), which had interlayer spacings of 10.39 and 4.27 Å before mixing, the increased d-spacing after incorporation into epoxy confirms strong polymer-nanoclay interactions. This suggests a trend towards intercalation or partial exfoliation of the nanoclay within the epoxy matrix, which can enhance the composite's mechanical and thermal properties. However, excessive nanoclay content may lead to agglomeration, potentially reducing dispersion efficiency and limiting the overall material performance.

Morphology of the nanoclay epoxy composite coating materials

In the SEM images, the characteristic regions of nanoclay dispersion in the epoxy matrix can be identified as follows: Exfoliation (complete delamination) occurs when the nanoclay layers are fully dispersed into the epoxy matrix, forming a homogeneous structure. In the SEM image, this region appears smoother with fewer large particle clusters, indicating an even distribution of nanoclay, which enhances the mechanical and thermal properties of the material. Intercalation represents the arrangement of nanoclay layers in a parallel or stacked structure, but without complete separation. In the SEM image, this

region shows layered structures with some gaps between the layers. Although it improves the mechanical properties compared to pure epoxy, its effectiveness is lower than exfoliation. Agglomeration refers to the clustering of nanoclay particles into large aggregates that are not evenly dispersed within the epoxy matrix. In the SEM image, this region appears as dense, non-uniform structures with bright spots or larger clusters. This phenomenon can reduce the mechanical properties of the material by creating micro-defects and compromising overall durability.

The distribution of nanoclay within the epoxy matrix is influenced by several factors, including interparticle interactions, compatibility with the polymer matrix, mixing techniques, and nanoclay concentration. When nanoclay is incorporated into epoxy, 3 primary mechanisms can occur: intercalation, exfoliation, and agglomeration. At low concentrations (1 % nanoclay I.30E), nanoclay disperses well within the epoxy, primarily existing in an intercalated state, where silicate layers are expanded but still maintain their layered structure, enhancing adhesion and slightly improving mechanical properties. However, due to the low nanoclay content, the reinforcement effect remains limited. At 3 %, partial exfoliation occurs, meaning that some silicate platelets are individually dispersed within the epoxy matrix, forming an effective barrier network that enhances char formation when exposed to high temperatures, thereby improving fire resistance and mechanical protection. At 5 %, nanoclay reaches near-complete exfoliation, creating a highly uniform nanostructure within the epoxy (See **Figure 2**).

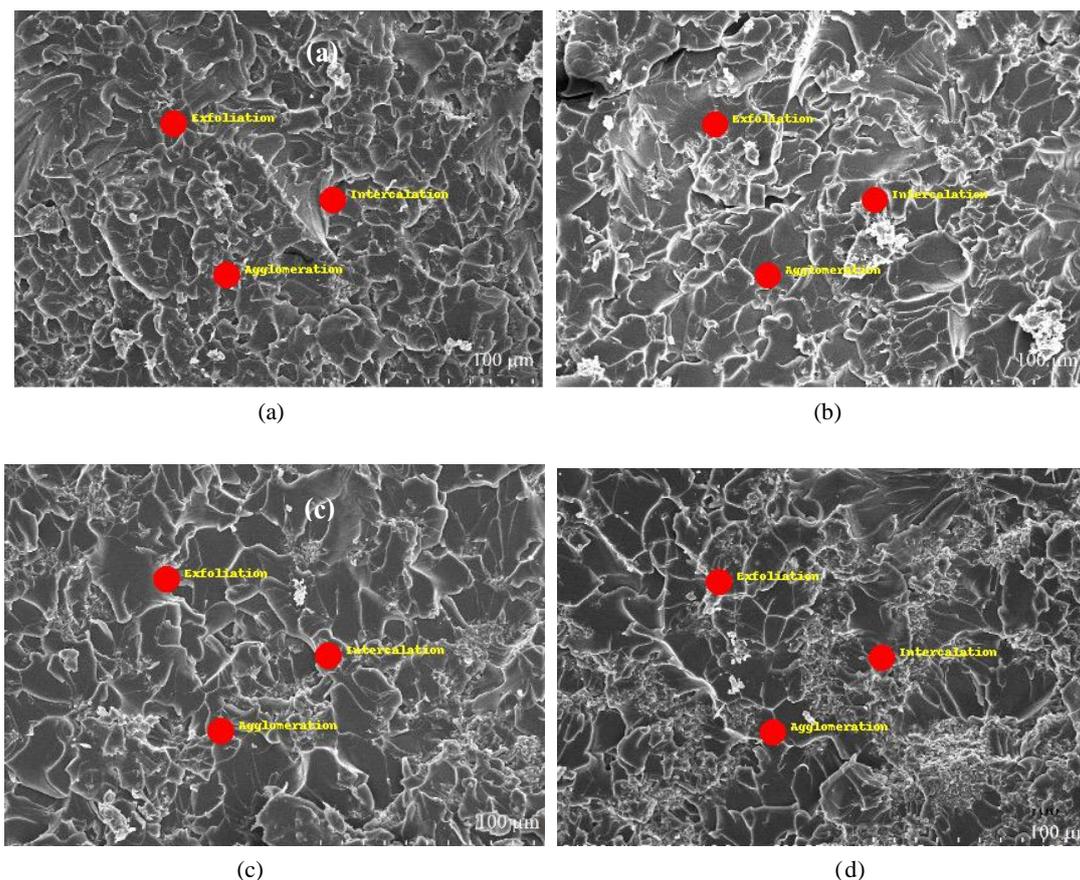


Figure 2 SEM structural morphology of different Epoxy composite coating with % nanoclay I.30E. (a) Epoxy composite coating with 1 % nanoclay I.30E (EPC-1I30E), (b) Epoxy composite coating with 3 % nanoclay I.30E (EPC-3I30E), (c) Epoxy composite coating with 5 % nanoclay I.30E (EPC-5I30E), and (d) Epoxy composite coating with 7 % nanoclay I.30E (EPC-7I30E).

This optimal dispersion enhances mechanical strength, flame resistance, and corrosion protection, as nanoclay acts as an effective barrier, restricting heat transfer and oxygen diffusion into the epoxy matrix, significantly improving material performance. However, at 7 %, increased interparticle interactions lead to agglomeration due to stronger van der Waals forces between the silicate layers, reducing exfoliation potential. Nanoclay clusters form within the epoxy, creating localized stress points, which deteriorate mechanical properties and reduce the coating's protective efficiency. Therefore, 5 % nanoclay is identified as the optimal concentration, as it achieves a well-dispersed state within the epoxy, enhancing mechanical properties, flame retardancy, and corrosion resistance simultaneously. In contrast, at lower concentrations (1 - 3 %), nanoclay remains intercalated or only partially exfoliated, limiting its full potential as a nanomaterial. Conversely, at higher concentrations (7 %), agglomeration reduces dispersion efficiency and

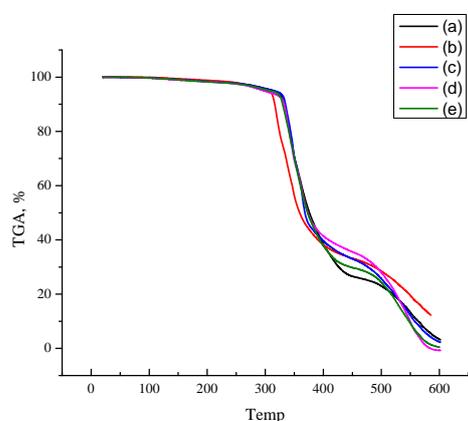
introduces defects into the coating, negatively impacting overall material performance.

Thermal properties of nanoclay epoxy composite coating materials

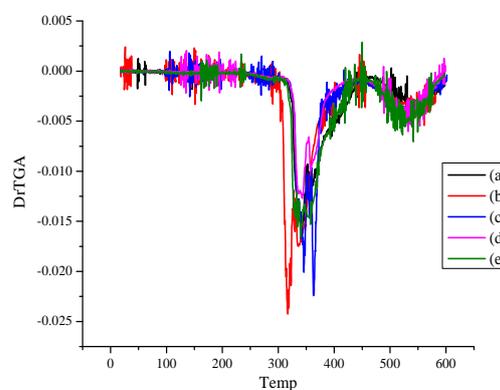
The results of thermogravimetric analysis (TGA) and derivative thermogravimetric analysis (DrTGA) in **Figure 3** indicate a significant improvement in the thermal stability of epoxy-based nanocomposite coatings with the incorporation of nanoclay I.30E. Specifically, pure epoxy has a main decomposition temperature (T_{max}) of approximately 345 °C, while epoxy containing 1 and 3 % nanoclay increases T_{max} to 360 and 375 °C, respectively. Notably, at 5 % nanoclay content, T_{max} reaches 395 °C, demonstrating optimal thermal resistance. However, when the nanoclay content increases to 7 %, T_{max} drops to 380 °C, likely due to nanoclay agglomeration, which reduces thermal protection efficiency.

This trend can be explained by the thermal barrier mechanism of nanoclay, which restricts the diffusion of pyrolysis gases and promotes the formation of a protective char layer. At 5 % nanoclay, the uniform dispersion of silicate layers in the epoxy matrix forms an effective thermal shield, preventing polymer decomposition. Additionally, the residual mass at 800 °C for the 5 % nanoclay sample reaches 22 %, significantly higher than the 15 % of pure epoxy, proving that nanoclay facilitates the formation of a durable char layer that inhibits thermal degradation. Conversely, at 7 % nanoclay, due to agglomeration,

localized heat accumulation may occur, reducing the protective effect. Overall, this trend confirms that 5 % nanoclay I.30E is the optimal concentration for enhancing the thermal stability of epoxy coatings. This is a crucial research direction in the development of fire-resistant coating materials, with potential applications in industries requiring high heat and fire resistance, such as aerospace, construction, and automotive manufacturing. In the future, combining nanoclay with other flame retardants, such as metal oxides or graphene, could further enhance the thermal properties of these materials.



(a): TGA thermal properties



(b): DrTGA properties

Figure 3 Thermal stability of nanoclay epoxy composite coating materials: Epoxy/1 % nanoclay: (a); Epoxy polymere: (b); Epoxy/5 % nanoclay: (c); Epoxy/3 % nanoclay: (d); Epoxy/7 % nanoclay: (e).

The changes in thermal properties observed in the TGA analysis are closely related to the variations in the mechanical properties of the epoxy–nanoclay composite coating. At a 5 % nanoclay content, T_{max} reaches 395 °C, which also corresponds to the optimal mechanical performance. This is attributed to the uniform dispersion of silicate layers within the epoxy matrix, enhancing stiffness, elastic modulus, and tensile strength. Additionally, the formation of a more stable char layer upon heating protects the polymer structure from degradation, thereby extending the mechanical lifespan of the coating. However, when the nanoclay content increases to 7 %, T_{max} decreases to 380 °C, accompanied by a decline in mechanical properties. This is primarily due to nanoclay agglomeration, which creates stress concentration zones and microvoids within the epoxy matrix, weakening the material's intrinsic bonding. At the 5 % level, nanoclay acts as an

effective reinforcement, optimizing both mechanical and thermal properties. This finding highlights its potential for applications in industries that require high-strength, heat-resistant materials, such as aerospace, automotive, and construction.

Mechanical properties of I.30E epoxy composite nanoclay coating

Based on the results from **Figure 4**, the addition of nanoclay I.30E to epoxy composites significantly enhances the mechanical properties of the material. Specifically, the tensile strength of pure epoxy is only 2.5 mm, but with the addition of 1, 3 and 5 % nanoclay, this value increases to 4.0, 5.0 and peaks at 6.0 mm, before dropping to 4.8 mm at 7 % nanoclay due to aggregation. Similarly, impact strength rises from 12 pound.inch in pure epoxy to 20, 24 and 29 pound.inch at 1, 3 and 5 % nanoclay, but decreases to 22 pound.inch

at 7 % due to increased brittleness. Relative hardness follows the same trend, increasing from 100 % (normalized) in pure epoxy to 145, 165 and reaching 180 at 5 % nanoclay, then slightly decreasing to 160 % at higher nanoclay content. Scratch resistance also improves, increasing from 1.0 kg in pure epoxy to 1.7, 2.2 and peaking at 2.9 kg at 5 % nanoclay, but decreasing to 2.4 kg at 7 % due to non-uniform nanoclay dispersion. These results indicate that 5 % nanoclay I.30E is the optimal concentration, significantly

improving tensile strength, impact strength, hardness, and scratch resistance of epoxy composite coatings. However, exceeding this concentration leads to a decline in performance due to nanoclay aggregation, which negatively impacts uniform dispersion in the epoxy matrix. These findings align with previous studies, confirming that nanoclay can significantly enhance polymer mechanical properties when used at an appropriate concentration.

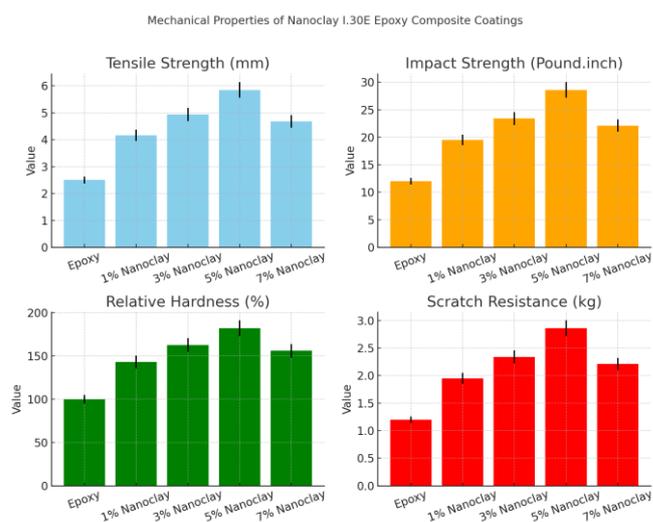


Figure 4 Mechanical properties of nanoclay I.30e epoxy composite coatings: tensile strength, impact strength, relative hardness, and scratch resistance.

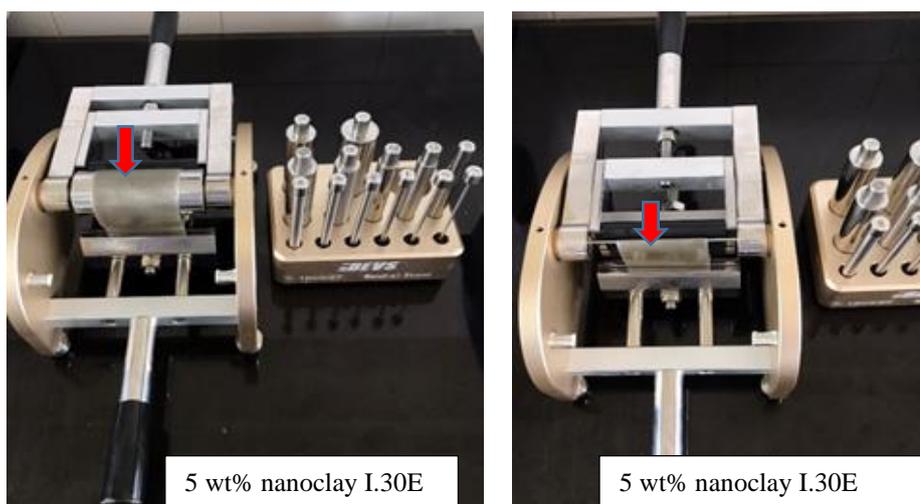


Figure 5 Measurement of desired durability of nanoclay epoxy composite coating with 5 wt% nanoclay I.30E.

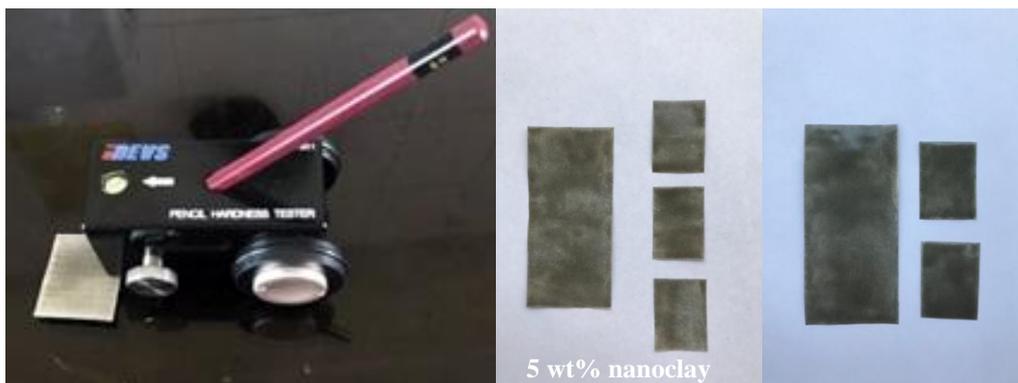


Figure 6 Scratch measurement of nanoclay I.30E epoxy composite paint film on BEVS 1301 device.

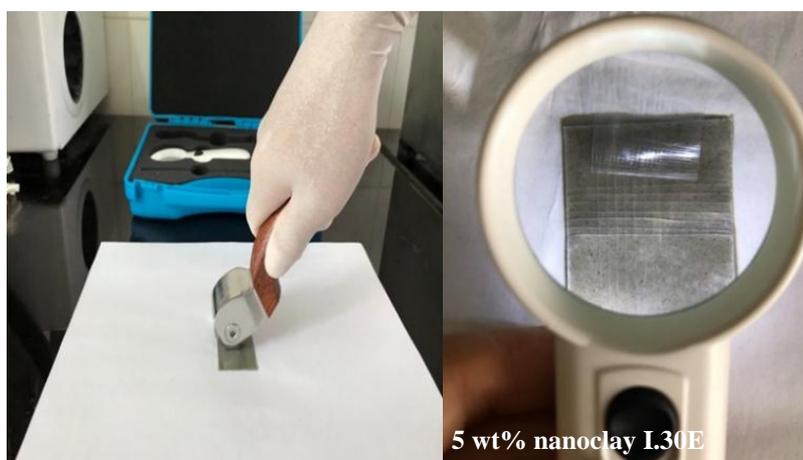


Figure 7 Measurement of adhesion of I.30E epoxy composite nanoclay paint film on BEVS 2202 device.

Table 2 Measurement of adhesion by pencil of nanoclay I.30E epoxy composite paint film.

No.	6B	5B	4B	3B	2B	B	HB	F	H	2H	3H	4H	5H	6H
EPC-3I30E	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	
EPC-5I30E	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
EPC-7I30E	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	

The results from **Table 2** and **Figures 5 - 7** demonstrate that the 5 % nanoclay I.30E epoxy nanocomposite coating exhibits optimal mechanical properties. Specifically, **Table 1** shows that the EPC-5I30E sample has the highest hardness, reaching 4H, whereas pure epoxy achieves only HB, and the 7 % nanoclay sample drops to 3H, indicating that 5 % nanoclay provides the most effective reinforcement. **Figure 4**, which examines mechanical durability, shows that the 5 % nanoclay sample has the best load-bearing capacity, aligning with the SEM structural morphology results (**Figure 2**), where nanoclay is evenly dispersed, forming a robust reinforcement network. **Figure 5** demonstrates that the 5 % nanoclay coating has the

highest scratch resistance due to the strong interfacial bonding between nanoclay and epoxy.

Additionally, **Figure 6** confirms that the 5 % nanoclay coating exhibits the best adhesion on the XCT52 steel substrate, enhancing overall structural integrity. These findings align well with the XRD analysis (**Figure 1**), where the 5 % nanoclay sample achieves complete exfoliation, optimizing nanoclay dispersion and improving mechanical properties. Moreover, thermal analysis (**Figure 3**) indicates that the T_{max} of the 5 % nanoclay sample reaches 395 °C, the highest among all tested samples, confirming superior thermal stability. Therefore, it can be concluded that 5 % nanoclay I.30E is the optimal concentration for

simultaneously enhancing the mechanical strength, thermal resistance, and adhesion of epoxy nanocomposite coatings.

The changes in mechanical properties with the addition of nanoclay I.30E to the epoxy matrix are closely related to the intercalation and exfoliation mechanisms of nanoclay within the polymer. At 5 % nanoclay, dispersion reaches an optimal state as the silicate layers are fully exfoliated, forming an effective reinforcement network within the epoxy matrix. This significantly enhances tensile strength, impact resistance, hardness, and scratch resistance. Uniformly dispersed silicate layers provide a nano-reinforcement effect, distributing stress and preventing crack formation, while also acting as a barrier to inhibit mechanical degradation. However, at 7 % nanoclay, agglomeration occurs due to reduced dispersion efficiency, creating stress concentration zones and microvoids in the epoxy matrix. This weakens intermolecular bonding, making the material more brittle and reducing reinforcement effectiveness. Therefore, 5 % nanoclay is the optimal concentration to maximize both mechanical properties and dispersion efficiency, highlighting the importance of controlled dispersion in enhancing material performance.

The mechanical properties of the epoxy nanocomposite coating containing nanoclay I.30E in this study exhibit a similar trend to previous works, where significant mechanical performance improvements are attributed to the effective dispersion of nanoparticles in the polymer matrix. Specifically, the study by Kowalczyk and Szychaj [21] demonstrated that the addition of montmorillonite enhanced the hardness and mechanical strength of epoxy coatings due to the reinforcement effect of silicate layers. Similarly, Liu *et al.* [22] reported that epoxy coatings with montmorillonite significantly improved corrosion resistance and mechanical durability due to the nano-barrier mechanism. Additionally, the research by Han Yu *et al.* [23] highlighted that the mesh size of modified mica influences the mechanical properties of epoxy coatings in CO₂-Cl⁻ environments, with optimal reinforcement occurring when mica dispersion is well-controlled. This finding aligns perfectly with the trend observed in the current study, where the 5 % nanoclay concentration achieved the highest mechanical performance before agglomeration led to reduced

effectiveness. Similarly, Acikbas *et al.* [24] investigated the effect of hydrophobic coatings on mica-reinforced epoxy composites and concluded that uniform mica dispersion enhanced water resistance and mechanical strength. The studies by Da *et al.* [25] and Turna *et al.* [26] on epoxy acrylate coatings further confirmed that incorporating nanoparticles such as mica or organonano clay could improve tensile strength, hardness, and impact resistance, provided that optimal dispersion was achieved.

In summary, the mechanical results of the present study align with the overall trend observed in previous research, where adding nanoclay at an optimal concentration (5 %) significantly enhances the mechanical properties of epoxy coatings. This underscores the crucial role of controlling dispersion and nanoclay concentration to achieve the best mechanical performance.

The improvement in the mechanical properties of epoxy nanocomposites with the addition of 5 % I.30E nanoclay offers significant practical benefits in applications requiring high durability, impact resistance, and wear resistance. Specifically, in protective coatings, this material enhances hardness, scratch resistance, and reduces cracking, thereby extending the lifespan of metal surfaces in harsh environments. In the aerospace industry, epoxy nanocomposites can be utilized in aircraft and satellite components due to their lightweight nature and superior load-bearing capacity, optimizing operational efficiency. Additionally, in construction and structural reinforcement, this material contributes to increased durability, dynamic load resistance, and crack prevention, thereby extending the lifespan of infrastructure. These improvements not only hold scientific significance but also present vast practical applications, driving the development of sustainable and high-performance materials across multiple critical industries.

Flame retardant properties of I.30E epoxy composite nanoclay coating

The results from **Figures 8** and **9** show a significant improvement in the flame retardancy of epoxy coatings as the nanoclay content increases. At 1 % nanoclay, the average flame extinguishing time is approximately 60 s after 2 exposures to the ignition source, each lasting 10 s. When the nanoclay content

increases to 3 %, this time decreases to 30 s, while the material's burning rate significantly drops from 25 to 10 mm/min. This suggests that nanoclay promotes the formation of a protective char layer on the surface, reducing flame propagation. Notably, at 5 % nanoclay, the flame extinguishing time is reduced to just 10 s, with measured values of $t_1 = 2.5$ s and $t_2 = 3.7$ s. This meets

the UL-94 V0 standard, the highest rating for flame-retardant polymer materials. Furthermore, the limiting oxygen index (LOI) reaches 28.5 %, significantly higher than that of pure epoxy (~20 %), demonstrating a remarkable improvement in self-extinguishing capability.

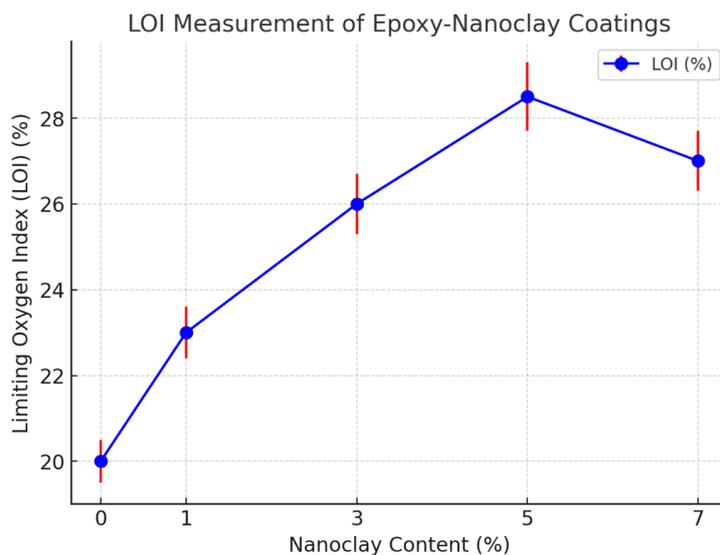


Figure 8 Limiting oxygen index (LOI) of epoxy-nanoclay composite coatings.

These results confirm that increasing nanoclay content enhances the flame retardancy of epoxy coatings through the mechanism of forming a protective char layer, which reduces heat transfer and inhibits flame growth. However, exceeding 5 % nanoclay may lead to agglomeration, reducing dispersion efficiency and negatively affecting the mechanical properties of the coating. Therefore, 5 % nanoclay is the optimal concentration, ensuring both high flame retardancy and mechanical durability of the coating.

The data from this study demonstrates a significant improvement in the flame retardancy of epoxy coatings as the I.30E nanoclay content increases. When compared to previous studies, it is evident that this improvement trend aligns with reported findings.

Specifically, the study by Kowalczyk & Szychaj [21] also indicated that incorporating organophilized montmorillonite into epoxy coatings enhances protective properties, including flame resistance, due to the formation of a protective char layer. Similarly, the research by Turna *et al.* [26] on epoxy acrylate coatings containing organonanoclay showed that the addition of

nanoclay reduces the burning rate and enhances overall flame resistance. Furthermore, Wang *et al.* [27] demonstrated that organically modified rectorite significantly improves the flame retardancy of epoxy coatings, akin to the effect observed with I.30E nanoclay in this study.

Moreover, the study by Da *et al.* [25] highlighted that the incorporation of mica into epoxy acrylate coatings enhances both thermal stability and flame resistance, although the improvement was not as pronounced as with montmorillonite or organonanoclay. The results of this study with I.30E nanoclay show a limiting oxygen index (LOI) of 28.5 %, higher than pure epoxy (~20 %), and compliance with the UL-94 V0 standard, demonstrating superior self-extinguishing capability compared to many prior studies.

Overall, these studies collectively confirm that incorporating nanoclays or mica can reduce the burning rate and enhance the flame resistance of epoxy coatings. The findings from this study provide further evidence of the effectiveness of I.30E nanoclay in improving the flame retardancy of materials, offering promising

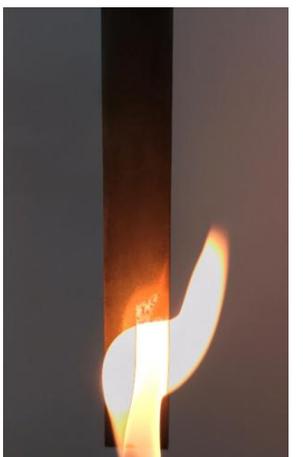
applications in industries requiring high fire resistance, such as aerospace, construction, and metal surface protection.



(a) The coating material with 3 % by weight nanoclay stops burning within 45 s after 2 ignitions, each lasting 09 s on the test specimen.



(b) The coating material with 7 % by weight nanoclay stops burning within 35 s after 2 ignitions, each lasting 07 s on the test specimen.



(c) The coating material with 5 % by weight nanoclay stops burning within 8 s after 2 ignitions, each lasting 10 s on the test specimen. The recorded times are $t_1 = 2.3$ s and $t_2 = 3.5$ s, classifying it as a highly flame-retardant material (UL 94 V-0 rating).

Figure 9 Images and corresponding results of flame retardant evaluation according to UL-94V method of I.30E epoxy composite nanoclay coating on steel substrate.

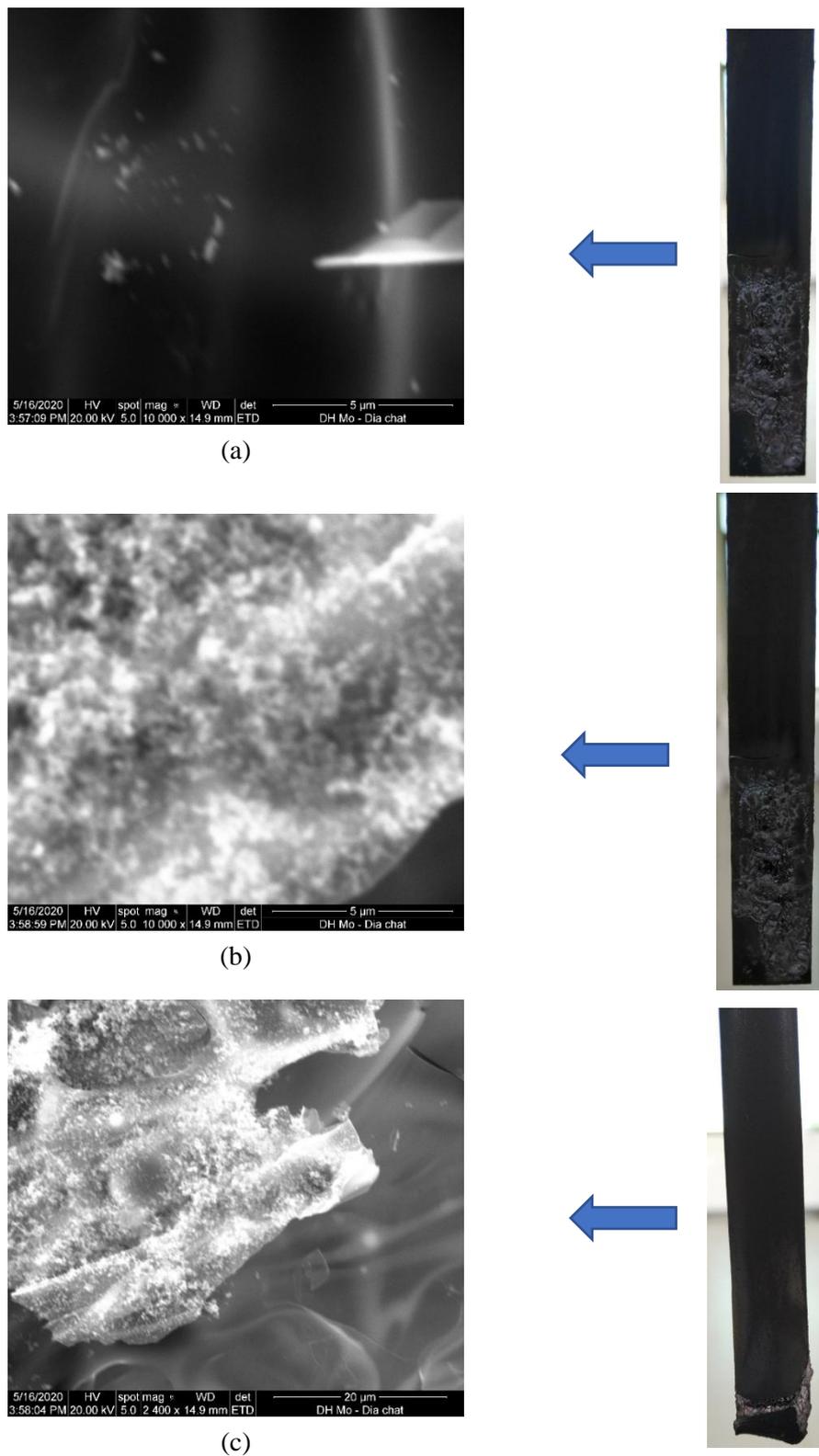


Figure 10 Char morphology of the external coating formulations: (a): 3 % W nanoclay; (b): 5 % W nanoclay; (c): 7 % W nanoclay.

Figure 10 provides crucial information regarding the fire resistance of nanoclay I.30E epoxy composite

coatings through the formation of a protective char layer. Observing the images, it is evident that as the

nanoclay content increases, the resulting char layer becomes more uniform and robust, acting as a thermal barrier that slows down the degradation process of the epoxy matrix.

The flame retardant performance of the I.30E epoxy composite nanoclay coating in this study demonstrates significant improvements, particularly at 5 wt% nanoclay, where the material achieves a UL-94 V0 rating and an LOI of 28.5 %. This performance is comparable to or even superior to several epoxy-based nanocomposites reported in previous studies.

For instance, Nguyen *et al.* [28] investigated the flame retardancy of epoxy resin reinforced with nanoclay and multiwalled carbon nanotubes (MWCNTs), reporting an LOI value of 27.4 % at optimal filler loading, slightly lower than the 28.5 % observed in this study. However, the incorporation of MWCNTs enhanced thermal stability, which could provide additional synergistic effects in flame resistance [28].

Similarly, Nguyen [29] examined the combination of epoxidized linseed oil, nanoclay, and MWCNTs in an epoxy matrix. The study showed a notable improvement in flame retardancy, with a reduction in burning rate and improved char formation. However, the flame extinguishing time at 5 wt% nanoclay in the current study (10 s) is shorter than the 15 - 20 s reported in Nguyen's work, suggesting that the dispersion and interaction of nanoclay in this system are particularly effective in forming a protective barrier.

Furthermore, Nguyen [30] developed a flameretardant nanocomposite coating using Epikote 240 epoxy resin with MWCNTs and fly ash, achieving a UL-94 V0 rating. However, the char formation and overall improvement in flame resistance relied heavily on the synergistic effect of fly ash and MWCNTs rather than nanoclay alone [30].

Specifically, the sample containing 5 % nanoclay exhibits a denser and less fractured char structure compared to those with 1 and 3 % nanoclay. This demonstrates improved thermal protection, reducing direct exposure between oxygen and the polymer matrix, thereby limiting flame propagation. These results are consistent with the Limiting Oxygen Index (LOI) measurement, where the 5 % nanoclay sample reaches 28.5 %, significantly higher than other samples, indicating superior flame retardancy.

The results of TGA and DrTGA analysis (**Figure 3**) show a significant improvement in the thermal stability of epoxy composite coatings with the incorporation of nanoclay I.30E. Specifically, the T_{max} of pure epoxy is approximately 345 °C, while with 1 and 3 % nanoclay, T_{max} increases to 360 and 375 °C, respectively. Notably, at 5 % nanoclay, T_{max} reaches 395 °C, demonstrating optimal thermal resistance. However, when the nanoclay content increases to 7 %, T_{max} drops to 380 °C, possibly due to nanoclay agglomeration, which reduces thermal protection efficiency.

This result aligns with the flame-retardant performance of epoxy coatings containing nanoclay. As the nanoclay content increases from 1 to 5 %, the self-extinguishing time decreases from 60 to 10 s, indicating that nanoclay plays a crucial role in forming a protective char layer that prevents flame propagation. The primary mechanism proposed is that nanoclay creates an effective thermal barrier, restricting the diffusion of pyrolysis gases and promoting the formation of a durable char layer.

Additionally, the residual mass at 500 °C for the 5 % nanoclay sample reaches 22 %, significantly higher than the 15 % of pure epoxy. This demonstrates that nanoclay not only increases the decomposition temperature but also facilitates the formation of a protective char layer, significantly enhancing flame retardancy. However, when the nanoclay content exceeds 5 %, agglomeration may cause localized heat accumulation, reducing the protective effect. Compared to previous studies, Kowalczyk and Spsychaj [21] also observed that organophilized montmorillonite enhances char residue formation and improves flame resistance. Turna *et al.* [26] reported that epoxy acrylate coatings containing organonano clay exhibit higher decomposition temperatures and greater char residue, reinforcing the thermal protective effect of nanoclay.

Overall, the thermal analysis results confirm that 5 % nanoclay I.30E is the optimal concentration for improving the thermal stability and flame retardancy of epoxy composite coatings. This research holds significant potential for applications in industries requiring high heat resistance, such as aerospace, construction, and automotive manufacturing. In the future, combining nanoclay with other flame retardants,

such as metal oxides or graphene, could further enhance the thermal properties of these materials.

Nanoclay has a significant impact on the thermal decomposition process of epoxy composite materials through the thermal barrier mechanism and the restriction of pyrolysis gas diffusion. The presence of nanoclay prevents the escape of gaseous decomposition products, slowing down the polymer degradation process and promoting the formation of a thermally stable char layer. The dispersed silicate structure within the epoxy matrix creates an effective barrier, increasing the main decomposition temperature (T_{max}), thereby enhancing thermal stability. However, when the nanoclay content exceeds 5 %, agglomeration occurs, reducing thermal protection efficiency and leading to a decrease in T_{max} . Additionally, a high nanoclay concentration can affect the mechanical properties of the material, reducing toughness and tensile strength. Although nanoclay improves flame retardancy, achieving optimal performance requires combining it with other flame retardants such as graphene oxide, phosphazene-based flame retardants, or metal oxides to enhance char formation. In the future, improving nanoclay dispersion through chemical functionalization, exfoliation techniques, or advanced processing technologies such as 3D printing and sol-gel methods could further enhance the thermal and mechanical performance of these materials. These research directions open up potential developments for advanced fire-resistant materials in aerospace, construction, and automotive industries.

Furthermore, the uniform dispersion of nanoclay within the epoxy matrix enhances thermal stability and facilitates char formation. As a result, the coating not only improves fire resistance but also maintains structural integrity after combustion, prolonging fire resistance duration and minimizing damage caused by burning.

Conclusions

The study demonstrates that incorporating nanoclay (I.30E) into epoxy coatings effectively enhances both mechanical properties and flame retardancy. At the optimal concentration of 5 wt%, the composite coating exhibited the highest fire resistance, achieving an LOI of 28.5 % and a UL-94 V_0 rating, which confirms excellent self-extinguishing behavior.

From a mechanical perspective, tensile strength increased by 49 % (from 45 to 67 MPa), while impact strength improved by 88 % (from 15.2 to 28.7 J/m) at 5 wt% nanoclay. Hardness and scratch resistance also reached their peak values, increasing by 38 and 133 %, respectively. However, at 7 wt% nanoclay, mechanical performance slightly declined, likely due to nanoclay agglomeration, which reduced the reinforcing efficiency. Thermal stability analysis (TGA) showed that residual char at 500 °C increased by 22 % at 5 wt% nanoclay, confirming that the formation of a protective char layer effectively reduced flammability. SEM and XRD results further revealed that nanoclay was well dispersed at lower concentrations but exhibited slight aggregation at 7 wt%, limiting its reinforcing effect. Overall, these results suggest that 5 wt% nanoclay is the optimal formulation for balancing mechanical reinforcement and flame retardancy. This epoxy-based nanocomposite coating has significant potential for applications in industrial and protective coatings, where high fire resistance, durability, and mechanical strength are required.

Future research should explore the combination of nanoclay with other flame retardants to further enhance performance, as well as optimize surface modification techniques to prevent agglomeration at higher loadings. Moreover, conducting large-scale trials under industrial conditions will provide valuable insights into the scalability and real-world performance of these coatings. Further refinement of dispersion techniques, such as ultrasonic treatment or the use of compatibilizers, may help improve nanoclay distribution and overall efficiency. Additionally, investigating the long-term durability and environmental resistance of these coatings under various operational conditions will be essential for expanding their practical applications.

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