Polímeros
ISSN 1678-5169 (Online)

https://doi.org/10.1590/0104-1428.20250032

Properties of epoxy polymer materials reinforced with boric acid and nano silica

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Abstract

Boric acid (BA) and nano silica (NS) were used to improve the properties of composite materials based on epoxy resin. The structural and thermal characteristics of materials were evaluated by using Scanning Electron Microscopy (SEM), Transmission Electron Microscopy (TEM), Thermogravimetric Analysis (TG), Derivative Thermogravimetry (dTG), and Differential Calorimetry (DSC). The mechanical properties of polymer films were evaluated through the parameters of impact strength, flexibility, adhesion, and hardness. The results showed that the content of 4.0 wt% BA increased the impact strength of the epoxy polymer film (increased by 38%) and increased the thermal properties of epoxy composite materials (increased by 14%). Combining 4.0 wt% BA and 1.0 wt% NS, the thermal properties of the epoxy composite polymer material increased by 18.5%. The epoxy coating was reinforced with 4.0 wt% BA and 1.0 wt% NS achived high thermal stability with the strongest decomposition temperature at 494 °C.

Keywords: *boric acid, nano silica, thermal properties, mechanical properties, epoxy coatings.*

Data Availability: Research data is available upon request from the corresponding author.

How to cite: Le Huy, C. H., Thanh, A. T., & Nhat, H. N. (2025). Properties of epoxy polymer materials reinforced with boric acid and nano silica. *Polímeros: Ciência e Tecnologia*, *35*(4), e20250047. https://doi.org/10.1590/0104-1428.20250032

1. Introduction

Epoxy resins have been widely used in coatings, composites, adhesives, and molding materials due to their excellent chemical resistance, strong adhesion, and anti-corrosion properties. However, owing to the presence of many aromatic rings in their chemical structure, epoxy resins are typically hard and brittle. Various approaches have been developed to enhance the mechanical and thermal properties of epoxy resins, including the incorporation of nanoparticles and thermally stable compounds. Heat-resistant and environmentally friendly additives, such as antimony oxides, borate compounds, and phosphorus-based compounds have been widely applied in polymer materials[1-7]. In study[1], zinc borate was incorporated into an ethylene-vinyl acetate (EVA) copolymer, showing its role as a smoke suppressant and combustion-rate reducer. In another study[2], the addition of 1.0 wt% zinc borate to polypropylene resulted in the formation of a protective char layer during combustion, thereby improving its flame retardancy.

Study[3] reported that a combination of aluminum powder and boric acid enhanced both the thermal stability and mechanical properties of epoxy polymer materials. Similarly, study[4] found that the incorporation of 5.0 wt% zinc borate and 1.0 wt% nano silica significantly improved the mechanical properties and increased the thermal stability of epoxy composites by 4.7%. In study[5], polypropylene was reinforced with varying amounts of boric acid (1.25 wt%, 2.5 wt%, 3.75 wt%, and 5.0 wt%), in combination with 2.0 wt% montmorillonite (MMT) nano clay, ammonium polyphosphate (APP), and pentaerythritol (PER). The composition containing 2.5 wt% boric acid exhibited the most effective fire resistance performance.

To improve the thermal stability and flame retardancy of polymer materials, halogen-based and phosphorus-based flame retardants have been commonly used. However, the application of halogen-based compounds is increasingly restricted due to the release of toxic by-products during combustion[6-8]. In contrast, phosphorus-based flame retardants offer both high fire resistance and environmental friendliness[8-21].

Numerous studies have also explored the use of nanocomposite materials reinforced with silica and clay. These studies have shown that the incorporation of appropriate amounts of nano silica or nano clay can enhance the physical and mechanical properties of polymer materials, as well as improve other performance characteristics[21-29].

Despite the extensive literature on the reinforcement of polymer materials, no published studies to date have examined the combined effects of boric acid and nano silica on the properties of epoxy composite materials.

In this study, the effects of boric acid and nano silica on the mechanical properties and thermal resistance of polymer materials and coatings based on Epotec YD011X75 epoxy resin and G5022 hardener were investigated.

2. Materials and Methods

2.1 Materials

The epoxy resin used in this study was Epotec YD011X75 (Aditya Birla Chemicals, Thailand), with an epoxy equivalent weight (EEW) ranging from 460 to 490 g/eq. The corresponding hardener was a polyamide-based curing agent, G5022 (Korea). Nano silica was used in the form of fumed silica (S5505, Sigma-Aldrich). Xylene (China) was used as the solvent.

Boric acid was used as a flame-retardant additive, is a fine white powder with a density of 1.435 g/cm^3 and a melting point of 170.9 °C. Additional materials included iron oxide (China), zinc phosphate (France), and talc filler powder (Talc 1250, Taiwan). BYK-110 (USA) was used as a wetting and dispersing additive, while Bentone SD1 (Belgium) was incorporated as a rheological modifier.

2.2 Preparation of samples

2.2.1 Dispersing boric acid (BA) into epoxy resin

A total of 100.0 g of Epotec YD011X75 epoxy resin was weighed into a 250 mL beaker, followed by the addition of 10.0 g of xylene as a solvent. The mixture was stirred at a speed of 500 rpm for 10 minutes to reduce the resin viscosity. Subsequently, 2.0 g of boric acid (BA) was added to the resin solution. The stirring speed was then increased to 2000 rpm and maintained for 60 minutes. The resulting resin mixture was subjected to ultrasonic treatment at a frequency of 50 kHz for 45 minutes. The BA content varied at 2.0 wt%, 4.0 wt%, and 6.0 wt% relative to the weight of the Epotec YD011X75 epoxy resin.

2.2.2 Disperse boric acid (BA) and nano silica into epoxy resin

A total of 100.0 g of epoxy resin and 1.0 g of nano silica were weighed into a 250 mL beaker. Subsequently, 10.0 g of xylene was added as a solvent. The mixture was stirred at 500 rpm for 15 minutes, then the stirring speed gradually increased to 2000 rpm and maintained for 60 minutes. Ultrasonic treatment was then applied at a frequency of 50 kHz for 45 minutes.

Following this, 4.0 g of boric acid (BA) was added to the mixture, which was stirred at 500 rpm for 10 minutes. The stirring speed was then gradually increased to 2000 rpm and maintained for an additional 60 minutes, followed by ultrasonic vibration at a frequency of 50 kHz for 30 minutes.

2.3 Devices and analysis methods for polymer composite materials

The dispersion process was carried out using mechanical stirrers (IKA RW-20, Germany) and an ultrasonic device (Vibram Cell, model CV334, USA). Surface morphology was examined using a Scanning Electron Microscope (SEM, JEOL JSM-IT200, Japan). The thermal properties of the polymer composite materials were analyzed using Thermogravimetric Analysis and Differential Scanning Calorimetry (TG-DSC-DTG) on a Setaram Labsys Evo system (TG-DSC 1600 °C).

The properties of epoxy paints and coatings were evaluated following the TCVN 9014:2011 standard[33]. Mechanical properties of the coating films were assessed based on relevant ISO, ASTM, and JIS standards. Specifically, the flexibility of the films was tested according to ISO 1519:2002 using an Erichsen tester (model 266). Impact strength was evaluated following ISO 6272:2002 (Erichsen, model 304). Adhesion was measured following the ASTM D3359-93 standard (Erichsen, model 295). Pencil hardness was assessed according to the JIS K5400-90 standard. The dry film thickness was measured using a MiniTest 3100 device in compliance with TCVN 9406:2012.

3. Results and Discussions

3.1 Effects of boric acid on the mechanical properties of epoxy composite materials

The Epotec YD011X75 epoxy resin was cured using the G5022 curing agent at a mass ratio of 100:33 (wt/wt) (epoxy resin:curing agent), as reported in study[30]. The curing process occurs through a chemical reaction between the epoxy groups of the resin and the amine groups of the curing agent, resulting in a crosslinked thermosetting network.

The curing degree was evaluated by determining the gel content, following the method described in study[30]. In this method[30], 100 parts by mass of Epotec YD011X75 epoxy resin were mixed with 33 parts by mass of G5022 curing agent. The gel content is the insoluble fraction of the cured material after extraction with acetone solvent and shows the extent of crosslinking. The gel content determination was performed in a manner consistent with that described in study[29].

After curing, the samples were stabilized at room temperature for 72 hours prior to characterization.

Boric acid (BA) was incorporated into the epoxy resin at a concentration of 2.0 wt% (as described in Section 2.2) using mechanical stirring followed by ultrasonic dispersion. The effectiveness of BA dispersion within the epoxy matrix was evaluated through Scanning Electron Microscopy (SEM). The SEM micrograph of the Epotec YD011X75/G5022/2.0 wt% BA epoxy composite is presented in Figure 1.

The SEM image in Figure 1 shows that boric acid (BA) was uniformly dispersed within the Epotec YD011X75/G5022 epoxy polymer matrix.

The effects of varying BA content on the mechanical properties of the Epotec YD011X75/G5022 epoxy coating film were evaluated through measurements of impact strength, adhesion, pencil hardness, and flexibility. The corresponding results are summarized in Table 1.

According to ISO 1519:2002 (Erichsen, model 266), the flexibility of polymer coating films is determined by bending the films over cylindrical metal shafts with diameters ranging from 2 mm to 20 mm. The flexibility is expressed as the smallest shaft diameter over which the coating film can be bent without sustaining visible damage.

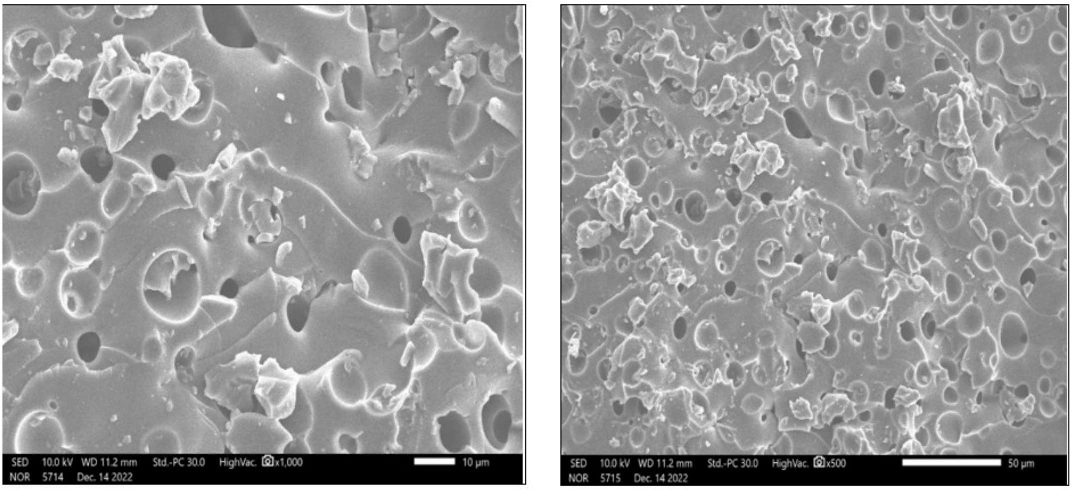

Figure 1. SEM image of the Epotec YD011X75/G5022/2.0%BA epoxy polymer.

Table 1. Mechanical properties of the Epotec YD011X75/G5022/BA epoxy coating film samples.

Sample	Epotec Epoxy Polymer	Dry film thickness (µm)	Impact strength (kg.cm)	Flexibility (mm)	Pencil hardness	Adhesion
BA0	YD011X75/G5022	128.38	32.5	4	6H	5B
BA2	YD 011X75/G5022/2.0%BA	94.42	40	2	4H	5B
BA4	**YD 011X75/G5022/4.0%BA**	**104.56**	**45**	**2**	**4H**	**5B**
BA6	YD 011X75/G5022/6.0%BA	118.72	42.5	2	5H	5B

The impact strength of the coating films was evaluated following ISO 6272:2002 (Erichsen, model 304), which measures the resistance of the film to mechanical impact. The value is reported in kg.cm, representing the maximum height (in centimeters) from which a 1.0 kg weight can freely fall onto the coated specimen without causing damage. For example, an impact strength of 45 kg.cm indicates that the coating can withstand the free fall of a 1.0 kg mass from a height of 45 cm without failure.

Adhesion was assessed according to ASTM D3359-93 (Erichsen, model 295) by making a grid of 1 mm × 1 mm cut on the coating surface, applying adhesive tape, and then evaluating the percentage of coating removed upon tape detachment.

Pencil hardness was measured following JIS K5400-90, which determines the hardest pencil capable of scratching the surface of the film, thereby indicating its surface hardness.

Dry film thickness was measured according to ASTM D1005 or TCVN 9406:2012 (MiniTest 3100) using an electronic thickness gauge with a magnetic induction probe, suitable for magnetic metal substrates such as iron and steel. Prior to measurement, the instrument was calibrated using a standard reference film. The probe was gently pressed against the surface until the reading stabilized (approximately 2-3 seconds).

The results summarized in Table 1 show that the unmodified Epotec YD011X75/G5022 epoxy coating film (sample BA0) exhibited excellent adhesion (5B), high pencil hardness (6H), and acceptable flexibility (bending diameter of 4 mm). However, its impact strength was relatively low at 32.5 kg.cm. This is attributed to the inherent brittleness of epoxy resins, which, due to their highly crosslinked structure and high aromatic content, possess limited flexibility[26-30].

To enhance the mechanical performance, boric acid (BA) was introduced as a reinforcing filler. Its effect on the mechanical properties was clearly observed. The sample containing 4.0 wt% BA (BA4) showed a significant increase in impact strength, rising from 32.5 kg.cm to 45 kg.cm (an improvement of 38%). This enhancement is attributed to the ability of BA to act as a reinforcing agent, contributing to the formation of additional physical interactions between the epoxy matrix and the curing agent[29,30]. The presence of an optimal BA content improved the interfacial bonding and dispersion, thereby enhancing the mechanical strength (as seen in BA2 and BA4 samples). However, when the BA content was increased to 6.0 wt% (BA6), the mechanical performance deteriorated. This reduction is likely due to the excess BA disrupting the network structure of the epoxy resin, resulting in regions lacking sufficient resin to form effective bonds, which led to decreased cohesion and poorer mechanical integrity.

Based on the results in Table 1, the optimal boric acid content was determined to be 4.0 wt% (BA4), which significantly enhanced the mechanical properties of the Epotec YD011X75/G5022 epoxy coating. Therefore, the BA4 sample was selected for further investigation into the thermal properties of the epoxy composite and was compared to the unmodified sample (BA0).

3.2 Effects of boric acid on the thermal properties of the Epotec YD011X75/G5022 epoxy material

3.2.1 Thermal properties of the Epotec YD011X75/G5022 epoxy material

The thermal properties of the Epotec YD011X75/G5022 epoxy material were evaluated using thermogravimetric and derivative thermogravimetric (TG-dTG) analysis. Thermogravimetric analysis (TG, %) measures the change in sample mass as a function of increasing temperature, thereby indicating the material's thermal stability and decomposition behavior. Derivative thermogravimetry (dTG, %/min) quantifies the rate of mass loss over time, allowing for more precise identification of decomposition stages.

The results of the TG-dTG analysis are presented in Figure 2.

The results presented in Figure 2 indicate that the BA0 sample (epoxy composite without boric acid) began to decompose at approximately 100 °C, corresponding to the evaporation of residual solvent. The onset of significant thermal degradation, defined by a 5.0 wt% mass loss, occurred within the temperature range of 180-210 °C. The temperature at which the sample reached 50 wt% mass loss was 341 °C. The maximum rate of decomposition, recorded at 9.11 %/min, occurred at 377 °C.

The thermal resistance of epoxy resins originates from their crosslinked thermosetting structure, which is formed through the reaction between epoxy groups and amine groups in the curing agent. The high aromatic ring content in the epoxy backbone contributes significantly to the material's thermal stability[4,26-29].

In comparison, a previous study[4] reported that epoxy composite materials based on DER 671X75 resin and T31 curing agent began to decompose between 150 °C and 158 °C due to solvent evaporation, and showed a maximum decomposition temperature of 339.96 °C. This comparison highlights the superior thermal stability of the Epotec YD011X75/G5022 epoxy system used in this study.

Thermal properties are a critical parameter reflecting the thermal stability and degradation behavior of thermosetting polymers, which is of great significance for applications in high-temperature and extreme environments. Thermoset epoxy resins are characterized by a chemically cross-linked network structure, along with a high content of aromatic rings, both of which contribute to their excellent thermal stability.

The results obtained from TG and dTG analyses in this study are in good agreement with those reported in the literature, including study[4] and previous works[26-29], thereby confirming the reliability of the thermal behavior observed for the Epotec YD011X75/G5022 epoxy system.

3.2.2 Thermal properties of the Epotec YD011X75/G5022/4.0 wt% BA epoxy material

The incorporation of 4.0 wt% boric acid (BA) was found to enhance the mechanical properties of the Epotec YD011X75/G5022 epoxy coating film, as previously demonstrated in Table 1. To further evaluate its thermal behavior, TG-dTG analysis was performed on the BA4 sample (Epotec YD011X75/G5022/4.0 wt% BA epoxy polymer), and the results are presented in Figure 3.

Chemical structure of acid boric was shown in Figure 4.

As shown in Figure 3, the BA4 sample began to decompose at approximately 100 °C, corresponding to the evaporation of residual solvent. The onset of 5.0 wt% mass loss occurred at 142 °C. Additionally, the decomposition of boric acid was observed to begin at approximately 170 °C, a temperature at which boric acid (H_3BO_3) undergoes dehydration to form metaboric acid (HBO_2)[31,32].

The dTG curve revealed that the peak decomposition rate occurred at 430 °C, with a maximum mass loss rate of 10.2%/min. This shows an enhancement in thermal resistance compared to the unmodified epoxy system (BA0), suggesting that the incorporation of BA contributes to improved thermal stability, likely due to its flame-retardant and reinforcing characteristics.

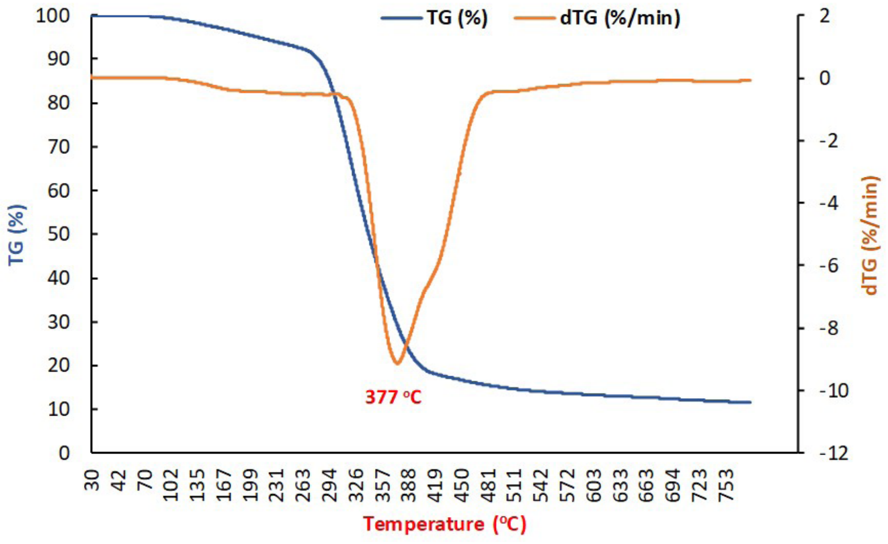

Figure 2. TG-dTG curve of the Epotec YD011X75/G5022 epoxy material.

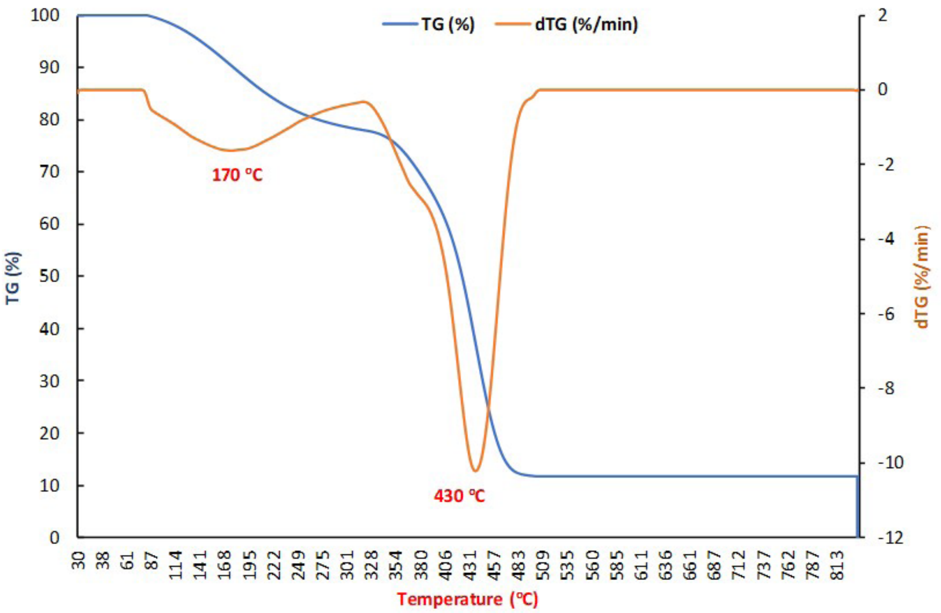

Figure 3. TG-dTG analysis of the Epotec YD011X75/G5022/4.0%BA epoxy polymer material.

The thermal stability of boric acid can be attributed to its flame-retardant mechanism, which primarily acts in the gas phase[31,32]. When the temperature reaches approximately at 170 °C, boric acid (H_3BO_3) undergoes thermal decomposition to form metaboric acid (HBO_2) and water, according to the following reaction:

$$H_3BO_3 \rightarrow HBO_2 + H_2O \quad (1)$$

The released water plays a critical role in suppressing combustion by diluting flammable gases and reducing the temperature in the combustion zone. As the temperature continues to rise above 300 °C, metaboric acid further decomposes to form tetraboric acid (also known as pyroboric acid, $H_2B_4O_7$), while releasing more water:

$$4\ HBO_2 \rightarrow H_2B_4O_7 + H_2O \quad (2)$$

At even higher temperatures, $H_2B_4O_7$ decomposes into boron trioxide (B_2O_3) and water:

$$H_2B_4O_7 \rightarrow 2B_2O_3 + H_2O \quad (3)$$

Boron trioxide is a thermally stable, non-flammable compound that can form a glass layer on the material surface, effectively acting as a barrier to heat and oxygen. Additionally, the release of water and the reduction of available oxygen at the combustion interface contribute to the inhibition of flame propagation[31,32].

As a result of this flame-retardant mechanism, the incorporation of 4.0 wt% boric acid (BA) significantly enhanced the thermal stability of the Epotec YD011X75/G5022 epoxy composite. The maximum decomposition temperature increased from 377 °C for the unmodified sample (BA0) to 430 °C for the BA4 sample, representing an improvement of approximately 14%.

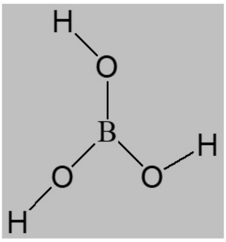

Figure 4. Chemical structure of boric acid.

3.3 Effects of nano silica on the mechanical and thermal properties of the Epotec YD011X75/G5022 epoxy material

Nano silica was incorporated into the Epotec YD011X75 epoxy resin using a combination of mechanical stirring and ultrasonic dispersion. Specifically, the mixture was initially stirred at 500 rpm for 15 minutes, followed by an increase in stirring speed to 2000 rpm for 60 minutes. The dispersion process was then enhanced by ultrasonic treatment for 45 minutes at a frequency of 50 kHz. This method was adapted from several previously published studies[4,26,28,29].

The dispersion quality of nano silica within the epoxy matrix was confirmed by transmission electron microscopy (TEM). The TEM image showing the distribution of 1.5 wt% nano silica in the Epotec YD011X75 epoxy resin is presented in Figure 5.

The TEM image in Figure 5 shows that nano silica particles were well dispersed within the Epotec YD011X75 epoxy resin matrix at the nanoscale, forming a uniform nanocomposite structure. The combination of mechanical stirring and ultrasonic vibration proved to be highly effective for dispersing nano silica into the epoxy resin. These findings are consistent with those reported in previous studies[4,26-30].

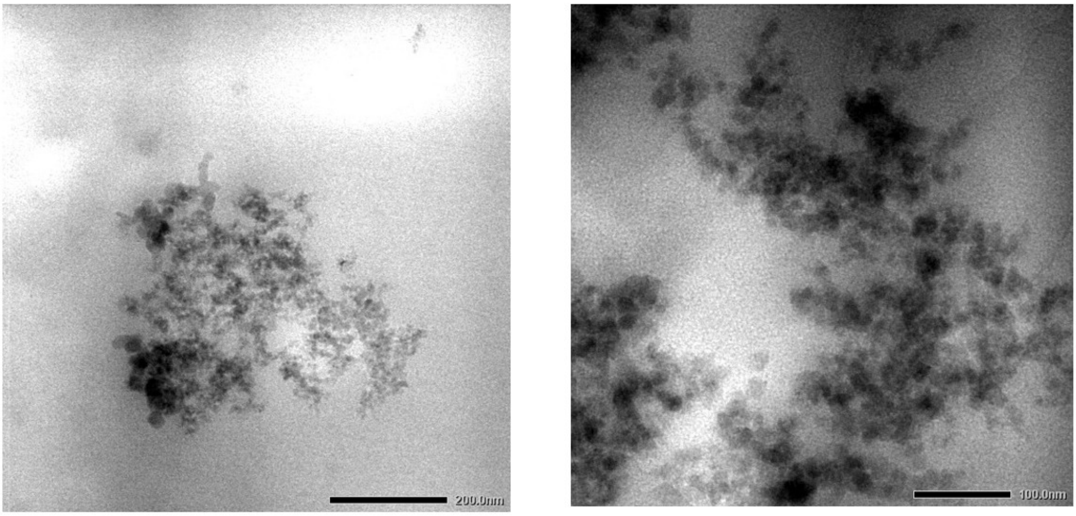

Figure 5. TEM image of the Epotec YD011X75/G5022/1.5% nano silica epoxy polymer.

Table 2. The mechanical properties of the Epotec YD011X75/G5022/nano silica epoxy nanocomposite coating films.

Sample	Epotec Epoxy Polymer	Dry film thickness (μm)	Impact strength (kg.cm)	Flexibility (mm)	Pencil hardness	Adhesion
BA0	YD011X75/G5022	128.38	32.5	4	6H	5B
BA0_Si0.5	YD011X75/G5022/0.5%NS	114.5	40	3	6H	5B
BA0_Si1.0	YD011X75/G5022/1.0%NS	**125.7**	**50**	**2**	**5H**	**5B**
BA0_Si1.5	YD011X75/G5022/1.5%NS	108.8	47.5	2	5H	5B
BA0_Si2.0	YD011X75/G5022/2.0%NS	121.4	45	2	5H	5B

Following ultrasonic dispersion, the samples were stored under stable conditions for at least 72 hours before TEM imaging. The image confirms the absence of aggregation, showing that the nano silica particles remained uniformly distributed within the epoxy matrix.

The incorporation of silica nanoparticles significantly influenced the properties of the epoxy composite materials. Maintaining the nanoscale dispersion of silica was essential to achieving the desired enhancements in mechanical performance, which is a key feature of epoxy-based nanocomposites[4,26-30]. The epoxy resin acts as the continuous phase (matrix), effectively encapsulated and bonding with the dispersed nano silica particles, thereby improving the overall mechanical and physical properties of the resulting nanocomposite.

In this study, nano silica was added at various concentrations of 0.5 wt%, 1.0 wt%, 1.5 wt%, and 2.0 wt%, corresponding to the sample codes BA0_Si0.5, BA0_Si1.0, BA0_Si1.5, and BA0_Si2.0, respectively.

The mechanical properties of the Epotec YD011X75/G5022/nano silica epoxy nanocomposite coating films are summarized in Table 2.

The results of the mechanical property evaluation presented in Table 2 indicate that the incorporation of nano silica significantly enhanced the performance of the epoxy films. The nano silica-reinforced samples (BA0_Si0.5, BA0_Si1.0, BA0_Si1.5, and BA0_Si2.0) exhibited improved mechanical properties compared to the unreinforced BA0 sample. Among these, the BA0_Si1.0 sample, containing 1.0 wt% nano silica, demonstrated the most superior mechanical performance. Specifically, its impact strength increased from 32.5 kg.cm to 50 kg.cm, representing a 54% enhancement.

Moreover, the incorporation of 1.0 wt% nano silica also improved the flexibility of the Epotec YD011X75/G5022 epoxy film. These findings are in strong agreement with previous studies[4,26-30], which have reported similar reinforcing effects of nano silica in polymer matrices. Nano silica acts as a reinforcing filler by dispersing within the epoxy matrix and mitigating stress concentrations under load.

However, when the nano silica content was increased to 1.5 wt% and 2.0 wt%, a decline in mechanical performance was observed. This deterioration is attributed to the insufficient amount of epoxy resin to effectively encapsulate the increased quantity of silica nanoparticles. Furthermore, the high surface activity associated with the Si–O bonds promote the agglomeration of adjacent nanoparticles, leading to the formation of larger silica clusters. These agglomerated regions, which lack sufficient resin to ensure proper dispersion and bonding, result in localized stress points and. So, a reduction in impact strength[4,26-30].

3.4 Effects of boric acid and nano silica on the mechanical and thermal properties of the Epotec YD011X75/G5022/BA/nano silica epoxy composite material

The results from previous sections demonstrated that both boric acid (BA) and nano silica individually enhanced the mechanical and thermal properties of the Epotec YD011X75/G5022 epoxy system. When used in combination, boric acid and nano silica exhibited a synergistic effect, further improving the overall performance of the composite material.

To evaluate this combined effect, a composite formulation reinforced with 4.0 wt% boric acid and 1.0 wt% nano silica was prepared. The mechanical properties of the resulting epoxy coating film were determined and are summarized in Table 3.

The results presented in Table 3 indicate that the BA4_Si1.0 sample exhibited the most enhanced mechanical performance among all tested formulations. Specifically, the Epotec YD011X75/G5022 epoxy film reinforced with 4.0 wt% boric acid and 1.0 wt% nano silica achieved an impact strength of 52.5 kg.cm, a flexibility value of 2 mm, a pencil hardness of 4H, and adhesion rated at 5B.

All measured mechanical properties of the BA4_Si1.0 epoxy coating film meet or exceed the requirements specified in TCVN 9014:2011 for protective coatings. Notably, these values are higher than those observed for the BA0_Si1.0 and BA0 formulations, confirming the synergistic reinforcing effect of combining boric acid and nano silica.

Boric acid and nano silica individually act as effective reinforcing fillers and, when dispersed uniformly and properly bonded within the epoxy matrix, they contribute to significant improvements in mechanical strength and integrity. In the BA4_Si1.0 formulation, the appropriate ratio between the epoxy resin, boric acid, and nano silica facilitated the formation of a well-integrated network structure, enhancing the filler-matrix interaction. This optimized microstructure played a critical role in improving the overall mechanical properties of the epoxy coating film[4,26-30].

3.4.1 Thermal properties of the Epotec YD011X75/G5022/4.0 wt% BA/1.0 wt% nano silica epoxy polymer material

The thermal behavior of the Epotec YD011X75/G5022 epoxy polymer reinforced with 4.0 wt% boric acid and 1.0 wt% nano silica (BA4_Si1.0 sample) was evaluated through thermogravimetric analysis. The TG-dTG results are presented in Figure 6.

The results presented in Figure 6 show that the BA4_Si1.0 sample began to decompose at approximately 100 °C, which corresponds to the evaporation of residual solvent. The dTG curve revealed a decomposition stage occurring in the 140-180 °C range, consistent with the thermal decomposition of boric acid observed previously in Figure 3.

Table 3. The Mechanical properties of the Epotec YD 1001X75/G5022/4.0% BA/1.0% nano silica epoxy composite films.

Sample	Epotec Epoxy Polymer	Dry film thickness (µm)	Impact strength (kg.cm)	Flexibility (mm)	Pencil hardness	Adhesion
BA0	YD011X75/G5022	128.38	32.5	4	6H	5B
BA4	YD 11X75/G5022/4.0%BA	104.56	45	2	4H	5B
BA0_Si1.0	YD011X75/G5022/1.0%NS	125.7	50	2	5H	5B
BA4_Si1.0	**YD 011X75/G5022/4.0%BA/1.0%NS**	**114.9**	**52.5**	**2**	**4H**	**5B**

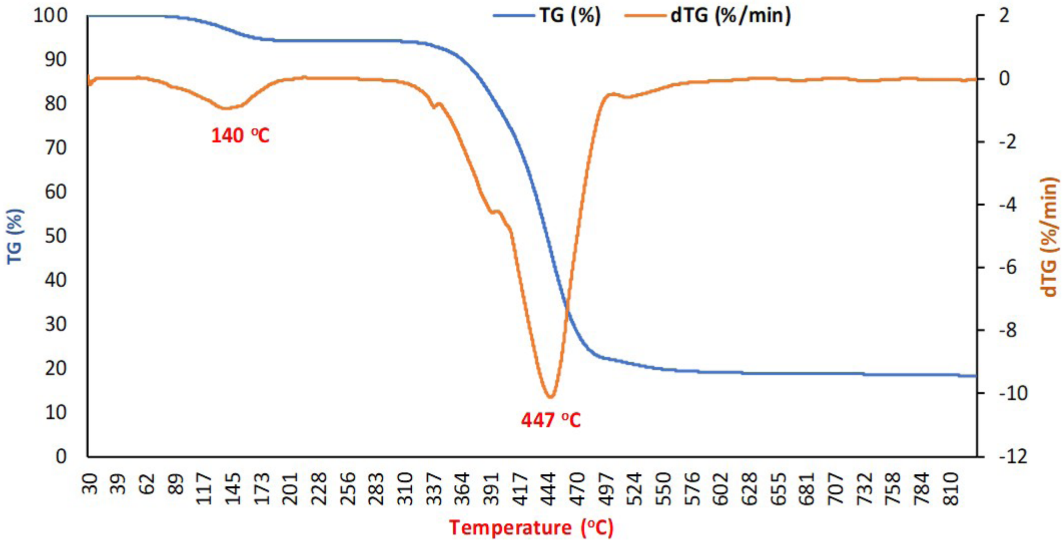

Figure 6. TG-dTG analysis of the Epotec YD011X75/G5022/4.0%BA/1.0% nano silica epoxy polymer material.

Table 4. The thermal properties of the Epotec YD011X75/G5022 epoxy polymer composite.

Sample	Epotec Epoxy Polymer	Temperature °C				
		$T_{5\%}$	$T_{20\%}$	$T_{50\%}$	T_{max}	dTG (%/min)
BA0	**YD001X75/G5022**	180-210	303	341	377	-9.11
BA4	**YD011X75/G5022/4.0%BA**	142	270	423	430	-10.2
BA4_Si1.0	**YD011X75G5022/4.0%BA/1.0%NS**	166	396	443	447	-10.1

$T_{5\%}$, $T_{20\%}$, $T_{50\%}$ - the rate of 5 wt%, 20 wt%, 50 wt% mass loss; T_{max} - the strongest decomposition temperature; dTG (%/min) - decomposition rate.

The maximum decomposition rate was observed at 447 °C, with a peak mass loss rate of 10.1%/min, indicating an improvement in thermal stability compared to the BA0 and BA4 samples.

A summary of the thermal analysis results for all epoxy material samples is provided in Table 4.

The results presented in Table 4 indicate that the thermal stability of the Epotec YD011X75/G5022 epoxy materials progressively increased with the incorporation of 4.0 wt% boric acid (BA) and 1.0 wt% nano silica. Specifically, the temperature corresponding to the maximum decomposition rate increased from 377 °C for the unmodified epoxy (BA0 sample), to 430 °C for the BA4 sample (an improvement of 14%), and further to 447 °C for the BA4_Si1.0 sample (an improvement of 18.5%).

The improvement in thermal resistance due to nano silica can be attributed to its barrier and insulation mechanism. Nano silica forms a protective layer on the surface of the material, which hinders the diffusion of oxygen needed for combustion. Additionally, it keeps heat within the structure and restricts the release of volatile compounds during pyrolysis, thereby enhancing thermal stability[4,26-30].

Meanwhile, the flame-retardant effect of boric acid primarily occurs in the gas phase, as explained by its stepwise decomposition reactions. These include the release of water vapor and the formation of thermally stable boron oxides, which inhibit combustion through heat absorption and oxygen dilution[31,32].

The combination of 4.0 wt% boric acid and 1.0 wt% nano silica showed a synergistic effect, significantly improving the thermal stability of the Epotec YD011X75/G5022 epoxy polymer composite by 18.5%, compared to the unmodified system.

3.5 Mechanical and thermal properties of the Epotec YD011X75/G5022/4.0 wt% BA/1.0 wt% nano silica epoxy paint

The epoxy paint formulation was prepared using the grinding method. The composition of the Epotec YD011X75/G5022/4.0 wt% BA/1.0 wt% nano silica epoxy paint (denoted as the PBA4_Si sample) is detailed in Table 5.

The resin phase consisted of the Epotec YD011X75 epoxy resin, boric acid (BA), nano silica, talc powder (Talc 1250), iron oxide, zinc phosphate, xylene solvent, dispersing and wetting additives (BYK 110), and rheological additives (Bentone SD1). The curing agent used in the formulation was G5022, a polyamide-based hardener.

The PBA4_Si epoxy paint shows excellent corrosion resistance and thermal stability. To achieve these properties, the paint formulation incorporates a variety of functional components aimed at enhancing both corrosion protection and heat resistance.

Table 5. The composition of the PBA4_Si epoxy paint.

Ingredient	Content (%)
Epotec YD 011X75 epoxy resin	100
Boric acid (BA)	4
Nano silica (S5505)	1
Talc powder (Talc 1250)	20
Iron oxide	70
Zinc phosphate	65
Xylene	70
Bentone SD1	1.5
BYK 110	1.5
Hardener G5022	33

The epoxy resin (Epotec YD011X75), which forms the base of the system, has inherent chemical resistance and strong adhesion to substrates, contributing significantly to the paint's protective capabilities. Additionally, iron oxide and zinc phosphate pigments are included to improve corrosion resistance by forming a passive barrier and inhibiting rust formation. Talc fillers (Talc 1250) provide enhanced shielding and contribute to the mechanical integrity and protective barrier of the coating. Crucially, boric acid (BA) and nano silica are incorporated as multifunctional additives. These components not only reinforce the physical and mechanical properties of the epoxy coating but also play a critical role in improving thermal resistance and flame retardancy.

Other supporting components include rheological additives such as Bentone SD1, which control viscosity and thixotropy during production and application, and wetting and dispersing agents (BYK 110), which ensure uniform pigment distribution and prevent sedimentation during storage. The xylene solvent serves as a viscosity modifier, facilitating optimal application performance[26-30].

The mechanical properties of the epoxy coating and the surface of the coating are presented in Table 6 and Figure 7.

The results presented in Table 6 and Figure 7 indicate that the epoxy paint film satisfies the required performance criteria outlined in the Vietnamese national standard TCVN 9014:2011 for epoxy coatings[33].

3.5.1 Thermal properties of the PBA4_Si epoxy coating film

The thermal behavior of the PBA4_Si epoxy coating film was evaluated using TG-DSC thermal analysis, and the results are presented in Figure 8.

Table 6. Mechanical properties of epoxy coating.

Sample	Impact strength (kg.cm)	Adhesion	Flexibility (mm)	Dry film thickness (µm)	Fineness (µm)	Pencil hardness	Curing time (hrs)	Drying time (hrs)
Epoxy Paint PBA4_Si	100	5B	3	56.04	25	5H	2	6

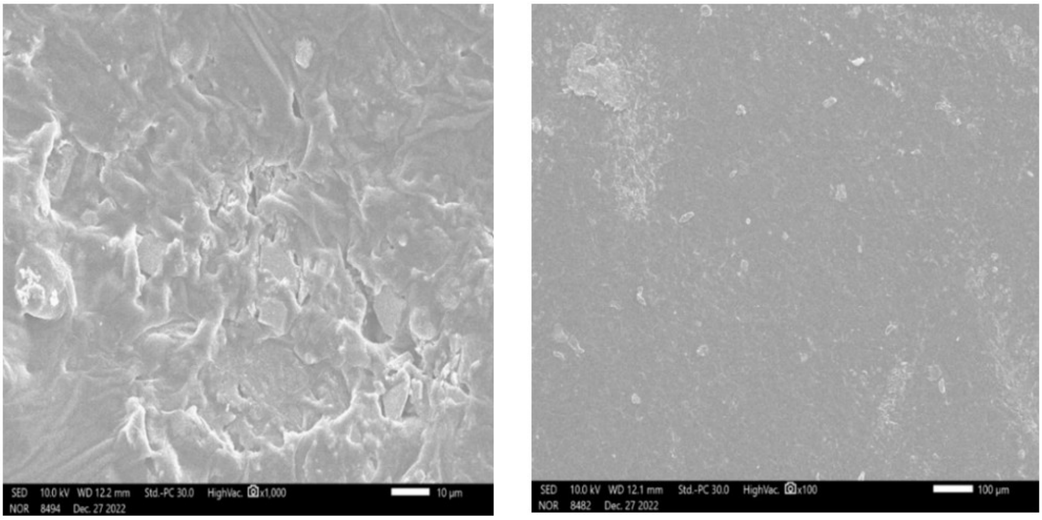

Figure 7. SEM image of the PBA4_Si epoxy coating film.

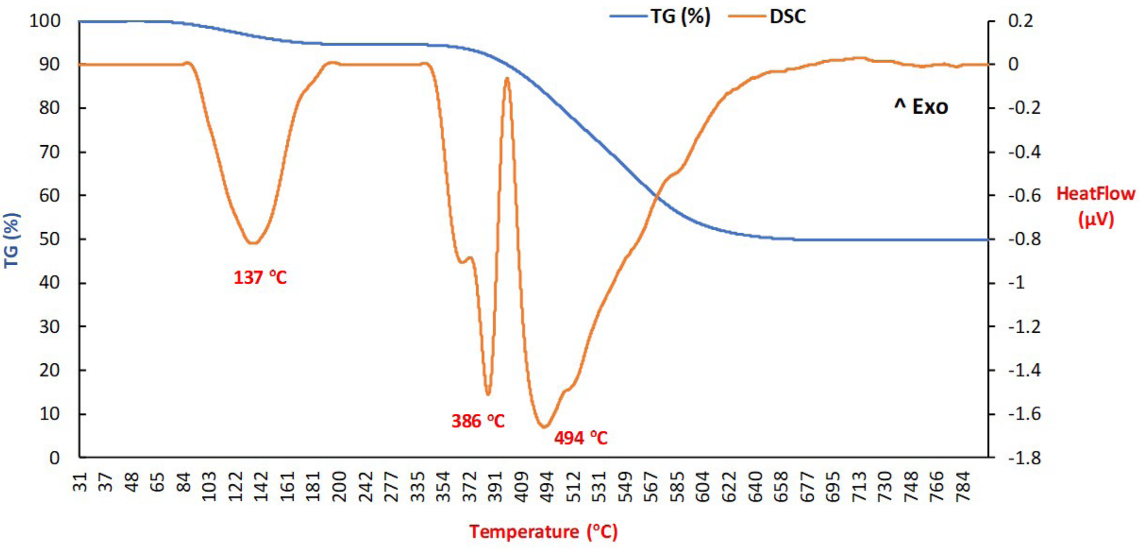

Figure 8. TG- DSC thermal analysis of the PBA4_Si epoxy coating film.

As shown in Figure 8, the DSC curve displays distinct endothermic peaks characteristic of the material's thermal decomposition. The decomposition of the PBA4_Si sample began at approximately 100 °C, corresponding to the evaporation of residual solvent. A notable endothermic peak was observed at 137 °C, extending to 180 °C, which aligns with the thermal decomposition of boric acid, as previously seen in Figure 3 and Figure 4.

The maximum decomposition temperature was recorded at 494 °C, showing a significant improvement in thermal stability. This enhancement can be attributed to the presence of 4.0 wt% boric acid and 1.0 wt% nano silica, which reinforce the epoxy matrix and contribute to improved resistance to thermal degradation. In addition to the functional fillers and pigments, these additives play a key role in enhancing the heat resistance of the epoxy paint system.

4. Conclusions

The effects of boric acid and nano silica reinforcement on the Epotec YD011X75/G5022 epoxy polymer composite were evaluated through the analysis of both mechanical and thermal properties. The incorporation of 4.0 wt% boric acid resulted in a 32% improvement in impact strength and a 14% increase in thermal resistance. When the composite was reinforced with 4.0 wt% boric acid and 1.0 wt% nano silica, the thermal stability was further enhanced, reaching an 18.5% increase compared to the unmodified epoxy system. Furthermore, the epoxy paint formulated from Epotec YD011X75/G5022 resin, reinforced with 4.0 wt% boric acid and 1.0 wt% nano silica, satisfied all mechanical property requirements for protective coatings and exhibited a strongest decomposition temperature of 494 °C, indicating high thermal stability and suitability for use in harsh or high-temperature environments.

5. Author's Contribution

- **Conceptualization** – Cuong Huynh Le Huy; Hao Nguyen Nhat; An Truong Thanh
- **Data curation** – Cuong Huynh Le Huy; Hao Nguyen Nhat; An Truong Thanh
- **Formal analysis** – Cuong Huynh Le Huy
- **Funding acquisition** – NA.
- **Investigation** – NA.
- **Methodology** – Cuong Huynh Le Huy; Hao Nguyen Nhat; An Truong Thanh
- **Project administration** – Cuong Huynh Le Huy
- **Resources** – Cuong Huynh Le Huy; Hao Nguyen Nhat; An Truong Thanh
- **Software** – NA.
- **Supervision** – Cuong Huynh Le Huy
- **Validation** – Cuong Huynh Le Huy
- **Visualization** – Cuong Huynh Le Huy
- **Writing – original draft** – Cuong Huynh Le Huy
- **Writing – review & editing** – Cuong Huynh Le Huy

Acknowledgements

This work was supported by Ho Chi Minh City University of Industry and Trade (HUIT).

References

1. Ramadhoni, B., Ujianto, O., & Nadapdap, M. (2017). Rigid polyurethane nanocomposites prepared by direct incorporation: effects of nanoclay, carbon nanotubes and mixing speed on physical and morphological properties. *Progress in Polymer Science*, *319*, 012041. https://doi.org/10.1088/1757-899X/319/1/012041.
2. Shen, K. K., & O'Connor, R. (1998). *Flame retardants: borates.* In G. Pritchard (Ed.), *Plastics additives: an A-Z reference* (pp. 268-276). Dordrecht: Springer. https://doi.org/10.1007/978-94-011-5862-6_30
3. Kiliaris, P., & Papaspyrides, C. D. (2010). Polymer/layered silicate (clay) nanocomposites: an overview of flame retardancy. *Progress in Polymer Science*, *35*(7), 902-958. https://doi.org/10.1016/j.progpolymsci.2010.03.001.
4. Cuong, H. L. H., & Thanh, A. T. (2022). Characteristics of mechanical properties and thermal behavior of epoxy nanocomposites and coatings by zinc borate, nano silica, and hardener. *ACS Omega*, *7*(43), 38299-38310. https://doi.org/10.1021/acsomega.2c02462. PMid:36340107.
5. Demirhan, Y., Yurtseven, R., & Usta, N. (2021). The effect of boric acid on flame retardancy of intumescent flame-retardant polypropylene composites including nano clay. *Journal of Thermoplastic Composite Materials*, *36*(3), 1187-1214. https://doi.org/10.1177/08927057211052327.
6. Hoang, D., Kim, J., & Jang, B. N. (2008). Synthesis and performance of cyclic phosphorus-containing flame retardants. *Polymer Degradation & Stability*, *93*(11), 2042-2047. https://doi.org/10.1016/j.polymdegradstab.2008.02.017.
7. Levchik, S., Bocchini, S., & Camino, G. (2010). *Halogen-containing flame retardants.* In C. A. Wilkie, & A. B. Morgan (Eds.), *Fire retardancy of polymeric materials* (pp. 58-80). Boca Raton: CRC Press. https://doi.org/10.1201/9781003380689-4.
8. van der Veen, I., & de Boer, J. (2012). Phosphorus flame retardants: properties, production, environmental occurrence, toxicity and analysis. *Chemosphere*, *88*(10), 1119-1153. https://doi.org/10.1016/j.chemosphere.2012.03.067. PMid:22537891.
9. Hörold, S. (1999). Phosphorus flame retardants in thermoset resins. *Polymer Degradation & Stability*, *64*(3), 427-431. https://doi.org/10.1016/S0141-3910(98)00163-3.
10. Camino, G., Costa, L., & di Cortemiglia, M. P. L. (1991). Overview of fire retardant mechanisms. *Polymer Degradation & Stability*, *33*(2), 131-154. https://doi.org/10.1016/0141-3910(91)90014-I.
11. Wu, Q., Lü, J., & Qu, B. (2003). Preparation and characterization of micro capsulated red phosphorus and its flame-retardant mechanism in halogen-free flame retardant polyolefins. *Polymer International*, *52*(8), 1326-1331. https://doi.org/10.1002/pi.1115.
12. Liu, Y., & Wang, Q. (2006). Melamine cyanurate-microencapsulated red phosphorus flame retardant unreinforced and glass fiber reinforced polyamide 66. *Polymer Degradation & Stability*, *91*(12), 3103-3109. https://doi.org/10.1016/j.polymdegradstab.2006.07.026.
13. Qiu, S., Ma, C., Wang, X., Zhou, X., Feng, X., Yuen, K. K. R., & Hu, Y. (2018). Melamine-containing polyphosphazene wrapped ammonium polyphosphate: A novel multifunctional organic-inorganic hybrid flame retardant. *Journal of Hazardous Materials*, *344*, 839-848. https://doi.org/10.1016/j.jhazmat.2017.11.018. PMid:29190581.
14. Xi, W., Qian, L., Huang, Z., Cao, Y., & Li, L. (2016). Continuous flame retardant actions of two phosphate esters with expandable graphite in rigid polyurethane foams. *Polymer Degradation & Stability*, *130*, 97-102. https://doi.org/10.1016/j.polymdegradstab.2016.06.003.
15. Rao, W.-H., Xu, H.-X., Xu, Y.-J., Qi, M., Liao, W., Xu, S., & Wang, Y.-Z. (2018). Persistently flame retardant flexible polyurethane foams by a novel phosphorus-containing polyol. *Chemical Engineering Journal*, *343*, 198-206. https://doi.org/10.1016/j.cej.2018.03.013.
16. Jin, S., Qian, L., Qiu, Y., Chen, Y., & Xin, F. (2019). High-efficiency flame retardant behavior of bi-DOPO compound with hydroxyl group on epoxy resin. *Polymer Degradation & Stability*, *166*, 344-352. https://doi.org/10.1016/j.polymdegradstab.2019.06.024.
17. Xie, C., Du, J., Dong, Z., Sun, S., Zhao, L., & Dai, L. (2016). Improving thermal and flame-retardant properties of epoxy resins by a new imine linkage phosphorous-containing curing agent. *Polymer Engineering and Science*, *56*(4), 441-447. https://doi.org/10.1002/pen.24270.

18. Zhu, Z.-M., Wang, L.-X., & Dong, L.-P. (2019). Influence of a novel P/N-containing oligomer on flame retardancy and thermal degradation of intumescent flame-retardant epoxy resin. *Polymer Degradation & Stability*, *162*, 129-137. https://doi.org/10.1016/j.polymdegradstab.2019.02.021.
19. Levchik, S. V., & Weil, E. D. (2006). A review of recent progress in phosphorus-based flame retardants. *Journal of Fire Sciences*, *24*(5), 345-364. https://doi.org/10.1177/0734904106068426.
20. Carja, I.-D., Serbezeanu, D., Vlad-Bubulac, T., Hamciuc, C., Coroaba, A., Lisa, G., López, C. G., Soriano, M. F., Pérez, V. F., & Romero Sánchez, M. D. (2014). A straightforward, eco-friendly and cost-effective approach towards flame retardant epoxy resins. *Journal of Materials Chemistry. A, Materials for Energy and Sustainability*, *2*(38), 16230-16241. https://doi.org/10.1039/C4TA03197K.
21. Mir, M. A. N., Mohammad, R. K., Amir, B., & Moslem, H. (2012). Nanosilica reinforced epoxy floor coating composites: preparation and thermophysical characterization. *Current Chemistry Letters*, *1*(1), 13-20. Retrieved in 2025, May 12, from https://www.growingscience.com/ccl/Vol1/ccl_2012_2.pdf
22. Chozhan, C. K., Chandramohan, A., & Alagar, M. (2018). Surface modified clay reinforced silicon incorporated epoxy hybrid nanocomposites: thermal, mechanical and morphological properties. *Sage Journals*, *9*(1), 1-22. https://doi.org/10.1177/204124791800900101.
23. Pellegrin, P., Nguyen, T., Mermet, L., Shapiro, A., Gu, X., & Chin, J. (2009). *Degradation and nanoparticle release of epoxy/silica composites exposed to UV radiation* (Vol. *1*, pp. 90-93). Punjab: Nano Science and Technology Institute. Retrieved in 2025, May 12, from https://tsapps.nist.gov/publication/get_pdf.cfm?pub_id=902990
24. Woldemariam, M. H., Belingardi, G., Koricho, E. G., & Reda, D. T. (2019). Effects of nanomaterials and particles on mechanical properties and fracture toughness of composite materials: a short review. *AIMS Materials Science*, *6*(6), 1191-1212. https://doi.org/10.3934/matersci.2019.6.1191.
25. Liu, X., Dang, L., Nai, X., Dong, Y., & Li, W. (2018). Design and preparation of high-aspect-ratio zinc borate whiskers and their effects on mechanical properties of PP nanocomposite. *Research on Chemical Intermediates*, *44*(10), 5697-5709. https://doi.org/10.1007/s11164-018-3450-9.
26. Cuong, H. L. H., Dieu, T. V., Thanh, N. D., & Oanh, D. T. Y. (2017). Study the effects of nanosilica on mechanical properties of polymer coating film based on epoxy resin DER 671X75. *Vietnam Journal of Chemistry*, *55*(1), 12.
27. Nguyen, T. A., Nguyen, T. H., Nguyen, T. V., Thai, H., & Shi, X. (2016). Effect of nanoparticles on the thermal and mechanical properties of epoxy coatings. *Journal of Nanoscience and Nanotechnology*, *16*(9), 9874-9881. https://doi.org/10.1166/jnn.2016.12162.
28. Gu, H., Guo, J., He, Q., Tadakamalla, S., Zhang, X., Yan, X., Huang, Y., Colorado, H. A., Wei, S., & Guo, Z. (2013). Flame-retardant epoxy resin nanocomposites reinforced with polyaniline-stabilized silica nanoparticles. *Industrial & Engineering Chemistry Research*, *52*(23), 7718-7728. https://doi.org/10.1021/ie400275n.
29. Cuong, H. L. H., Thanh, A. T., & Bao, L. H. (2021). Effects of triphenyl phosphate (TPP) and nanosilica on the mechanical properties, thermal degradation of polymer nanocomposite materials, and coating based on epoxy resin. *Materials Research Express*, *8*(9), 095301. https://doi.org/10.1088/2053-1591/ac2098.
30. Cuong, H. L. H., & Thanh, A. T. (2023). Study on fabricating epoxy coatings reinforced with iron oxide flakes and nano silica. *Journal of Reinforced Plastics and Composites*, *42* (13-14), 724-740. https://doi.org/10.1177/07316844231162562.
31. Dogan, M., Dogan, S. D., Atabek Savas, L., Ozcelik, G., & Tayfun, U. (2021). Flame retardant effect of boron compounds in polymeic materials. *Composites. Part B, Engineering*, *222*, 109088. https://doi.org/10.1016/j.compositesb.2021.109088.
32. Bourbigot, S., Le Bras, M., Leeuwendal, R., Shen, K. K., & Schubert, D. (1999). Recent advances in the use of zinc borates in flame retardancy of EVA. *Polymer Degradation & Stability*, *64*(3), 419-425. https://doi.org/10.1016/S0141-3910(98)00130-X.
33. Vietnamese National Standard. (2011). *Epoxy paint standard (TCVN 9014:2011).* Vietnam.

Received: May 12, 2025
Revised: July 18, 2025
Accepted: Sept. 24, 2025

Editor-in-Chief: *Sebastião V. Canevarolo*