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**ORIGINAL RESEARCH**

# Thermal investigation of epoxy composite with boric acid reinforcement

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**Abstract**

The thermal behavior of epoxy composites with different amounts of fine boric acid powder was examined in this work. Boric acid was used as a flame-retardant filler, methyl ethyl ketone peroxide was used as the curing agent, and epoxy resin ED-20 was used as the matrix to create the composites. Under carefully monitored circumstances, the samples were created utilizing standard casting methods. Scanning Electron Microscopy (SEM), Thermo-gravimetric Analysis (TGA), and Fourier Transform Infrared Spectroscopy (FTIR) were used for morphological, thermal, and chemical characterizations, respectively. Boric acid particles were uniformly distributed throughout the epoxy matrix, as shown by SEM micrographs. According to thermal studies, adding boric acid raised the decomposition temperature and increased the char yield as the filler concentration increased. The chemical interactions between the epoxy resin and boric acid were verified by FTIR spectra. The objective of this study is to investigate the effect of fine boric acid powder as a flame-retardant filler on the thermal behavior of epoxy resin composites. It aims to evaluate how different concentrations of boric acid influence the thermal stability and decomposition characteristics of the composites. Additionally, the study seeks to analyze the morphological and chemical interactions between boric acid and the epoxy matrix using SEM, TGA, and FTIR techniques. These findings suggest that the thermal stability and flame-retardant properties of epoxy composites are considerably improved by the addition of fine boric acid. These advancements demonstrate the materials' potential for usage in applications requiring resistance to heat and flame.

**Keywords**: Boric Acid, SEM, FTIR, Composite, Flame-reterdancy.

## 1 | INTRODUCTION

Epoxy resins are renowned for their exceptional mechanical strength, chemical resistance, electrical insulating qualities, and great adhesion. Epoxy resins are frequently employed as building materials in a variety of sectors due to these characteristics. Epoxy polymers do, however, have some disadvantages. Generally speaking, they are highly flammable and have poor thermal stability. By adding specific additives to the polymer matrix, epoxy resins' thermal stability and flame resistance can be effectively increased. By improving the epoxy resin's performance, these additions can make it more appropriate for applications requiring fire resistance or high temperatures. (1) For this, several inorganic particles were utilized. Enhance epoxy resins' qualities among inorganic flame retardants, boric acid is one of the boron compounds that is recognized to be both ecologically benign and effective. Water is released along with boric acid, and as a result, boron oxide B2O and the metabolic acid HBO3 are formed (2). This procedure, Polymer surface and volatile product dilution degradation of polymers. Creation of inflammable physical barrier is provided by a protective layer of boron oxide (3) . Epoxy resin is one of the well-known thermosetting polymers which is used as a building material and construction sites. (4) Also epoxy resins are the predominant thermosetting polymers in the composites industry. They are usually show corrosion resistance properties which is important for different building and high level applications. They are applied as matrices in paints, coatings, adhesives, and composites. (5) In the growing market of epoxy resins, it is very important to develop systems based on epoxy resins because their flame retardant properties help them to manage safety as building materials. (6) Epoxy polymers are characterized by low thermal stability and high flammability. Nanoparticles are considered effective fillers to improve the thermal and functional properties of polymer composites. The term of “epoxy” is used to describe a range of monomers containing an epoxy group, while “epoxy resins” refers to a class of molecules containing at least two epoxy groups. The material obtained after the curing reaction is commonly referred to as “epoxy resin” even if it no longer contains epoxy groups (17). Figure 1 shows examples of widely used epoxy monomers. Di glycidyl ether of bisphenol A (DGEBA), which can be obtained from the reaction between bisphenol A and epichlorohydrin in the presence of sodium hydroxide, is one of the most common precursors for epoxy resins. (11) Multifunctional epoxy monomers are also widely used because they tend to increase cross-linking density. For instance, N, N, N’, N'-tetraglycidyl-4,4-methylenedianiline (TGMDA) is a typical monomer extensively used in aerospace composites. Cycloaliphatic resins are another class of epoxy resins of great interest (16). They have less tendency toward yellowing than aromatic resins. In addition, their low viscosity and electrical loss properties have made them useful commercially in electrical and electronic applications. Such examples can be seen for 3,4-epoxycyclohexylmethyl 3,4-epoxycyclohexanecarboxylate (ECC). (10) and high-value recycling routes for post-consumer textile waste.

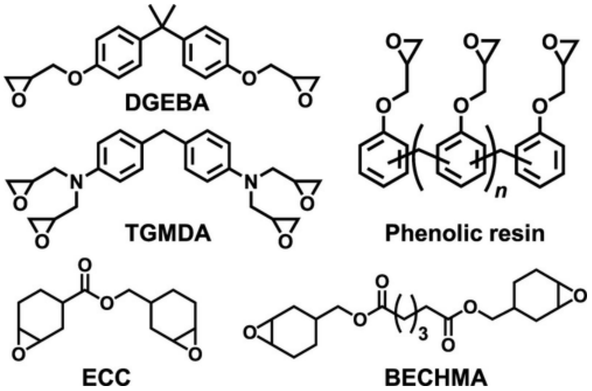


**Figure 1: Typical monomers used as a precursor for epoxy resins. (10)**

In this work, ED-20 epoxy resin and polyethylene polyamine as the curing agent were used to create epoxy composites for this investigation. As flame-retardant fillers, fine boric acid powder and aluminum nano-powder were added. Thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) were used to investigate the thermal properties of the resultant composites, and a systematic evaluation of their mechanical qualities was also conducted. The findings demonstrated that the epoxy composite’s mechanical and thermal stability significantly improved with the addition of fillers. The composite containing a mixture of boric acid and aluminum nano-powder showed the best flexural strength among the samples examined, while the composite filled with boric acid showed the maximum thermal stability.

## 2 | EXPERIMENTAL SECTION

### 2.1 | Materials

Epoxy polymers are characterized by low thermal stability and high flammability. One way to improve the thermal stability of the composite is adding epoxy resin as an additive. (11) Epoxy resins are the predominant thermosetting polymers in the composites industry. Here, collection of epoxy resin powder came from the local vendors. On the other hand, Boric acid also purchased from the local vendor of Nilkhet, Dhaka, Bangladesh. All the products are purchased after proper manufacturing and expiry date checking

### 2.2 | Methods

### 2.2.1 | Preparation of composite materials

Epoxy resin and different concentrations of boric acid were combined to create composites, both separately and in conjunction with methyl ethyl ketone peroxide (MEKP). Before mixing, the fillers' surface was pre-treated with polyethylene polyamine (PEPA) to improve the interfacial adhesion between the filler particles and the epoxy matrix. Then, using the hand lay-up method, the epoxy resin system was made with the necessary mass ratios and completely mixed. To get rid of trapped air bubbles, the resultant mixture was de-gassed under vacuum. Following the homogenization of all ingredients, the mixture was put in a platen press and compressed to 98.07 Pa in order to solidify the substance. The composites were post-cured in an oven at 50 °C for three hours and then at 80 °C for three hours after being cured overnight at room temperature.

**Table 1.** The compositions (wt. %) of the specimens.

| Samples | ED-20 | Boric acid |
|---|---|---|
| $H_3BO_3$ Boric acid powder | 0 | 100 |
| Epoxy resin + 5wt% $H_3BO_3$ | 95 | 5 |
| Epoxy resin + 10wt% $H_3BO_3$ | 90 | 10 |
| Epoxy resin | 100 | 0 |

### 2.2.2 | Characterization

**Thermal analysis**

Using Thermogravimetric Analysis (TGA) and Differential Scanning Calorimetry (DSC), the impact of boric acid content on the epoxy composites' heat stability was assessed. The findings showed that adding boric acid raised the temperature of decomposition and decreased the rate of weight loss. An SDT Q600 analyzer (TA

Instruments, USA) was used to perform thermogravimetric (TG) and differential scanning calorimetry (DSC) measurements. In both air and argon atmospheres, the samples' thermal behavior was examined throughout a temperature range of 20 to 900 °C at a heating rate of 10 °C $\mathrm{min}^{-1}$ and a gas flow rate of 100 mL $\mathrm{min}^{-1}$. For the analysis, about 10 mg of each sample were employed, and the samples were held in $Al_2O_3$ crucibles.

### Fourier transform infrared spectroscopy

The chemical bonding and interactions in the composites were determined using Fourier Transform Infrared (FTIR) spectroscopy. The presence of distinctive epoxy functional groups and peaks associated with boric acid was verified by the spectra. Absorption band shifts suggested that the filler and matrix were physically interacting. These findings imply that boric acid was successfully incorporated into the epoxy network. A Nicolet 5700 FTIR spectrometer (Thermo- Electron, USA) was used to record the Fourier transform infrared (FTIR) spectra of KBr pellets at room temperature. The spectra were recorded in the spectral region of 4000-400 $\mathrm{cm}^{-1}$ with a resolution of 4 $\mathrm{cm}^{-1}$.

## 3 | RESULTS AND DISCUSSION

### 3.1 | Scanning Electron Microscope (SEM) Test

The composite samples' surface morphology and microstructural features were investigated using scanning electron microscopy (SEM). To improve electrical conductivity, a small layer of gold was sputter-coated onto the specimens' cracked surfaces before imaging (9). To evaluate the filler particle dispersion and the interfacial adhesion between the filler and the epoxy matrix, SEM micrographs were taken at different magnifications. Important information about the composites' overall quality, homogeneity, and fracture behavior was revealed by this investigation (10). Boric acid powder is a polydisperse system, while methyl ethyl ketone peroxide is a crystal like system, as seen in the scanning electron microscopy image (Fig. S-2-1) (Fig. 2-2). According to the dispersed composition study of boric acid, 45 percent of the particles are smaller than 20 µm. Planar layers with a thickness of roughly 100 nm make up the scaly crystals that make up the powder particles. The zeolite particles are no larger than 1–3 µm.

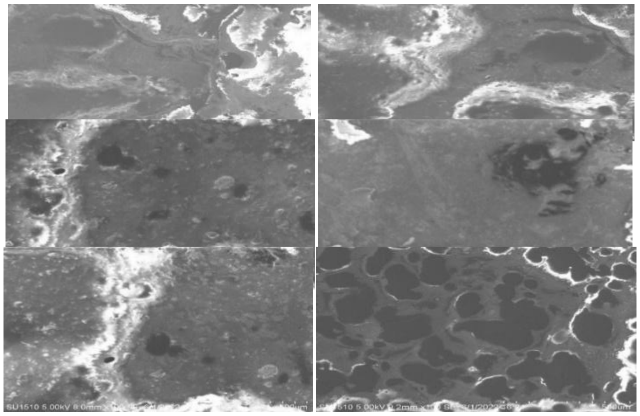


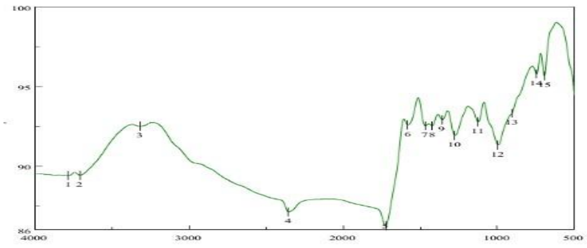
**Figure 2: S1-1,S1-2,S1-3,S1-6,S1-7,S 2-1,S 2-2,S2-3,S2-4,S3-1,S3-2,S3-3 SEM micrograph of the boric acid powder.**

According to the SEM study, over 45% of the boric acid particles are smaller than 20 µm, and they have a polydisperse, scaly morphology with planar layers that are roughly 100 nm thick. Through barrier and char-forming actions, this structure's vast surface area enhances thermal resistance by fostering good interfacial contact with the epoxy matrix. Methyl ethyl ketone peroxide's crystalline structure guarantees even curing and efficient crosslinking in the composite. The uniformly dispersed zeolite particles, which range in size from 1 to 3 µm, enhance structural stability. During degradation, the tight microstructure formed by the layered boric acid and tiny zeolite particles prevents heat and gas transfer. In comparison to neat epoxy, the composite is anticipated to provide improved flame retardancy, decreased degradation rate, and increased thermal stability.

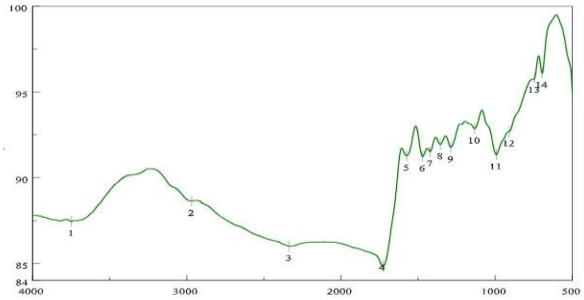
### 3.2 | Fourier Transform Infrared Spectroscopy (FTIR) Test

Organic, polymeric, and certain inorganic materials can be identified using the analytical method known as Fourier Transform Infrared Spectroscopy (FTIR) (11). It calculates how much infrared light at various wavelengths is absorbed by a sample. The resulting spectrum is the material's molecular fingerprint (12). In chemistry and material science, FTIR is frequently used for both qualitative and quantitative investigation (13).

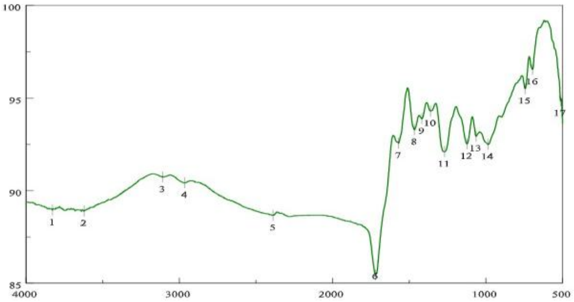
The FTIR spectra for the samples of boric acid, Sakhaptinsk zeolite, epoxy composite E/Z5/BA10, and neat epoxy E0 are shown in Fig. 1. The FTIR spectrum of Sakhaptinsk zeolite is characterized by intensive vibrations in the internal tetrahedron ties Si–O–Si and Si–O–Al [22]. The most intensive band is observed at 1040 cm-1 and corresponds to the asymmetric stretching vibrations Si–O–Si and Si–O–Al. The adsorption bands at 802 and 780 cm-1 are associated with the symmetric stretching vibrations Si–O–Si. The band at 468 cm-1 is attributed to the bending vibration O–Si–O. The strong wide band in the region 3250–3735 cm-1 is associated with the presence of zeolite water. The band at 3640 cm-1 cans be characteristic of hydrogen bond of OH groups of the water molecules. The bands in the region 3100–3400 cm-1 are attributed to the symmetric and asymmetric stretching modes of molecular water. The vibration band at 1625 cm-1 corresponds to the bending vibration of the zeolite water in the channels of the sample. The FTIR spectrum of boric acid is characterized by the wide band at 1405 cm-1 caused by asymmetric stretching vibrations of B–O. The band at 883 cm-1 is assigned to the symmetric stretching vibrations B–O. The bands at 1193 and 791 cm-1 are attributed to the vibrations of B–O–H in-plane bending and out-plane bending, respectively. The band at 648 cm-1 is attributed to the deformation vibration of atoms in B–O. The band at 548 cm-1 corresponds to the bending vibration of B–O–B.



**Wavenumber [cm-1] [S1]**



**Wavenumber [cm-1] [S2]**



**Wavenumber [cm-1] [S3]**

**Figure 3: Wavenumber S1, S2 and S3 [cm-1].**

The most intensive band is observed at 1040 cm$^{-1}$ and corresponds to the asymmetric stretching vibrations Si–O–Si and Si–O–Al. The adsorption bands at 802 cm$^{-1}$ and 780 cm$^{-1}$ are associated with the symmetric stretching vibrations Si–O–Si. The band at 468 cm-1 is attributed to the bending vibration O–Si–O. The strong wide band in the region 3250–3735 cm$^{-1}$ is associated with the presence of zeolite water. The band at 3640 cm-1 cans be characteristic of hydrogen bond of OH groups of the water molecules. The bands in the region 3100 to 3400 cm$^{-1}$ are attributed to the symmetric and asymmetric stretching modes of molecular water. The vibration band at 1625 cm$^{-1}$ corresponds to the bending vibration of the zeolite water in the channels of the sample. The FTIR spectrum of boric acid is characterized by the wide band at 1405 cm$^{-1}$ caused by asymmetric stretching vibrations of B–O. The band at 883 cm$^{-1}$ is assigned to the symmetric stretching vibrations B–O. The bands at 1193 and 791 cm$^{-1}$ are attributed to the vibrations of B–O–H in-plane bending and out-plane bending, respectively. The band at 648 cm$^{-1}$ is attributed to the deformation vibration of atoms in B–O. The band at 548 cm$^{-1}$ corresponds to the bending vibration.

### 3.3 | TGA & DSC results

Table 2 shows the TGA analysis results of boric acid powder, pure epoxy polymer, and epoxy composites with various percentages of boric acid performed in the presence of air.

Table 2. The TGA results of the neat epoxy polymer and boric acid-based epoxy composites at different percentages of boric acid were carried out in the presence of methyl ethyl ketone peroxide. The values of temperature for a fixed weight loss are 5% and 50% and the residue is at 600°C. The process of thermal degradation of the studied samples in epoxy resin occurs in several stages. There is no difference in thermal degradation of neat epoxy and 1 wt% of boric acid-based composite.

The process of thermal degradation of the studied samples occurs in several stages. The onset temperature of degradation is between 200-400 °C for all samples, steadily shifting between 400-600 °C. From this figure also we can say that thermal stability increases with increasing boric acid content. (16)

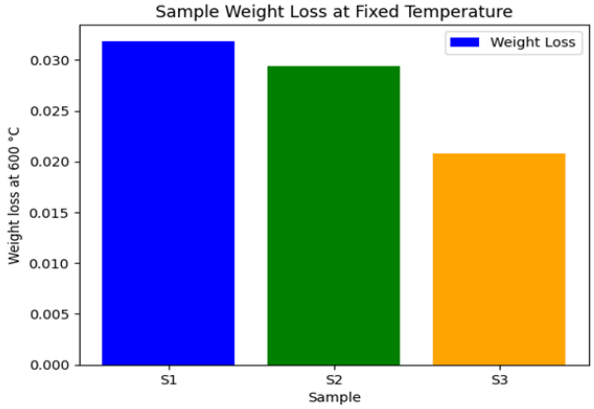
However, the addition of 5 and 10 wt% of boric acid shows very good improvement in the thermal behavior of the composites. The best result was obtained for the sample with a filler content of 10 wt%. The temperature of 50% weight loss for this sample is higher by 58°C than that for the neat epoxy polymer.

**Table 2:** Degradation of mass of the sample at different temperature.

| **Samples** | **Degradation of Temperature (°C)** | | | | |
|---|---|---|---|---|---|
| | **$T_{10\%}$** | **$T_{30\%}$** | **$T_{50\%}$** | **$T_{70\%}$** | **$T_{90\%}$** |
| **$H_3BO_3$ Boric acid powder ($S_0$)** | 110 | 139 | - | - | - |
| **Epoxy resin + 5wt% $H_3BO_3$ ($S_1$)** | 278 | 350 | 447 | 497 | 540 |
| **Epoxy resin + 10wt% $H_3BO_3$ ($S_2$)** | 279 | 378 | 476 | 531 | 800 |
| **Epoxy resin ($S_3$)** | 271 | 347 | 436 | 488 | 524 |

**Table 3:** degradation of mass of sample S1, S2 and S3 at fixed temperature.

| **Sample** | **Thermal Stability at $600^0$ C (Mass)** |
|---|---|
| S1 | 0.031864 |
| S2 | 0.029386 |
| S3 | 0.020753 |



Automated Moreover, the residue at 600°C of the sample contained 10 wt % of boric. The decomposition temperature of the epoxy composites at different stages increased upon the addition of boric acid. From the figure, neat epoxy and 1% boric acid composites are not very different in thermal degradation, but 2.5, 5 and 10% boric acid-based composites show very good improvements in the thermal degradation, these results show, the boric acid content effect in epoxy composites

## 4 | CONCLUSION

In this study, epoxy composites were prepared with a total loading of 15% by mass of boric acid and a combination of methyl ethyl ketone peroxide and boric acid. The findings demonstrated that, mostly as a result of water release, the mass loss of all epoxy composites during the initial phases of decomposition was lower than that of pure epoxy. However, the heat resistance of epoxy composites was enhanced at high temperatures by the addition of boric acid, either alone or in conjunction with methyl ethyl ketone peroxide. The highest intermediate temperature and residue during thermal and thermos-oxidative decomposition, as well as the lowest level of maximal decomposition rate when heated in air, were demonstrated by a mixture of 5 wt % zeolites and 10 wt % boric acids. The higher production of carbonaceous residues and the barrier effect in the presence of fillers may be responsible for the outcomes. The thermal stability of the epoxy composites was investigated by thermo-gravimetric analysis, scanning electron microscope and Fourier transform infrared spectroscopy. When compared to the clean epoxy polymer, the epoxy composites' mechanical and thermal stability improved with the inclusion of all the investigated fillers.

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