

Synthesis of Thermal Resistant and Transparent Al₂O₃ Reinforced Epoxy-Based Composite Coatings

Gaurav Verma¹, Kamal Sharma², Aayush Gupta^{3,*}

Abstract

This study explores the influence of thermal treatment on the structural, optical, and surface wettability characteristics of epoxy-based composite coatings reinforced with 3 wt.% aluminum oxide (Al₂O₃) nanoparticles. The coatings were synthesized via a dip-coating method on carbon fiber substrates and subjected to controlled thermal treatments at 300 °C, 400 °C, and 500 °C to assess their thermal resilience. X-ray diffraction (XRD) analysis confirmed the structural integrity of the composite coatings across all thermal conditions, with no signs of crystallization or phase transitions. Fourier transform infrared (FTIR) spectroscopy revealed consistent Al–O vibrational signatures, although some redshift and peak disappearance indicated temperature-dependent changes in the polymer matrix and interfacial bonding. Contact angle measurements demonstrated a stable hydrophobic behavior with negligible variation (~69.4°–69.7°) even after high-temperature exposure, indicating preserved surface chemistry and nanoparticle distribution. Notably, optical transmittance in the visible region exhibited a dramatic enhancement, increasing from 7–10% in untreated samples to over 50% following thermal treatment, peaking at ~60% after treatment at 400 °C. This significant improvement in transparency, coupled with thermal and surface stability, underscores the suitability of epoxy Al₂O₃ composite coatings for multifunctional applications in harsh environments, particularly where transparency and stable wettability are critical—such as in aerospace, automotive, and optical devices.

Keywords: Al₂O₃, epoxy coatings, nanocomposite materials, thermal stability, contact angle

INTRODUCTION

In recent years, hydrophobic surfaces have attained significant consideration of scientific community due to their distinctive features e.g., self-cleaning, anti-icing, and anti-corrosion. The ability to repel water due to high contact angle distinguishes these surfaces with other engineering materials [1,2]. For such hydrophobic applications at low temperatures, epoxy resins became a popular choice due to excellent mechanical properties, durability, and easy processing [2]. However, thermal stability of epoxy based coatings limits their utility at elevated temperatures. To mitigate such an issue, different inorganic materials (such as metal oxides) have been added into epoxy to restrict their thermal degradation. Aluminum oxide (Al₂O₃, alumina) is a popular ceramic due to its exceptional thermal stability, hardness, and chemical inertness for aerospace, electronics, and automotive performances [3,4]. Considering these characteristics of Al₂O₃, it could be an excellent choice to enhance the mechanical and thermal properties of epoxy based composite coatings. Some research groups investigated the effects of Al₂O₃ nanoparticles on the hydrophobicity of epoxy based composite coatings. It was observed that the hydrophobicity of composite coatings was directly correlated to the concentration of Al₂O₃ nanoparticles which was ascribed to the formation of a rough, textured surface that traps air pockets [1][5].

*Author for Correspondence

Aayush Gupta
 E-mail: aayush.gupta@gla.ac.in

¹Research Scholar, Department of Mechanical Engineering, GLA University, Mathura, Uttar Pradesh, India

²Professor, Department of Mechanical Engineering, GLA University Mathura, Uttar Pradesh India

³Assistant Professor Research, Department of Mechanical Engineering, GLA University Mathura, Uttar Pradesh India

Received Date: May 07, 2025

Accepted Date: May 21, 2025

Published Date: May 26, 2025

Citation: Gaurav Verma, Kamal Sharma, Aayush Gupta. Synthesis of Thermal Resistant and Transparent Al₂O₃ Reinforced Epoxy-Based Composite Coatings. Journal of Polymer & Composites. 2025; 13 (3): 178–183p.

Besides hydrophobicity, Al₂O₃ nanoparticles can also participate in improving the overall thermal stability of epoxy based composite coatings. It was found that the incorporation of ceramic particles (i.e., Al₂O₃) would increase the glass transition temperature of epoxy based composite coatings enhancing its thermal resistance [6,7]. These results support the utility of ceramic reinforced epoxy based coatings in aerospace and automotive industries. Agglomeration is considered as a major limitation of nanoparticles limiting their uniform distribution within matrix. Uniform distribution of Al₂O₃ nanoparticle in epoxy as matrix would exhibit a rough and textured surface supporting the enhanced hydrophobicity of composite coatings [8,9].

In the present study, we have incorporated Al₂O₃ nanoparticles into an epoxy matrix to develop epoxy-ceramic coatings and investigated their thermal resistance behavior. We hypothesize that the addition of Al₂O₃ nanoparticles will not only improve the thermal stability of the epoxy coatings and maintain their hydrophobic properties.

EXPERIMENTAL SECTION

For the synthesis of composites coatings, a multi-step methodology was adopted in which an ethanol based suspension was prepared by using Al₂O₃ (purity = 99.9%; avg. particle size = 80 nm; Intelligent Materials Pvt. Ltd., India) particles through sonication. This solution was then mixed with an epoxy resin solution (Araldite CY 230-1) at a 3-weight percentage loading of Al₂O₃ nanoparticles. The mixture was thoroughly homogenized and degassed to remove entrapped air using a desiccator and vacuum pump. Afterward, a hardener (Aradur HY 951) was added, and the resulting solution (epoxy+Al₂O₃) was applied to a carbon fiber substrate using the dip coating technique. The coating was cured at room temperature (RT) for 24 hours, followed by post-curing at 80 °C for 2 hours. To check the thermal resistance of the epoxy-Al₂O₃ coating, the coating was subjected to three different temperatures of 300, 400 and 500 °C in a muffle furnace. The coatings were labeled as E-3A, E-3A300, E-3A400 and E-3A500 for RT, 300, 400 and 500 °C, respectively. X-ray diffraction (XRD), Fourier Transform Infrared (FTIR) spectroscopy and contact angle measurements were used to characterize the coatings. A PANalytical diffractometer was used for measuring the XRD patterns (with Cu K α radiation) in the 2 θ range of 10-70°. FTIR spectroscopy was done using a Shimadzu spectrophotometer for analyzing the chemical composition of the coatings. Optical transmittance was recorded on UV-Vis. spectrophotometer (Hitachi U-3900H). The ACAM-NSC drop-shape analyzer was used for contact angle measurements. For this, distilled water drops were placed on the coated surfaces, and the contact angles were measured by using image analysis.

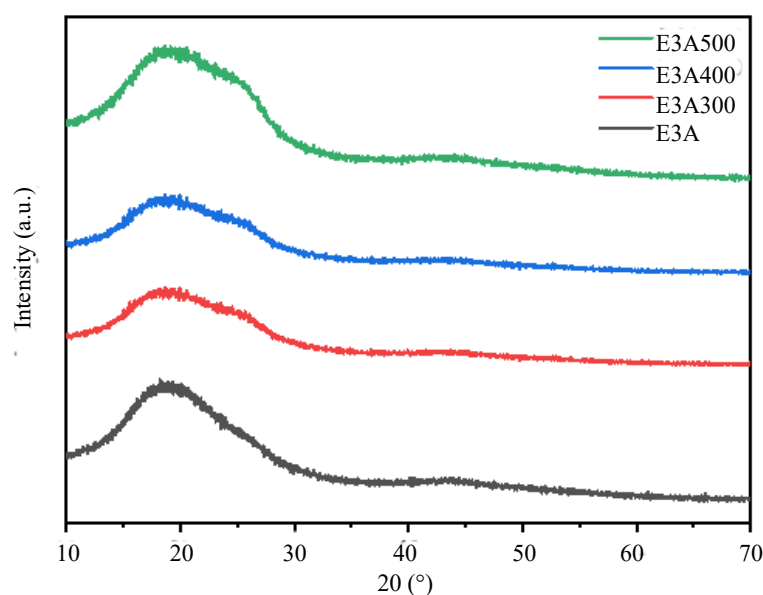


Figure 1. XRD patterns of epoxy-Al₂O₃ coatings with different thermal treatments.

RESULTS AND DISCUSSION

XRD Analysis

Figure 1 presents the XRD patterns of various thermally treated epoxy- Al_2O_3 coatings, ranging from 300 °C to 500 °C. The analysis shows that the thermal treatment did not make any significant changes in the XRD patterns of the coatings, suggesting good structural integrity of the coatings. This observation is in agreement with the amorphous nature of the coatings, as well as the homogeneous distribution of alumina nanoparticles within the epoxy matrix [8,9]. The absence of any new or altered diffraction peaks suggest that no phase transformations, crystallization, or significant structural reorganization took place due to elevated temperatures [6]. The characteristic peak at $\sim 25^\circ$ in XRD patterns of all samples corresponds to the (002) crystallographic plane of the base carbon fiber [10]. The presence of this peak throughout the thermal treatment process confirms that the carbon fiber structure remained intact, even in the presence of the epoxy- Al_2O_3 coating [11].

FTIR Analysis

Figure 2 presents the FTIR spectra of the epoxy- Al_2O_3 coatings subjected to various thermal treatments. The presence of peaks at $\sim 823\text{ cm}^{-1}$ in all the coatings indicates the stretching vibrations of the Al-O bonds within the Al_2O_3 lattice. The stretching vibrations of the C-O-C group in the epoxy matrix are observed between $1000\text{--}1300\text{ cm}^{-1}$, while the C=O bonds exhibit stretching peaks between $1700\text{--}1750\text{ cm}^{-1}$ [9]. On the other hand, the peaks at $\sim 550\text{ cm}^{-1}$ and $1600\text{--}1700\text{ cm}^{-1}$ are attributed to bending vibrations of the Al-O-Al linkages and O-H bonds, respectively [12]. FTIR spectra exhibited a vibrational shift to lower wavenumbers in composite coatings upon heat treatment at different temperatures. It could be associated with the temperature dependent structural modifications of polymer matrix, Al_2O_3 particles, and interfacial interactions. With the increase in treatment temperature (300-500°C), the polymer matrix i.e., epoxy resin underwent thermal degradation resulting to the breakage of C-O. It could lead to the decreased bond strength and hence observed lower wavenumber shift [13,14]. The lower wavenumber shift could also be associated with the weakening or breaking of hydrogen bonds of polymer matrix and at interfacial region [15]. As a result of heat treatment, the structural characteristics (vibrational features of bonds) of composites could also be affected by the thermal characteristics (thermal conductivity and thermal expansion) of polymer matrix (i.e., epoxy) and ceramic (Al_2O_3) reinforcement by inducing localized stresses [16]. Lastly, the increased mobility of polymer chains at higher temperatures can alter the interactions between the chains and with the Al_2O_3 particles, contributing to the observed redshift in the FTIR spectrum [13,14]. These reasons could also be the factor behind the omission of some bands in heat-treated coatings between $1200\text{--}1500\text{ cm}^{-1}$.

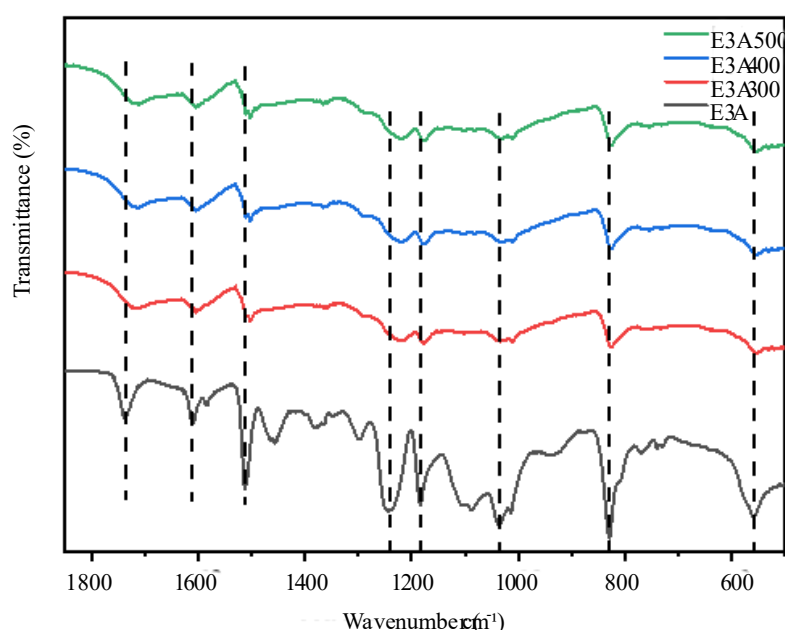


Figure 2. FTIR spectra of epoxy- Al_2O_3 coatings with different thermal treatments.

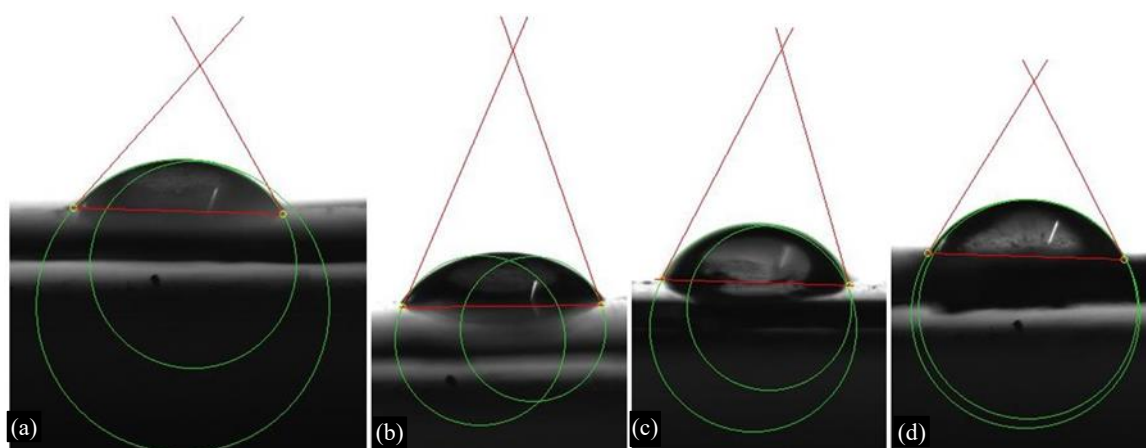


Figure 3. The contact angle of (a) E3A, (b) E3A300, (c) E3A400 and (d) E3A500.

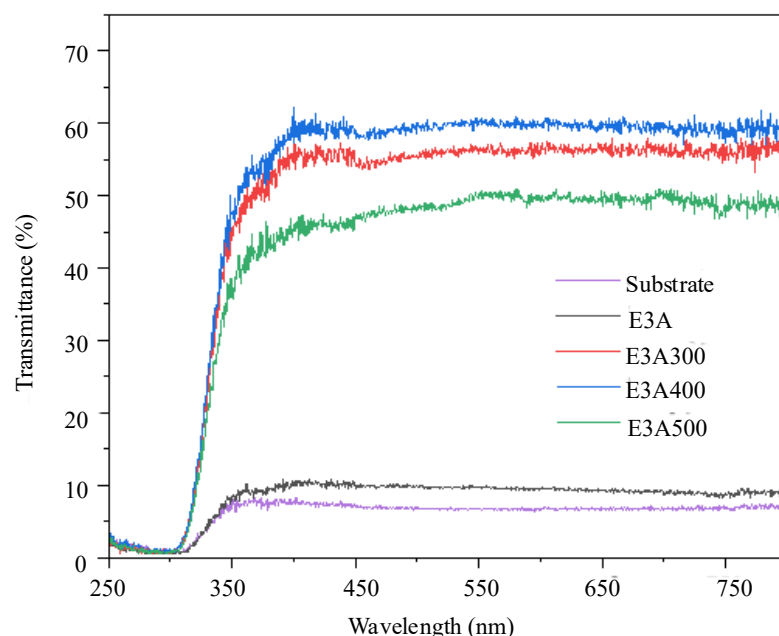


Figure 4. Transmittance spectra of epoxy- Al_2O_3 coatings with different thermal treatments.

Contact Angle Analysis

Figure 3 shows the contact angle measurements for different samples of epoxy- Al_2O_3 coatings. These measurements indicate that the contact angles for all samples vary slightly, i.e., $69.4 \pm 0.13^\circ$ for the E3A sample, while for E3A300, E3A400 and E3A500, it came out to be 69.4 ± 0.21 , 69.6 ± 0.26 , and $69.7 \pm 0.18^\circ$, respectively. The unchanged contact angle of the epoxy- Al_2O_3 coating at various temperatures can be attributed to several factors. The coating's stable surface chemistry, smooth morphology, and small, uniformly distributed Al_2O_3 particles may contribute to its hydrophobicity. Additionally, the inherent hydrophobicity of the epoxy resin itself could play a role. A combination of these factors likely leads to the consistent contact angle observed at room temperature and after exposure to elevated temperatures [17,18].

Optical Transmittance Analysis

Optical transmittance study revealed that the prepared polymer-based coating was not completely transparent in the visible region of the electromagnetic spectra. Figure 4 suggested that the heat treatment enhanced the transparent feature of the coating significantly. The substrate and E3A exhibited 7-10% transmittance in the visible region, while E3A300-E3A500 exhibited $>50\%$ transparency in the

same region. Among these heat-treated samples, it was observed that treatment at 400 °C (E3A400) yielded maximum transmission (~60%) as compared to other E3A300 (<50%) and E3A500 (~55%) samples. These observations opened a gate to explore the possibility of transmittance optimization for filler concentration and heat treatment of epoxy-based coatings.

CONCLUSIONS

This study investigates the thermal stability of the contact angle in epoxy-Al₂O₃ coatings subjected to elevated temperatures. The coatings were exposed to temperatures of 300°C, 400°C, and 500°C, and the contact angle was measured at each stage. Results indicate that the contact angle remains approximately unchanged across the tested temperature range. The stability is attributed to the inherent thermal resistance of the epoxy resin and the inert nature of Al₂O₃ particles, which prevent significant changes in surface morphology and chemistry. The preservation of surface roughness and minimal oxidation of the epoxy matrix likely contribute to the consistent contact angle observed. These findings suggest that transparent (visible region) epoxy-Al₂O₃ coatings maintain their wettability characteristics even under high-temperature conditions, making them suitable for applications where thermal resistance and stable surface properties are desired.

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