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Research Paper

MICROSTRUCTURAL CHARACTERIZATION OF SIC-AL₂O₃ REINFORCED PLA COMPOSITES FABRICATED FOR FUSED DEPOSITION MODELING

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Abstract

Owing to its ease of processing and biodegradability, polylactic acid (PLA) is an instrumental thermoplastic in fused deposition modelling (FDM). Besides, the properties, such as mechanical performance and thermal stability are inadequate, thereby limiting the use of neat PLA for advanced engineering applications. In this study, a hybrid PLA composite is developed by reinforcing the neat PLA with silicon carbide (SiC) and aluminum oxide (Al₂O₃) nanoparticles. Further, the developed hybrid PLA composite is characterized to investigate its microstructural properties, followed by the preparation of the components through controlled drying, blending, and filament extrusion. The significant properties of the prepared composites are investigated using X-ray diffraction (XRD), field-emission scanning electron microscopy (FESEM), and energy-dispersive spectroscopy (EDS). The inclusion of SiC and Al₂O₃ nanoparticles improved the crystallinity of the composites, as inferred by the XRD analysis. Enhanced interfacial particle dispersion and interactions between reinforcements and the PLA matrix are observed through FESEM analysis. The presence of carbon, silicon, aluminum, and oxygen is detected through EDS analysis. Thus, the hybrid ceramic composite yields improved microstructural and properties of PLA, making it suitable for developing composite filaments which are desirable for advanced FDM-based engineering applications.

Keywords: Polylactic Acid (PLA), Silicon Carbide (SiC), Aluminum Oxide (Al₂O₃), Hybrid Composites, Additive Manufacturing, Fused Deposition Modeling (FDM), Microstructural Characterization.

1. Introduction

Owing to shorter production cycles, improved design flexibility and ability to

fabricate intricate geometries, the implementation of additive manufacturing (AM) has emerged as a promising

manufacturing technology [1]. Among the many AM techniques, fused deposition modelling (FDM) has gained substantial attention due to its compatibility with thermoplastic polymers [2]. The thermoplastic filaments are melted and deposited layer by layer, ultimately yielding three-dimensional specimens with the required geometries and sufficient dimensional accuracy. FDM has accelerated the fabrication of high-performance polymer composites for various applications in the aerospace, automotive, and biomedical sectors [3]. Owing to their ease of processing, biodegradability and adequate dimensional stability, the implementation of polyactic acid (PLA) thermoplastic polymers is instrumental in FDM [4]. Also, compared to conventional petroleum-based polymers, PLA is more sustainable [5]. However, neat PLA offers limited mechanical performance, thermal stability, and wear resistance, thereby limiting its use for advanced engineering applications [6]. The performance of PLA, including mechanical, tribological, physicochemical, and thermal properties, can be improved by incorporating ceramic reinforcements into the polymer matrix [7]. Among many ceramic fillers, silicon carbide (SiC) offers high hardness, exceptional wear resistance, and thermal stability, while aluminum oxide (Al_2O_3) offers better corrosion resistance, chemical stability, and stiffness [8-9]. The reinforcement of these fillers into the PLA matrix could eventually enhance the thermal stability, stiffness, interfacial bonding and the overall structural integrity of the components fabricated through FDM [10-11]. It is reported that the reinforcement of

SiC alone in the PLA matrix enhances particle-matrix interaction and improves wear resistance and load transfer [12]. On the other hand, improved stiffness and hardness are achieved by reinforcing Al_2O_3 alone in the PLA matrix [13]. Unlike single-filler systems, the hybrid reinforcement of SiC & Al_2O_3 enables a synergetic interaction across both fillers and could lead to balanced, superior properties [14].

Moisture can make PLA extremely hygroscopic and vulnerable to hydrolytic degradation during thermal processing, thereby affecting the final quality [15]. Further, the ceramic nano-fillers retain surface moisture and can lead to reduced interfacial bonding within the polymer matrix. Therefore, appropriate drying of PLA granules and ceramic reinforcements before blending and extrusion is indispensable for achieving the uniform dispersion and defect-free filament fabrication. Further, better dimensional consistency and process stability are achieved through proper extrusion process parameters, such as screw speed and barrel temperature [16]. On the other hand, the influence of ceramic reinforcements on the structural performance of PLA composites fabricated via AM can be assessed through microstructural and physicochemical characterization. The analyses, such as X-ray diffraction (XRD), field-emission scanning electron microscopy (FESEM), and energy-dispersive spectroscopy (EDS), are being used for investigating the distribution of particles, morphology, crystallinity, interfacial compatibility and elemental composition of reinforced PLA composites [17]. Despite numerous studies

investigating ceramic-reinforced PLA composites, the effect of hybrid reinforcement with SiC & Al₂O₃ on the microstructural properties of PLA polymer composites used in AM applications is rarely explored [18]. The present study focuses on developing a hybrid PLA composite by reinforcing the neat PLA with SiC and Al₂O₃ nanoparticles. Further, the developed hybrid PLA composite is characterized to investigate its microstructural properties, followed by the preparation of the components through FDM. The influence of hybrid ceramic reinforcements on the structural, chemical, and morphological behavior is investigated using XRD, FESEM, and EDS analyses. The findings of this study provide potential insights and contribute towards the fabrication of high-performance PLA composites, which are desirable for advanced FDM-based engineering applications.

2. Materials and Methods

2.1 Materials

In this study, commercial-grade polylactic acid (PLA) and ceramic nanopowders were used as the matrix and reinforcements, respectively. The pure PLA granules (grade FY804) were sourced from 2M Biotech (India), and nano-scale silicon carbide (SiC) and aluminum oxide (Al₂O₃) powders were obtained from Saveer Matrixnano Pvt. Ltd. (India). These PLA granules approximately had a density of 1.24 g·cm⁻³, melt flow index (MFI) of 3.0 g/10 min (ISO 1133-A), glass transition temperature (T_g) of 60 °C, melting point of 155 °C (DSC), tensile strength of 45 MPa (ISO 527-1), tensile modulus of 3500 MPa (ISO 527-1), and

elongation at break $\leq 5\%$. The granules were supplied as pellets <3 mm in size. The SiC and Al₂O₃ fillers were provided as nanopowders (average particle size “as supplied” by the vendor). Silicon carbide is an extremely hard ceramic (Vickers hardness on the order of 2300–2600) with high thermal conductivity (~60–120 W/m·K), and Al₂O₃ is also very hard (tensile hardness up to ~15–22 GPa) with excellent thermal stability (service temperatures above ~1500 °C).

2.2 Composite Preparation and Filament Extrusion

As PLA is extremely hygroscopic and vulnerable to hydrolytic degradation during thermal processing, the PLA granules and the ceramic nano-fillers (SiC and Al₂O₃) were dried in a vacuum oven at 40°C for 1 hour to remove moisture [19]. Surface moisture on nano-fillers can lead to poor interfacial bonding; this vacuum drying enhances uniform dispersion and processing stability during fabrication, eventually resulting in defect-free filament fabrication [19]. Post-drying, the neat PLA granules and composite reinforcements (SiC & Al₂O₃) were blended at varying reinforcement ratios of 0:0, 75:25, 50:50, and 25:75, while maintaining a constant total filler loading of 3 wt. % relative to the PLA matrix.

A double-cone blender was used for performing the blending operation, as the double-cone blunder has the scope of achieving homogeneous dispersion of ceramic reinforcements throughout the PLA matrix. For ensuring the uniform microstructural properties, and to inhibit the nano-particle agglomeration, controlled blending was imperative. For achieving the

controlled blending, a rotational speed of 20 rpm having 20 min per cycle is maintained, for a total of 12 blending cycles per batch [20]. The blended feedstock was processed further through a screw-type electric filament extruder. This extruder contains a cylindrical barrel (hollow) connected with external electric heaters, and the screw was driven through a geared motor. The blended composite feedstock was supplied to the extruder through the hopper, where it is subjected to forward melting by screw rotation (friction shear) and external heat [21]. The melted composite stock (in molten state) was subsequently channeled to the nozzle and is subjected to extrusion for forming continuous filaments. The employed extrusion mechanism is in line with the earlier reported conventional extrusion techniques for thermoplastic filament production [21-22].

It is imperative to optimize the extrusion parameters for achieving consistent and high-quality filaments. Inconsistent filament diameters can hamper the consistent deposition during printing, whereas a uniform filament diameter helps ensure dimensional accuracy, steady material flow, and print quality during AM [23-26]. In this study, screw rotational speed and barrel temperature were optimized for attaining filaments of diameter 1.75 mm, enabling an improved structural consistency and process reliability, which are well required for FDM. The obtained filaments were characterized by XRD, FESEM, and EDS to investigate the influence of hybrid ceramic reinforcements (SiC & Al₂O₃) on the structural and microstructural properties of the PLA composites.

3. Characterization Techniques

X-ray Diffraction (XRD)

XRD analysis is used to unveil the phases, assess crystallinity and detect structural modifications in the developed hybrid composites (PLA-SiC & Al₂O₃) [27]. The obtained diffraction patterns are analyzed not only to identify the presence of ceramic reinforcements (SiC & Al₂O₃) in the PLA matrix but also to examine the effect of the fillers on the crystallinity behavior of PLA. Examining the modifications induced in diffraction angles and peak intensities enables understanding of the phase interactions and structural stability of the fabricated composites.

Field Emission Scanning Electron Microscopy (FESEM)

FESEM analysis is instrumental for understanding interfacial bonding, surface morphology, filler dispersion, and microstructural features of the fabricated composites. Also, FESEM can provide comprehensive information on agglomeration, void formation, particle distribution, and fracture features [28]. In this study, FESEM is performed, and the micrographs are examined to evaluate the uniform dispersion of nanoparticles (SiC & Al₂O₃) within the PLA matrix and to detect defects (agglomerates, voids, pores, etc.), which could impact the overall performance of the fabricated composites.

Energy Dispersive Spectroscopy (EDS)

The integration of EDS with FESEM enables the elemental composition and computational analysis of composites [29]. In this study, EDS is performed to endorse the elemental composition and distribution of ceramic reinforcements (SiC & Al₂O₃)

with the PLA matrix. The obtained data is examined to identify the existence of carbon, oxygen, silicon, and aluminum elements, thus confirming the successful distribution of ceramic reinforcement (SiC & Al_2O_3) nanoparticles within the fabricated composites.

4. Results and Discussion

The present study focuses on developing a hybrid PLA composite by reinforcing the neat PLA with SiC and Al_2O_3 nanoparticles. Further, the developed hybrid PLA composite is characterized to investigate its microstructural properties, followed by the preparation of the components through FDM. The influence of hybrid ceramic reinforcements on the structural, chemical, and morphological behavior is investigated using XRD, FESEM, and EDS analyses. The findings of this study provide potential insights and contribute towards the fabrication of high-performance PLA composites, which are desirable for advanced FDM-based engineering applications.

4.1 XRD Analysis

XRD analysis is conducted to identify the phases, assess crystallinity and detect structural modifications of neat PLA, SiC nano-powder, Al_2O_3 nano-powder, hybrid composites (PLA-SiC & Al_2O_3) with varying reinforcement ratios (0:0, 75:25, 50:50, and 25:75) of SiC & Al_2O_3 . The obtained diffraction patterns are analyzed not only to identify the presence of ceramic reinforcements (SiC & Al_2O_3) in the PLA matrix, but also to examine the effect of the fillers on the PLA crystallinity [30-31]. The neat PLA exhibited a broad diffraction peak $\sim 22.5^\circ$, which not only confirms the semi-

crystallinity nature but also the existence of both crystalline and amorphous regions in the PLA granules. As the PLA granules are untreated, they lead to limited crystalline ordering, as confirmed by the low-intensity, broad diffraction peak, refer Figure 4.1. On the other hand, pure SiC nano-powder exhibited several sharp diffraction peaks at $\sim 26.76^\circ$, 35.75° , 38.26° , 50.27° , 60.13° , and 71.90° , endorsing the highly crystalline behavior and phase purity of the ceramic reinforcement (SiC nano-powder), as shown in Figure 4.2. Akin to this, the intense impulsive diffraction peaks at $\sim 25.96^\circ$, 35.53° , 43.74° , 57.88° , 66.90° , and 68.57° signify the highly crystalline nature of aluminum oxide (Al_2O_3) nano-powder, as shown in Figure 4.3.

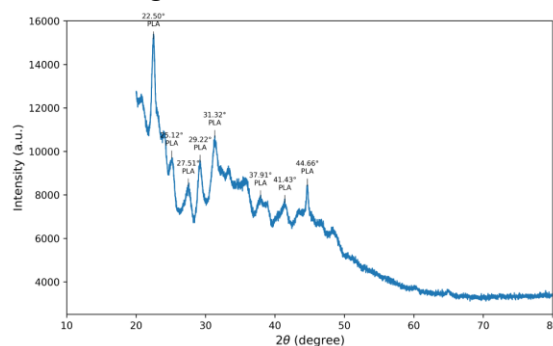


Figure 4.1. XRD pattern of pure PLA granules

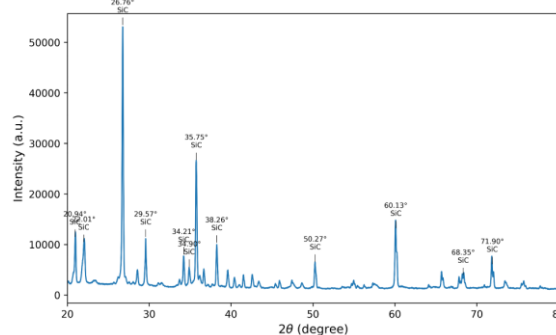


Figure 4.2. XRD pattern of silicon carbide (SiC) nanopowder

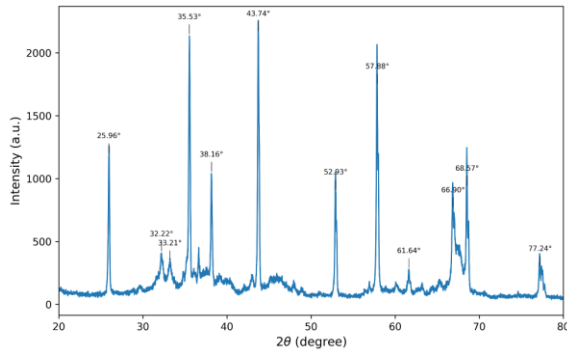


Figure 4.3. XRD pattern of aluminum oxide (Al₂O₃) nanopowder

On the other hand, the hybrid composite (PLA-SiC & Al₂O₃) with the reinforcement ratio of 75% of SiC 25% Al₂O₃ nano particles displayed a core pattern with additional peaks, where the retained core pattern confirms the characteristic of PLA matrix and the additional diffraction peaks correspond to the inclusion of ceramic reinforcements (SiC & Al₂O₃). The diffraction peaks at ~27.01°, 35.58°, and 60.15° resemble the inclusion of ceramic reinforcements (SiC), whereas the diffraction peaks at ~25.16°, 33.31°, 38.01°, 43.64°, and 57.71° resemble the inclusion of ceramic reinforcements (Al₂O₃). At first glance, these diffraction peaks confirm the successful inclusion of both ceramic reinforcements (SiC & Al₂O₃) in the PLA matrix, as shown in Figure 4.4. The hybrid composite (PLA-SiC & Al₂O₃) with the reinforcement ratio of 50% of SiC 50% Al₂O₃ nano particles leads to an increase in the intensity/magnitude of the diffraction peaks when compared with the neat PLA, indicating an effective dispersion and retention of the crystalline nature of both the ceramic reinforcements (SiC & Al₂O₃). The diffraction peaks at ~27.06°, 35.57°, and 60.32° correspond to SiC, whereas the diffraction peaks at 25.21°, 33.36°, 38.04°,

43.69°, 44.66°, 57.89°, and 68.49°, correspond to Al₂O₃, refer Figure 4.5. Additionally, the overlapping diffraction peak at ~35° confirms the presence of both ceramic reinforcement phases (SiC & Al₂O₃) within the PLA matrix (refer Figure 4.5). The hybrid composite (PLA-SiC & Al₂O₃) with the reinforcement ratio of 25% of SiC 75% Al₂O₃ nano particles, exhibited more pronounced alumina behavior, which is confirmed by the diffraction peaks at ~25.16°, 38.04°, 43.65°, 44.66°, 52.81°, 57.83°, and 66.85°, while hardly one diffraction peak about SiC at ~26.98° is visible, refer Figure 4.6.

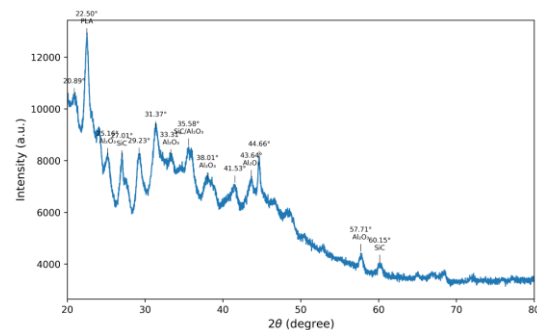


Figure 4.4. XRD pattern of PLA + 2.25 wt.% SiC + 0.75 wt.% Al₂O₃

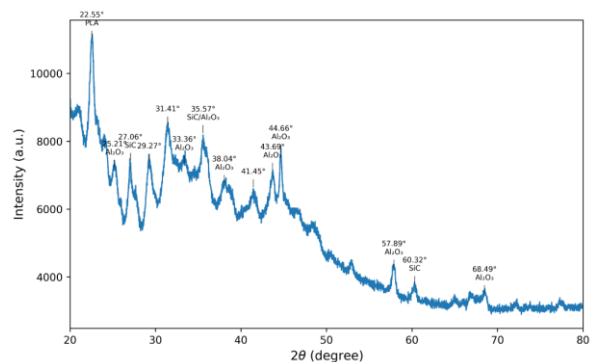


Figure 4.5. XRD pattern of PLA + 1.5 wt.% SiC + 1.5 wt.% Al₂O₃

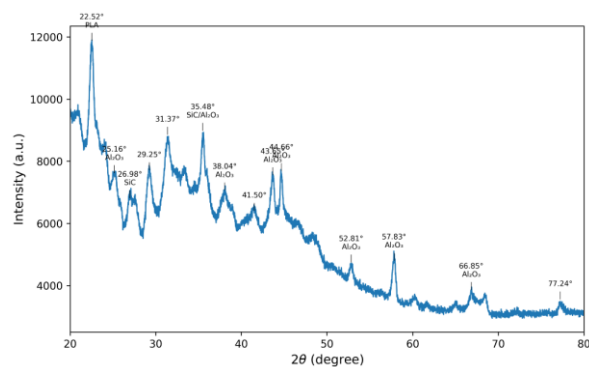


Figure 4.6. XRD pattern of PLA + 0.75 wt.% SiC + 2.25 wt.% Al₂O₃

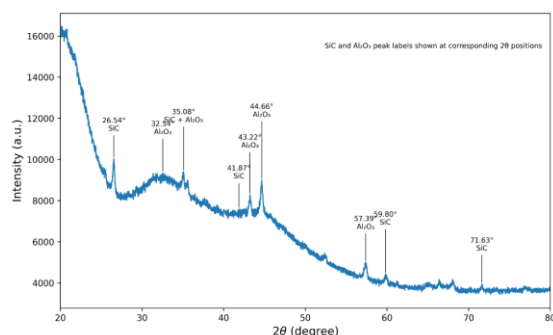


Figure 4.7. XRD pattern of PLA+1.5 wt.% SiC + 1.5 wt.% Al₂O₃ extruded filament

Thus, it can be inferred that the inclusion of ceramic reinforcement (SiC & Al₂O₃) nanoparticles enhances the crystalline nature of the hybrid composite compared with neat PLA. Because ceramic nanoparticles are heterogeneous, they promote crystal growth and enhance crystallinity within the PLA matrix. Additionally, because the ceramic reinforcements (SiC & Al₂O₃) are sharp, they displayed a more intense impulsive diffraction peak than neat PLA. The improved crystallinity would further enhance the mechanical strength, thermal stability and dimensional accuracy of AM components. Also, the additional peaks are hardly visible, confirming that there are no secondary phases in the ceramic reinforcements. This confirms the exceptional phase compatibility of the PLA matrix with the ceramic reinforcements (SiC

& Al₂O₃). The findings of the XRD investigation demonstrate the effectiveness of ceramic nano-reinforcements (SiC & Al₂O₃) in improving the physicochemical properties of PLA composites, which are desirable for advanced FDM-based engineering applications [32-34].

4.2 FESEM Analysis

FESEM analysis is conducted to identify the interfacial bonding, surface morphology, void formation, particle distribution, filler dispersion, void formation, and particle distribution of neat SiC nano-powder and Al₂O₃ nano-powder. The obtained micrographs are analyzed not only to assess dispersion quality but also to understand interfacial characteristics [35-37]. The micrographs of SiC nano-powder reveal irregularly shaped particles with an angular morphology and a relatively uniform distribution. Also, a minor agglomeration with rough surface texture is identified at the nanoscale, indicating the appropriateness of the ceramic reinforcement (SiC) for integration with the PLA matrix for fabricating components, refer Figure 4.8. On the other hand, the ceramic composite (Al₂O₃) displayed a homogeneous distribution and has the potential to form minor agglomerates due to the particles' high surface energy. Figure 4.9 illustrates the morphology of Al₂O₃, which supports its nanoscale nature and its appropriateness for integration with the PLA matrix. Thus, it can be inferred that the ceramic nano-reinforcements (SiC & Al₂O₃) displayed adequate dispersion with marginal clustering throughout the PLA matrix. Also, a satisfactory interfacial adhesion between the PLA matrix and the reinforcements is

observed, indicating effective stress transfer between the PLA matrix and the fillers. Additionally, homogeneous filler distribution is achieved, which assists the mechanical and thermal performance. Though minor agglomeration is reported in a few localized regions, the absence of cracks and voids indicates that blending and extrusion are effective. The findings of the

FESEM analysis demonstrate the effective and uniform dispersion of ceramic nano-reinforcements (SiC & Al_2O_3) within the PLA matrix, leading to homogeneous filler distribution, good interfacial adhesion, and minimal agglomeration, which are desirable for advanced FDM-based engineering applications [35-36].

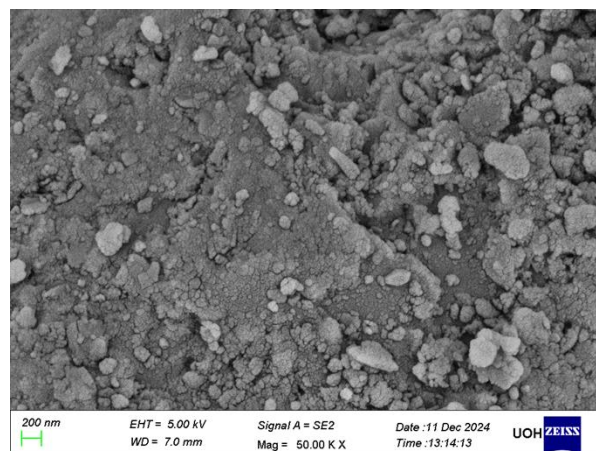
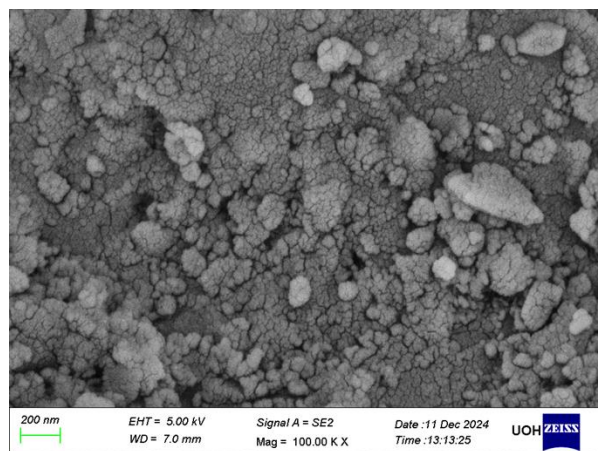


Figure 4.8. FESEM micrograph of silicon carbide (SiC) nanopowder

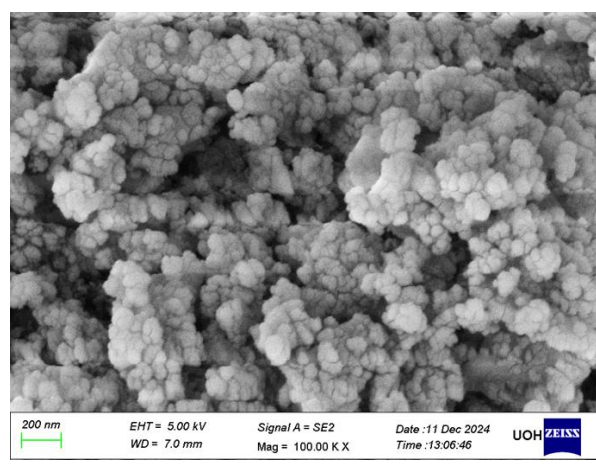
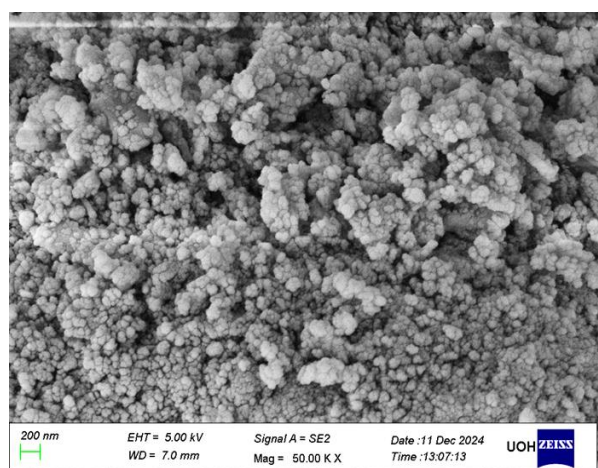


Figure 4.9. FESEM micrograph of aluminum oxide (Al_2O_3) nanopowder

4.5 Energy Dispersive Spectroscopy (EDS) Analysis

Energy Dispersive Spectroscopy (EDS) analysis was performed to confirm the

elemental composition, chemical purity, and successful incorporation of the ceramic reinforcements employed in the present study. EDS is a widely used analytical

technique for elemental identification and compositional analysis of nanomaterials and polymer composites. The EDS spectra obtained for the Al_2O_3 and SiC nanopowders are presented in Fig. 4.22 and Fig. 4.23, respectively.

The EDS spectrum of the Al_2O_3 nanopowder exhibited prominent peaks corresponding to aluminum (Al) and oxygen (O), confirming the characteristic elemental composition of aluminum oxide. The strong peak observed at approximately 1.49 keV corresponds to the Al $K\alpha$ emission line, while the peak near 0.52 keV corresponds to the O $K\alpha$ emission line [37]. The high intensity of these peaks indicates the predominance of aluminum and oxygen within the analyzed sample. **Fig. 4.11** presents the EDS spectrum of Al_2O_3 nanopowder.

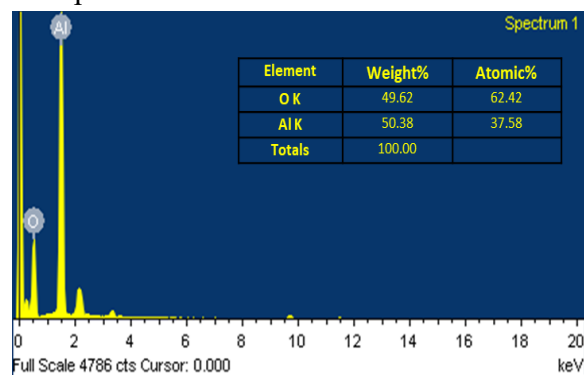


Fig. 4.11. EDS spectrum of Al_2O_3 nanopowder

No significant impurity peaks were observed in the spectrum, demonstrating the high purity of the Al_2O_3 nanoparticles. The elemental composition obtained through EDS is in agreement with the expected stoichiometric composition of aluminum oxide, thereby confirming the successful utilization of high-quality Al_2O_3 nanoparticles as reinforcement material in the PLA hybrid composites.

The EDS spectrum of the SiC nanopowder revealed distinct peaks corresponding to silicon (Si) and carbon (C), which are the principal constituent elements of silicon carbide. The prominent peak observed at approximately 1.74 keV corresponds to the Si $K\alpha$ emission line, while the peak appearing near 0.28 keV corresponds to the C $K\alpha$ emission line [44]. Additionally, a minor oxygen peak was observed around 0.52 keV, which may be attributed to slight surface oxidation of the nanoparticles or atmospheric exposure during sample handling. **Fig. 4.12** presents the EDS spectrum of SiC nanopowder.

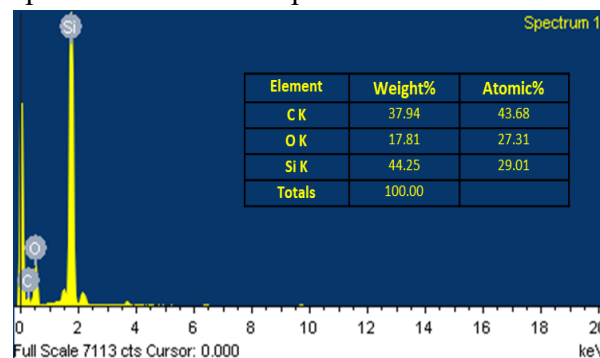


Fig. 4.12. EDS spectrum of SiC nanopowder. The dominance of silicon and carbon peaks confirms the chemical composition and high purity of the SiC nanopowder. The absence of significant foreign elemental peaks indicates that the reinforcement material is free from major contaminants and suitable for composite fabrication.

The EDS results collectively confirm the presence of the key constituent elements required for the fabrication of the PLA hybrid composites. The Al_2O_3 nanoparticles exhibited strong aluminum and oxygen peaks, whereas the SiC nanoparticles displayed prominent silicon and carbon peaks. These observations verify the successful elemental composition of both

ceramic reinforcements and demonstrate their suitability for incorporation into the PLA matrix.

The successful elemental confirmation obtained through EDS analysis supports the XRD findings and further validates the purity and phase integrity of the reinforcement materials. The presence of aluminum, oxygen, silicon, and carbon in their respective spectra confirms that the selected ceramic nanoparticles retained their chemical composition during processing and can effectively contribute to the enhanced physicochemical properties of the developed hybrid composites.

Overall, EDS analysis successfully confirmed the elemental composition and purity of the reinforcement materials and validated the presence of SiC and Al₂O₃ nanoparticles within the developed composite system. The results demonstrate the effectiveness of the material selection, blending, and extrusion processes in achieving successful incorporation of ceramic nanoparticles into the PLA matrix, thereby supporting the development of advanced hybrid composites for additive manufacturing applications [37].

5. Conclusion

In this study, a hybrid PLA composite (PLA-SiC & Al₂O₃) was developed at a constant reinforcement loading of 3 wt.% by reinforcing the neat PLA with silicon carbide (SiC) and aluminum oxide (Al₂O₃) nanoparticles with varying reinforcement ratios (0:0, 75:25, 50:50, and 25:75) of SiC & Al₂O₃. The developed hybrid PLA composites are characterized to investigate its microstructural properties, followed by the preparation of the components through

controlled drying, blending, and filament extrusion. The significant properties of the prepared composites are investigated using X-ray diffraction (XRD), field-emission scanning electron microscopy (FESEM), and energy-dispersive spectroscopy (EDS). The inclusion of SiC and Al₂O₃ nanoparticles improved the crystallinity of the composites, as inferred by the XRD analysis. Enhanced interfacial particle dispersion and interactions between reinforcements and the PLA matrix are observed through FESEM analysis. The presence of carbon, silicon, aluminum, and oxygen is detected through EDS analysis. Thus, the hybrid ceramic composite yields improved microstructural properties of PLA, making it suitable for developing composite filaments which are desirable for advanced FDM-based engineering applications.

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