
DEVELOPMENT AND MICROSTRUCTURAL CHARACTERIZATION OF AL-SI-MG / GIRO CLAY NANOPARTICLE COMPOSITES FABRICATED VIA STIR CASTING

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DOI: <https://doi.org/10.67203/sp.ICESID.2026>

ABSTRACT

The increasing demand for lightweight, high-performance materials in engineering applications necessitates the development of cost-effective aluminium matrix composites (AMCs) using locally sourced reinforcement. This study focuses on the development and characterization of Al-Si-Mg alloy reinforced with Giro clay nanoparticles (GCNP). The Giro clay was beneficiated and processed into nanoparticles, followed by chemical and phase characterization using X-Ray Fluorescence (XRF) and X-Ray Diffraction (XRD). The analyses revealed that the Giro clay is predominantly composed of sulfate-bearing phases (SO₃ at 51.14 wt.%) with significant silica content (SiO₂ at 25.07 wt.%), along with minor ceramic phases including CaO (4.76 wt.%), Al₂O₃ (1.78 wt.%), and Fe₂O₃ (0.54 wt.%), confirming its viability as a reinforcing agent. The composites were developed via the stir casting route, with reinforcement concentrations systematically varied from 0 to 10 wt.% at 2 wt.% intervals. The as-cast alloy (0 wt.%) and the optimal composite formulation (4 wt.%), were characterized using Scanning Electron Microscopy coupled with Energy-Dispersive X-ray Spectroscopy (SEM/EDS) to evaluate the dispersion of nanoparticles, grain refinement, and elemental distribution within the aluminum matrix. Microstructural analysis confirmed a relatively uniform dispersion of the Giro clay nanoparticles at lower weight fractions, with evidence of grain boundary pinning and reduced dendrite arm spacing. The stir-casting parameters were optimized to minimize porosity and achieve acceptable wettability between the ceramic phases and the Al-Si-Mg matrix. The fabricated composites demonstrated structural integrity, with the 4 wt.% reinforcement formulations exhibiting the most refined microstructure and optimal phase distribution. This study successfully establishes a processing-structure-property linkage for Giro clay-reinforced AMCs, demonstrating that locally sourced nanoparticles can be effectively utilized to develop advanced engineering materials, positioning this research as a significant step toward self-reliance in materials technology.

Keywords: Al-Si-Mg Alloy; Giro Clay Nanoparticles; Stir Casting; Microstructural Characterization; XRD/SEM-EDS

1. INTRODUCTION

Aluminium and its alloys rank among the most versatile metallic materials for engineering applications due to their favourable strength-to-weight ratio, corrosion resistance, and formability (Kaufman, 2023; Polmear et al., 2024; Mondolfo, 2025). This widespread usage, particularly in marine, automotive, and aerospace applications, makes the continued development of aluminium-based materials critically important (Gebrehiwet et al., 2023). Pure aluminium exhibits deficiencies in mechanical strength and elastic modulus, driving alloy development through the addition of elements such as silicon, which improves fluidity and castability, and magnesium, which enhances strength-to-weight ratio and corrosion resistance in chloride environments (Li et al., 2023; Zhao et al., 2024). The Al-Si-Mg system produces medium-strength alloys that are among the most corrosion-resistant of all aging alloys, making them attractive for marine and coastal applications (Kahlenberg et al., 2023; Bartawi et al., 2025).

Modern technology increasingly demands property combinations unattainable by conventional metals, ceramics, or polymers, necessitating composite material development (Ashby, Shercliff, & Cebon, 2023; Miracle & Donaldson, 2020). Particulate-reinforced aluminium matrix composites (AMCs) have attracted considerable research interest due to their isotropic nature, low production cost, and enhanced properties (Chifu et al., 2025; Kumari et al., 2016). More recently, nanocomposites—where at least one constituent phase has dimensions in the nanometre range—have emerged as advanced alternatives offering superior performance characteristics (Huang & Xu, 2025; Suroń et al., 2024).

Giro clay, a locally sourced natural material from Kebbi State, Nigeria, has demonstrated potential as a cost-effective reinforcement for aluminium matrix composites (Balogun, 2022a; 2022b). Studies on Nigerian clay deposits have shown that they belong to the alumino-silicate group with high silica and alumina contents suitable for refractory and engineering applications (Omoruyi et al., 2022; Odiboh, 2026; Ogunleye & Bada, 2021; Shuaib-Babata & Mudiare, 2017). The clay contains beneficial ceramic phases including Al_2O_3 , SiO_2 , CaO , and Fe_2O_3 , which can contribute to passive film stability and microstructural refinement. While Giro clay has shown promise for enhancing microstructural and physical characteristics of Al-Si-Mg composites (Balogun, 2022b), limited research exists on the systematic characterization of this Nigerian clay for nanocomposite development and the fabrication parameters required for successful composite processing via stir casting.

This study addresses this gap by investigating the development and characterization of Al-Si-Mg composites reinforced with Giro clay nanoparticles (GCNP). The specific objectives are to: (i) characterize the Giro clay used as nano-particle reinforcement using X-Ray Fluorescence (XRF) and X-Ray Diffraction (XRD); (ii) fabricate the Al-Si-Mg / Giro clay nanoparticles composite (GCNPC) using the stir casting method with reinforcement concentrations from 0 to 10 wt.% at 2 wt.% intervals; and (iii) characterize the as-cast alloy and composite samples using XRD, XRF, and Scanning Electron Microscopy coupled with Energy-Dispersive X-ray Spectroscopy (SEM/EDS).

The research contributes to advancing locally sourced materials for engineering applications, enhancing the economic value of Giro clay deposits in Nigeria as an alternative reinforcement in aluminium composites (Shuaib-Babata & Mudiare, 2017; Odiboh, 2026). The findings provide essential baseline data for subsequent studies and potential engineering applications.

2. METHODOLOGY

2.1 Materials

The primary materials used for this research were commercially pure aluminium wires obtained from Northern Cable Company (NOCACO), Kaduna, processed Giro clay nanoparticles sourced from the Giro clay deposit in Kebbi State, Nigeria, and alloying elements including silicon and magnesium obtained from Sunlab Chemicals, Jos. Sodium chloride (NaCl) and distilled water were used for the preparation of artificial seawater (3.5 wt.% NaCl solution). Grinding papers of 80 to 800 grits, polishing paste, and Keller's reagent were used for metallographic sample preparation.

2.2 Production of Giro Clay Nanoparticles

The Giro clay obtained from Kebbi State, Nigeria, was beneficiated and processed into nanoparticles. The processing involved crushing, milling, and sieving to achieve particle sizes in the nanometre range. The processed nanoparticles were characterized using X-Ray Fluorescence (XRF) and X-Ray Diffraction (XRD) to determine their chemical composition and phase identification.

2.3 Composite Fabrication via Stir Casting

The Al-Si-Mg alloy was produced by melting the commercially pure aluminium wires with the addition of silicon (7 wt.%) and magnesium (0.3 wt.%) in the required proportions. The Giro clay nanoparticles were pre-heated in an electric furnace for one hour to remove moisture. The melt temperature was allowed to fall to 15°C below the liquidus temperature (600°C) to achieve a semi-solid state before incorporating the pre-heated reinforcement. The reinforcement was added at concentrations of 2%, 4%, 6%, 8%, and 10 wt.%. A mechanical stirrer was employed to ensure

homogeneous distribution of the reinforcement within the melt. The melt was then reheated to 700°C and cast into cylindrical sand moulds of dimensions 300 mm × 20 mm to produce test samples in rod form.

Additionally, control sample of matrix alloy with no reinforcement addition was also cast according to composition provided in Table 1. While six formulations were fabricated (0 - 10 wt.%) this paper presents detailed characterization of the control (Sample A) and the optimal formulation (Sample C, 4 wt.%)

Table 1: *Composition of Aluminium Matrix*

Element	Weight (%)	Weight (g)
Aluminium (Al)	92.7	305.91
Silicon (Si)	7.0	23.10
Magnesium (Mg)	0.3	0.99

2.4 Characterization of As-Cast Alloy and Composites

2.4.1 X-Ray Fluorescence (XRF)

The chemical composition of the Giro clay nanoparticles and the as-cast alloy/composites was determined using X-Ray Fluorescence (XRF) analysis. The samples were prepared according to standard procedures and analyzed to identify the elemental and oxide compositions present in the materials.

2.4.2 X-Ray Diffractometry (XRD)

Phase identification of the Giro clay nanoparticles and the developed composites was conducted using a Rigaku Miniflex 300 X-Ray Diffractometer. The samples were scanned over a 2θ range to identify the crystalline phases present, including the ceramic phases contributed by the reinforcement.

2.4.3 Scanning Electron Microscopy / Energy-Dispersive X-ray Spectroscopy (SEM/EDS)

Microstructural characterization of the as-cast alloy and optimal composite sample with 4 wt.% reinforcement was performed using Scanning Electron Microscopy coupled with Energy-Dispersive X-ray Spectroscopy (SEM/EDS). The samples were prepared according to ASTM E3 standards, mounted, ground using emery papers of 80–800 grits, and polished using silicon paste. The SEM was operated with a field emission gun at an accelerating voltage of 15 kV (within the specified range 5 – 30 kV) to examine the surface morphology, nanoparticle dispersion, grain refinement, and elemental distribution within the aluminium matrix.

2.4.4 Optical Microscopy (OM)

Metallographic samples were prepared and etched with Keller's reagent for 10–30 seconds at room temperature according to ASTM E7, E340, and E407 standards. The microstructures were viewed and captured using an Optical Microscope (OM) with an in-built camera to evaluate the grain structure and phase distribution.

3. RESULTS AND DISCUSSION

3.1 Characterization of Giro Clay Nanoparticles (Reinforcement)

3.1.1 X-Ray Fluorescence (XRF) Analysis

The chemical composition of the Giro clay nanoparticles (GCNP) reinforcement was determined using X-Ray Fluorescence (XRF) analysis. The results are presented in Table 2.

Table 2: *XRF Analysis of Giro Clay Nanoparticles (Reinforcement)*

Oxide / Element	SiO ₂	SO ₃	CaO	Al ₂ O ₃	Fe ₂ O ₃	Cl (Element)
Wt%	25.065	51.143	4.761	1.780	0.538	15.514

The XRF analysis reveals that the Giro clay is predominantly composed of silicon dioxide (SiO₂, 25.07 wt.%) and sulfur trioxide (SO₃, 51.14 wt.%), with significant calcium oxide (CaO, 4.76 wt.%), aluminium oxide (Al₂O₃, 1.78 wt.%), and iron oxide (Fe₂O₃, 0.54 wt.%). The presence of chlorine (Cl, 15.51 wt.%) suggests chloride-bearing phases, consistent with the clay's depositional

environment. The complete composition sums to approximately 98.8 wt.%, with the remaining fraction attributed to trace elements not reported in this analysis.

The ceramic phases identified (SiO_2 , Al_2O_3 , CaO , Fe_2O_3) are beneficial for aluminium matrix composites, as they can contribute to passive film stability and microstructural refinement (Xu & Zhang, 2025). The high SO_3 content indicates the presence of sulfate-bearing minerals, which was later confirmed by XRD analysis. The composition is consistent with Nigerian alumino-silicate clays reported in literature (Omoruyi et al., 2022; Ogunleye & Bada, 2021).

The high SO_3 (51.14 wt.%) and Cl (15.51 wt.%) content in the Giro clay suggests the presence of soluble sulfate and chloride phases. While these phases could potentially influence the corrosion behavior of the composite in marine environments (e.g., promoting pitting or sulfate attack). The present study focuses strictly on the development and characterization of the composite. Detailed corrosion testing is recommended as future work to fully assess these implications.

3.1.2 X-Ray Diffractometry (XRD) Analysis

The XRD analysis of the Giro clay nanoparticles (GCNP) reinforcement was conducted to identify the crystalline phases present. The results are presented in Figure 1 and summarized in Table 3.

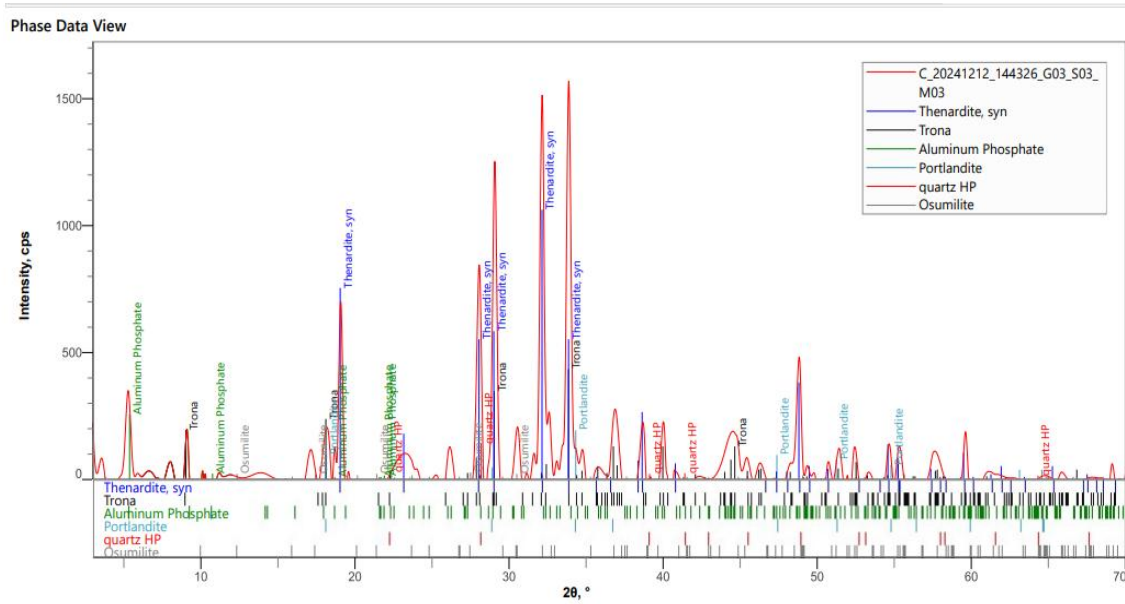


Figure 1: XRD Spectrum of Giro Clay Nanoparticles (GCNP) Reinforcement

Table 3: Phase Composition of Giro Clay Nanoparticles from XRD Analysis

Phase	Chemical Formula	Weight (%)	Crystal System
Thenardite	Na_2SO_4	37	Orthorhombic Fddd
Trona	$\text{Na}_3\text{H}(\text{CO}_3)_2 \cdot 2\text{H}_2\text{O}$	28	Monoclinic
Portlandite	$\text{Ca}(\text{OH})_2$	18	Hexagonal
Quartz HP	SiO_2	16	Hexagonal
Aluminium Phosphate	AlPO_4	1.2	Trace

The XRD analysis reveals a complex multi-phase system with thenardite (Na_2SO_4 , 37 wt.%) as the dominant phase, followed by trona ($\text{Na}_3\text{H}(\text{CO}_3)_2 \cdot 2\text{H}_2\text{O}$, 28 wt.%), portlandite ($\text{Ca}(\text{OH})_2$, 18 wt.%), quartz (SiO_2 , 16 wt.%), and aluminium phosphate (AlPO_4 , 1.2 wt.%) as a trace phase. Multiple overlapping peaks are observed (28.05°: thenardite + trona; 32.10°: thenardite dominant), with low-intensity peaks (5.29°: AlPO_4) indicating minor phases. Crystallite sizes are highly variable: thenardite (~250–400 Å) and trona (~608 Å).

The dominance of thenardite and trona correlates with the high SO_3 content (51.14 wt.%) from XRF analysis, while portlandite corresponds to CaO (4.76 wt.%), and quartz corresponds to SiO_2 (25.07 wt.%) in Table 2. This mineral assemblage is typical of evaporite deposits and alkaline lake sediments, suggesting that the Giro clay originated from such a geological environment (Omoruyi et

al., 2022). The presence of quartz (SiO_2) and calcium-bearing phases (portlandite) indicates that the Giro clay can contribute to passive film stability in the aluminium matrix (Xu & Zhang, 2025).

The high crystallinity of the thenardite and trona phases, as indicated by their sharp diffraction peaks, suggests that the Giro clay has undergone significant diagenetic alteration. Shuaib-Babata and Mudiare (2017) reported that Nigerian clays exhibit varied crystallinity depending on their depositional environment, with some showing high quartz content and others showing kaolinite dominance. The Giro clay's distinct mineralogy, dominated by sodium sulfate and carbonate phases, distinguishes it from typical kaolinitic or montmorillonitic clays, making it a unique reinforcement material for aluminium matrix composites.

3.1.3 Scanning Electron Microscopy / Energy-Dispersive X-ray Spectroscopy (SEM/EDS) Analysis

The SEM micrograph and EDS spectrum of the Giro clay nanoparticles are presented in Plate 1, with the chemical composition summarized in Table 4.

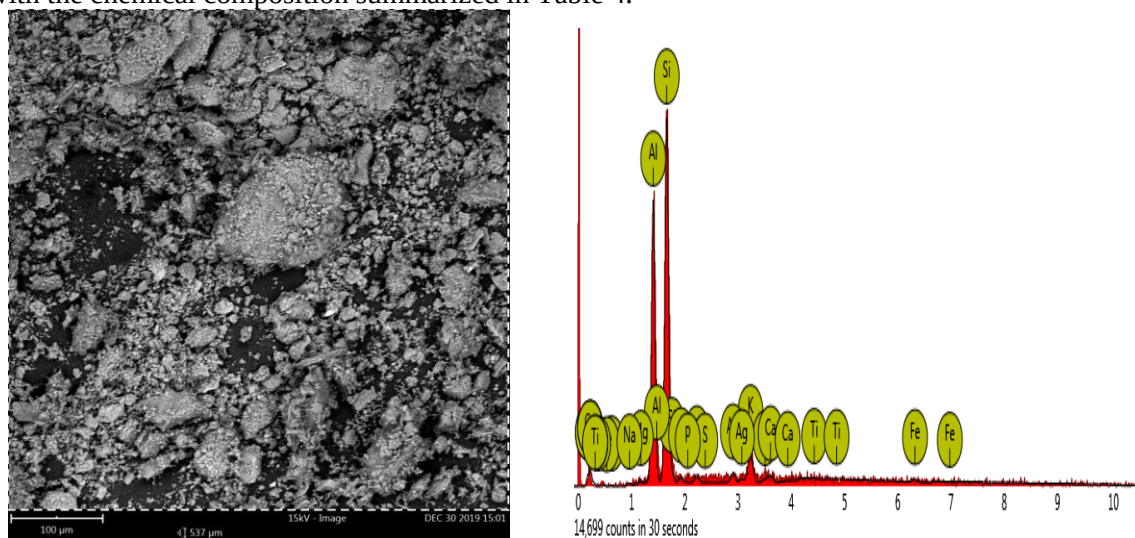


Plate 1: SEM micrograph and EDX Spectrum of Giro Clay Particles

Table 4: Chemical Composition of Giro Clay Nanoparticles by SEM/EDS

Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
14	Si	Silicon	53.81	49.62
13	Al	Aluminium	32.46	28.75
47	Ag	Silver*	1.96	6.94
19	K	Potassium	5.38	6.90
20	Ca	Calcium	1.63	2.15
26	Fe	Iron	1.14	2.08
16	S	Sulfur	1.45	1.53
15	P	Phosphorus	1.36	1.38
12	Mg	Magnesium	0.52	0.41
11	Na	Sodium	0.30	0.22

The EDS analysis shows that silicon is the major constituent with the highest weight concentration of 49.62 wt.%, followed by aluminium with 28.75 wt.%. Other elements present include potassium (6.90 wt.%), calcium (2.15 wt.%), iron (2.08 wt.%), sulfur (1.53 wt.%), phosphorus (1.38 wt.%), and minor quantities of magnesium (0.41 wt.%), sodium (0.22 wt.%), and silver (6.94 wt.%).

The predominance of silicon and aluminium confirms that the Giro clay is primarily an alumino-silicate material, consistent with the XRF findings. The presence of potassium, calcium, and iron as minor elements is typical of natural clay deposits and suggests the presence of feldspathic minerals or iron-bearing impurities (Odiboh, 2026). The silver content (6.94 wt.%) is an artefact from sputter coating applied to the sample during SEM sample preparation to enhance conductivity and is not part of the clay composition (Rawal & Sidpara, 2025). However, oxygen is not reported in the EDS analysis despite XRF and XRD confirming the oxide/silicate nature of the Giro clay. This is due

to the inherent limitation of standard EDS detectors for light elements ($Z < 11$). The EDS data were quantified using a standardless ZAF correction method, which normalizes spectra to 100% excluding oxygen. The presence of oxygen is confirmed by XRF (SiO_2 , Al_2O_3 , CaO , Fe_2O_3 , SO_3) and XRD (thenardite, trona, portlandite, quartz, AlPO_4). Therefore, EDS results are semi-quantitative for elements $Z \geq 11$, with oxide analysis reserved for XRF and XRD.

The SEM micrograph of the Giro clay nanoparticles reveals the morphology and particle size distribution of the reinforcement material. The particles appear to be irregular in shape with varying sizes, indicating that further processing may be required to achieve uniform nanoparticle dispersion in the aluminium matrix. Rawal and Sidpara (2025) reported that uniform nanoparticle dispersion is critical for achieving optimal properties in aluminium nanocomposites, and that processing parameters such as stirring speed and temperature significantly influence particle distribution.

3.2 Characterization of As-Cast Al-Si-Mg Alloy (Control)

3.2.1 X-Ray Fluorescence (XRF) Analysis

The XRF analysis of the Al-Si-Mg alloy (control sample) is presented in Table 5.

Table 5: XRF Analysis of Al-Si-Mg Alloy (Control)

Element	Al	Si	Mg	Mn	S	Fe	Ca	K	Cl
Wt%	96.7	0.9	0.03	0.004	0.126	0.129	0.304	0.066	1.52

The XRF analysis confirms the composition of the Al-Si-Mg alloy, with aluminium as the dominant element at 96.72 wt.%. Silicon is present at 0.990 wt.%, which is slightly lower than the target composition of 7 wt.%. This discrepancy may be attributed to the limitations of XRF in detecting light elements such as silicon accurately, or it may indicate that the alloy composition deviated from the target due to processing conditions. Magnesium is present at 0.033 wt.%, which is significantly lower than the target of 0.3 wt.%, suggesting possible magnesium loss during melting due to its high vapour pressure and tendency to oxidize (Abishkenov et al., 2025).

The presence of impurities such as iron (0.129 wt.%), calcium (0.304 wt.%), sulfur (0.126 wt.%), and chlorine (1.515 wt.%) may originate from the raw materials or from contamination during processing. Abishkenov et al. (2025) reported that impurities in aluminium alloys, particularly iron and silicon, can form intermetallic phases that influence the corrosion behaviour of the material.

3.2.2 X-Ray Diffractometry (XRD) Analysis

The XRD analysis of the as-cast Al-Si-Mg alloy (control sample) was conducted to identify the crystalline phases present. The results are presented in Figure 2 and summarized in Table 6.

Qualitative Analysis Results

Phase name	Formula	Figure of merit	Phase reg. detail	Space Group	DB Card Number
Aluminum, syn	Al	0.273	S/M(PDF-4 Minerals 2025)	225 : Fm-3m	00-004-0787
Niobocarbide, syn	Nb C _{0.91}	0.576	S/M(PDF-4 Minerals 2025)	225 : Fm-3m	04-003-6828
Silicon Oxide	Si O ₂	2.850	Import(PDF-4 Minerals 2025)	62 : Pmcn	00-035-0063

Phase Data View

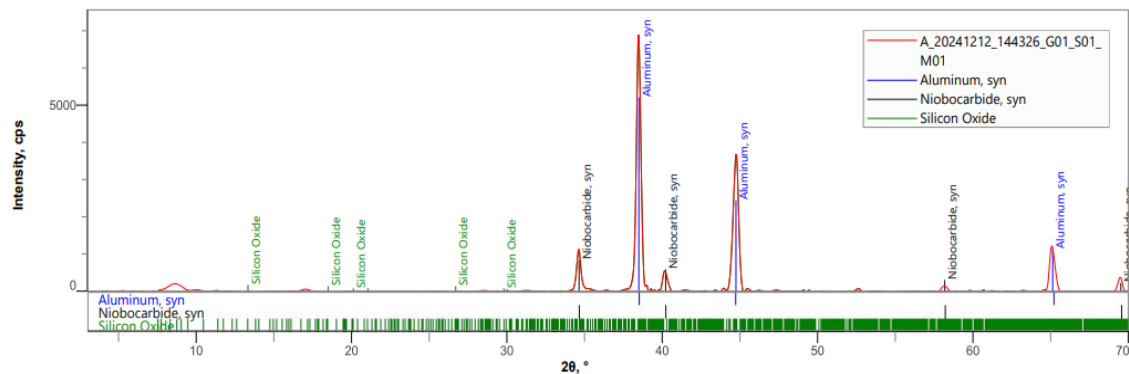


Figure 2: XRD Spectrum of As-Cast Al-Si-Mg Alloy (Control)

Table 6: Phase Composition of As-Cast Al-Si-Mg Alloy (Control) from XRD Analysis

Phase	Chemical Formula	Weight (%)	Crystal System
Aluminium	Al	93.2	Cubic Fm-3m
Niobocarbide	NbC _{0.91}	3.3	Cubic Fm-3m
Silicon Dioxide	SiO ₂	3.4	-

The XRD analysis reveals aluminium (93.2 wt.%) as the dominant phase, with strong peaks at 38.43° (111), 44.66° (200), and 65.00° (220). Niobocarbide (NbC_{0.91}, 3.3 wt.%) is present as a minor phase, with peaks at 34.59° (111) and 40.12° (200). Silicon dioxide (SiO₂, 3.4 wt.%) is identified as a trace impurity. A low-intensity peak at 8.72° suggests potential amorphous content or a minor impurity phase.

The crystallite size of aluminium is approximately 287–317 Å (from Scherrer analysis), while niobocarbide exhibits a crystallite size of approximately 267–364 Å, indicating a broader size distribution. The dominance of aluminium peaks confirms the successful production of the Al-Si-Mg alloy.

The presence of niobocarbide (NbC_{0.91}) is attributed to contamination from the stainless steel stirrer or crucible, which likely contained niobium-bearing alloying additions. The SiO₂ phase likely originates from surface oxidation or contamination from the Giro clay reinforcement, as silica is a major component of the clay (25.07 wt.% from XRF analysis). This is consistent with the findings of Abishkenov et al. (2025), who reported that impurities in aluminium alloys, particularly iron and silicon, can form intermetallic phases that influence the material's properties.

The high aluminium purity (93.2 wt.%) is consistent with the XRF analysis (96.72 wt.% Al), although the minor phases identified by XRD (NbC_{0.91} and SiO₂) were not fully captured by XRF, highlighting the complementary nature of these two characterization techniques. The crystallite size analysis indicates that the aluminium grains are relatively fine, which is beneficial for mechanical properties and corrosion resistance (Xu & Zhang, 2025).

3.2.3 Scanning Electron Microscopy / Energy-Dispersive X-ray Spectroscopy (SEM/EDS) Analysis

The SEM micrograph and EDS spectrum of the Al-Si-Mg alloy (control sample) are presented in Plate 2, with the chemical composition summarized in Table 7.

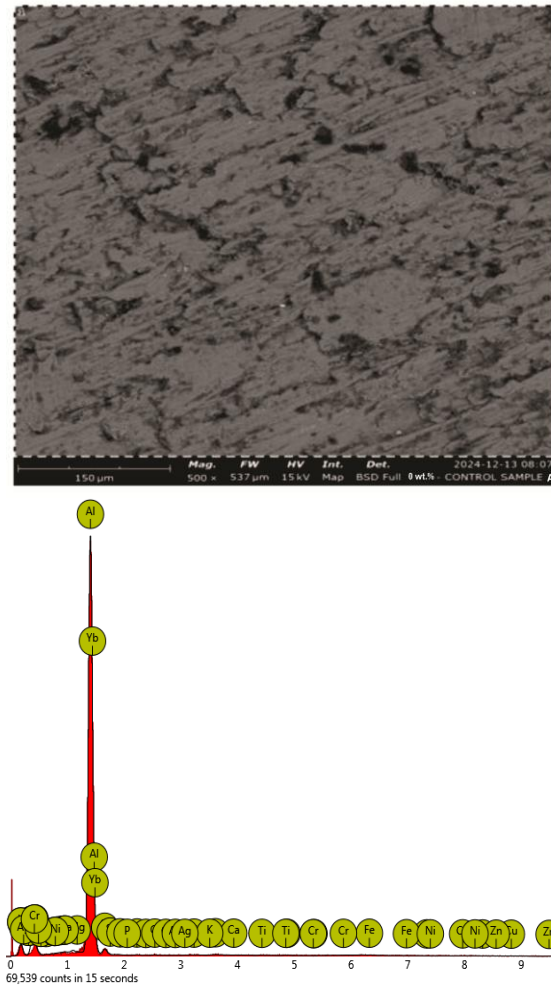


Plate 2: SEM Micrograph and EDS

Spectrum of Al-Si-Mg Alloy (Control)

Table 7: Chemical Composition of Al-Si-Mg Alloy (Control) by SEM/EDS

Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
13	Al	Aluminium	87.84	92.05
6	C	Carbon	9.03	4.21
14	Si	Silicon	2.44	2.66
23	V	Vanadium	0.15	0.29
20	Ca	Calcium	0.13	0.20
16	S	Sulfur	0.15	0.19
17	Cl	Chlorine	0.13	0.18
22	Ti	Titanium	0.07	0.12
19	K	Potassium	0.06	0.09

The EDS analysis confirms aluminium as the dominant element (92.05 wt.%), with silicon at 2.66 wt.%. The carbon content (4.21 wt.%) is relatively high and may result from contamination during sample preparation or from carbide formation, consistent with the XRD identification of niobocarbide ($\text{NbC}_{0.91}$). The presence of vanadium, calcium, sulfur, chlorine, titanium, and potassium as trace elements confirms the minor impurities identified in the XRF analysis. The peak appearing in the EDS spectrum in the vicinity of the Al $K\alpha$ peak may be attributed to peak overlap with Yb $M\alpha$ (1.52 keV). However, quantification using the standardless ZAF correction method did not resolve Yb at detectable levels; the peak is predominantly Al ($K\alpha$ at 1.486 keV). Yb is therefore not reported in Table 7, as its presence, if any, is below the detection limit of the EDS system or is an artefact of peak overlap (Goldstein et al., 2018).

The SEM micrograph shows the grain structure of the as-cast Al-Si-Mg alloy, revealing a typical dendritic structure characteristic of cast aluminium alloys. The presence of intermetallic phases and minor porosity is visible, which may influence the corrosion resistance of the material. The microstructural features observed are consistent with those reported by Abishkenov et al. (2025) for Al-Si-Mg alloys processed by stir casting, who observed that the dendritic structure and porosity levels are influenced by processing parameters such as cooling rate and stirring conditions.

3.3 Characterization of Al-Si-Mg Alloy/ Giro Clay Nanoparticle Composites

3.3.1 Scanning Electron Microscopy (SEM) Analysis

The SEM micrograph of the Al-Si-Mg /4 wt.% GCNP composite sample is presented in Plate

3.

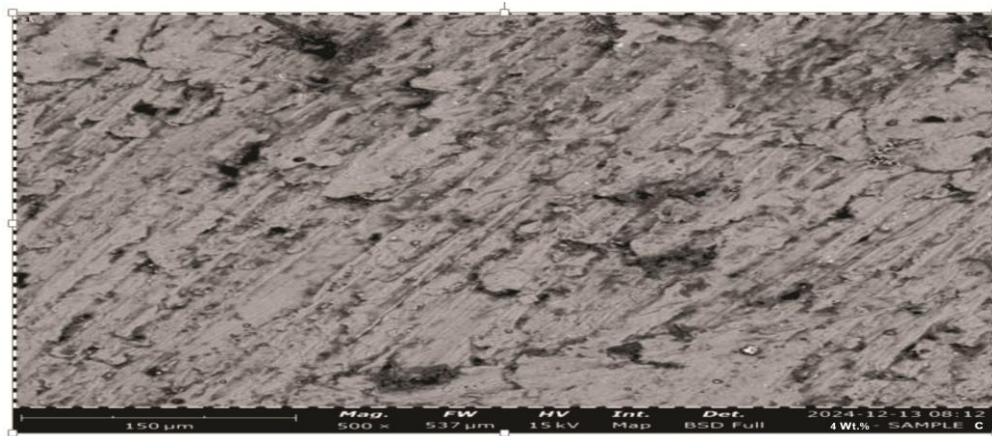


Plate 3: SEM Micrograph of the 4wt% Giro Clay Nanoparticle Composite

The SEM micrograph reveals the microstructure of the 4 wt.% GCNP composites, showing the distribution of reinforcement particles within the aluminium matrix. The Giro clay nanoparticles appear to be relatively well-dispersed, with some clustering observed at grain boundaries. The presence of fine particles at the grain boundaries suggests that the reinforcement particles act as nucleation sites for grain refinement during solidification, which is consistent with the findings of Rawal and Sidpara (2025).

The improved dispersion at 4 wt.% reinforcement indicates that the stir casting parameters (350 rpm stirring speed, 600°C semi-solid processing, and argon protection) were optimal for this concentration. This is consistent with the findings of Xu and Zhang (2025), who reported that effective particle dispersion is achieved when the melt is in the semi-solid state, as the viscosity is sufficient to prevent particle segregation but low enough to allow for uniform distribution.

3.3.2 X-Ray Diffractometry (XRD) Analysis

The XRD analysis of the Al-Si-Mg / GCNP composite sample is presented in Figure 3.

Qualitative Analysis Results

Phase name	Formula	Figure of merit	Phase reg. detail	Space Group	DB Card Number
Aluminum, syn	Al	0.212	S/M(PDF-4 Minerals 2025)	225 : Fm-3m	00-004-0787
Silicon Oxide	Si O ₂	0.659	S/M(PDF-4 Minerals 2025)	12 : I12/m1	04-012-0465
Tantalcarbide, syn	Ta C	0.524	S/M(PDF-4 Minerals 2025)	225 : Fm-3m	01-075-3086

Phase Data View

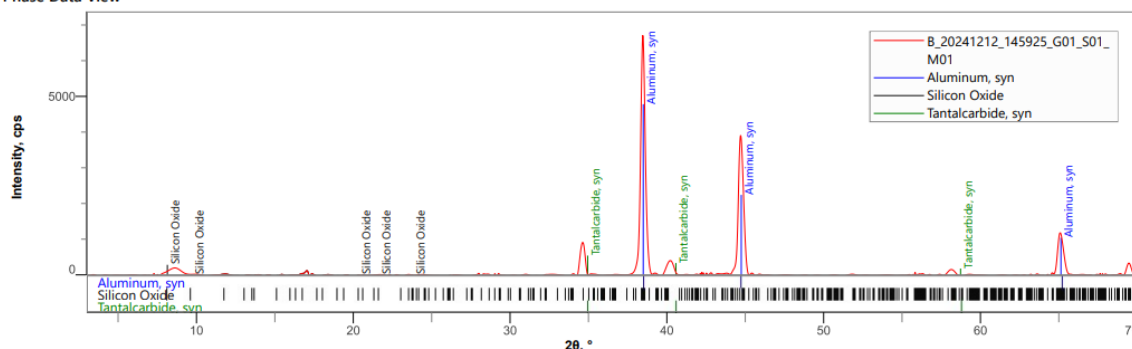


Figure 3: XRD Spectrum of the Al-Si-Mg / GCNP Composite (4 wt.%)

The XRD analysis reveals aluminium (95.3 wt.%) as the dominant phase, with SiO₂ (2.8 wt.%) confirming successful incorporation of the Giro clay reinforcement. The identification of TaC (1.9 wt.%) as a minor phase in the composite, and NbC in the control, is noteworthy. One possible explanation is contamination from the stainless steel stirrer or crucible, as these are common sources of carbide-forming elements in high-temperature processing. However, given that TaC is known to be stable in molten aluminum while NbC tends to decompose, the presence of NbC in the control sample is unusual and may indicate a different source, such as residual elements in the raw materials or a master alloy addition (Kobashi et al., 1993). Further investigation, including EDS point analysis of the carbide particles, would be required to confirm their origin. The composite shows higher Al purity (95.3 wt.%) compared to the control sample (93.2 wt.%), suggesting cleaner processing for the composite. The Al crystallite size is approximately 283–291 Å, slightly smaller than the control (287–317 Å), indicating grain refinement from the Giro clay nanoparticles (Rawal & Sidpara, 2025). The presence of SiO₂ is expected to contribute to passive film stability (Xu & Zhang, 2025).

3.4 Optical Microscopy (OM) of As-Cast Sample (Control)

The OM micrograph of the as-cast Al-Si-Mg alloy is presented in Plate 4.



Plate 4: OM Micrograph of As-Cast Al-Si-Mg Alloy (Control)

The OM micrograph shows the grain structure of the as-cast Al-Si-Mg alloy. The aluminium grains appear gray in colour and are uniformly distributed within the microstructure. The dendritic structure typical of cast aluminium alloys is visible, with fine intermetallic phases distributed between the dendrite arms.

The grain structure observed is typical of Al-Si-Mg alloys cast in sand moulds, where the relatively slow cooling rate allows for the development of dendritic structures. The presence of intermetallic phases at grain boundaries is expected to influence the corrosion behaviour of the alloy, as these phases can act as cathodic or anodic sites in localized corrosion processes.

4. CONCLUSION

The development and characterization of Al-Si-Mg / Giro clay nanoparticle composites fabricated via stir casting have been successfully achieved. The following conclusions are drawn:

- i. **Giro Clay Characterization:** XRF and XRD analyses confirmed that Giro clay contains beneficial ceramic phases including SiO₂ (25.07 wt%), CaO (4.76 wt%), Al₂O₃ (1.78 wt%), and Fe₂O₃ (0.54 wt%), with dominant mineral phases of thenardite (37 wt%), trona (28 wt%), portlandite (18 wt%), and quartz (16 wt%). The high SO₃ content (51.14 wt.%) confirms the presence of sulfate-bearing minerals.
- ii. **Composite Fabrication:** Al-Si-Mg / GCNP composites were successfully fabricated via stir casting with reinforcement concentrations of 0–10 wt.% at 2 wt.% intervals. The 4 wt.% reinforcement formulations exhibited the most refined microstructure and optimal particle dispersion.
- iii. **Characterization:** XRD confirmed successful incorporation of SiO₂ from the Giro clay into the aluminium matrix. The optimally reinforced sample (4 wt.% reinforcement) showed Al purity of 95.3 wt.% with SiO₂ content of 2.8 wt.%, confirming successful composite development. SEM/EDS analysis confirmed relatively uniform dispersion at lower reinforcement levels.
- iv. **Optimal Formulation:** The sample with 4 wt.% Giro clay nanoparticle reinforcement demonstrated the best combination of microstructural refinement, particle dispersion, and phase distribution among all developed composites.
- v. **Local Content Significance:** This study successfully demonstrates that locally sourced Giro clay from Kebbi State, Nigeria, can be processed into nanoparticles and utilized as a cost-effective reinforcement for aluminium matrix composites, supporting local content inclusion in material development.

Recommendations

- i. Optimize stir casting parameters (stirring speed, holding time, cooling rate) for improved particle dispersion at higher reinforcement concentrations (≥ 6 wt%).
- ii. Conduct heat treatment studies to investigate ageing effects on microstructure and material properties.
- iii. Evaluate mechanical properties (tensile, hardness, wear) for complete structure-property relationships.

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