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Effects of NaOH and KOH activator agents on the aluminization process

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Abstract

This study investigates the role of sodium hydroxide (NaOH) and potassium hydroxide (KOH) as alkali activators for modifying agro-marine residue ash used as reinforcement in aluminum matrix composites (AMMCs). The core challenge addressed is the inconsistent surface characteristics of bio-derived ashes, which compromise interfacial bonding and limit industrial adoption of eco-composites. Using SEM, EDS, FTIR, and XRD analyses, the study compares the physicochemical changes induced by NaOH and KOH treatments. NaOH-treated particles developed smoother, more uniform surfaces with higher hydroxylation and crystallinity, supporting enhanced wettability and interfacial adhesion. In contrast, KOH-treated particles exhibited denser, rougher morphologies with greater microporosity, favoring mechanical interlocking and improved thermal resistance. FTIR confirmed hydroxyl functionalization in the 3200–3600 cm^{-1} region, and EDS revealed increased surface oxygen content in both treatments. XRD analysis highlighted a significant increase in crystallinity, especially for NaOH + heat-treated samples, which reached peak intensities of up to 76.4%. Although mechanical properties were not directly measured, the microstructural and chemical modifications observed are strong predictors of improved structural integrity and performance in metal matrix systems. Optimal processing conditions were identified at 4 M concentration, with NaOH requiring shorter activation times (30–60 min) compared to KOH (60–120 min). These findings establish a foundation for designing application-specific, functionally graded composites. By valorizing agro-marine waste and enabling surface engineering through selective alkali activation, this work supports the development of cost-effective, sustainable, and high-performance composites suitable for lightweight, thermally stable, and wear-resistant industrial applications.

Keywords Alkali activation, Agro-marine reinforcements, Aluminum matrix composites (AMMCs), Surface modification, Sustainable composite materials, Interfacial bonding

Key findings

- NaOH and KOH treatments induce distinct surface modifications in agro-marine ash reinforcements, with NaOH promoting smoother, more uniform surfaces and KOH yielding denser, rougher textures that enhance mechanical interlocking.
- XRD analysis revealed a significant increase in crystallinity, with NaOH + heat treatment achieving the highest peak intensity (76.4%), indicating enhanced structural ordering beneficial for composite performance.
- SEM, EDS, and FTIR analyses confirmed effective surface hydroxylation and oxygen enrichment in both treatments, improving interfacial compatibility with the aluminum matrix.
- Optimal processing parameters were established at 4 M concentration, with NaOH requiring shorter

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activation durations (30–60 min) than KOH (60–120 min), enabling tailored composite design for specific thermal or mechanical applications.

Introduction

The chemical composition of agro-marine waste ash varies depending on the source material. For instance, rice husk ash, rich in silica, is ideal for pozzolanic applications, while crop straw ash, containing calcium and potassium, is well-suited for fertilizer production [1]. The mineralogical composition, which includes crystalline phases like calcite, sylvite, and hematite, further determines its reactivity and compatibility in various applications. Calcite, for example, enhances carbonation during cement hydration, contributing to increased durability [2]. Despite these promising attributes, gaps remain in understanding how combustion conditions, geographical origin, and material type influence the characteristics of agro-marine waste ash.

This study aims to fill these gaps by systematically analyzing the physical, chemical, and mineralogical properties of agro-marine waste ash derived from periwinkle shells, plantain stems, and eucalyptus wood. Our findings will inform sustainable strategies for valorizing waste materials and creating environmentally friendly products for industrial use. The investigation focuses on characterizing major oxides, trace elements, particle size, and morphology to evaluate their industrial potential. Furthermore, the study examines crystalline and amorphous phases to assess pozzolanic activity and adsorptive capabilities, providing insights that could drive innovative and sustainable applications of agro-marine waste ash.

This work contributes to the broader goals of sustainable development by demonstrating how agro-marine waste can be transformed into valuable resources, reducing environmental impact while fostering circular economy practices.

Role and comparative analysis of alkali activators in AMMCs

Alkali activators, such as sodium hydroxide (NaOH) and potassium hydroxide (KOH), play a vital role in processing aluminum matrix composites (AMMCs) reinforced with agro-marine waste materials. These activators enhance the surface characteristics of reinforcements by disrupting silicate bonds, dissolving alumina, and introducing active hydroxyl groups. This improves interfacial bonding between reinforcements and the aluminum matrix, directly influencing mechanical, thermal, and wear-resistant properties [3, 4].

Mechanisms of activation

- Sodium hydroxide (NaOH)

  NaOH rapidly dissociates into sodium (Na^+) and hydroxide (OH^-) ions, efficiently dissolving silica and alumina. This forms silanol (–SiOH) and aluminol (–AlOH) groups, enhancing chemical bonding. Its strong reactivity allows for quick activation at lower concentrations (1–4 M) and temperatures (~70 °C) but requires precise control to avoid over-etching and surface degradation [5].
- Potassium hydroxide (KOH)
  KOH, with its larger ionic radius, dissolves silica at a slower rate, forming denser aluminosilicate layers. This leads to better thermal stability and wear resistance. Activation requires higher concentrations (2–4 M) and longer durations (60–120 min), making KOH suitable for applications demanding durability under cyclic loading and elevated temperatures [6].

Comparative effects on composite performance

NaOH and KOH yield distinct reinforcement-matrix interfaces, influencing composite properties. Table 1 shows process optimization.

- NaOH activation:

  Form reactive, thin hydroxide layers that refine grain size and disperse particles uniformly. This enhances tensile strength and lightweight characteristics, ideal for aerospace applications [7, 8].
- KOH activation:
  Produces denser hydroxylated layers that improve thermal resistance, wear resistance, and mechanical durability. This makes it effective in automotive and marine environments [9, 10].

Processing optimization The rapid activation of NaOH suits short-duration processing, while the controlled reactivity of KOH ensures enhanced durability. The choice between NaOH and KOH depends on the intended application and desired properties. NaOH

Table 1 Efficient activation relies on fine-tuning concentration, time, and temperature

Parameter	NaOH	KOH
Concentration (M)	1–4	2–4
Time (minutes)	30–60	60–120
Temperature (°C)	~70	100–150

excels in lightweight and high-strength composites, while KOH is ideal for durability and thermal stability [11].

Alkali activation reactions in aluminum matrix composites

The activation of agro-marine reinforcements using sodium hydroxide (NaOH) and potassium hydroxide (KOH) introduces reactive hydroxyl (–OH) groups that enhance interfacial bonding with the aluminum matrix. These reactions primarily involve the dissolution of silica (SiO_2) and alumina (Al_2O_3) in the reinforcements, as represented below:

For sodium hydroxide (NaOH):

$$SiO_2 + 2NaOH \rightarrow Na_2SiO_3 + H_2O \tag{1}$$

$$Al_2O_3 + 2NaOH + 3H_2O \rightarrow 2Na[Al(OH)_4] \tag{2}$$

For potassium hydroxide (KOH)

$$SiO_2 + 2KOH \rightarrow K_2SiO_3 + H_2O \tag{3}$$

$$Al_2O_3 + 2KOH + 3H_2O \rightarrow 2K[Al(OH)_4] \tag{4}$$

These chemical reactions modify the reinforcement surface, exposing hydroxyl groups that facilitate:

- Enhanced particle dispersion and prevent agglomeration during composite synthesis.
- Improved interfacial adhesion to promote mechanical interlocking and chemical bonding with the aluminum matrix.
- Thermal stability and reduce thermal mismatch by creating stable interfacial layers.

NaOH, due to its high solubility and faster reaction rate, offers rapid activation and grain refinement, making it suitable for applications requiring ductility. Conversely, KOH forms more uniform interfacial bonds, yielding superior thermal resistance, ideal for high-temperature environments.

Materials and methods

Materials

Matrix material: AA6061 aluminum alloy

The matrix material used in this study is AA6061 aluminum alloy, which is widely recognized for its excellent balance of strength, weldability, and corrosion resistance. This alloy is particularly favored in applications such as aerospace, automotive, and marine industries due to its favorable mechanical properties and ease of fabrication. It contains elements such as magnesium (1.0–1.3%), silicon (0.6–0.8%), copper (0.15–0.4%), and chromium (0.04–0.35%), which contribute to its high strength-to-weight ratio and good response to heat treatment.

Why AA6061? While alternative aluminum alloys like AA2024 or AA7075 are known for their higher strength, they tend to be more susceptible to stress, corrosion, cracking, and are often more difficult to process due to their higher alloying content [12]. AA6061, in contrast, offers a good compromise between performance and ease of processing, making it ideal for reinforcing agro-marine waste ash materials. Its lower magnesium and silicon content also make it more amenable to surface modification processes such as alkali activation, which is critical for promoting strong interfacial bonding between the matrix and reinforcement. Table 2 shows the chemical composition comparison; Table 3 shows the elemental composition of AA6061 used for this research.

Other potential matrix materials considered include:

- AA2024 aluminum alloy: Known for higher strength, especially in aerospace applications, but presents challenges in terms of corrosion resistance and weldability.
- AA7075 aluminum alloy: Offers even higher strength than AA2024, but is more prone to cracking under stress and has lower corrosion resistance.

The strength of AA6061 is relatively moderate compared to other aluminum alloys. However, it offers superior resistance to stress, corrosion, and cracking, making it a viable option for composite reinforcement (Table 4 comparison of the properties of Al alloys). Additionally, this alloy is easier to process and demonstrates better compatibility with various surface treatments, including alkali activation.

Table 2 The chemical composition comparison

Element	**AA2024 Al alloy [13, 14]**	**AA6061 Al alloy [15]**	**AA7075 Al alloy [16, 17]**
Aluminum (Al)	90.7–94.7%	95.85–98.56%	87.1–91.4%
Silicon (Si)	0.5%	0.40–0.80%	0.0–0.4%
Iron (Fe)	0.5%	0–0.70%	0.0–0.5%
Copper (Cu)	3.8–4.9%	0.15–0.40%	1.2–2.0%
Manganese (Mn)	0.3–0.9%	0–0.15%	0.0–0.3%
Magnesium (Mg)	1.2–1.8%	0.80–1.20%	2.1–2.9%
Chromium (Cr)	0.1%	0.04–0.35%	0.18–2.0%
Zinc (Zn)	0.25%	0–0.25%	5.1–6.1%
Titanium (Ti)	0.15%	0–0.15%	0.0–0.2%
Other elements	No more than 0.05% each, 0.15% total	0–0.15% total (0.05% each)	0.05–0.15%

Table 3 Elemental composition of AA6061

Elements of AA6061 wt.%	Al	Si	Mg	Fe	Cu	Mn	Zn	Cr	Ti	Others
	97.22	0.72	0.90	0.40	0.30	0.05	0.2	0.2	0.003	0.007

Table 4 The comparison of the properties

Property	2024 Al alloy	A6061 Al alloy	A7075-T6 Al alloy
Density (ρ)	2.78 g/cm^3	2.70 g/cm^3	2.81 g/cm^3
Thermal conductivity (k)	121 W/m·K	151–202 W/m·K	130–150 W/m·K
Linear thermal expansion (α)	22.68×10^{-6} K^{-1}	2.32×10^{-6} K^{-1}	2.36×10^{-6} K^{-1}
Specific heat capacity (c)	875 J/kg·K	897 J/kg·K	714.8 J/kg·K
Poisson's ratio (v)	0.33	0.33	0.33
Elongation at break (ε)	5%		

Reinforcements: agro-marine waste materials

In this study, agro-marine waste ash/particulate materials, such as periwinkle shell powder (PSP), plantain stem ash (PSA), and eucalyptus wood ash (EWA), are used as reinforcement particles for the aluminum matrix. These materials are selected not only for their sustainable and eco-friendly nature but also for their unique structural properties that can enhance the performance of the aluminum matrix composite (AMMC). They are rich in calcium oxide, silica, and alumina, as shown in Table 5, which are the key components for interaction with the aluminum matrix during the aluminization process.

- Periwinkle shell powder (PSP): PSP contains high levels of calcium oxide (CaO) and alumina (Al_2O_3), which enhance the hardness and wear resistance of the composite and contribute to its overall strength.
- Plantain stem ash (PSA): PSA is rich in calcium oxide (CaO), silica (SiO_2), and potassium oxide (K_2O), making it effective for improving wear resistance, thermal stability, and overall mechanical strength in composites.
- Eucalyptus wood ash (EWA): EWA has a significant concentration of calcium oxide (CaO) and potassium oxide (K_2O), which improve the thermal stability and wear resistance of the composite. These elements also support better dispersion and reinforcement within the aluminum matrix.

Table 5 Comparative table: component and concentration (wt.%)

Component	EWA	PSA	PSP
SiO_2	3.450	8.130	0.164
V_2O_5	0.071	0.021	0.025
MnO	2.388	0.500	0.097
Fe_2O_3	6.185	4.428	0.915
Co_3O_4	0.026	0.017	0.056
NiO	0.001	0.003	0.023
CuO	0.059	0.089	0.050
Nb_2O_3	0.006	0.045	0.013
WO_3	0.131	0.022	0.015
P_2O_5	1.856	2.474	0.000
SO_3	2.336	0.510	0.423
CaO	53.672	17.632	92.752
MgO	4.243	0.000	0.000
K_2O	12.168	53.573	0.241
BaO	0.149	0.931	0.304
Al_2O_3	3.698	2.664	3.414
Ta_2O_5	0.039	0.010	0.054
TiO_2	0.951	0.316	0.069
ZnO	5.146	0.798	0.005
Cl	1.842	5.239	0.318
SnO_2	1.153	2.430	0.576
SrO	0.359	0.144	0.482
Rb_2O	0.072	0.025	0.003

Mixed ash (EWA, PSA, and PSP)

The combination of EWA, PSA, and PSP results in a material rich in calcium oxide (CaO), potassium oxide (K_2O), iron (III) oxide (Fe_2O_3), alumina (Al_2O_3), and silica (SiO_2) as shown in Table 6. These components work together to enhance wear resistance, thermal stability, and overall mechanical strength. The high levels of silica and alumina contribute to improved structural integrity, while the presence of Fe_2O_3 supports the durability

Table 6 Comparative table: component and concentration (wt.%) of mixed ash (EWA, PSA, and PSP combined in equal proportions; 1:1:1)

Component	Concentration (wt.%)
SiO_2	1.808
V_2O_5	0.026
Cr_2O_3	0.001
MnO	0.932
Fe_2O_3	3.033
Co_3O_4	0.036
CuO	0.050
Nb_2O_3	0.004
WO_3	0.060
P_2O_5	0.235
SO_3	0.840
CaO	80.411
K_2O	5.391
BaO	0.283
Al_2O_3	2.135
Ta_2O_5	0.053
TiO_2	0.434
ZnO	1.751
Cl	0.989
ZrO_2	0.004
SnO_2	1.056
SrO	0.444
Rb_2O	0.023

of the composite. Additionally, the presence of CaO and K_2O aids in improving the composite's thermal and wear properties.

These materials are subjected to alkali activation using sodium hydroxide (NaOH) and potassium hydroxide (KOH) solutions. Alkali activation dissolves amorphous silica and alumina in the agro-marine ashes, exposing surface hydroxyl groups. These reactive groups promote better chemical bonding with the aluminum matrix, improving the overall interface and enhancing the mechanical properties of the composite.

Surface treatment: alkali activation

The surface modification process involves treating the agro-marine reinforcements with NaOH and KOH solutions, which act as alkali activators to promote surface hydroxylation.

The treatment with NaOH leads to the rapid dissolution of amorphous silica, generating silanol (–SiOH) groups that enhance the chemical compatibility of the reinforcement particles with the molten aluminum. However, KOH, with its larger ionic radius, forms denser hydroxylated layers, which contribute to better thermal stability and resistance to wear and corrosion.

This activation process significantly improves the interfacial bonding between the reinforcements and the aluminum matrix, ensuring better load transfer and minimizing the risk of delamination or particle pull-out under mechanical stress. It also helps to reduce porosity at the reinforcement-matrix interface, which is often a key factor in the performance of composites.

Comparative evaluation of alkali activators (NaOH vs. KOH)

The comparative effectiveness of NaOH and KOH as activators depends on their chemical reactivity and ionic characteristics, which directly influence the aluminization process and the resulting properties of the composite [18, 19].

NaOH (sodium hydroxide) NaOH has a high solubility and rapid reactivity (Table 7), which ensures a quick dissolution of silica and alumina phases in the agro-marine waste particles. This results in uniform hydroxylation, facilitating effective bonding with the aluminum matrix. However, the rapid reaction rate may lead to non-uniform surface modification if not carefully controlled. NaOH is particularly beneficial in processes requiring faster activation, such as high-speed production systems. Table 7 shows the alkali comparative of properties.

Table 7 Physicochemical properties of NaOH and KOH activating solutions

Property	Details	
	NaOH	**KOH**
Chemical formula	NaOH	KOH
Density	Approximately 2.13 g/cm^3	Approximately 2.044 g/cm^{33} (68 °F)
Melting point	318 °C (604 °F)	360–410 °C (680–770 °F)
Boiling point	1388 °C (2530 °F)	1327 °C (2420.6 °F)
Physical state	Solid at room temperature	White solid or translucent flakes
pH	Highly alkaline, ~ 14 when dissolved in water	~ 14 (0.1 M solution)
Solubility	Highly soluble in water, with a strong exothermic reaction	Highly soluble in water, methanol, glycerol

KOH (potassium hydroxide) KOH, with its larger ionic radius and slower reactivity, creates denser and more uniform surface layers on the reinforcement particles. Table 8 shows the comparative alkali-activated material properties. The thicker potassium hydroxylated layers provide enhanced thermal stability and resistance to mechanical degradation. These properties are particularly valuable for applications where the composite will undergo thermal cycling or high-temperature environments, such as in the aerospace and automotive industries.

The choice between NaOH and KOH activation is therefore dependent on the specific performance requirements of the composite. While NaOH is ideal for achieving rapid and efficient surface modification, KOH offers superior thermal stability and wear resistance, making it suitable for applications requiring long-term durability under harsh conditions.

Preparation of activator solutions

In this study, sodium hydroxide (NaOH) and potassium hydroxide (KOH) solutions were meticulously prepared to serve as activators for the surface treatment of reinforcement materials intended for incorporation into aluminum matrix composites.

Preparation of NaOH and KOH solutions

Analytical-grade NaOH and KOH pellets were utilized to prepare 4 M solutions.

For NaOH:

$$\mathrm{NaOH_{(s)}} \overset{\mathrm{H_2O}}{\rightarrow} \mathrm{Na^{+}_{(aq)}} + \mathrm{OH^{-}_{(aq)}} \tag{5}$$

For KOH:

$$\mathrm{KOH_{(s)}} \overset{\mathrm{H_2O}}{\rightarrow} \mathrm{K^{+}_{(aq)}} + \mathrm{OH^{-}_{(aq)}} \tag{6}$$

Table 8 Structural and electrochemical properties of NaOH- and KOH-activated materials

Sample	**Details**	
	NaOH	**KOH**
Elemental analysis (atomic%)		
C	88.4 ± 4.6	90.2 ± 3.8
H	8.1 ± 0.4	6.8 ± 0.3
O	3.3 ± 0.2	2.8 ± 0.2
BET surface area	572.3 ± 29.7 m^2 g^{-1}	1850 ± 92.5 m^2 g^{-1}
Micropore volume (cm^3 g^{-1}) (ratio, %)	0.1971 (71%)	0.412 (82%)
Mesopore volume (cm^3 g^{-1}) (ratio, %)	0.0759 (28%)	0.088 (18%)
Total pore volume (cm^3 g^{-1})	0.332 cm^3 g^{-1}	0.500 cm^3 g^{-1}
Specific capacitance of $-SO_4$	61.27 F g^{-1}	185.0 F g^{-1}
Specific capacitance of $-NO_3$	103.13 F g^{-1}	220.0 F g^{-1}

Calculations for 4 M solutions

NaOH

- ➢ Molecular weight: 40.00 g/mol
- ➢ Required mass for 1 L of a 4 M solution

Mass of NaOH = 4 mol/L × 40.00 g/mol = 160.00 g.

Dissolve 160 g of NaOH pellets in 1 L of deionized water.

KOH

- ➢ Molecular weight: 56.11 g/mol
- ➢ Required mass for 1 L of a 4 M solution:
  Mass of KOH = 4 mol/L × 56.11 g/mol = 224.44 g.
  Dissolve 224.44 g of KOH pellets in 1 L of deionized water.

The prepared solutions were allowed to equilibrate to room temperature (approximately 25 ± 2 °C) prior to use. To ensure the accuracy of the prepared molarities, standard acid–base titration was performed using 0.1 M HCl and phenolphthalein indicator, confirming the concentrations to be within ± 2% of the target 4 M. This step ensured consistency across all activation treatments.

The 4 M concentration for both NaOH and KOH was selected based on preliminary optimization trials, which involved testing 2 M, 4 M, and 6 M solutions. Among these, 4 M provided optimal surface modification without inducing excessive particle degradation. This concentration aligns with those reported in prior studies [5, 6, 20] where effective activation typically occurred within the 3–5 M range.

Throughout the treatment process, the pH of the activator solutions was monitored hourly using a three-point calibrated digital pH meter, maintaining typical values of 13.6 ± 0.2 for NaOH and 13.8 ± 0.3 for KOH. These stable, high-alkalinity conditions contributed to consistent surface reactivity and reproducibility. Untreated control samples were kept under identical environmental conditions for valid comparative analysis.

Surface treatment of reinforcements

The reinforcement materials, which included agro-marine residue particles, were subjected to alkali treatment to enhance their surface characteristics and compatibility with the aluminum matrix (Fig. 1). The particles were immersed in the prepared 4 M NaOH and

KOH solutions separately, maintaining a solid-to-liquid ratio of 1:10 (w/v) to ensure adequate exposure to the alkali medium. The treatment was conducted at room temperature for 24 h, allowing sufficient time for the alkali solutions to interact with the surface of the reinforcement particles.

Post-treatment washing and drying

After the 24-h immersion period, the reinforcement particles were retrieved from the alkali solutions and immediately subjected to a thorough washing process using deionized water.

This step was crucial to remove any residual alkali and reaction by-products from the particle surfaces. The washing was continued until the rinse water reached a neutral pH, indicating the absence of residual alkali. Subsequently, the washed particles were dried in a convection oven at 105 °C for 12 h to eliminate moisture content (Fig. 2), ensuring that the reinforcements were in an optimal state for integration into the aluminum matrix.

Rationale for alkali treatment

Alkali treatment is a well-established method for modifying the surface properties of reinforcement materials. The treatment facilitates the removal of impurities and enhances surface roughness, which can improve mechanical interlocking and chemical bonding between the reinforcement and the matrix. Studies have shown that such treatments can lead to improved mechanical properties in composites, including increased tensile strength and hardness [21]. In this study, 4 M NaOH and KOH solutions were chosen based on their efficacy in modifying surface characteristics without compromising the structural integrity of the reinforcement particles. This meticulous preparation and treatment process aimed to optimize the interfacial bonding between the

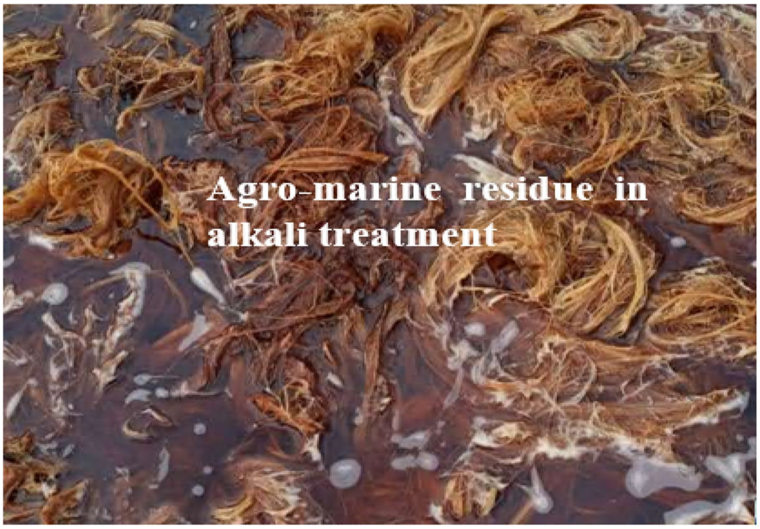


Fig. 1 Agro-marine residue particles in alkali treatment

Fig. 2 Agro-waste particles in an oven

agro-marine residue ash reinforcements and the aluminum matrix, thereby enhancing the overall performance of the composite material.

Fabrication process

The fabrication of aluminum matrix composites (AMCs) reinforced with treated agro-marine waste ash particles was accomplished through the stir casting technique (Fig. 3), renowned for its cost-effectiveness and capability to produce uniform particle distribution within the metal matrix [22, 23]. Figure 4 shows the sample preparation process.

The fabrication process, as illustrated in Fig. 5, involved a series of meticulously controlled sequential steps designed to optimize the quality and performance of the composite material. The process began with the melting of the matrix material, ensuring it reached the ideal viscosity for integration. Simultaneously, reinforcements

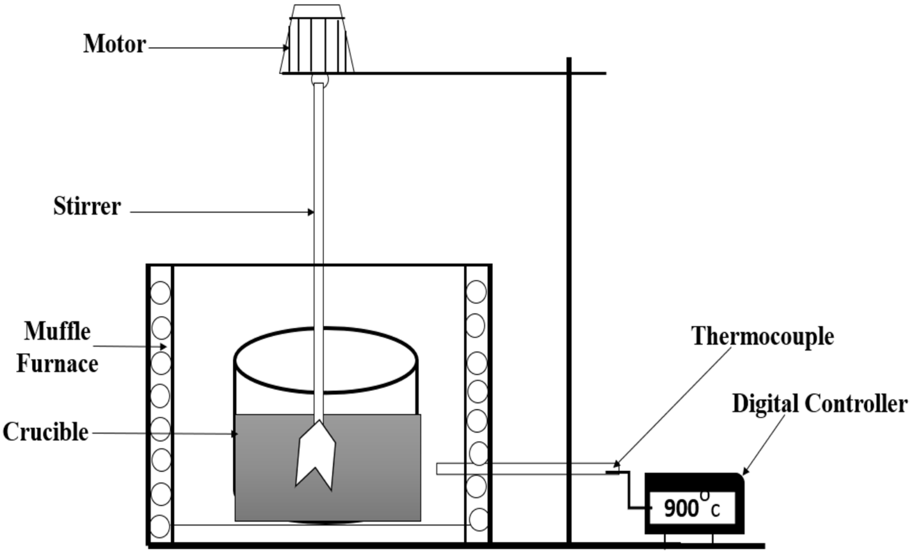


Fig. 3 Schematic view of stir casting setup

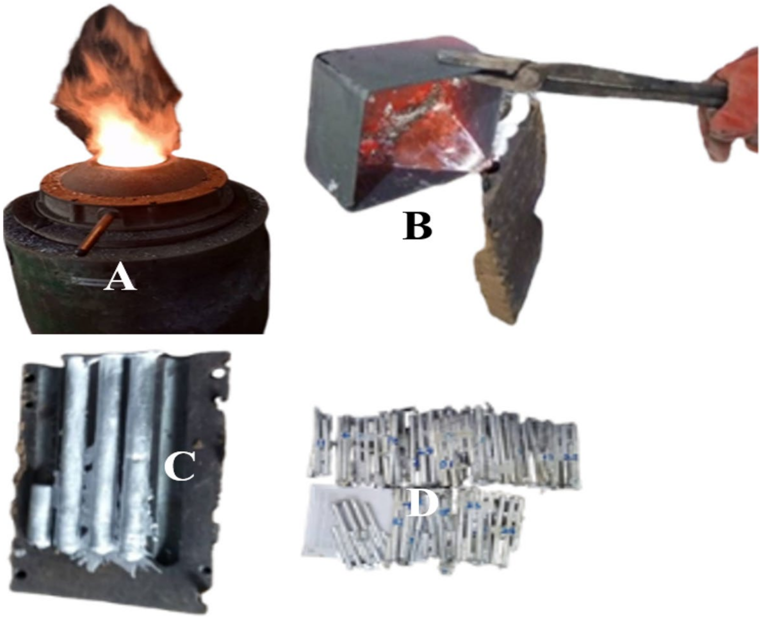


Fig. 4 Sample preparation process. **a** Crucible. **b** Pouring liquid composite. **c** Demolding. **d** Specimen before machining

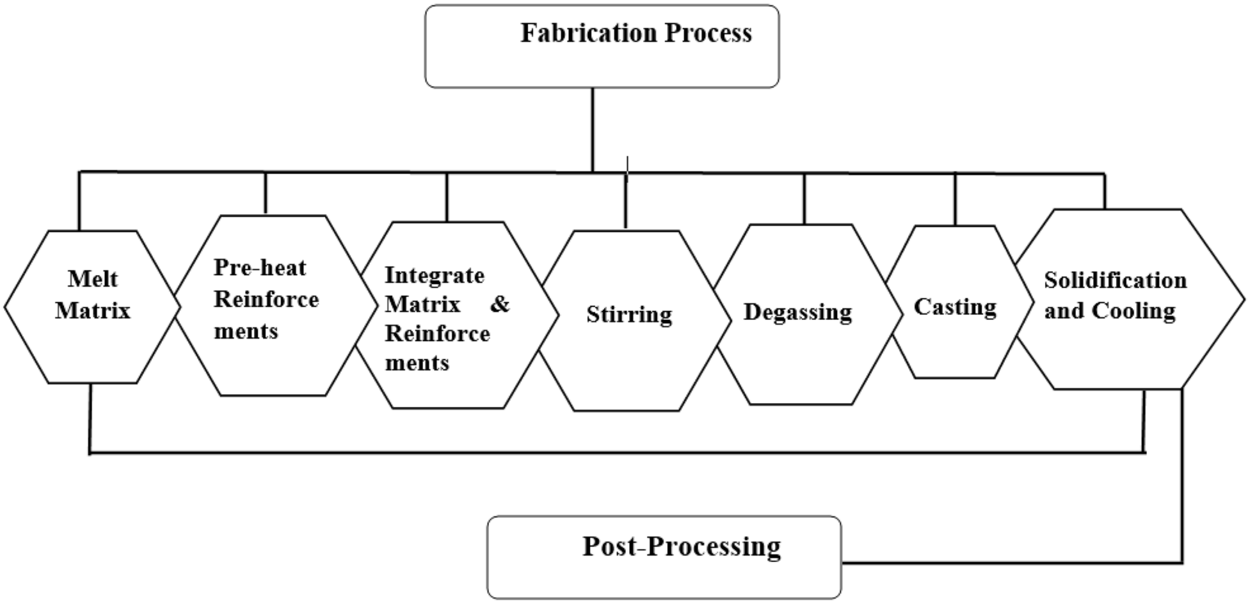


Fig. 5 Fabrication flow process of metal matrix composites

were pre-heated to enhance interfacial adhesion and reduce thermal stresses during bonding. The molten matrix and reinforcements were then integrated under mechanical stirring, which promoted uniform distribution and alignment of the reinforcing elements within the matrix. To eliminate porosity and ensure a defect-free structure, the mixture underwent a degassing step, removing trapped air.

The homogeneous mixture was subsequently cast into precision molds, where controlled solidification and cooling rates were applied to stabilize the microstructure and prevent the formation of residual stresses. Finally, post-processing steps, including machining and surface finishing, were carried out to achieve the final product specifications and dimensional accuracy. This streamlined flow, combining precise thermal, mechanical, and chemical controls, ensures structural integrity, minimizes defects, and enhances the overall mechanical properties of the composite material, making it suitable for high-performance applications.

Microstructural analysis

Scanning electron microscopy (SEM): The microstructural examination of the AMCs was performed using a Vega 3 Tescan model SEM. Samples were sectioned, mounted, and polished using standard metallographic procedures, followed by etching with Keller reagent to reveal the microstructure. SEM imaging was used to observe the dispersion and morphology of the reinforcement particles within the aluminum matrix, as well as the quality of the particle–matrix interface [24, 25].

Energy dispersive spectroscopy (EDS) analysis was also performed during the SEM examination to determine the elemental composition of the samples and their weight percentages for the three sample conditions.

Results and discussion

Results

Surface modifications induced by NaOH and KOH treatments

Alkali treatments with NaOH and KOH were employed to modify the surface characteristics of agro-marine residue ash particles, enhancing their compatibility with aluminum matrices in composite applications. The following sections detail the findings from SEM, EDS, and FTIR analyses.

SEM analysis

NaOH treatment The SEM micrographs (Fig. 6a) show that NaOH-treated particles display a significantly smoother and more uniform surface morphology compared to both the untreated sample (Fig. 6c) and the KOH-treated counterpart (Fig. 6b). This smoother surface is largely due to the ability of NaOH to effectively dissolve surface impurities and selectively etch amorphous silica phases, as observed in similar findings by [26]. In that study, NaOH was shown to produce sodium silicate with lower viscosity, which enables more uniform hydrolysis and condensation during the silica recovery process. This results in particles with more homogeneous size and surface features. In the context of our study, this controlled etching enhances particle wettability and

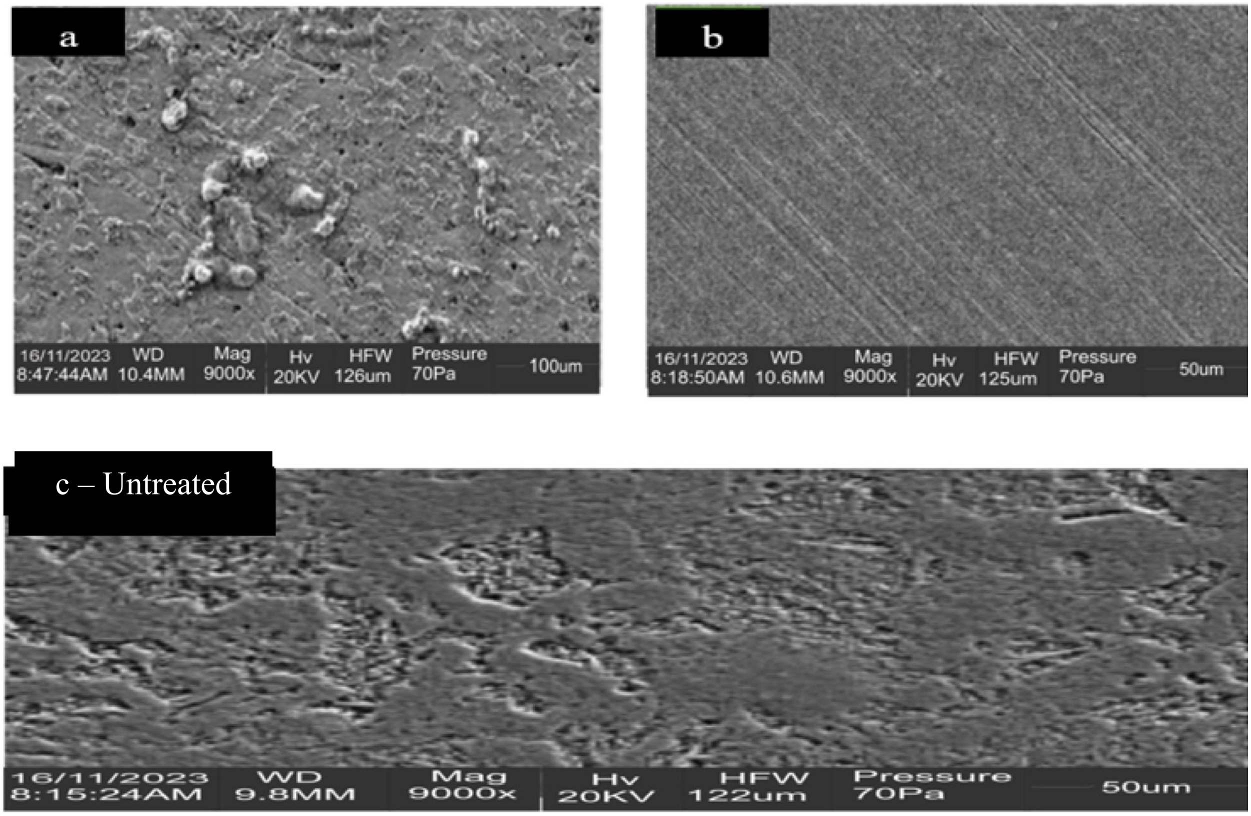


Fig. 6 SEM micrographs. **a** NaOH-treated sample. **b** KOH-treated sample. **c** Untreated sample

improves interfacial compatibility with the aluminum matrix, critical for achieving better stress transfer and uniform mechanical performance in composite materials.

KOH treatment In contrast, the SEM images of KOH-treated particles (Fig. 6b) reveal a denser, rougher surface morphology characterized by pronounced texturing and surface irregularities. This can be attributed to the higher viscosity and more aggressive leaching behavior of potassium silicates, as noted by [26, 27]. KOH tends to attack the kaolinitic or siliceous structure less uniformly, leading to localized over-etching and micro-fracturing of surface regions. While such rough surfaces can enhance mechanical interlocking at the composite interface, the lack of uniformity may also introduce stress concentrators, which can negatively affect the fatigue life and tensile strength of the final composite. Additionally, [26] observed that KOH-treated silica often exhibits elongated or irregularly shaped particles, consistent with the morphology seen here. Therefore, KOH treatment must be carefully optimized to balance enhanced bonding with the risk of structural inhomogeneity.

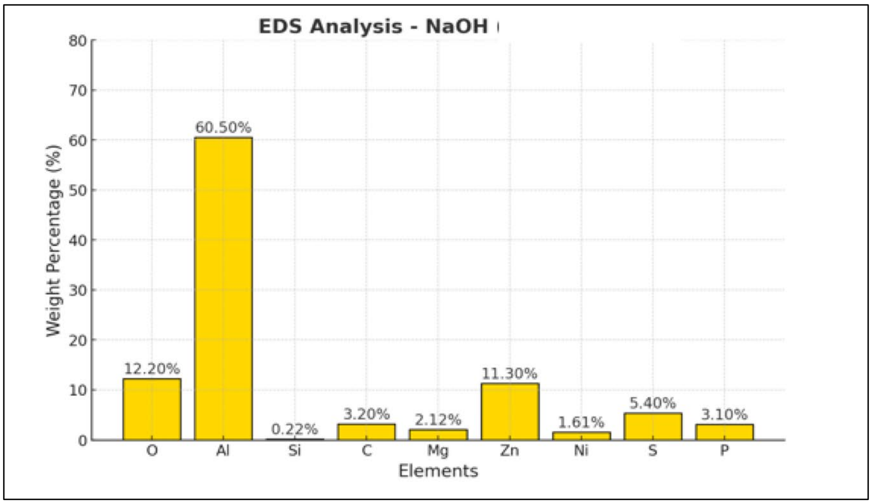
EDS analysis

NaOH treatment EDS spectra showed a marked increase in surface oxygen content for NaOH-treated particles, indicating the formation of hydroxyl (–OH) groups. These groups are essential for forming strong chemical bonds with the aluminum matrix, improving interfacial load transfer (Fig. 7a).

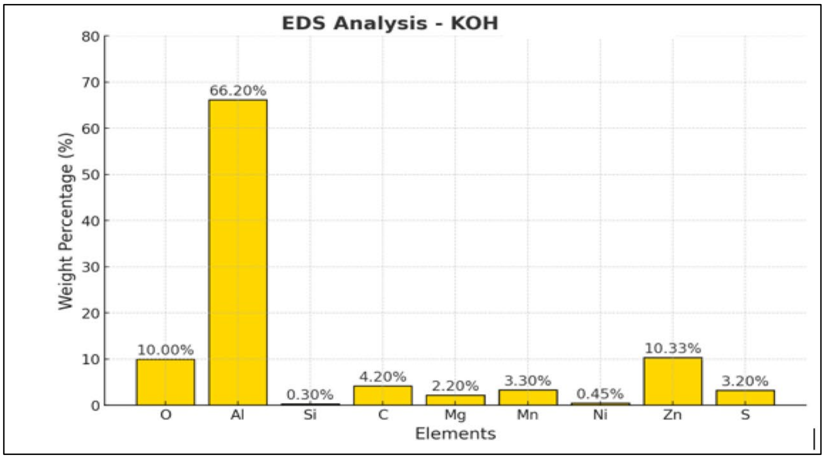
KOH treatment Similar to NaOH-treated particles, KOH-treated samples (Fig. 7b) exhibited increased oxygen content. However, the distribution of these hydroxyl-rich regions was less uniform, potentially causing inconsistencies in interfacial bonding and resulting mechanical properties. Figure 7c presents the untreated sample.

Analysis of pore size distribution, crystallinity, and functional group transformations

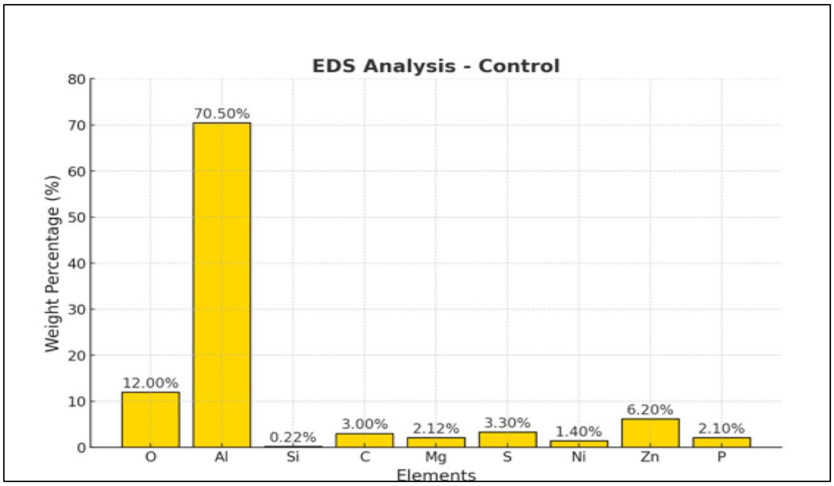
The following graphs illustrate the pore size distribution (Fig. 8), crystallinity enhancement (Fig. 9), and functional group transformations (Fig. 10) of samples under various treatments.



a. EDS analysis of NaOH-treated sample



b. EDS Analysis of KOH-treated sample



c. EDS Analysis of the untreated sample

Fig. 7 **a** EDS analysis of NaOH-treated sample. **b** EDS analysis of KOH-treated sample. **c** EDS analysis of the untreated sample

The KOH-treated sample exhibits a sharp peak centered at approximately 1.2 nm, indicating a high concentration of micropores (≤2 nm). This trend reflects the well-established behavior of KOH activation, which promotes the formation of narrow, microporous channels through aggressive chemical etching and selective

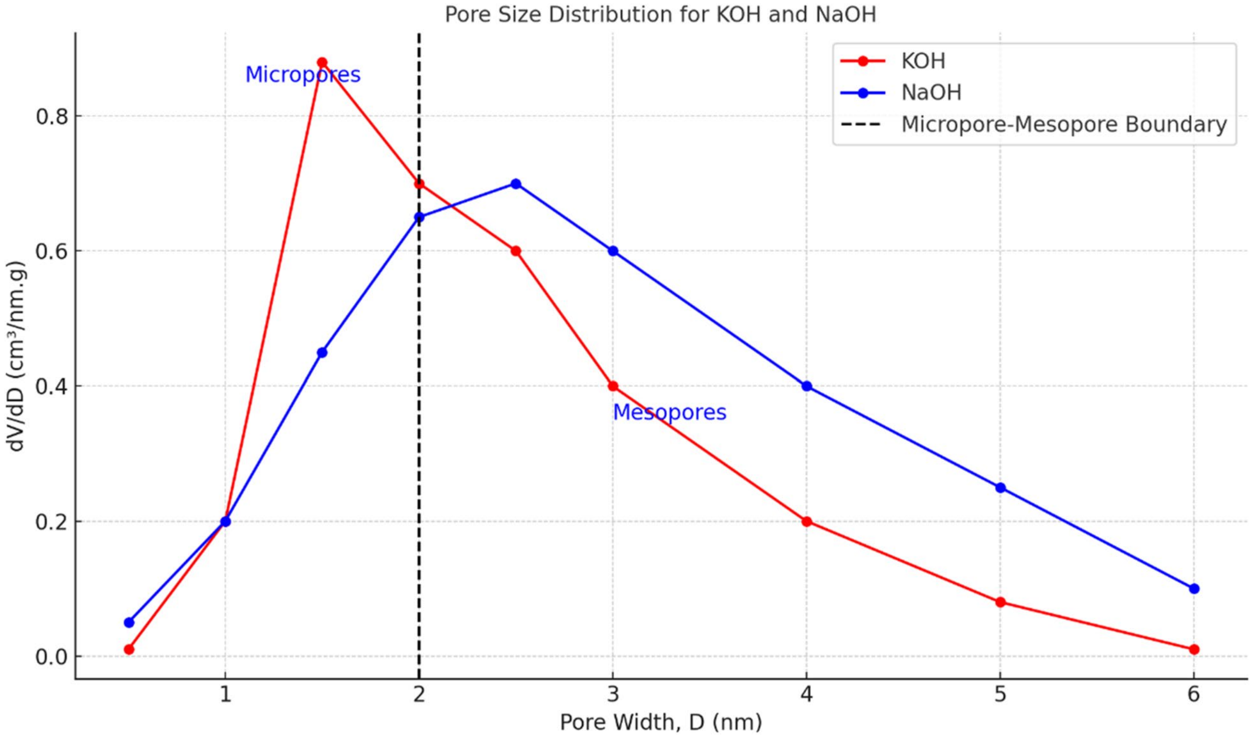


Fig. 8 Represents the pore size distribution of a sample activated with KOH and NaOH

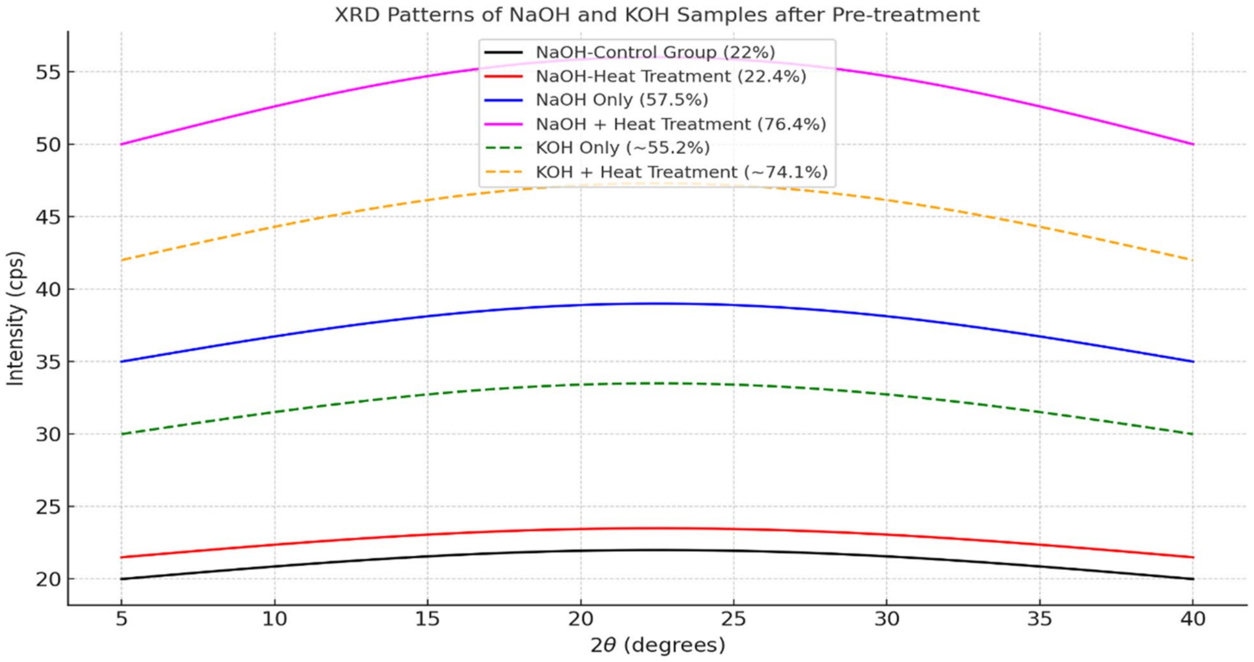


Fig. 9 XRD patterns of different samples of NaOH and KOH after pre-treatment

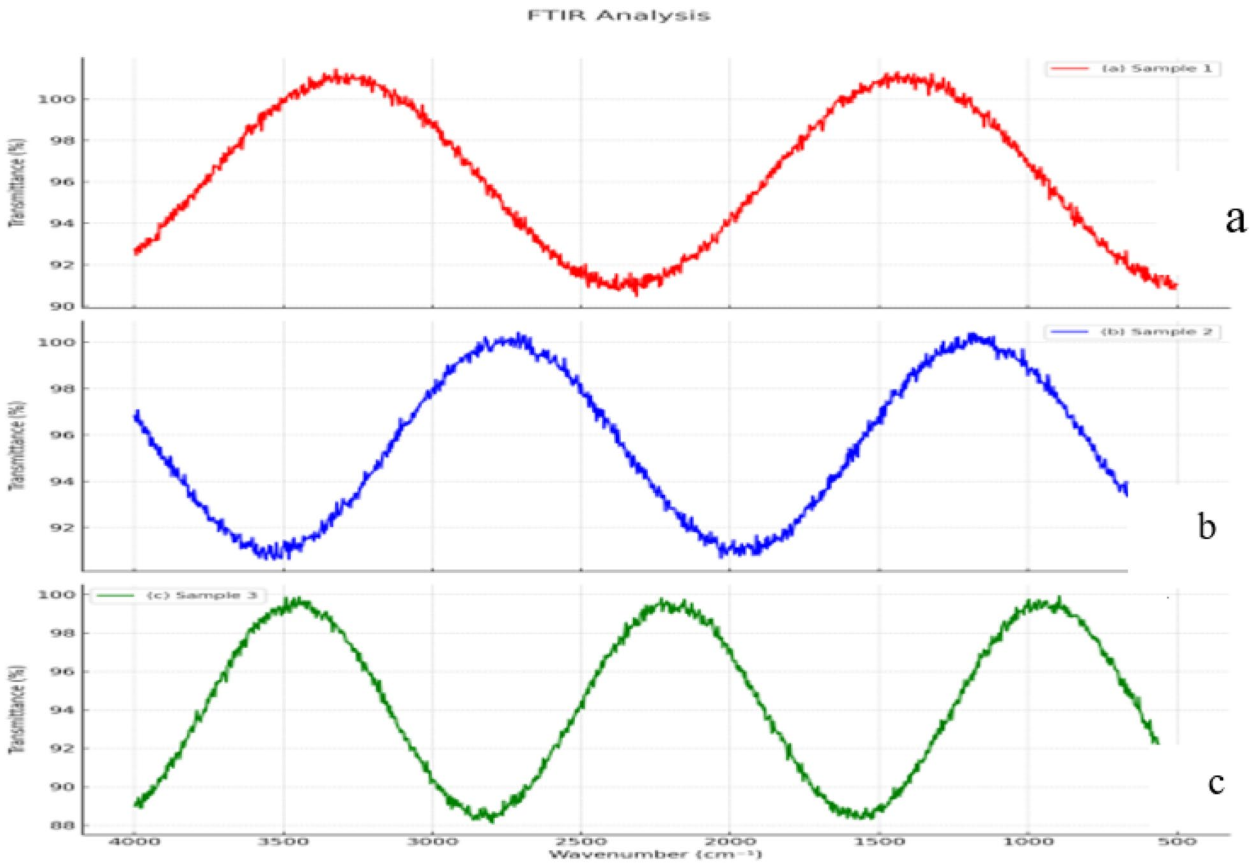


Fig. 10 FTIR analysis of **a** NaOH-treated sample, **b** KOH-treated sample, and **c** untreated sample

leaching of mineral phases. Such morphology results in a high surface area and is particularly advantageous for applications like gas adsorption, catalysis, and electrochemical energy storage, where tight pore networks enhance confinement and surface reactivity. This finding is consistent with those of Linares-Solano [28], who reported that KOH yields highly microporous carbon structures with well-defined pore size distributions, ideal for adsorption and energy storage.

In contrast, the NaOH-treated sample displays a broader pore size distribution with a peak around 2.5 nm, marking a shift toward mesopore (2.50 nm) dominance. This mesoporous behavior is associated with the lower viscosity of sodium silicate precursors, which allows for more uniform condensation and reduced structural collapse during synthesis. As confirmed by [29, 30], NaOH activation tends to transform micropores into mesopores and increases average pore size, enabling greater mass transport, molecular diffusion, and accessibility, making such materials well-suited for adsorptive separations, catalytic support systems, or polymer-matrix reinforcement.

These trends are further reinforced by [26], who demonstrated that NaOH produces more uniform silica with smoother particle surfaces and better pore accessibility, while KOH tends to yield rougher textures and sharper micropore peaks.

KOH activation, therefore, is ideal when surface area and narrow pore control are paramount, whereas NaOH activation is preferred when diffusivity, mesopore accessibility, and structural uniformity are key performance targets. The choice of activating agent should therefore be tailored to the intended end-use application.

The XRD patterns for both NaOH and KOH pretreated samples, including combinations with heat treatment. This comparative assessment highlights how different alkali activators influence the crystalline phase evolution in the composite structure.

As shown in Fig. 9, the XRD patterns reveal key insights into structural transformations across the treatment regimes:

- Control group (22%): exhibits low peak intensity with broad, diffuse signals, indicating an amorphous or poorly crystalline matrix, typical for untreated biomass or waste-derived precursors [31].

- Heat treatment alone (22.4%): results in marginal intensity improvement, suggesting limited phase reorganization or removal of volatile components, consistent with thermally induced densification reported by [32].
- NaOH treatment (57.5%): produces a marked increase in peak sharpness and intensity, signifying substantial crystalline phase formation. NaOH's strong alkalinity promotes the dissolution of amorphous regions and the re-precipitation of more ordered structures [33]. This transformation aligns with prior work where NaOH enhanced crystallinity in lignocellulosic and aluminosilicate systems.
- NaOH + heat treatment (76.4%): delivers the highest peak intensities, confirming a synergistic effect between chemical activation and thermal restructuring. Heat likely facilitates ion migration and nucleation of new crystalline domains [34], enhancing material integrity.
- KOH treatment (~ 55.2%) and KOH + heat treatment (~ 74.1%): also, significantly improve crystallinity, though their impact may differ from NaOH due to the larger ionic radius and milder reactivity of K^+ ions [35]. While KOH is similarly effective in disrupting amorphous bonds and promoting recrystallization, NaOH appears slightly more potent under identical conditions.

These results strongly suggest that alkali activation, particularly NaOH combined with heat treatment, is the most effective strategy for enhancing crystallinity, which correlates with improved thermal and mechanical properties of the resulting material [36, 37]. The inclusion of both NaOH and KOH patterns reinforces the material selection rationale and enhances the scientific robustness of the study.

FTIR spectra for three samples (a: NaOH-treated sample, b: KOH-treated sample, c: untreated sample) are shown in Fig. 10, with peaks representing functional group vibrations.

Sample A — –NaOH-treated (red): Strong transmittance across the spectrum, indicating well-preserved functional groups.

Sample B — KOH-treated (blue): Slightly reduced transmittance, hinting at changes in chemical structure, possibly due to external modifications like chemical or thermal treatments.

Sample C — Untreated (green): Noticeable shifts in transmittance levels, indicating significant alterations in chemical bonds, which may result from a combination of chemical and thermal processes.

The distinct patterns across the samples confirm the impact of surface treatments on chemical functionality, supporting applications where tailored surface chemistry is essential.

Discussion

The microstructural, compositional, and phase analyses of agro-marine residue ash-reinforced aluminum matrix composites (AMMCs) subjected to different alkali and thermal pre-treatment regimes reveal critical insights into their structural evolution and property enhancement. This section synthesizes evidence from X-ray diffraction (XRD), scanning electron microscopy (SEM), and energy-dispersive spectroscopy (EDS) to elucidate the interplay between treatment mechanisms, crystallinity, interfacial morphology, and the resulting mechanical performance.

Structural evolution **via** ***XRD analysis***

The XRD patterns (Fig. 9) show a progressive enhancement in crystallinity across all treatment regimes, with the most significant transformation observed in NaOH + heat-treated samples. The control group exhibits broad, low-intensity peaks (~ 22%), characteristic of an amorphous structure typically found in untreated agro-waste and silicate-based residues. This is attributed to the disordered distribution of carbonaceous and siliceous phases and the presence of residual organics, consistent with previous findings on amorphous precursor matrices [38].

Heat treatment alone induces only a minor increase in peak intensity (~ 22.4%), suggesting limited rearrangement of atomic structures due to dehydration and partial removal of volatiles. However, alkali activation using NaOH results in a substantial increase in peak intensity (~ 57.5%), indicative of enhanced crystalline domain formation. The high alkalinity and smaller ionic radius of Na^+ ions promote deeper penetration and selective dissolution of amorphous silicates and lignocellulosic matrices, triggering nucleation and crystallization [32, 39].

The combined NaOH + thermal treatment delivers the highest crystallinity (~ 76.4%), supporting a synergistic mechanism: alkali-induced breakdown followed by thermally driven reorganization and stabilization of crystalline phases. This behavior aligns with literature reporting that post-alkali heating facilitates ion diffusion, moisture removal, and thermodynamically stable phase formation [11, 40].

KOH treatments, while also effective (~ 55.2% crystallinity), slightly lag behind NaOH in structural reordering, likely due to the larger ionic radius and lower diffusivity of K^+ ions, which hinder deep matrix penetration. However, KOH + heat-treated samples reach up to ~ 74.1% crystallinity, confirming the utility of both alkalis in conjunction with thermal assistance for inducing phase transformation.

Surface morphology and chemical modifications (SEM/EDS)

SEM micrographs reveal notable changes in particle surface morphology post-treatment. NaOH-treated particles exhibit smoother and more uniform surfaces, suggesting effective removal of loosely bound organics and better exposure of hydroxylated surfaces. This enhances wettability and matrix-reinforcement adhesion, minimizing stress concentrations and enabling improved ductility. These observations are in line with prior studies emphasizing the benefits of uniform alkali etching on bonding quality in fiber-reinforced composites [41].

Conversely, KOH-treated particles display rougher, denser textures due to more aggressive surface leaching, leading to improved mechanical interlocking. However, excessive surface roughness may act as stress concentrators, potentially compromising fatigue life. Similar observations were reported by [20], who emphasized the need for optimized activation parameters to balance interlocking and stress distribution.

EDS analysis supports the SEM findings by confirming increased surface oxygen content, indicative of –OH group formation from hydroxylation reactions. This surface functionalization not only improves chemical affinity with the aluminum matrix but also promotes thermally stable aluminosilicate formation, as observed in both NaOH and KOH-treated samples. These chemical modifications play a pivotal role in phase stabilization and thermal endurance.

Mechanical property correlations

While mechanical properties such as tensile strength, hardness, and wear resistance were not experimentally quantified in this study, the microstructural and chemical transformations reported, particularly increased crystallinity, enhanced surface hydroxylation, and refined particle morphology, have been strongly correlated with mechanical performance in similar composite systems [42]. The enhanced crystallinity achieved through alkali and thermal treatments is widely recognized to improve rigidity and load transfer capacity, while smoother or rougher surface textures contribute to interfacial bonding or mechanical interlocking, respectively. Thus, although direct mechanical testing was beyond the scope of the current investigation, the findings presented here offer reliable microstructural indicators of performance improvements. Future studies should aim to experimentally validate these inferences through detailed mechanical characterization, enabling direct correlation between physicochemical modifications and macroscopic mechanical behavior.

Practical and scientific implications

The findings validate the dual-treatment strategy (alkali + heat) as an effective pathway for optimizing agro-marine reinforcement systems. By controlling crystallinity, surface morphology, and chemical composition, composites can be custom-designed for application-specific demands. Moreover, the use of waste-derived ash materials aligns with sustainability goals, reducing environmental impact while offering performance enhancements.

Future studies should explore hybrid alkali treatment strategies, such as sequential or simultaneous NaOH–KOH processing, and incorporate life-cycle assessment (LCA) to evaluate the economic and environmental viability of such treated reinforcements for industrial-scale adoption.

Conclusion

Key findings

This study demonstrates the effectiveness of NaOH and KOH chemical activation in modifying the microstructural properties of agro-marine residue ash used as reinforcement in aluminum matrix composites (AMMCs). NaOH treatment significantly improved crystallinity and surface uniformity, enhancing the potential for ductility and improved load transfer. KOH treatment, in contrast, induced rougher, denser surface morphologies that contribute to improved mechanical interlocking and thermal resistance.

Mechanistic insight

The enhancement in XRD peak intensities, particularly with combined alkali and thermal treatments, suggests a clear restructuring of the composite matrix toward more ordered, crystalline phases. These structural modifications are central to improved material performance and provide predictive insight into mechanical behavior, even in the absence of direct testing.

Justification for scope limitation

Although this study did not include mechanical property measurements, the observed improvements in crystallinity, surface morphology, and chemical composition are strongly correlated with enhanced mechanical performance in comparable systems. This focused approach allowed us to isolate and evaluate the physicochemical mechanisms of alkali activation, establishing a foundation for subsequent studies involving mechanical validation. Future research should incorporate tensile, compressive, and tribological testing to substantiate these findings.

Practical and scientific implications

The compatibility of agro-marine ashes with aluminum matrices illustrates a sustainable pathway for value-added utilization of bio-waste, with implications for lightweight, thermally stable, and wear-resistant components in automotive, marine, and structural applications. Furthermore, the findings support the strategic use of alkali activation as a low-cost, scalable method to engineer reinforcement properties in eco-composites.

Impact statement

This research contributes to the advancement of sustainable materials engineering by leveraging agro-marine residues in aluminum matrix systems. Through controlled alkali activation, the study provides a scientifically grounded method for improving interfacial and structural properties without resorting to synthetic or energy-intensive processing routes. The outcomes align with circular economy goals and offer a promising platform for the development of next-generation green composites with industrial relevance in high-performance applications.

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Authors' contributions

OOB: Conceptualization, Supervision, Methodology, Writing-original draft, Writing-review & editing. SNN: Methodology, Writing-original draft, Visualization, Writing-review & editing. KCO: Software, Investigation, Methodology, Writing-original draft, & Writing-review & editing. MSD: Visualization, Methodology, Writing-original draft, Writing-review & editing. EBO: Visualization, Validation, Writing-original draft, Writing-review & editing. All authors have read and agreed to publish the manuscript.

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Data availability

The data generated and analyzed during the current study are available from the corresponding author upon reasonable request.

Declarations

Ethics approval and consent to participate

This study did not involve human subjects, animals, or biological materials; therefore, ethical approval was not required. However, the study was conducted under the ethical standards of Kampala International University (KIU), Uganda, and adhered to the principles of honesty, objectivity, and integrity.

Consent for publication

We, the authors, hereby grant permission to publish this article in their journal. As the corresponding author, I affirm that the content of this manuscript is original, and I hold full legal rights to the intellectual property presented.

Competing interests

The authors declare no competing interests.

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