ALKALI-ACTIVATED PASTE BASED ON BIOMASS ASH

PASTA ATIVADA POR ALCALIS À BASE DE CINZAS DE BIOMASSA

PASTA ALCALINAMENTE ACTIVADA BASADA EN CENIZA DE BIOMASA

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ABSTRACT

The increasing demand for sustainable alternatives in civil construction has spurred the development of cementitious materials with reduced environmental impact. In this context, alkali-activated materials have emerged as a promising solution, enabling the partial replacement of Portland cement with industrial by-products, such as biomass ash and fly ash. This paper investigates the use of biomass ash from the ceramic industry (CBA) and the cellulose industry (PBA), in binary mixtures with fly ash (FA), in the production of alkaline-activated cementitious pastes. Mini-slump, setting time, compressive strength, water absorption and scanning electron microscopy (SEM) tests were performed. CBA with a high calcium content (86.23%), showed greater reactivity and better mechanical performance in the pastes. In contrast, PBA, characterized by a high silica content (65.9%) and a low calcium content (6.54%), exhibiting higher compressive strength (30,66 MPa), but lower workability indices. The results demonstrate that these ashes are viable alternatives to Portland cement, contributing to sustainability and the circular economy by reusing industrial waste. However, the variability in ash properties requires strict control to ensure consistency and optimization of composites.

KEYWORDS

Alkali activated materials; Binary pastes; Fly ash.

RESUMO

A crescente demanda por alternativas sustentáveis na construção civil tem estimulado o desenvolvimento de materiais cimentícios com menor impacto ambiental. Nesse contexto, os materiais álcali-ativados têm surgido como uma solução promissora, possibilitando a substituição parcial do cimento Portland por subprodutos industriais, como cinzas de biomassa e cinzas volantes. Este trabalho investiga o uso de cinzas de biomassa da indústria cerâmica (CBA) e da indústria de celulose (PBA), em misturas binárias com cinzas volantes (FA), na produção de pastas cimentícias álcali-ativadas. Foram realizados ensaios de mini-slump, tempo de pega, resistência à compressão, absorção de água e microscopia eletrônica de varredura (MEV). CBA com alto teor de cálcio (86,23%), apresentou maior reatividade e melhor desempenho mecânico nas pastas. Em contrapartida, PBA, caracterizado por alto teor de sílica (65,9%) e baixo teor de cálcio (6,54%), apresentou maior resistência à compressão (30,66 MPa), porém menores índices de trabalhabilidade. Os resultados demonstram que essas cinzas são alternativas viáveis ao cimento Portland, contribuindo para a sustentabilidade e a economia circular por meio do reaproveitamento de resíduos industriais. No entanto, a variabilidade nas propriedades das cinzas requer controle rigoroso para qarantir a consistência e a otimização dos compósitos.

PALAVRAS-CHAVE

Materiais Álcali-ativados: Pastas binárias: Cinza volante.



RESUMEN

La creciente demanda de alternativas sostenibles en la construcción civil ha impulsado el desarrollo de materiales cementantes con menor impacto ambiental. En este contexto, los materiales alcalinamente activados han surgido como una solución prometedora, permitiendo la sustitución parcial del cemento Portland por subproductos industriales, como las cenizas de biomasa y las cenizas volantes. Este trabajo investiga el uso de ceniza de biomasa proveniente de la industria cerámica (CBA) y de la industria de la celulosa (PBA), en mezclas binarias con ceniza volante (FA), para la producción de pastas cementicias alcalinamente activadas. Se realizaron ensayos de asentamiento en mini-slump, tiempo de fraguado, resistencia a la compresión, absorción de agua y microscopía electrónica de barrido (SEM). La CBA, con un alto contenido de calcio (86,23%), mostró una mayor reactividad y mejor desempeño mecánico en las pastas. En contraste, la PBA, caracterizada por un alto contenido de sílice (65,9%) y bajo contenido de calcio (6,54%), presentó una resistencia a la compresión más elevada (30,66 MPa), pero menores índices de trabajabilidad.Los resultados demuestran que estas cenizas son alternativas viables al cemento Portland, contribuyendo a la sostenibilidad y a la economía circular mediante la reutilización de residuos industriales. Sin embargo, la variabilidad en las propiedades de las cenizas requiere un control riguroso para garantizar la consistencia y la optimización de los compuestos.

PALABRAS CLAVE

Materiales alcalinamente activados; Pastas binarias; Ceniza volante.

1. INTRODUCTION

Ordinary Portland Cement (OPC), a fundamental material in civil construction, is produced from a mixture of limestone (calcium carbonate) and clay (aluminum and iron silicates). During production, the calcination of calcium carbonate (CaCO₃) generates calcium oxide (CaO) and releases carbon dioxide (CO₂) into the atmosphere (Castro, 2021). According to the National Cement Industry Union (SNIC, 2024), Brazil produced an average of more than 50 million tonnes of OPC annually between 2015 and 2023. In 2017, the country became the largest producer in Latin America, accounting for 1.3% of global production (Federal Government, 2018). For each tonne of OPC manufactured, approximately 0.5–0.6 tonnes of CO₂ are emitted (Cement Technology Roadmap, 2019).

The uncontrolled emission of CO2, along with other gases, significantly contributes to environmental issues such as global temperature variations, changes in atmospheric composition, and impacts on human health due to the dispersion of pollutant particles (Silva et al., 2016). In light of this, researchers are exploring sustainable alternative solutions to mitigate environmental impacts. Recent studies emphasize the urgency of transitioning to renewable energy sources, as the energy sector accounts for nearly three-quarters of global greenhouse gas emissions (Kyei et al., 2025).

A promising alternative is Alkali-Activated Materials (AAM), which stand out for not requiring the calcination of precursors. These materials exhibit high initial mechanical strength, fire resistance, and low water absorption (França et al., 2022). The term AAM denotes a class of cementitious materials produced through the reaction between aluminosilicate precursors and alkaline activators (Kong; Kurumisawa, 2023).

In the production of AAM, various industrial wastes or by-products can be used as precursors. A relevant example is biomass ash, a by-product of the red ceramic and cellulose industries, among others. The red ceramic industry, a crucial sector for civil construction, generates Ceramic Industry Biomass Ash (CBA) during the process of burning wood in the heat treatment of pieces (Aguiar et al., 2022). In turn, the pulp industry, which occupies a prominent position in the Brazilian industrial scenario, also generates similar waste, such as Pulp Biomass Ash (PBA).

The characterization of these residues is essential to determine their viability as precursors in AAMs. For

PBA, Oliveira (2023) found that the material obtained from the waste disposal basin of a pulp plant in Maranhão contained 6,54% calcium oxide (CaO), a characteristic which, combined with its pozzolanic activity, indicates significant potential for the production of cementitious materials. In contrast, CBA, derived from the kilns of the ceramic industry in the municipality of Crateús, Ceará, contained 86,23% CaO (Mendonça, 2023). This variation in calcium content suggests that the two ashes can serve different functions in the production of AAMs.

The use of PBA and CBA, in combination with other supplementary materials, can lead to the development of AAMs with physical and mechanical properties suitable for practical applications. Fly ash (FA), a by-product generated from coal combustion in thermoelectric power plants, is well known for its pozzolanic activity, which makes it an effective material for producing high-performance cements and concretes (Costa, 2015; Cirino, 2021). Therefore, the incorporation of these residues in binary mixtures represents a promising strategy for the production of AAMs with optimized properties for civil construction.

Thus, this study aims to analyze the physical and mechanical properties of binary pastes formed by the reaction between PBA and FA and between CBA and FA, evaluating the potential of these combinations for the production of AAM with lower environmental impacts and sufficient physical and chemical characteristics for practical applications in the construction sector.

2. ALKALI ACTIVATED MATERIALS

The term MAA refers to a group of cementitious materials formed by the reaction of precursors with alkaline activators (Kong; Kurumisawa, 2023). The precursors are typically aluminosilicate powders, while the activator is an alkaline solution that promotes the formation of a hardened binder through the generation of hydrated aluminosilicate phases (Provis, 2018).

According to Costa (2022), the cementitious matrix of AAMs varies in both structure and chemical composition, with calcium content being a determining factor in silicate formation. Precursors rich in calcium tend to produce a calcium–aluminosilicate hydrate (C-A-S-H) gel, whereas low-calcium precursors lead to the formation of a highly cross-linked and structurally disordered sodium–aluminosilicate hydrate (N-A-S-H) gel (Bernal et al., 2014).

3. MATERIAL AND METHODS

3. 1. Materials

The precursors used in the preparation of the pastes were biomass ash from the ceramics industry (CBA), biomass ash from the pulp industry (PBA), with high and low calcium contents, respectively, and fly ash from thermoelectric power plants (FA). Sodium hydroxide (NaOH) and sodium silicate (Na₂SiO₃) were employed as alkaline activators.

The CBA was obtained from a ceramics industry producing ceramic blocks, located in Crateús, Ceará, Brazil. The wood used by the factory is sourced locally and includes Mimosa caesalpiniifolia and Mimosa tenuiflora (Mendonça, 2023). The CBA was sieved through a 45 µm mesh prior to characterization. Particle size distribution was determined by laser diffraction using a Malvern Mastersizer 2000 instrument, following ISO 13320 (2020) and ABNT NBR 12653 (2015). Specific gravity was measured according to ABNT NBR 16605 (2017), and chemical composition was analyzed via energy-dispersive X-ray spectroscopy (EDS) to evaluate the material's properties.

PBA was obtained from the pulp processing industry, specifically from the waste disposal basin of Suzano Celulose e Papel, located in Imperatriz, Maranhão, Brazil. Characterization of the material included particle size distribution by laser diffraction, following ISO 13320 (2020); determination of specific gravity using the gas pycnometry technique in accordance with ABNT NBR 12154 (2022); and chemical composition analysis via X-ray fluorescence. The calcium content in the ash was 6.54% (Oliveira, 2023).

Finally, the fly ash (FA) was obtained from the Pecém Thermoelectric Plant, located in Pecém, Ceará, Brazil. The specific gravity of the FA was determined according to ABNT NBR 16605 (2017), and its chemical composition was analyzed using X-ray fluorescence.

The physical and chemical properties of FA, CBA, and PBA are summarized in Table 1.

Chemical	Materials		
Composition (%)	FA	CBA	PBA
Al2O3	14,49	-	8,4
SiO2	41,49	2,34	65,9
P2O5	0,44	2,49	0,92
SO3	0,29	1,54	4,71
CI	-	-	0,05
K20	4,54	3,61	1,72
CaO	9,54	86,23	6,54
TiO2	3,26	-	0,83

MnO	0,17	-	0,08
Fe2O3	24,95	-	3,5
ZnO	0,14	-	0,02
SrO	0,55	-	0,04
MgO	-	3,78	1,18
Na2O	-	-	0,67
NiO	-	-	0,01
CuO	0,11	-	0,01
Rb2O	-	-	< 0,01
Y2O3	-	-	< 0,01
ZrO2	-	-	0,11
Nb2O5	-	-	< 0,01
Specific gravity	2,18	2,87	2,59
F45 µm (%) Sample	-	12	12
D10% (μm)	1,61	4,918	1,51
D50% (μm)	7,38	14,384	5,12
D90% (μm)	30,5	53,489	25,96

Table 1: Physical and chemical characterizations of FA, CBA and PBA.

Source: The Authors.

Sodium hydroxide was supplied in solid form and dissolved before use to prepare a 6 mol/L solution. Sodium silicate was supplied as an aqueous solution, with a composition of 14.38% Na_2O , 31.83% SiO_2 , 46.21% total solids, and a specific gravity of 1.58 g/cm³.

3. 2. Methods

3. 2. 1. Preparation of mixtures

The proportions of each precursor were determined experimentally, and the intervals were selected according to the observed behavior of the pastes. Based on these results, the values presented in Table 4 were established.

Mixture	FA (%)	CBA (%)	FA (%)	PBA (%)
CBA20	80	20	-	-
CBA40	60	40	-	-
PBA20	-	-	80	20
PBA40	-	-	60	40

Table 2: Mixtures.
Source: The Authors.

Proportions of 20% and 40% of each ash were selected, as pastes containing 60% biomass ash exhibited insufficient workability for molding and testing.

The liquid-to-solid ratio was fixed at 0.45, with the liquid phase comprising the combined activators (sodium hydroxide and sodium silicate in equal proportions) and the solid phase consisting of fly ash and biomass ash. The only variables considered were the relative contents of FA, CBA, and PBA.

The mixing sequence consisted of 1 minute of manual mixing, followed by 1.5 minutes in a mortar mixer, an additional 1 minute of manual mixing, and a final 1.5 minutes in the mortar mixer, totaling 5 minutes. The specimens were cast in $4 \times 4 \times 4$ cm cubic acrylic molds, with three specimens produced per mold.

3. 2. 2. Tests and analysis

Characterization in the fresh state was carried out using the mini-slump test. The spreading diameter was measured with a caliper at 30 seconds, 1 minute, 2 minutes, 3 minutes, 4 minutes, and 5 minutes, in order to monitor the short-term evolution of workability.

The setting time was determined using the Vicat apparatus, following ABNT NBR 16607 (2018). Measurements were initially taken every 30 minutes and then every 5 minutes. The initial setting time was identified when the needle penetrated 6 ± 2 mm into the paste. Figure 1 presents the equipment used in the test.



Figure 1: Physical and chemical characterizations of FA, CBA and PBA. **Source:** The Authors.

The compressive strength test was conducted in accordance with NBR 13279 (2005). Specimens were subjected to compression after 28 days. The water absorption rate was adapted from Luukkonen (2019). Test specimens were heated to 50 °C in an oven and the dry weight was measured. All faces were waterproofed with a liquid membrane, as shown

Figure 2, except one, which was in contact with water (2 mm depth).

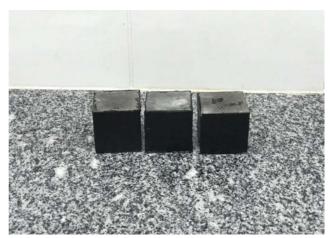


Figure 2: Waterproofed test especimens

Source: The Authors.

Subsequent weighings were performed at predetermined intervals: 1 min, 5 min, 10 min, 20 min, 30 min, 60 min, 1 day, 2 days, 4 days, 6 days, and 8 days. Water absorption was then calculated according to Equation (1).

$$I = \frac{m_t}{a \times d} \tag{1}$$

Where I = amount of water absorbed (mm), mt = weight of the sample (g), a = area of the exposed surface (mm²), and d = density of water (g/mm³).

Microstructural analysis was performed using Scanning Electron Microscopy (SEM) at the Central Analytical Facility of the Federal University of Ceará (UFC). The equipment used was a FEI company, model QUANTA FEG 450.

4. RESULTS

4. 1. Characterization of pastes in the fresh state

Figure 3 illustrates the variation in spread diameter over time for three of the four pastes evaluated using the minislump test. The CBC40 paste exhibited insufficient fluidity to allow measurement.

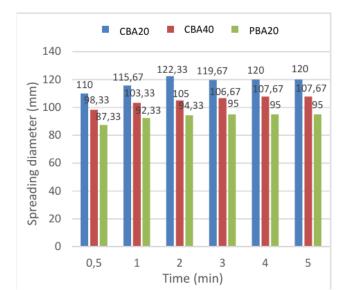


Figure 3: Mini slaughter test. **Source**: The Authors.

The final measured diameters were 95 mm (PBA20), 107.7 mm (CBA40), and 120 mm (CBA20). The CBA40 and CBA20 pastes reached these values at the fourth minute, whereas CBC20 stabilized at the third minute. This indicates that the CBA mixtures exhibited higher fluidity, which is consistent with expectations, as PBA is a finer ash with a larger surface area, resulting in increased water demand and reduced initial flow. A similar trend was reported by Jurado-Contreras et al. (2022), who observed that finer biomass fly ash particles tend to decrease workability due to their high reactivity and surface energy.

The CBA40 paste, containing 20% more biomass ash than CBA20, exhibited a 10.25% reduction in final spread diameter. In the pastes with CBC, the increase of CBC40 compared to PBA20 significantly reduced the workability, making PBA40 impossible to measure, as shown in Figure 4. These results indicate that both ashes displayed similar trends in response to variations in paste content.



Figure 4: Mini slump result CBA40. **Source**: The Authors.

Teixeira et al. (2015) reported that increasing the biomass ash content reduces workability due to particle irregularity and high surface area. This behavior was observed in both PBA and CBA pastes; however, only PBA is finer than FA, suggesting that additional factors influenced the workability of the CBA mixtures.

However, the high calcium content of CBA negatively affects paste workability. Consequently, increasing the CBA content from 20% to 40% reduces workability, as the calcium content of FA is lower than that of CBA.

4. 2. Settingtime

With the same percentages of biomass ash and fly ash, the CBA had a longer initial setting time at both FA and BA contents.

Increasing the biomass ash content by 20% reduced the setting time for both CBA and PBA pastes. The reduction was 32.87% for PBA and 22.35% for CBA. In the case of CBA, this decrease is attributed to its higher ash content and greater proportion of free calcium, which accelerates hydration and consequently shortens the setting time. According to Karadağ et al. (2025), biomass ashes with elevated calcium levels—such as hazelnut shell ash significantly accelerate the final setting time, whereas ashes with lower reactivity may delay it depending on their chemical composition and replacement ratio. For PBA, the reduced setting time is due to its smaller particle size, which increases surface area and accelerates the reaction, consistent with Caetano et al. (2023), who reported faster setting for finer ash particles in alkaliactivated systems.

The results of the setting time tests are presented in Figure 5.

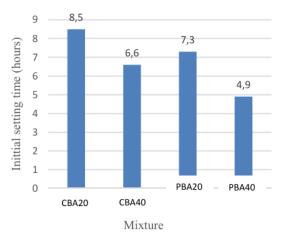


Figure 5: Setting time. **Source**: The Authors.

4. 3. Compressive strength

The strengths of CBA and PBA pastes varied with increasing biomass ash content. The CBA paste exhibited a 21% reduction in strength at 20% BA, whereas the PBA paste showed a 14% increase at the same proportion. Among the different biomass ash contents, the mixture containing 80% FA and 20% CBA displayed the highest strength, while the mixture with 60% FA and 40% PBA achieved the best strength index. Figure 6 presents the results of the compressive strength tests.

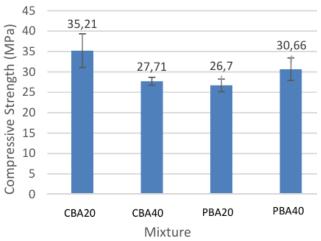


Figure 6: Compressive strength at 28 days. **Source**: The Authors.

Silica (SiO₂) and alumina (Al₂O₃) combine to form aluminosilicates, which provide the essential framework for mechanical strength (Davidovits, 2008). PBA contains 65% SiO₂, FA 42.20%, and CBA only 2.34%. Increasing the proportion of PBA raises the SiO₂ content, thereby enhancing strength. In contrast, increasing the CBA content decreases the SiO₂ concentration, which impedes the formation of aluminosilicates.

Risson et al. (2025) analyzed eucalyptus ash with 2.1% silica, observing behavior similar to CBA, with a reduction in strength as the ash content increased. Liang et al. (2025) characterized wood biomass fly ash with elevated SiO₂ content and noted that despite its chemical richness, strength gains were limited due to poor reactivity and particle morphology. Elawadly and Sanad (2025) observed both positive and negative variations in 28-day compressive strength with increasing sugarcane bagasse ash content, emphasizing the complex interplay between ash dosage, microstructure, and hydration kinetics.

4. 4. Water absorption

The water absorption process exhibited three distinct phases. The first phase occurred within the first hour, with minimal absorption; the highest value was observed for PBA40 (2.08 mm). The second phase, spanning 1 to 96 hours, showed the highest absorption for PBA40 (8.19 mm) and the lowest for CBA40 (3.30 mm). In the third phase, after 96 hours, all pastes reached saturation, with no significant further absorption.

CBC exhibited a significant increase in water absorption with increasing ash content, whereas CBIC showed no noticeable change. The low workability of the PBA40 paste may have contributed to the formation of additional voids. Similar behavior was observed by Irshidat et al. (2022), who reported that the incorporation of municipal solid waste incineration ashes in alkaliactivated binders can lead to increased porosity and water absorption, especially when particle morphology and mix rheology are not optimized.

Figure 7 presents the water absorption rates, calculated using Equation (1), as a function of the square root of time.

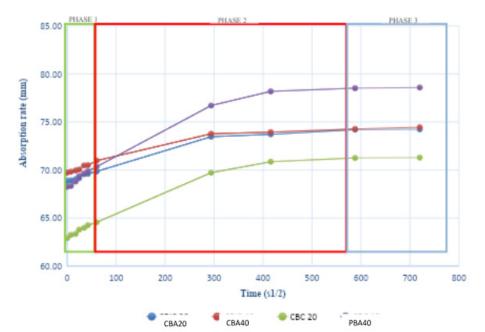


Figure 7: Water absorption. **Source**: The Authors.

4. 5. Scanning microstructure

Figures 8 and 9 present the results of scanning electron microscopy (SEM) analyses for the CBA20, CBA40, CBC20, and CBC40 matrices. In all samples, sodium oxide crystals, microcracks, aluminosilicate gels, fly ash particles, and porous regions were observed, as illustrated in the corresponding figures. Enlarged images (Figures 8b, 8d, 9b, and 9d) highlight these features.

The porous regions result from unreacted or partially reacted fly ash particles, which often exhibit a spherical morphology and smooth surfaces, characteristics typical of mineral coal fly ash (Oliveira, 2023). CBA40 and PBA40, with lower fly ash content than their CBA20 and PBA20 counterparts, display fewer pores and unreacted particles.

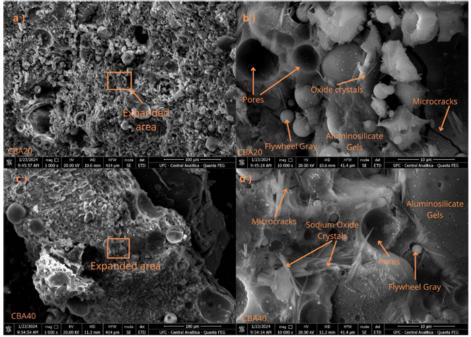


Figure 8: Microscopy CBA20, CBA40.

Source: The Authors.

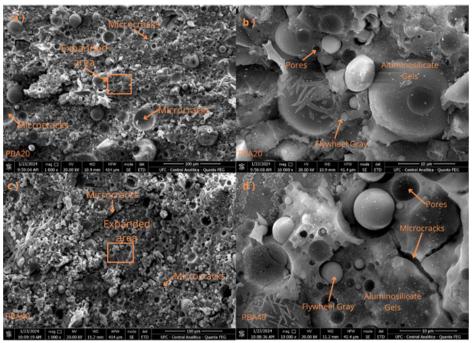


Figure 9: Microscopy PBA20, PBA40.

Source: The Authors.

The formation of aluminosilicate gels was observed in all pastes. These gels can be classified as C-A-S-H (high calcium), N-A-S-H (low calcium), or C-N-A-S-H (intermediate calcium) (Costa, 2022). The CBA20 and CBA40 matrices, belonging to an intermediate calcium system, form C-N-A-S-H gels. CBIC exhibits a high calcium content (86.25%), while fly ash contains low calcium (9.54%). Consequently, the CBC20 and CBC40 pastes, composed of CBC and fly ash with low calcium content, form N-A-S-H gels.

Finally, the presence of sodium oxide is linked to the high calcium content of CBA, which competes with sodium in polymerization. Calcium binds to silicates and aluminates, leaving excess sodium or potassium outside the matrix (Davidovits, 2008).

5. CONCLUSION

This study investigated alkali-activated pastes composed of biomass ash and fly ash, focusing on flow properties, setting time, compressive strength, water absorption, and microstructure. The main contributions of the work are summarized as follows:

 The flow analysis indicated that mixtures containing ceramic industry ash (CBA) exhibit better workability than pastes with cellulose biomass ash (PBA). This behavior is attributed

- to differences in particle fineness and calcium content, which influence the handling properties of the pastes;
- Increasing the biomass ash content reduced the setting time, with a more pronounced effect in pastes containing CBA. This finding is critical for determining appropriate curing procedures and application methods, as setting time is a key factor for the effective use of the pastes;
- CBA and PBA pastes exhibited distinct behaviors.
 The compressive strength of CBA pastes decreased with increasing ash content, whereas PBA pastes showed an increase in strength under the same conditions. These results indicate that the selection of biomass ash can significantly influence the material's suitability for specific applications;
- Water absorption varied among the pastes, with PBA exhibiting greater changes as the ash content increased. The low workability of the PBA40 paste may have contributed to this behavior. These findings suggest that material selection should consider not only strength but also durability and permeability;
- Microstructural analysis revealed the formation of aluminosilicate gels, indicating that an appropriate combination of materials promotes the development of the cementitious matrix. However, the presence of microcracks suggests

that adjustments in workability or curing may be required to prevent structural defects. The occurrence of unreacted particles in both pastes underscores the importance of optimizing the mixture formulation to enhance material performance. Additionally, the formation of sodium oxide crystals highlights the need for careful selection of the activator, considering both quality and dosage.

The results demonstrated that alkali-activated pastes can contribute to environmental sustainability while providing materials with acceptable mechanical properties, depending on the type and proportion of ash used.

Future studies should investigate their behavior under high temperatures, acidic environments, long-term durability, and varying ash contents. Such analyses will improve understanding of their performance and feasibility for applications in the construction sector.

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