

Research Article

Effect of Rice Hull Ash on the Geopolymerization of a Kaolinite Clay from Togo

Anove Komla Mawoulikplim^{1,*} , Kpetemey Amen¹ ,
Tchanate Kolani N'Djoibini¹ , Degbe Koffi Agbegnigan¹ ,
Babakoua Dilami Diana¹ , Tchegueni Sanonka¹ , Douty Damdjoin¹ ,
Fiaty Koffi² , Drogui Patrick³ , Tchangbedji Gado¹ 

¹Waste Management Treatment and Recovery Laboratory (GTVD), Lome University, Lome, Togo

²Automatic Control Chemical and Pharmaceutical Engineering Laboratory (LAGEPP), Claude Bernard Lyon-1 University, Villeurbanne Cedex, France

³Water Earth Environment Center, The National Institute for Scientific Research (INRS), Qu ébec, Canada

Abstract

Geopolymers have recently emerged as a promising class of inorganic aluminosilicate polymer materials. They present a viable alternative to Portland cement, which is well known for its significant contribution to greenhouse gas emissions. To support the reduction of these emissions, this study aims to develop geopolymer cements by investigating the impact of rice husk ash on the geopolymerization of local kaolinite clay using an alkaline solution. Rice husk ash (with a SiO₂ content of 91.6%), obtained by calcining rice husks at 600 °C, serves as a source of amorphous silica. The GP₀ geopolymer material, derived from clay calcined at 750 °C and activated with a 12N sodium hydroxide solution, exhibits a compressive strength of 9.9 MPa. This mechanical strength was enhanced by incorporating rice husk ash, which produces sodium silicate solutions as activators. The addition of 10% rice husk ash increased the compressive strength from 9.9 MPa to 15.9 MPa. The sodium silicate solution derived from the ash proved to be an effective alkaline activator in the geopolymer synthesis. Consequently, rice husk ash can potentially replace commercial sodium silicate solutions, contributing to the formulation of more eco-friendly materials. However, further research is needed to optimize the mechanical properties of these geopolymers.

Keywords

Clay, Ash, Geopolymerization, Alkaline Solution, Environment

1. Introduction

Geopolymers have recently emerged as a new class of inorganic polymers and are considered promising candidates for various applications [1]. These polymers are synthesized through the polycondensation of monomeric or oligomeric

species of aluminum and silicon in solutions activated by an alkali metal. Geopolymer precursors can be derived from various aluminosilicate sources, such as metakaolin, volcanic slag, and ash [2, 3].

*Corresponding author: manove05@gmail.com (Anove Komla Mawoulikplim)

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In general, any powder with high levels of silica and alumina, coupled with an amorphous phase, can serve as a geopolymer precursor. The selection of these precursors often depends on factors like availability, cost, and the specific application. A broad range of materials can be activated, including both calcined and uncalcined clays [4-6], as well as industrial or agricultural by-products [7, 8].

Rice is a major global food crop, and its production generates substantial waste, notably rice husk. In Togo, a significant rice-producing country in Africa, the large volume of rice husk generated often lacks commercial value and is typically burned in open spaces, leading to pollution problems. To address these issues and conserve resources, efforts have been made to combust rice husks under controlled conditions to produce a material rich in silica [4, 9]. This silica is crucial for synthesizing sodium silicate solutions, which are vital in geopolymer production.

The manufacture of commercial sodium silicate solutions is energy-intensive, involving the treatment of silica sand and sodium carbonate at approximately 1400 °C [4]. This process is costly due to the high energy requirements and results in considerable air pollution from emissions such as carbon dioxide, nitrogen oxides, and sulfur oxides. Therefore, utilizing silica derived from rice husk ash, combined with sodium hydroxide pellets and distilled water, to produce sodium silicate solutions for geopolymer cements presents an attractive alternative.

This study focuses on the valorization of local materials from Togo, specifically kaolinite clay and rice husk ash, in the synthesis of geopolymer binders. It examines the impact of incorporating rice husk ash on the physico-chemical and mechanical properties of geopolymer binders.

2. Material and Methods

2.1. Sampling and Preparation of Kaolinite Clay

Kaolinite clay was sourced from Afagnan (6°29'39" N, 1°37'56" E), a locality in southern Togo, approximately 95 km from Lomé. Upon arrival at the laboratory, the clay was air-dried on the bench for 72 hours. It was then crushed and ground to a particle size of 75 µm to produce clay powder. To enhance the reactivity of the clay powder [10], it was calcined at 750 °C with a heating rate of 10 °C/min for 2 hours using an SNOL 15/1200 model electric furnace.

2.2. Ash from Rice Husks

Rice husk ash was obtained through the incineration of rice husks collected from the rice-growing region of Kovié located 30 km northwest of Lomé. The rice husks were incinerated at 600 °C for 2 hours in an SNOL 15/1200 electric furnace. The resulting ash was then ground to a particle size of 75 µm.

2.3. Preparation of Alkaline Solutions

Activator solutions, which are alkaline silicate solutions, were prepared from a reference solution S₀, which consists of 12 M sodium hydroxide. This solution was prepared by dissolving sodium hydroxide pellets with a purity of 99% in distilled water. Three silicate solutions (S₁, S₂, and S₃) were formulated by adding rice husk ash in specific proportions to the S₀ solution. These solutions contained 62.5 g, 125 g, and 187.5 g of rice husk ash per liter of S₀, respectively.

2.4. Formulation and Nomenclature of Geopolymer Materials

The synthesis protocol was consistent across all four geopolymers developed. Geopolymer pastes were prepared by mixing the clay calcined at 750 °C with the activating solution at a mass ratio (clay/activating solution) of 1.25. The mixtures were kneaded for 10 minutes with a glass rod and then cast into cylindrical plastic molds (30 mm diameter, 30 mm height). The geopolymer paste samples were dried for 24 hours at room temperature before demolding. The samples were then cured at room temperature for 28 days prior to characterization.

For nomenclature, the following designations were used: GP₀, GP₁, GP₂, and GP₃, corresponding to the geopolymers synthesized with solutions S₀ (control without ash), S₁, S₂, and S₃, respectively. The percentages of ash in these geopolymers were 0%, 5%, 10%, and 15% by mass.

2.5. Physico-Chemical and Mechanical Characterizations

Various characterization techniques were employed to study the formation mechanisms, structure, and morphology of the synthesized geopolymer materials.

Compression tests were performed using a Controls Vitesse device equipped with a 1500 kN sensor. The test involved applying opposing axial forces to the specimen, which was placed between the platens of a press. The upper platen descended at a rate of 2 mm/min. Samples, which were 30 mm in diameter and 20 mm in height, were tested at 28 days of age. Three samples per material type were tested.

Post-compression, fragments of each sample were crushed, and the powders were used for X-ray diffraction (XRD) and Fourier transform infrared (FTIR) analyses.

FTIR analysis was conducted using a BRUKER VERTEX 70 spectrometer. Samples were finely ground, mixed with 95% by mass of KBr, and pressed to form transparent pellets for infrared analysis. The absorption bands were examined within the range of 4000 to 400 cm⁻¹.

The mineralogical composition was determined by X-ray diffraction (XRD) using a BRUKER D8 diffractometer. The analysis was performed over the range of 5° to 70°, and the crystalline phases present in the materials were identified

using QualX software version 2.24 with the COD 1906 database.

3. Results and Discussion

3.1. Characterization of Raw Materials

Chemical Composition

Elemental analysis via Inductively Coupled Plasma (ICP) was conducted to determine the chemical composition of the kaolinite clay and rice husk ash. The results are summarized in Table 1.

For kaolinite clay, the predominant oxides are SiO₂ (43.8%)

and Al₂O₃ (34.9%), confirming its aluminosilicate nature. The higher loss on ignition (LOI) observed in the clay supports its kaolinitic composition [11].

The rice husk ash, obtained by calcining rice husks at 600 °C for 2 hours, has an ash content of 26%. The white-colored ash was analyzed by ICP and found to be highly silica-rich (91.6%). It also contains 1.49% K₂O and has a LOI of 3.7%, indicating some residual unburnt material. The alumina content is relatively low (1.28%). This high silica content suggests that the ash can be a valuable additive or precursor for geopolymer binder synthesis when used in appropriate quantities [12, 13].

Table 1. Elemental Chemical Composition of Rice Husk Ash and Kaolinite Clay.

Material	SiO ₂ (%)	Al ₂ O ₃ (%)	CaO (%)	Fe ₂ O ₃ (%)	K ₂ O (%)	MgO (%)	TiO ₂ (%)	Na ₂ O (%)	LOI (%)
Kaolinite Clay	43.80	34.90	0.14	3.60	0.24	0.15	1.39	0.10	15.70
Rice Husk Ash	91.60	1.28	0.52	0.53	1.49	0.31	0.09	0.10	3.70

3.2. Characterization of Engineered Geopolymers

3.2.1. Physical and Mechanical Properties

The physical and mechanical properties of the engineered geopolymers were evaluated, including bulk density, water-accessible porosity, and compressive strength.

Figure 1 shows the bulk density of the geopolymer materials made from kaolinite clay with varying rice husk ash content. The density ranges from 1.51 to 1.64 g/cm³. An increase in bulk density was observed with higher ash content up to 10%, but a decrease in density was noted for the sample

with 15% ash. This variation in density is related to the material's porosity.

Figure 2 illustrates the water-accessible porosity of the geopolymers. Porosity, which represents the volume of open pores in a material, was measured to be between 22.17% and 30.12%. An inverse relationship is observed between ash content and porosity: as the amount of ash increases, the porosity decreases. This trend aligns with the observed changes in bulk density; denser materials tend to have lower porosity.

Porosity affects the water absorption rate and mechanical resistance of the material. All tested materials demonstrated good porosity, remaining below 40% [9].

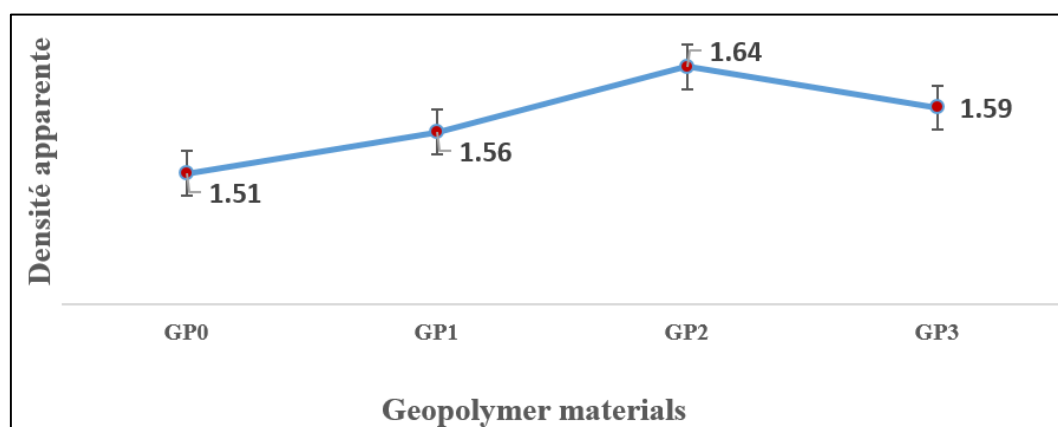


Figure 1. Bulk density of geopolymers.

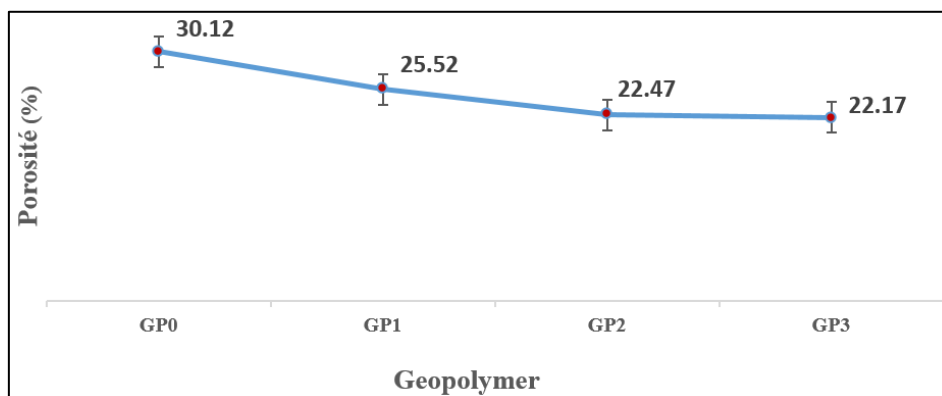


Figure 2. Water-accessible porosity of geopolymer materials.

The compressive strength of materials made from kaolinite clay and rice husk ash, measured after 28 days, is presented in Figure 3. The compressive strength ranges from 9.9 to 15.9 MPa. It is observed that the compressive strength increases with the amount of rice husk ash added to the materials [4, 12].

This trend in mechanical strength correlates well with the observed variations in bulk density and porosity. Denser materials, which are less porous, absorb less water and exhibit higher mechanical strength. These factors combined help assess the impact of rice husk ash on the material properties. The addition of rice husk ash increases the amorphous silica content in the aluminosilicate mixture and enhances the release of silicate species during the alkaline dissolution phase [13-15]. The highly concentrated alkaline solutions lead to the formation of a gel with an extensive three-dimensional aluminosilicate network, which contributes to the material's

strength by binding the particles together.

However, the mechanical properties deteriorate when the ash content exceeds 10%. Specifically, the material containing 15% ash (GP₃) exhibits a decrease in both density and compressive strength. This reduction may be attributed to the formation of zeolites or the presence of undissolved ash within the GP₃ material, which compromises its structural integrity.

The compressive strength of the materials produced ranges from 10 to 19 MPa, aligning with values reported in previous studies [9, 16]. According to Kamaloo [16], for metakaolin-based geopolymers, the compressive strength for SiO₂/Al₂O₃ molar ratios similar to those used in this study should fall between 10 and 49 MPa. Additionally, Seick [9] found that for geopolymers with calcined clay and rice husk ash, with SiO₂/Al₂O₃ molar ratios between 2.5 and 3, the compressive strength ranges from 14 to 26 MPa.

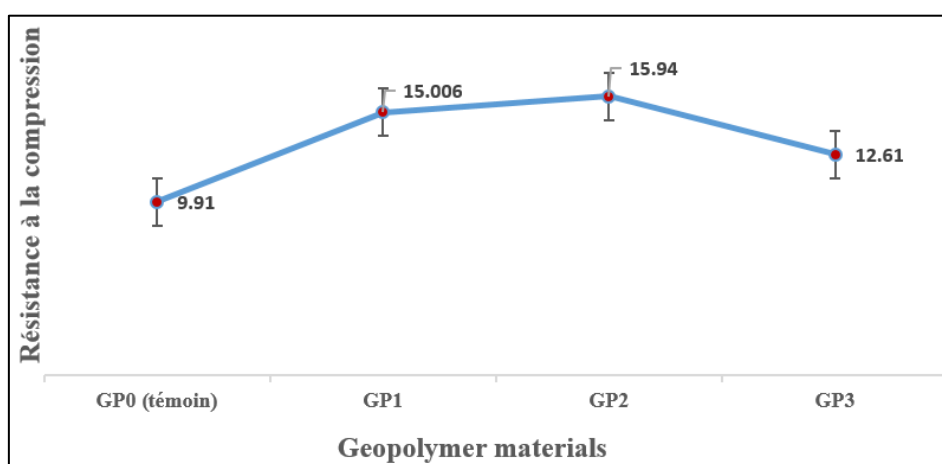


Figure 3. Compressive strength of engineered geopolymer materials.

3.2.2. Structural and Mineralogical Characterizations of the Materials Produced

To understand the mineralogical profile of the geopolymers after consolidation, infrared analysis and X-ray diffraction

(XRD) were performed on two formulations: the control material without rice husk ash (GP₀) and the formulation with the optimal physical and mechanical properties, GP₂, which contains 10% rice husk ash.

Fourier Transform Infrared (FTIR) Spectroscopy Analysis

The FTIR spectra (Figure 4) reveal distinct differences between the GP₀ and GP₂ materials, primarily in the intensity and position of the absorption bands. As the silica content increases, which is associated with the formation of the geopolymer gel, the intensity of these bands decreases.

- 1) *Si-O-X Bonds*: The primary peaks in the spectra are found at 976 cm⁻¹ for GP₂ and 974 cm⁻¹ for GP₀. These peaks are attributed to Si-O-X bonds, where X can be Si, Al, K, or Na [17, 18]. The slight shift and intensity differences in these peaks reflect the variation in the silicate network structure due to the different silica content.
- 2) *Hydroxyl Group Vibrations*: Peaks at 3460 cm⁻¹ and

1655 cm⁻¹ for GP₂, and 3471 cm⁻¹ and 1660 cm⁻¹ for GP₀, correspond to the -OH group vibrations. These peaks indicate the presence of water of hydration either within the cavities or on the surface of the geopolymers [19, 20].

- 3) *Si-O-Si and Si-O Bonds*: Peaks around 735 cm⁻¹ for GP₂ and 665 cm⁻¹ for GP₀ are associated with Si-O-Si and Si-O bonds, respectively. These bands are indicative of zeolite formation [21].
- 4) *Carbonate Groups*: The peaks at 1440 cm⁻¹ and 1471 cm⁻¹ in both spectra are attributed to the asymmetric stretching mode of the O-C-O bonds in CO₃²⁻ groups [22].

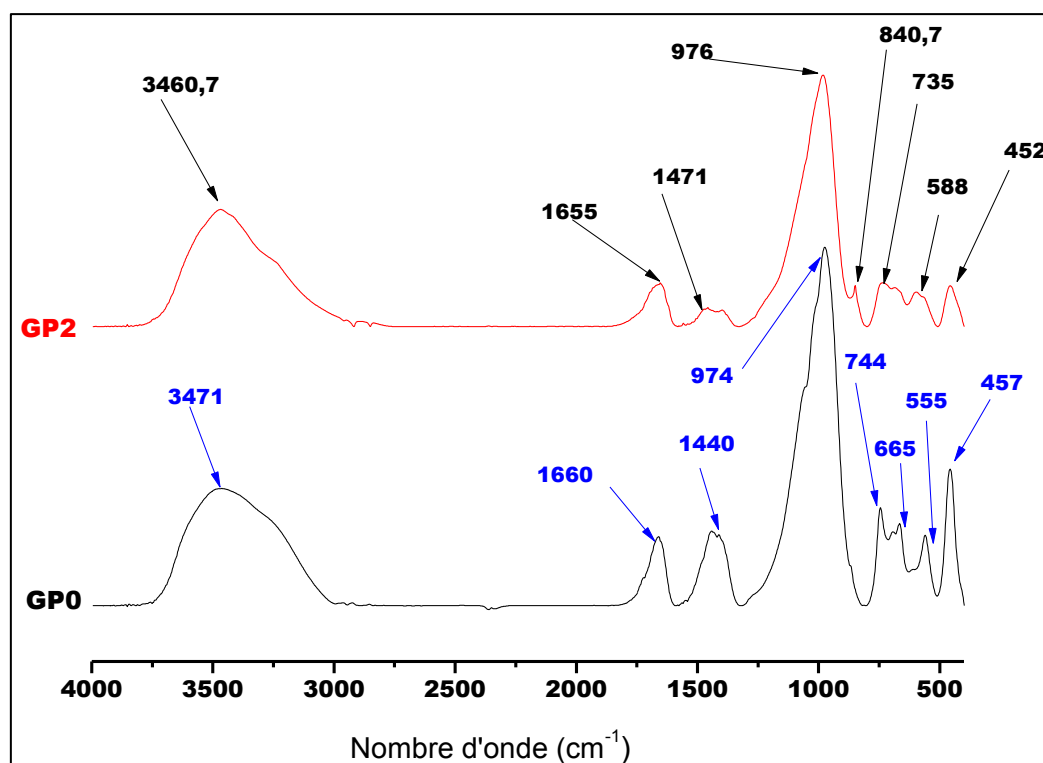


Figure 4. Infrared of geopolymer materials GP₀ and GP₂.

X-ray Diffractogram of the Two Materials GP₀ and GP₂

The X-ray diffraction (XRD) diffractograms for the materials GP₀ and GP₂ are presented in Figure 5. The analysis reveals the formation of zeolites, specifically zeolite A, faujasite, and hydrosodalite. The nature and degree of crystallinity of these zeolites are influenced by the composition of the materials.

- 1) *Zeolite Formation*: In the diffractograms, zeolite A is prominently observed in samples containing rice husk ash (GP₂), while it is present in lower quantities or not at all in the control material (GP₀). This indicates that the addition of rice husk ash contributes to the formation of zeolite A.
- 2) *Hydrosodalite Crystallinity*: The crystallinity of hy-

dro-sodalite decreases as the rice husk ash content increases. This trend suggests that the incorporation of rice husk ash affects the stability and formation of hydro-sodalite within the geopolymer matrix.

- 3) *Amorphous Phases*: The diffractograms also show broad domes or halos extending from 20° to 40° (2θ), indicative of the presence of amorphous phases. These halos signify partial dissolution of the initial amorphous phase in the raw materials and the formation of new amorphous phases within the geopolymer materials [23]. The presence of these amorphous phases can affect the overall properties of the geopolymers.

The degree of crystallinity observed in the XRD analysis is significant as it can influence the mechanical and physical

properties of the geopolymer binders. A higher degree of crystallinity could lead to improved structural integrity and

mechanical performance.

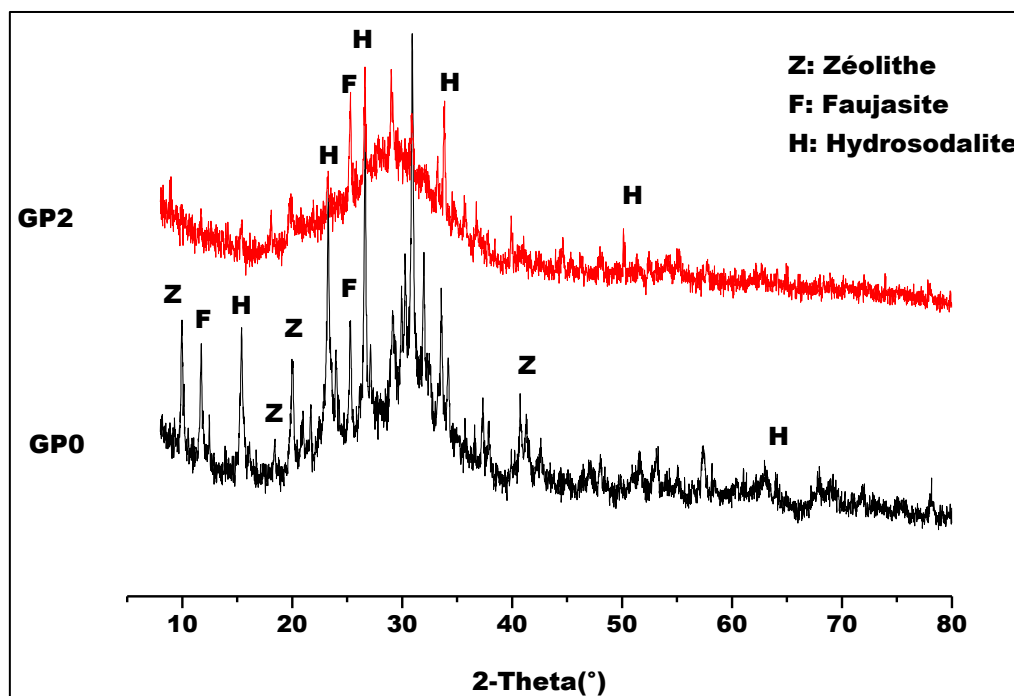


Figure 5. X-ray diffractogram of the two materials GP_0 and GP_2 .

4. Conclusion

This study aimed to enhance the use of local materials in the production of ecological cements by developing and characterizing geopolymer materials based on kaolinite clay and rice husk ash. A comprehensive physico-chemical characterization was first performed on the raw materials, which included kaolinite clay and rice husk ash. The elemental analysis confirmed that kaolinite clay is predominantly composed of aluminosilicates, while rice husk ash is rich in amorphous silica, with a content of 91.6%. Different formulations of geopolymer materials were prepared by varying the proportion of rice husk ash (5%, 10%, and 15%) added to the calcined kaolinite clay. The prepared materials were then characterized using X-ray diffraction (XRD), infrared spectroscopy (FTIR), compressive strength tests, and porosity measurements. The inclusion of rice husk ash improved the mechanical properties of the geopolymer materials. Specifically, adding 10% rice husk ash increased the compressive strength from 9.9 MPa to 15.9 MPa. The experimental results indicated that rice husk ash effectively enhances the reactivity of the calcined clay and contributes to the formation of stronger geopolymer materials. The geopolymer materials, activated with alkaline solutions, showed consolidation at room temperature within 24 hours, demonstrating their potential as viable alternative cements. The study highlights that

kaolinite clay, when processed appropriately, can serve as a valuable source of aluminosilicate for geopolymer production. The addition of rice husk ash, a by-product of rice cultivation, not only improves the mechanical properties of the geopolymers but also contributes to waste recovery and resource optimization. Utilizing these local resources helps reduce reliance on Portland cement, thereby decreasing CO_2 emissions associated with traditional cement production. Furthermore, the recycling of rice husk ash aligns with sustainable practices, contributing to the protection of our ecosystem and benefiting future generations. The transformation of kaolinite clay and rice husk ash into geopolymer materials offers a promising avenue for sustainable construction practices. This approach not only utilizes local resources but also supports ecological and economic goals by reducing environmental impact and enhancing waste recovery.

Abbreviations

XRD	X-Ray Diffraction
FTIR	Fourier Transform Infrared
GP	Geopolymer

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Author Contributions

Anove Komla Mawoulikplim: Conceptualization, Data curation, Formal Analysis, Funding acquisition, Investigation, Methodology, Resources, Software, Supervision, Validation, Visualization, Writing – original draft, Writing – review & editing

Kpetemey Amen: Data curation, Formal Analysis, Software, Validation, Visualization, Writing – review & editing

Tchanate Kolani N'Djoibini: Visualization, Writing – review & editing

Degbe Koffi Agbegnigan: Data curation, Formal Analysis, Methodology, Software, Supervision, Validation, Visualization, Writing – review & editing

Babakoua Dilami Diana: Data curation, Visualization, Writing – review & editing

Tchegueni Sanonka: Conceptualization, Data curation, Formal Analysis, Funding acquisition, Investigation, Methodology, Resources, Software, Supervision, Validation, Visualization, Writing – original draft, Writing – review & editing

Douty Damdjoin: Data curation, Visualization

Fiaty Koffi: Funding acquisition, Investigation, Resources

Drogui Patrick: Funding acquisition, Investigation, Resources

Tchangbedji Gado: Funding acquisition, Investigation, Resources, Supervision, Validation, Visualization

Conflicts of Interest

The authors declare no conflicts of interest.

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