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SUSTAINABLE GEOPOLYMER BINDERS: MECHANICAL AND DURABILITY INSIGHTS INTO FLY ASH-BASED COMPOSITES

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Abstract

Geopolymers can be regarded as a type of sustainable and green building material, with the potential to significantly reduce carbon emissions when compared with conventional alternatives. The present study analyses the performance of geopolymeric binder-based composites that have been obtained by means of alkaline activation of fly ash, which is an abundant industrial waste. The present research is centred upon the evaluation of mechanical properties, such as compressive strength and flexural strength, as main characteristics of their durability. A further objective of the project was to investigate the influences of various factors on the final properties of the material. The experimental findings suggest that the optimised mixture displays enhanced performance with regard to durability and sustainability. This suggests that the material has considerable potential for use in a variety of construction applications. The study provides detailed insight into the potential of this innovative material, which contributes to the development of environmentally friendly construction technologies.

Key words*: alkaline activator, Circular Economy, fly ash, geopolymer binder.*

INTRODUCTION

The increasing environmental concerns surrounding the excessive use of Portland cement (PC) in construction have led researchers to explore alternative, sustainable binders with lower carbon footprints (Provis & Bernal, 2014; Davidovits, 2015). The utilisation of geopolymer binders as a substitute for conventional materials has been identified as a potential solution for mitigating climate change, with their capacity to incorporate industrial by-products such as fly ash, blast furnace slag and metakaolin, leading to a substantial reduction in carbon dioxide (CO_2) emissions (Duxson et al., 2007; Zhang et al., 2009). Of particular interest are fly ash-based geopolymers, which have proven to be a subject of considerable interest due to their widespread availability and favourable mechanical and durability characteristics (Temuujin et al., 2009; Heah et al., 2011). It is estimated that Protland cement production is responsible for approximately 8% of global CO_2 emissions. This is primarily attributable to the limestone calcination process and the significant amount of energy consumption inherent in manufacturing processes (Gartner & Hirao, 2015; Andrew, 2018). In contrast, the synthesis of geopolymeric binders entails the alkaline activation of aluminosilicate-rich precursors, resulting in the formation of a three-dimensional amorphous network, which exhibits enhanced mechanical and chemical properties (Xu & van Deventer, 2000; Chindaprasirt et al., 2007). The use of fly ash, a by-product of coal combustion, further enhances sustainability by diverting waste materials from landfills while reducing the demand for virgin resources (Van Riessen et al., 2013; Abhishek et al., 2020).

Geopolymerization is a chemical process involving the dissolution of aluminosilicate precursors in an alkaline medium, followed by the formation of oligomers that subsequently condense into a rigid network (Phair & van Deventer, 2001; Fernández-Jiménez et al., 2005). The reaction kinetics and final properties of geopolymer composites are influenced by factors such as the precursor composition, activator type and concentration, curing temperature, and water-to-solid ratio

(Provis & van Deventer, 2009; Zhang et al., 2009). The amorphous to semi-crystalline nature of geopolymer binders provides excellent mechanical strength, chemical resistance, and long-term durability (Kumar et al., 2010; Fernández-Jiménez et al., 2019). Fly ash-based geopolymer composites exhibit high compressive strength, reaching up to 80 MPa depending on the synthesis conditions (Sathonsaowaphak et al., 2009; Ryu et al., 2013). Their mechanical performance is attributed to the compact microstructure and strong bonding within the aluminosilicate gel matrix (Lloyd & Rangan, 2010; Sukmak et al., 2013). In addition, geopolymer composites demonstrate enhanced flexural and tensile strength compared to PC-based counterparts due to their dense microstructure and reduced porosity (Lee & van Deventer, 2004; Deb et al., 2014).

Durability is a critical parameter for assessing the long-term performance of construction materials. Fly ash-based geopolymers exhibit superior resistance to sulfate attack, acid corrosion, and freeze-thaw cycles compared to conventional cementitious materials (Bakharev, 2005; Olivia & van Riessen, 2011). The low calcium content and dense matrix of geopolymers hinder the formation of expansive ettringite, thus mitigating degradation due to sulfate exposure (Lloyd et al., 2012; Ismail et al., 2013). Additionally, their reduced permeability and high resistance to chloride penetration make them suitable for marine and aggressive environments (Pan et al., 2012; Pasupathy et al., 2021).

The integration of geopolymer binders into the circular economy framework is crucial for achieving sustainable construction practices. Circular economy principles emphasize the reduction, reuse, and recycling of materials to minimize environmental impact and resource depletion (Ghisellini et al., 2016; Kirchherr et al., 2017). Fly ash-based geopolymers align with this concept by utilizing industrial waste as raw materials, thereby reducing landfill disposal and promoting the valorization of secondary resources (Pacheco-Torgal et al., 2012). Moreover, the long service life and recyclability of geopolymer composites contribute to a closed-loop material flow, further enhancing sustainability (Rahman & Rasul, 2020). By adopting circular economy strategies, the construction industry can significantly decrease its reliance on virgin materials while improving environmental and economic performance. Despite the notable strides made in the field of developing fly ash-based geopolymer binders, challenges persist in the optimisation of their formulation, the assessment of their long-term performance, and the expansion of their production capacities for large-scale implementation (Bernal et al., 2011; Provis, 2018). The present article aims to provide a comprehensive analysis of the mechanical and durability characteristics of fly ash-based geopolymer composites, utilising various types of aggregates. The article places particular emphasis on recent advancements in synthesis techniques, performance evaluation, and factors that influence the mechanical properties of the material. The discussion will contribute to the ongoing efforts to promote geopolymer technology as a sustainable alternative to traditional cement-based materials using Romanian local raw materials.

MATERIALS AND METHODS

The raw materials used in this study were sourced from local Romanian industries to promote sustainability and regional resource utilization. The primary aluminosilicate precursor, fly ash (FA), was obtained from a Romanian coal-fired power plant. The chemical composition of the fly ash was analyzed using X-ray fluorescence (XRF) (Table 1).

Table 1. Fly ash sample chemical composition

Oxides	Percentage (%)
SiO_2	46.94
Al_2O_3	23.83
Fe_2O_3	10.08
CaO	10.72
MgO	2.625
SO_3	0.45
Na_2O	0.62
K_2O	1.645
P_2O_5	0.25
TiO_2	0.92
Cr_2O_3	0.02
Mn_2O_3	0.06
ZnO	0.02
SrO	0.03
L.O.I.	2.105

The cumulative distribution of fly ash particles from a Romanian thermal power plant was determined using a HELOS RODOS/L, R5 instrument (Sympatec, Germany) in order to analyse the particle size of the material utilised in the development of alkali-activated geopolymers. The physical characteristics of the fly ash are presented in Figure 1 (Particle size distribution) and Figure 2 (Distribution density of the particles), offering a better visualisation representation.

In order to obtain the alkaline activator (AA) solution, a commercially purchased sodium silicate (Na_2SiO_3) solution was combined with a sodium hydroxide (NaOH) solution (purity ≥ 98%) of 6M, 8M, and 10M molar concentrations in various mass ratios at room temperature. Following the completion of the preparatory stage, the alkaline activator solution was stored under laboratory conditions (23°C) in a closed container for a period of 24 hours in order to allow maturation. In this study, two types of fine aggregates were utilised in the preparation of geopolymer samples: polygranular sand and recycled glass aggregates with a diameter of 0/4 mm (Figure 3). Each aggregate was used independently to evaluate its influence on the material's performance. The selected aggregates were thoroughly mixed with the binder to ensure uniform distribution and proper interaction within the geopolymer matrix. The use of recycled glass aimed to enhance sustainability while maintaining the structural integrity of the material. The activation process and curing conditions were optimized to promote effective geopolymerization and achieve a stable hardened structure.

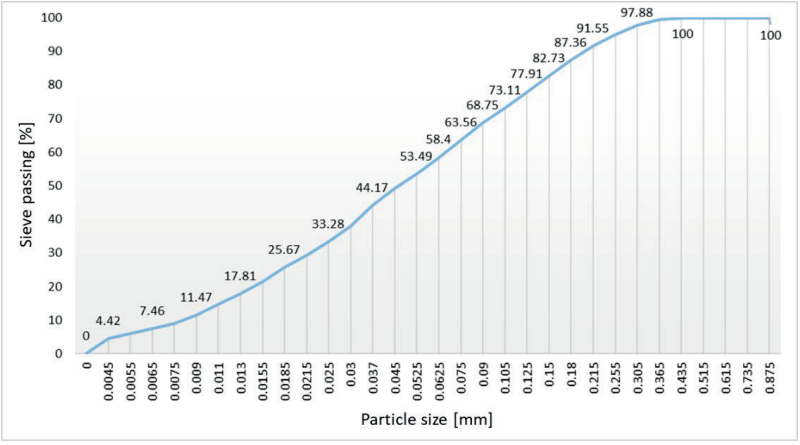

Figure 1. Fly ash particle size distribution

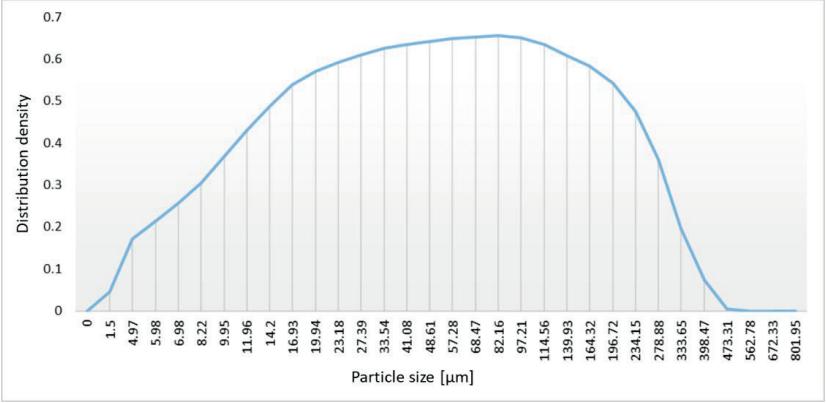

Figure 2. Fly ash distribution density of the particles

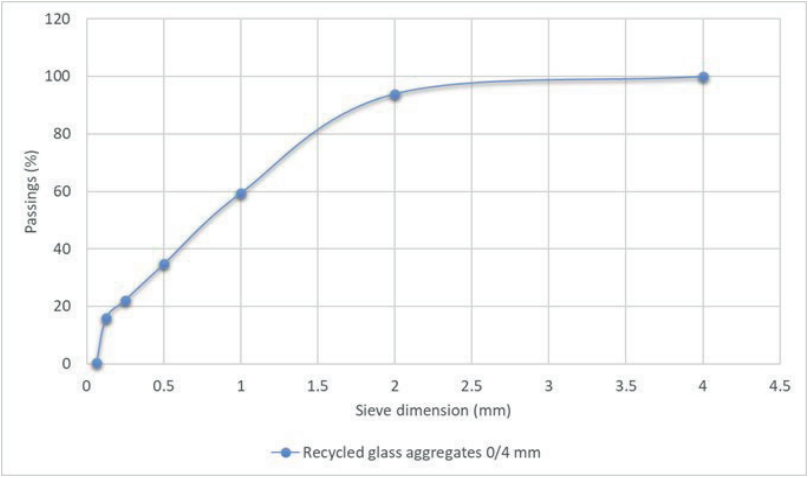

Figure 3. Particle size analysis of the recycled glass aggregates (granular class 0/4 mm)

The geopolymer samples were formulated using two different binder-to-aggregate ratios of 1:1 and 1.25:1 to assess their influence on the material's properties. In the alkaline activation system, sodium silicate (Na_2SiO_3) was dissolved in a solution of sodium hydroxide (NaOH), at a ratio of 2:1 and 1:2, respectively.

To evaluate the influence of alkalinity on the geopolymerisation process, the NaOH solution was prepared in a range of molar concentrations: 6M, 8M, and 10M. The fly ash-to-alkaline activator ratio was fixed at 0.9 to ensure a consistent binder composition. The prepared mixtures were homogenized to achieve a uniform consistency before being cast into molds and subjected to curing under controlled conditions (70°C for 24 hours).

The specimens were demolded after 24 hours and cured at elevated temperatures to study the impact of thermal curing on strength development. Based on preliminay research, curing conditions were optimized to achieve maximum compressive strength and durability resistance (Lăzărescu et al., 2020).

The mix design ratios of the geopolymer samples are presented in Table 2 (samples produced using polygranular standardized sand - PS and samples produced using recycled glass agreggates - GA). For each mixture, a color coding was considered. Each result was obtained from the combination of a minimum of three independently tested samples.

All tests were conducted at the age of 7 days, primarily because geopolymer samples typically gain most of their strength and resistance during this period, particularly due to the heat treatment applied. Research indicates that heat curing accelerates the polymerization process, allowing geopolymers to reach their maximum strength within the first week (Lăzărescu et al., 2024). This time frame was critical for evaluating the material's early-stage properties, as the heat treatment enhances the formation of the geopolymer matrix and strengthens the material. Compressive strength was selected as the main parameter for evaluation due to its direct correlation with the material's structural integrity.

RESULTS AND DISCUSSIONS

The results are presented in graphical form, with each graph, with specific colour code representing different mixtures. The colour coding facilitated the identification of variations in the mixture. Throughout the analysis, the molar concentration of the NaOH solution remained constant, ensuring that the observed differences in the results are solely attributable to the variations in the other components of the mixture (Figures 4-6).

Table 2. Mix design ratio of the alkali-activated geopolymer samples

Type of aggregate	Code	Code colour	NaOH solution concentration	Binder:Aggregate ratio	Na_2SiO_3 / NaOH ratio
Polygranular standardized sand (PS)	P1(PS)		10M	1:1	1:2
	P2(PS)				2:1
	P3(PS)			1.25:1	1:2
	P4(PS)				2:1
	P5(PS)		8M	1:1	1:2
	P6(PS)				2:1
	P7(PS)			1.25:1	1:2
	P8(PS)				2:1
	P9(PS)		6M	1:1	1:2
	P10(PS)				2:1
	P11(PS)			1.25:1	1:2
	P12(PS)				2:1
Recycled glass Granular class 0/4 mm (RG)	P1(RG)		10M	1:1	1:2
	P2(RG)				2:1
	P3(RG)			1.25:1	1:2
	P4(RG)				2:1
	P5(RG)		8M	1:1	1:2
	P6(RG)				2:1
	P7(RG)			1.25:1	1:2
	P8(RG)				2:1
	P9(RG)		6M	1:1	1:2
	P10(RG)				2:1
	P11(RG)			1.25:1	1:2
	P12(RG)				2:1

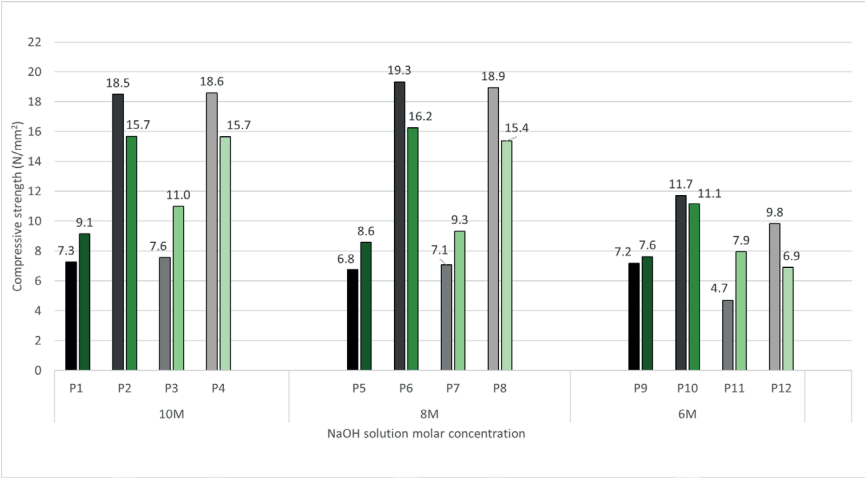

Figure 4. Graphical representation of the compressive strength variation in alkali-activated geopolymer samples (10M, 8M, and 6M)

The performance of the geopolymer samples, particularly in terms of compressive strength, was found to be significantly affected by the incorporation of polygranular sand, as indicated by the results of mechanical tests (Figure 4). Samples with Na_2SiO_3/NaOH ratio of 2:1 (P2(PS), P4(PS) -10M, P6(PS), P8(PS) – 8M and P10(PS), P12(PS) – 6M) exhibited higher values of compressive strength than those with a ratio of 1: 2, suggesting that a higher content of sodium silicate contributes to a better consolidated and denser geopolymer matrix. Regarding the influence of the molar concentration of the NaOH solution, a significant increase in compressive strength between 6M and 8M can be observed, but above this threshold, samples produced with 10M NaOH solution do not show an improvement in compressive strength, which indicates reaching a saturation level in the alkaline activation process.

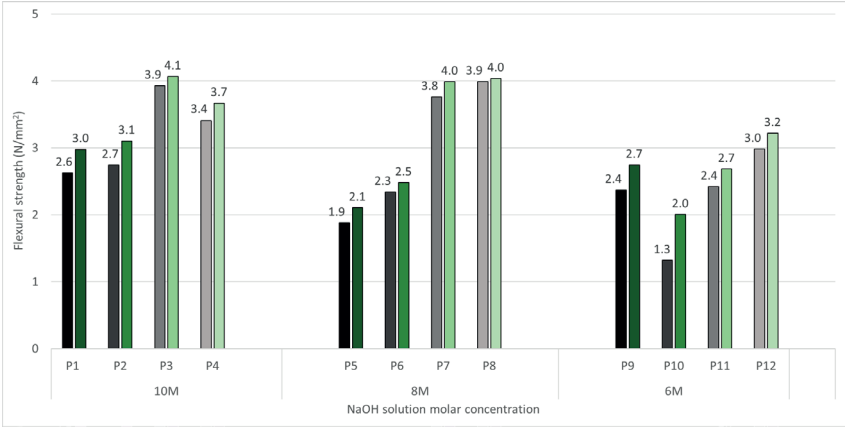

Figure 5. Graphical representation of the flexural strength variation in the alkali-activated geopolymer samples (10M, 8M, and 6M)

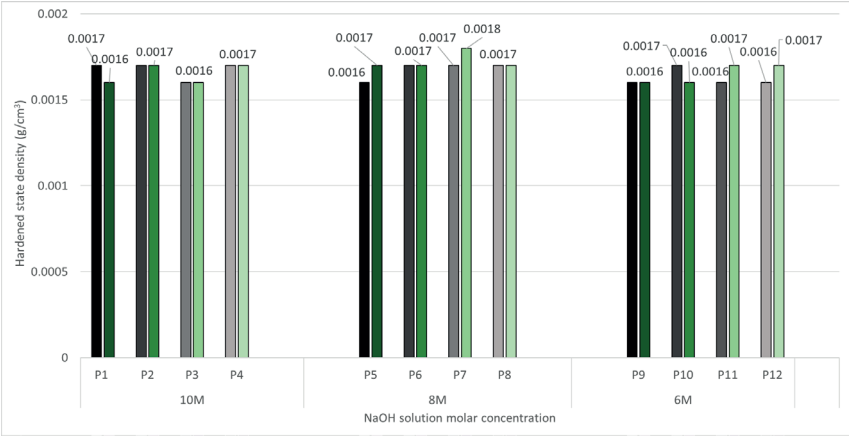

Figure 6. Graphical representation of the hardened state density variation in the alkali-activated geopolymer samples (10M, 8M, and 6M)

However, the flexural strength test results did not follow the same trend (Figure 5). Although the samples with an activator ratio of 2:1 had better compressive strength, the values obtained for flexural strength did not show a direct correlation with this ratio. This difference suggests that although a denser geopolymer matrix favors compressive behavior, the mechanisms of cracking and crack propagation under tensile stress are influenced by additional factors such as pore distribution, microstructure of the gel formed, and interfacial adhesion between component phases.

The mechanical performance of the samples exhibited variation depending on the type of aggregate utilised. Recycled glass aggregates demonstrated distinct behaviour in both compressive and flexural strength when compared to polygranular sand. For the 1:2 activator ratio, samples incorporating recycled glass (P1(RG), P3(RG) – 10M, P5(RG), P7(RG) – 8M, and P9(RG), P11(RG) – 6M) demonstrated higher compressive strength values. The enhancement in strength is attributed to the improved compaction of the geopolymer matrix, a consequence of the morphology of the glass particles, and the potential contribution of an additional pozzolanic reaction involving the glass particles. However, the general trend was maintained, and the samples with 2:1 activator ratio continued to show the best compressive performance regardless of the type of aggregate used.

In terms of the influence of the molar concentration of NaOH, the same significant increase was observed between 6M and 8M, with no significant improvements when using 10M NaOH solution.

However, the tensile behavior was different from that observed in compression. In contrast to the samples with polygranular sand, those with recycled glass aggregates exhibited higher values of tensile strength, for all the mixtures (P1-P12(RG)).

This result suggests that glass aggregates may contribute to improve the ability of the geopolymer to respond to tensile stresses, possibly through a more favorable stress distribution in the matrix and a more efficient interface between the binder and the aggregates. Despite the smooth surface of the glass particles, which could reduce interfacial adhesion, it is possible that this effect could be compensated by a better interaction at the microstructural level, limiting crack initiation and propagation under tensile stress.

Moreover, optimising the molar concentration of NaOH within the 6M to 8M range, where mechanical performance improvements become negligible beyond this threshold, may have significant sustainability implications. This optimisation enables a reduction in the quantity of alkaline substances required for geopolymer activation, thereby minimising both the consumption of chemical resources and the environmental impact associated with their production and handling. Within the framework of the circular economy, geopolymers emerge as a viable and environmentally friendly alternative to conventional building materials, as their mechanical properties benefit from the incorporation of recycled aggregates. This approach not only enhances material performance but also contributes to effective glass waste management.

A key aspect highlighted by this study is that, although the mechanical performance of the samples differs depending on the type of aggregates used and the activator ratios, the density in the hardened state remains relatively constant for all samples (Figure 6). This result is extremely important, as it suggests that the improvement in mechanical properties observed in some samples is not due to a simple variation in density, but to a complex interaction between the geopolymer matrix and the aggregates. Thus, optimization of material performance is achieved by changing the chemical composition and type of aggregates without significantly affecting the final density. This observation further supports the potential of using recycled aggregates in the development of sustainable geopolymers. Should the density remain consistent, irrespective of the type of aggregate utilised, it can be deduced that recycled glass has the potential to serve as a substitute for conventional aggregates without altering the material's fundamental characteristics. Additionally, the observation that recycled glass samples exhibited higher tensile strength values suggests that these aggregates contribute to a more uniform stress distribution and reduce premature cracking. These phenomena, if substantiated, may enhance the durability of constructed structures.

Furthermore, the consistency in sample density suggests that incorporating additional functionalization, such as the integration of TiO_2 nanoparticles, is unlikely to alter the material's density but rather enhance its functional properties. Thus, the utilisation of geopolymer materials as a medium for the creation of intelligent and environmentally sustainable solutions has been demonstrated to result in a dual effect, with a reduction in ecological impact through the reutilisation of discarded materials and an improvement in the functionality of these solutions, including self-cleaning properties and antibacterial protection. It is clear that the constant density of these materials offers a significant technological advantage. Indeed, these materials can be manufactured in a manner that does not demand substantial adjustments to existing processes. Consequently, the shift towards sustainable and intelligent geopolymers becomes a viable and readily implementable solution. This transformation has the potential to redefine the building materials industry by promoting structures that are more sustainable, efficient and environmentally friendly.

CONCLUSIONS

This study emphasises the considerable influence of aggregate type and activator ratios on the mechanical performance and durability of geopolymers, thereby validating their potential as a sustainable substitute for conventional building materials. Previous studies have shown that geopolymer concrete has higher initial costs due to the price of alkali activators and specialized production processes. However, due to its superior durability, low maintenance costs and significantly lower environmental impact, it is becoming a sustainable and cost-effective alternative to traditional concrete in the long term. A specific cost assessment can only be made on a case-by-case basis, considering both initial costs and long-term benefits. Furthermore, studies have shown that, when compared to ordinary Portland cement with similar mechanical properties, geopolymer concrete reduces CO_2 equivalent (CO_2eq) emissions by up to 50-60%, due to the use of industrial waste and the elimination of the Portland cement production process (Lăzărescu et al., 2024). Thus, in the long term, geopolymer concrete not only offers the benefits of durability and low maintenance but also contributes significantly to the goals of sustainability and reduced environmental impact.

This study has shown that, the use of recycled glass aggregates was found to be beneficial, improving the tensile strength and, in the case of an activator ratio of 1:2, even the compressive strength compared to polygranular sand. These results highlight that the right choice of aggregates can lead to optimized performance of geopolymeric materials, thus facilitating the transition towards more sustainable and environmentally friendly solutions. Furthermore, the recovery of waste glass contributes directly to reducing the impact on natural resources, promoting a circular economy model.

The study also demonstrated that the ratio of activators to NaOH molar concentration plays a crucial role in the formation of a stable and efficient geopolymer network. The significance of sodium silicate in establishing a compact structure is substantiated by the attainment of optimal mechanical strengths at an activator ratio of 2:1. An analysis of molar concentration of NaOH revealed a significant increase between 6M and 8M, with no such improvement observed at 10M, thus indicating the attaining of a saturation point in the activation process. This finding carries significant implications for sustainability as it facilitates optimisation of chemical consumption, leading to a reduction in costs and a diminished environmental impact within the manufacturing process.

It is anticipated that future research on geopolymer materials will concentrate on mechanical properties and sustainability. In addition, however, there is a growing recognition of the importance of exploring the development of smart, eco-innovative materials that can provide enhanced benefits to meet evolving efficiency and performance demands. The integration of titanium dioxide (TiO_2) nanoparticles has been identified as a potentially significant solution in this regard. This integration has the capacity to impart self-cleaning, antibacterial, and antimicrobial properties to geopolymers, thereby enhancing their applicability in environments subject to contamination or within urban infrastructure. The incorporation of TiO_2 into geopolymer materials has been shown to yield a photocatalytic property, enabling the degradation of organic pollutants when exposed to light. This property has the potential to mitigate the environmental impact of buildings, positioning geopolymers as a promising alternative to conventional materials. The functionalization of geopolymers with TiO_2 could lead to their advancement as smart materials that actively contribute to environmental protection and safety.

In this context, geopolymers are evolving beyond their traditional role as a sustainable alternative to conventional cement. They are increasingly recognised as a pivotal component in the development of innovative materials characterised by advanced, adaptable and environmentally friendly properties. The integration of the principles of the circular economy, utilising recycled aggregates, with cutting-edge nanomaterials-based technologies is pivotal in shaping a genuine transition towards smart, eco-innovative materials. These

materials have the potential to redefine standards in the domains of construction and infrastructure. This will not only reduce environmental impact, but also increase the durability, functionality, and safety of built structures, thus contributing to a more sustainable and smarter future.

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