

RESEARCH ARTICLE

REVISED Use of pumice stone and silica fume as precursor

material for the design of a geopolymer

[version 2; peer review: 2 approved, 2 approved with reservations]

Alexis Iván Andrade Valle^{1,2}, Tito Oswaldo Castillo Campoverde², Cristian Andrés Marcillo Zapata², María Gabriela Zúñiga Rodríguez¹⁰², Andrea Natalí Zárate Villacrés¹⁰², Marcelo David Guerra Valladares¹⁰², Mayte Lisbeth Mieles Mariño², Jefferson Javier Castillo Cevallos²

¹Engineering, Universitat Politecnica de Valencia, Valencia, Valencian Community, 46022, Spain ²Engineering, Universidad Nacional de Chimborazo, Riobamba, Chimborazo Province, 060150, Ecuador

V2 First published: 05 Jun 2024, 13:580 https://doi.org/10.12688/f1000research.147701.1 Latest published: 12 Aug 2024, 13:580 https://doi.org/10.12688/f1000research.147701.2

Abstract

Background

Geopolymers are alternative materials to cement because they require less energy in their production process; hence, they contribute to the reduction in CO2 emissions. This study aims to evaluate the possibility of using industrial residues such as silica fume (SF) to improve the physical and mechanical properties of a pumice stone (PS)-based geopolymer.

Methods

Through an experimental methodology, the process starts with the extraction, grinding, and sieving of the raw material to carry out the physical and chemical characterization of the resulting material, followed by the dosage of the geopolymer mixture considering the factors that influence the resistance mechanical strength. Finally, the physical and mechanical properties of the geopolymer were characterization of the pumice stone, design of the geopolymer through laboratory tests, application according to the dosage of the concrete, and analysis of the data through a multi-criteria analysis.

Results

It was determined that the optimal percentage of SF replacement is

Open Peer Review

Approval S	tatus 👌	/ / ?	?	
	1	2	3	4
version 2		~	?	?
(revision) 12 Aug 2024		view	view	view
version 1 05 Jun 2024	view	? view		

- 1. **Abdullah Zeyad**, Jazan University,, Jazan, Saudi Arabia
- 2. Dillshad K. H. Amen Bzeni (D, Salahadin University, Erbil,, Iraq
- 3. **Muhammad Tariq Bashir** (D), CECOS University of IT and Emerging Sciences, INTI International University, Peshawar, Pakistan
- 4. Zrar Safari ២, Soran University, Soran, Iraq

Any reports and responses or comments on the article can be found at the end of the article.

10%, which to improves the properties of the geopolymer allowing to reach a maximum resistance to compression and flexion of 14.10 MPa and 4.78 MPa respectively, showing that there is a direct relationship between the percentage of SF and the resistance.

Conclusions

Geopolymer preparation involves the use of PS powder with a composition rich in silicon and aluminum. The factors influencing strength include the ratio of sodium silicate to sodium hydroxide, water content, temperature, curing time, molarity of sodium hydroxide, and binder ratio. The results showed an increase in the compression and flexural strength with 10% SF replacement. The geopolymer's maximum compressive strength indicates its nonstructural use, but it can be improved by reducing the PS powder size.

Keywords

Alkali activation, molar concentration, geopolymer, silica fume, pumice powder

Corresponding author: Alexis Iván Andrade Valle (alexis.andrade@unach.edu.ec)

Author roles: Andrade Valle AI: Conceptualization, Formal Analysis; Castillo Campoverde TO: Investigation; Marcillo Zapata CA: Data Curation, Investigation; Zúñiga Rodríguez MG: Data Curation, Methodology, Validation; Zárate Villacrés AN: Validation, Writing – Original Draft Preparation, Writing – Review & Editing; Guerra Valladares MD: Resources, Validation; Mieles Mariño ML: Resources; Castillo Cevallos JJ: Resources

Competing interests: No competing interests were disclosed.

Grant information: The author(s) declared that no grants were involved in supporting this work.

Copyright: © 2024 Andrade Valle AI *et al*. This is an open access article distributed under the terms of the Creative Commons Attribution License, which permits unrestricted use, distribution, and reproduction in any medium, provided the original work is properly cited.

How to cite this article: Andrade Valle AI, Castillo Campoverde TO, Marcillo Zapata CA *et al.* Use of pumice stone and silica fume as precursor material for the design of a geopolymer [version 2; peer review: 2 approved, 2 approved with reservations] F1000Research 2024, **13**:580 https://doi.org/10.12688/f1000research.147701.2

First published: 05 Jun 2024, 13:580 https://doi.org/10.12688/f1000research.147701.1

REVISED Amendments from Version 1

- Introduction: More information was included in the introduction regarding previous relevant research and pumice stone characteristics.
- **Methods:** In the methods section, the numerical value for water to binder ratio has been clarified and explained, this was done in Table 4 and in the written explanations.
- **Results:** The results section includes two new figures (Figure 12 and 13), showing compression and flexural failure, and the text includes more discussion regarding the compressive strength results obtained.
- References: The reference list has been updates considering the new information included in the amended manuscript.

Any further responses from the reviewers can be found at the end of the article

Introduction

Concrete is the most commonly used construction material in civil engineering applications owing to its physicalmechanical properties, durability, and cost. These advantages compared to other materials imply an increase in the consumption of cement, whose industry is responsible for 8% of CO_2 emissions worldwide according to Ref. 1. This is due to the significant amount of energy required to produce clinker, which is its main component. The Environmental Investigation Agency (EIA)² states that strategies must be adopted to optimize the use of cement, such as energy efficiency techniques, changing fuels to those with less carbon, promoting technological innovations, and promoting the efficiency of materials, the latter to reduce the clinker-cement production ratio and its total demand. The EIA also emphasizes that the incorporation of alternative binder materials could be key to reducing emissions from cement production.

Although alternatives for reducing CO_2 emissions in cement production are growing, their large-scale implementation faces many challenges. These include research costs, development and adaptation of new technologies, and the need to modify existing regulations and standards. In addition, it is essential to involve all actors in the value chain, from cement producers to builders and end users, to ensure the transition to more sustainable construction. In this sense, education and awareness are key elements to promote adopting more sustainable practices in the construction sector.³

Therefore, a new alternative that reduces the use of cement is geopolymers, which are obtained by alkaline activation of materials rich in alumina and silica, such as natural pozzolans, fly ash, and other industrial wastes.⁴

References 5, 6, and 7 established that materials of volcanic origin favor alkaline activation due to the presence of aluminum silicates. The study seeks to establish whether the material of volcanic origin presents the necessary conditions to act as a precursor in the design of geopolymers.

Regarding the use of natural pozzolans such as pumice stone (PS) in the production of geopolymers,^{8,9} it can be concluded that PS in alkaline-activated geopolymers provides satisfactory compressive strength at 28 days of 12 MPa. Furthermore, Ref. 8 determined that it is possible to use geopolymers with structural and non-structural applications that can reach a compressive strength of up to 24 MPa at 28 days.

Reference 10 explored the deposits throughout Ecuador, the largest found in the provinces of Tungurahua, Cotopaxi, and Ibarra. A quantitative and qualitative microscopic analysis determined that the material presents amorphous in its constitution, with a high presence of aluminum silicates, which is the base requirement of the research.

Regarding the use of industrial waste, research has focused on the use of fly ash; however, other industrial wastes, such as silica fume (SF), have great potential as precursor materials. From SF it has been shown that its incorporation into geopolymers at 2% by weight means an increase in compressive strength, while at 4% it would decrease it according to Refs. 11, 12, however, Ref. 13 refers to SF incorporations of up to 5% to obtain satisfactory resistance results. On the other hand,¹⁴ concluded that up to 7% SF addition by weight increased the mechanical properties of the geopolymer, although an increase in the % reduced the resistance.

Therefore, the aim of this study was to characterize the physical and chemical properties of PS to produce a geopolymer with SF alkaline-activated with a solution of sodium silicate and sodium hydroxide at different concentrations.

Methods

In the present investigation a combination of Pumice Stone (obtained from the Cotopaxi deposit, specifically from the "PROFUTURO" quarry), and Silica Fume (obtained from the company "Ferrekret" in Guayaquil) was used as precursor material in replacement percentages of 2.5%, 5% and 7.5%. Moreover, the precursor material used contained approximately 86% of particles of a size smaller than 0.3 mm and 25% smaller than 0.075 mm.

A combination of two alkaline solutions was used as activators: sodium silicate (Na_2SiO_3) and sodium hydroxide (NaOH) in a ratio of 2.5. The concentration of these compounds in the aqueous solution was 8 Mol/l and 12 Mol/l. The reactive used to prepare the activating solutions were Na_2SiO_3 solution of 97% purity $(11\%Na_2O, 29.5\%SiO_2)$; NaOH in the form of flakes of 97% purity (97%NaOH, 0.005%Ca, 0.001%Fe). These were supplied by the RELUBQUIM company.

The mixtures were prepared by mixing the precursor material (combination of Pumice Stone with Silica Fume) and the different activating solutions (varying their molar concentration). The alkaline binder solution ratio was maintained constant and equal to 0.75 (where the alkaline solution includes distilled water and the chemical compounds: Sodium hydroxide NaOH and Sodium silicate Na₂SiO₃). The specimens obtained were transported to an oven at 60°C to begin to cure, here they were kept for 120 hours. To proceed to a second curing stage, the specimens were covered with a plastic bag and kept until the testing day at room temperature and humidity (approximately 18°C and relative humidity of 50%).

Once the curing time had elapsed, the 40×160 mm prismatic specimens and $50 \times 50 \times 50$ mm cubic specimens were tested to determine their flexural and compressive strength, respectively, for which the mortar standards^{15,16} were used, in addition, for the physical characterization of the mixtures (density and absorption), the Ref. 17 standard was used. The specimens after 7 days of curing were subjected to mechanical tests, while after 28 days of curing, the samples were subjected to both physical and mechanical tests (Figure 1).

Pumice Stone (PS)

The PS was obtained from the "Profuturo" mining area with code "201004," located in the city of Latacunga, Cotopaxi province, with UTM coordinates 764747.34 m E, 9893327. 94 m S, and 2760 m elevation. See Figure 2.

Silica fume

This industrial waste (Figure 3) was purchased from the company "Ferrekret" located in the city of Guayaquil, in a presentation of 5 kg, and was stored in the warehouses of the UNACH Civil Engineering Laboratories in a cool and ventilated environment. Table 1 shows the chemical composition of silica fumes.

Sodium silicate

This chemical substance was purchased from the company "Relubquim," located in the city of Quito. It was stored in the warehouses of UNACH Civil Engineering Laboratories, always guaranteeing a cool, ventilated, and dark environment to avoid evaporation. The technical specifications of Sodium Silicate are shown in Table 2.

Sodium hydroxide

This chemical substance was purchased from the company "Largo Rivera Herwin Roger-Novachem del Ecuador" in Quito. The sodium hydroxide used was solid in the form of flakes/lentils and was stored in the warehouse of the UNACH Environmental Engineering Laboratories, always guaranteeing a cool, ventilated, and dark environment to avoid reaction. See Figure 4. The technical specifications of Sodium Silicate are shown in Table 3.

Physical and chemical characterization of pumice stone powder

To physically and chemically characterize the pumice powder, the raw material was extracted with the help of a hammer tree mill, rotary drum, and a 0.6 mm diameter sieve for its mesh.

Physical characterization of pumice powder

One of the determined physical properties of the PS powder is the oven-dry density and absorption under¹⁸ procedures. This test began with the sample drying at a temperature of $110^{\circ}C \pm 5^{\circ}C$ until obtaining a mass constant, which was later covered with water and left to rest for 24 ± 4 h. Water was decanted, and the gravimetric method (pycnometer) and the corresponding formulas given by Ref. 19 were applied to determine the density and absorption of the material.

The granulometry and fineness modulus were also determined following the Ref. 19, the material began to be dried at a temperature of $110^{\circ}C \pm 5^{\circ}C$ until a constant mass. The necessary sieves (Sieve No. 4) were selected, and mechanical



Figure 1. Methodology diagram materials.





Figure 3. Silica fume (SF).

Table 1. SF chemical composition (Chemical composition of "Ferrekret").

Component	Unit	Composition
SiO ₂ _	%w/w	91.10
C free	%w/w	-
Sic	%w/w	-
MgO	%w/w	0.27
Fe ₂ O ₃ _	%w/w	0.13
Al ₂ O ₃ _	%w/w	6.82
CaO	%w/w	0.47
Na ₂ O	%w/w	0.96
K ₂ O	%w/w	0.34

Table 2. Technical specifications of Na₂ SiO₃.

Parameter	Units	Specifications
Alkalinity	%Na ₂ O	9.2% - 11%
Silicon dioxide	%SiO ₂	27.5% - 29.5%
Relationship	SiO ₂ /Na ₂ O	2.9 – 3.2
Concentration	°Be	41-43
Total solids	%m/m	-



Result name	Unit	Specifications
Appearance		Report
NaOH	%	≥97.0
Calcium	%	≤0.005
Chloride	%	≤0.005
Heavy metals (as Ag)	%	≤0.002
Iron (Fe)	%	≤0.001
Magnesium	%	≤0.002
Mercury (Hg)	ppm	≤0.1
Nickel (Ni)	%	≤0.001
Compound nitrogen	%	≤0.001
Phosphate (PO4)	%	≤0.001
Potassium (K)	%	≤0.02
Sodium carbonate	%	≤1

 Table 3. Technical specifications of NaOH (Certificate of analysis of "Largo Rivera Herwin Roger – No vachem

 del Ecuador").

sieving was conducted with 500 g of material to determine the percentage retained and the percentage that passed, which allowed the construction of a granulometric curve.

Chemical characterization of pumice powder

The mineral composition of the PS powder was obtained using X-ray diffraction (XRD), which is an analysis method that was conducted under a.²⁰ The pumice composition was obtained by performing X-ray diffractometry (XRD) and fineness modulus determination by dry sieving.

The SF replacement percentages for the preparation of the test tubes were defined by a bibliographic review in which it was verified that the percentages are by weight depending on the binder and vary from 2% to 10%. For this area in the present study, it was proposed that the SF replacement rates be 2.5%, 5%, 7.5%, and 10%.

Once the resistance factors and SF percentages were defined, the total volume of geopolymer used for each dosage was established. In this case, a total of six 50 mm cubes and six $40 \times 40 \times 160$ mm beams were necessary, giving a volume of 750 (cm³) and 1536 (cm³), respectively, and an average density of 2200 (kg/m³) obtained²¹ was taken as a reference and a waste of 5% was considered.

Table 5 shows the dosages obtained for a variation in SF from 0% (controls) to 10% at concentrations of 8 and 12 M.

Geopolymer mix design

To calculate the amount of materials involved in the manufacture of the geopolymer, it was necessary to identify the factors that influence the resistance of this material according to a preliminary documentary review. Table 4 lists the values adopted for each.

Factors	Adopted Value	References
Alkaline Concentration	8 M and 12 M	4
Alkaline Solution/Binder Ratio	0.75	4
Sodium Silicate/Sodium Hydroxide Ratio	2.5	13
Curing time	120 hours	15
Curing temperature	60°	16

Table 4. Definition of resistance factors.

SF (%)	Concentration of NaOH	Alkaline solution (Na₂SiO₃+NaOH)	Binder (kg)	PS powder (kg)	SF (kg)	Na₂SiO₃ (kg)	NaOH (kg)	Water weight (kg)
0	8 M	1,369	3,912	3,912	-	0.978	0.391	1,565
0	12 M	1,369	3,912	3,912	-	0.978	0.391	1,565
2.5	8 M	1,369	3,912	3,814	0.098	0.978	0.391	1,565
2.5	12 M	1,369	3,912	3,814	0.098	0.978	0.391	1,565
5.0	8 M	1,369	3,912	3,716	0.196	0.978	0.391	1,565
5.0	12 M	1,369	3,912	3,716	0.196	0.978	0.391	1,565
7.5 -	8 M	1,369	3,912	3,618	0.293	0.978	0.391	1,565
7.5	12 M	1,369	3,912	3,618	0.293	0.978	0.391	1,565
10	8 M	1,369	3,912	3,520	0.391	0.978	0.391	1,565
10	12 M	1,369	3,912	3,520	0.391	0.978	0.391	1,565

Table 5. Geopolymer dosing.

Mixing procedure

The process began with the preparation of an alkaline solution that served as an activator for the mixture, for which the sodium hydroxide solutions were first prepared at the already defined concentrations of 8 and 12 M, and then mixed with sodium silicate.

Sodium silicate and sodium hydroxide solutions were mixed at a ratio of 2.5 for 2 min to form the activating alkaline solution, and to this, the amount of water resulting from the water/binder ratio of 0.40 was added and mixed for 2 min. On the other hand, the PS powder was integrated with the SF for 3 minutes to pour the alkaline solution and mix for another 5 minutes until a homogeneous paste was obtained.

Once the mixture was ready, 50 mm cubes and $\times 40 \times 40 \times 160$ mm beams were manufactured according to the process established in Refs. 18, 19. For this, wooden molds were made, which were cleaned and greased, and the mixture was poured into them, compacted, and eliminated the empty spaces, leaving them to rest for 24 h at room temperature to later proceed with the demolding, labeling, and curing; for the latter. The specimens were placed in a Humboldt Mfg. Co for 120 h at a constant temperature of 60°C.

Once this process was completed, all the test tubes were covered with a plastic film and kept in an environment with a temperature of 18°C and a relative humidity of 50% until the testing times of 7 and 28 days were completed.

Physical and mechanical characterization of the geopolymer

Physical characteristics of the geopolymer

The density of the hardened geopolymer was determined to be Ref. 22 using 50 mm cubic test tubes. It was obtained by taking the dry mass in an oven for 24 h at a temperature of $110^{\circ}C \pm 5^{\circ}C$. Subsequently, the mass of the saturated test tube was measured by immersion for 48 h. Finally, the apparent submerged mass was determined by placing the test tube in the immersion basket. Moreover, the absorption was evaluated using density data.

Mechanical characteristics of the geopolymer

The compressive strength of the obtained geopolymer was evaluated under the guidelines of Ref. 16, using 50 mm cubes that were compacted in two layers, for which three test tubes were necessary for each test age and dosage. The test was carried out in a Humboldt brand universal machine by applying a load on the two faces of the cube at a speed increasing in the range of 900 to 1800 N/s. In addition, the flexural resistance was determined according to Refs. 15, 23, using $40 \times 40 \times 160$ mm beams that were supported on cylinders with their longitudinal axis perpendicular to these supports and leaving a 100 mm clearance to apply in the center of this, a load that increased at a speed of 50 ± 10 N/s. The test was carried out with a universal control machine, and both properties were determined at ages of 7 and 28 days.



Figure 5. The granulometric curve of pumice stone powder.

Results and discussion

Physical and chemical characteristics of pumice powder

From the results obtained in the physical characterization of the base material, the density of the PS powder was 2084.23 (kg/m³) which is lower than that of other investigations, such as that of Ref. 24, where a PS powder with a density of 2500 (kg/m³) is used. In addition, when comparing the granulometric curves (Figure 5) of the materials used in the present study, only 24.78% of the material passed through the sieve. N°200 (0.075 mm), whereas in the research by Ref. 25, 57% of the material used passes through the No. 200 sieve, which indicates that the material used is thicker, which can be attributed to the grinding and sieving process of the material. It should be noted that the fineness of the base material affects the resistance of the analyzed geopolymer, as mentioned by Ref. 26.

Applying the calculation process of number 5.4 of Ref. 19, for the determination of the modulus of fineness (MF) of the PS powder, it was determined that the modulus of fineness of the PS powder is 0.69; hence, it is considered a fine material.

However, in the chemical characterization of the PS powder (Figure 6 and Table 6), silicon (Si) and aluminum (Al) are common denominators in the composition of the powder. PS dust from the Cotopaxi quarry is suggested by the presence of aluminosilicates, which, according to Refs. 8, 9, 24 have the potential to be pioneers in the elaboration of geopolymers.

Table 6 summarizes the name of the compound, its chemical formula, and the percentage of PS powder.

Physical and mechanical characteristics of the geopolymer

From the physical characterization of the geopolymer, density values ranging from 1645 (kg/m³) to 1726 (kg/m³) 3 were obtained (Figure 7). This material turned out to be 25% lighter than the maximum density of 2200 kg/m³ obtained by other authors as Ref. 21 of 2200 (kg/m³).

The obtained geopolymer presented an average absorption percentage of 30.79%; that is, it presented many pores, and in the presence of water, its weight increased.

The compressive strength was evaluated at 7 and 28 d in two molarities, one of 8 M and the other of 12 M, the latter being the concentration that allows reaching a compressive strength of 14.10 MPa at 10% SF replacement (see Figures 8 and 9). These results agree with the study carried out by Ref. 25, from which they concluded that the compressive strength was linked to the molar concentration and that a sodium hydroxide concentration of 12 M is the most optimal. Reference 27 investigated the correlation of water-cement ratios with compressive strength; the results of the water-cement vs. binder activator ratio presented in this research show a difference in strength of around 10MPa. It could be because pumice stone is in a stage of development as a precursor material of the geopolymer; furthermore, it could be due to the conditioning for



Figure 6. PS powder diffraction gram.²⁸

Table 6. Technical s	pecifications	of Na ₂ SiO ₃ .
----------------------	---------------	---------------------------------------

Compound name	Chemical formula	Percentage
Cristobalite, syn	SiO ₂	1.9%
Anorthite	CaAl ₂ Si ₂ O ₈	3.8%
Albite, calcite, ordered	(Na, Ca) Al (Si, Al) ₃ O ₈	6.7%
Potassium aluminum silicate	K Al SiO ₄	4.3%



Silica Fume (SF) Content at 8M

Figure 7. Geopolymer density at the age of 28 days with dosages at 8 M.



Figure 8. Compressive strength curves at different % of SF with 8 M.



Figure 9. Flexural strength curves at different % of SF with 8 M.

the elaboration of the precursor material (pumice stone) as its treatment was carried out manually, so the size of particle generated was considerable, with a fineness modulus equal to 0.69.

Similarly, when comparing the resistance of the control specimens versus those that have 10% SF replacement, the resistance increases as the percentage of SF replacement increases, with an increase of 56.31% for the concentration of 8 M and 47.59% for that of 12 M. This demonstrates the influence that SF has on geopolymer pastes in terms of compressive strength.

For resistance to flexure, the best results were obtained under the same conditions as the resistance to compression, while the maximum value obtained was 4.78 MPa. Figures 10 and 11 show that the increase in resistance between



Figure 10. Compressive strength curves at different % of SF with 12 M.



Figure 11. Flexural strength curves at different % SF with 12 M.

the control specimens and those with 10% SF replacement was 28.14% and 17.78% for the 8 M and 12 M molarities, respectively. This shows the influence of SF on geopolymer pastes when it comes to flexural strength.

Figures 12 and 13 shows compression and flexural failure for the 12M geopolymer specimens respectively.



Figure 12. Compression test on cubic geopolymeric specimens 12 M (uniaxial compression failure).



Figure 13. Flexure test on prismatic geopolymeric specimens 12 M (flexural failure located in the zone of maximum traction of the cross section).

Conclusions

The PS powder used in the preparation of the geopolymer has a chemical composition that benefits its use as a precursor, because it is composed mostly of silicon and aluminum. Physically, it is a fine material with a particle size of less than 0.6 mm and a density of 2084.23 (kg/m³).

In the design of the geopolymer dosage, several factors influence the resistance, such as the ratio of sodium silicate to sodium hydroxide, water content, temperature, curing time, molarity of sodium hydroxide and alkaline solution, and binder ratio. The latter two are the most important factors to consider in terms of environmental and economic feasibility because the excessive use of the alkaline solution is counterproductive to the contribution to reducing CO_2 emissions, as the production of these activators requires considerable energy consumption, which represents high production costs.

The results of resistance to compression and flexure increased up to 47% and 16% respectively, this is if the test tubes with 10% replacement of SF are compared versus the control test tubes, both with 12M molarity, in this way it is evidenced that there is a directly proportional relationship between mechanical resistance and the increase in the percentage of SF replaced.

A maximum compressive strength of 14.1 MPa was reported, indicating that the geopolymer cannot be used as a structural material. However, this strength can be improved by reducing the size of the PS powder particles to a diameter of less than 0.075 mm to guarantee the dissolution and reaction of silica and aluminum, making room for polymerization of the entire material.

Regarding the physical properties of the geopolymer, the density of the specimens with SF was greater than that of the control specimens because the fineness and composition of the SF fulfill the function of filling voids, thus densifying the mixture. In addition, it contributes to the formation of the polymeric gel, which is 25% lighter if the maximum density obtained is compared with that of other authors. A high percentage of absorption caused by free water stored inside the test tubes that later evaporates during oven curing has also been reported, generating a considerable number of pores that cause a decrease in resistance.

Data availability

The dataset for this research has been deposited in Harvard Dataset repository and contains:

- Set of analysis results for the relevant experimental processes. Available at:

Andrade Valle, Alexis, 2024, "Data - Use of pumice stone and silica fume as precursor material for the design of a geopolymer", https://doi.org/10.7910/DVN/MHO9OV, Harvard Dataverse, V2.²⁹

Data are available under the terms of the Creative Commons Zero "No rights reserved" data waiver (CC0 1.0 Public domain dedication).

References

- Ulusu H, Aruntas Y, Gencel O: Investigation on characteristics of blended cements containing pumice. *Constr. Build. Mater.* 2016; 118: 11–19. Publisher Full Text
- Mallagray J, Anagnostou J, Stek M, et al.: Strengthening Sustainability in the Cement Industry. International Finance Corporation; 2020. Accessed: Apr. 15, 2024. Reference Source
- Gessa Perera A, Sancha Dionisio MDP: Alternatives pour la réduction des émissions de dioxide de carbone (CO2) dans la production de ciment. Proposition d'un modèle d'évaluation. Innovar. 2016; 26(60): 51–66.
 Publisher Full Text
- 4. Hardjito D, Wallah E: On the Development of Fly Ash-Based Geopolymer Concrete.
- Payá J, Monzó J, Borrachero M v, et al.: Activadores alternativos para cementos de activación alcalina. Revista ALCONPAT. 2022; 12(1): 16–31.
 Publisher Full Text
- 6. Mantilla HÁ, Reyes CAR, Alarcón OMC: **Propiedades** fisicomecánicas, desempeño y durabilidad de morteros

geopoliméricos a base de puzolana natural tipo piedra pómez. Inge CuC. 2021; **17**(1): 216–229. Publisher Full Text

- Andrade Valle AI, Marcillo Zapata CA, Brito Noboa JP, et al.: Estudio exploratorio de piedra pómez como agregado en la producción de concretos ligeros. Polo Del Conocimiento: Revista Científico -Profesional. 2024; 9(1): 860–871.
 Publisher Full Text
- Occhipinti R, et al.: Alkali activated materials using pumice from the Aeolian Islands (Sicily, Italy) and their potentiality for cultural heritage applications: Preliminary study. Constr. Build. Mater. 2020; 259: 120391–120391.
 Publisher Full Text
- Haddad RH, Lababneh ZK: Geopolymer composites using natural pozzolan and oil-shale ash base materials: A parametric study. 2019.
 Publisher Full Text
- Andrade A, Castillo T, Paredes M, et al.: Geopolymer Designed with Pumice Stone from Ecuador. Civ. Eng. Archit. 2022; 10(5): 1864–1880.
 Publisher Full Text

- 11. Das SK, et al.: Fresh, strength and microstructure properties of geopolymer concrete incorporating lime and silica fume as replacement of fly ash. J. Build. Eng. Nov. 2020; 32: 101780. **Publisher Full Text**
- Min FS, *et al.*: Effect of silica fume and alumina addition on the mechanical and microstructure of fly ash geopolymer concrete. 12. Arch. Metall. Mater. 2022; 67(1): 197-202. **Publisher Full Text**
- Kockal NU, Beycan O, Gulmez N: Effect of binder type and content on physical and mechanical properties of geopolymers. Sadhana Acad. Proc. Eng. Sci. Apr. 2018; 43(4): 1–9. 13. **Publisher Full Text**
- Premkumar R, Hariharan P, Rajesh S: Effect of silica fume and recycled concrete aggregate on the mechanical properties of 14. GGBS based geopolymer concrete Selection and peer-review under responsibility of the scientific committee of the Sustainable Materials and Practices for Built Environment. Effect of silica fume on the characterization of the geopolymer materials. 2022; 60: 211-215. **Publisher Full Text**
- Instituto Ecuatoriano de Normalización (INEN): NTE INEN 198: 15. Resistencia a la flexión y compresión de morteros. Accessed: Jul. 03, 2023.

Reference Source

- 16. Instituto Ecuatoriano de Normalización (INEN): NTE INEN 488: Cemento Hidráulico. Determinación de la Resistencia a la Compresión de 50 mm de Arista.
- UNE Normalización Española: UNE-EN 1015-10:2000/A1:2007 17. Métodos de ensayo de los morteros. Accessed: Apr. 16, 2024.
- Instituto Ecuatoriano de Normalización (INEN): NTE INEN 0856: Áridos. Determinación de la densidad, densidad relativa 18. (gravedad específica) y absorción del árido fino. 2010. Accessed: Apr. 15, 2024. **Reference Source**
- Instituto Ecuatoriano de Normalización: NTE INEN 0696: Aridos. 19.
- Análisis granulométrico de agregados fino y grueso. 2011. 20 ISO 9001:2015, Quality Management Systems. Accessed: Apr.
- 16.2024. **Reference Source**

- Osorio B: Nuevos conglomerantes sostenibles de activación con 21. residuos de tierra de diatomeas y cenizas de cáscara de almendra. 2021. Accessed: Apr. 16, 2024. **Reference Source**
- Millán-Corrales G, Magallanes-Rivera RX, González-López JR, et al.: Synthesis of an Alternative Hydraulic Binder by Alkali Activation 22. of a Slag from Lead and Zinc Processing. Waste Biomass Valorization. Jan. 2020; 11(1): 375-388. **Publisher Full Text**
- Instituto Ecuatoriano de Normalización: NTE INEN 2380: Cementos 23. hidráulicos. Requisitos de desempeño para cementos hidráulicos. Accessed: Apr. 16, 2024. **Reference Source**
- Mobasheri F, Shirzadi Javid AA, Mirvalad S, et al.: Durability and 24. Mechanical Properties of Pumice-based Geopolymers: A Sustainable Material for Future. Iran. J. Sci. Technol. Trans. Civ. Eng. Feb. 2022; 46(1): 223-235. **Publisher Full Text**
- Safari Z, Kurda R, Al-Hadad B, et al.: Mechanical characteristics of 25. pumice-based geopolymer paste. Resour. Conserv. Recycl. Nov. 2020; 162: 105055. **Publisher Full Text**
- Mehta A, Siddique R: An overview of geopolymers derived from 26. industrial by-products. Constr. Build. Mater. Nov. 2016; 127: 183-198. Publisher Full Text

- Rahimzadeh CY, Salih A, Barzinjy AA: **Systematic Multiscale Models** to Predict the Compressive Strength of Cement Paste as a Function of Microsilica and Nanosilica Contents, Water/Cement 27 Ratio, and Curing Ages. Sustainability. 2022; 14(3): 1723. **Publisher Full Text**
- Andrade A, Castillo T, Paredes M, et al.: Geopolymer Designed with 28. Pumice Stone from Ecuador. Civ. Eng. Archit. 2022; 10(5): 1864-1880. **Publisher Full Text**
- Andrade Valle A: Data Use of pumice stone and silica fume 29. as precursor material for the design of a geopolymer. Harvard Dataverse. 2024; V2. **Publisher Full Text**

Open Peer Review

Current Peer Review Status: 💉 💉 ?

Version 2

Reviewer Report 07 February 2025

https://doi.org/10.5256/f1000research.169994.r359271

© **2025 Safari Z.** This is an open access peer review report distributed under the terms of the Creative Commons Attribution License, which permits unrestricted use, distribution, and reproduction in any medium, provided the original work is properly cited. The author(s) is/are employees of the US Government and therefore domestic copyright protection in USA does not apply to this work. The work may be protected under the copyright laws of other jurisdictions when used in those jurisdictions.

? 🛛 Zrar Safari 匝

Soran University, Soran, Iraq

The manuscript presents a "Use of pumice stone and silica fume as a precursor material for the design of a geopolymer" The present study deals with using pumice as a precursor in the production of geopolymers and replacing silica fume as a partial replacement of pumice. This investigation examines geopolymer paste's strength and physical properties, specifically focusing on the influence of molarity and replacement percentage of SF on the mechanical and physical properties of combined SF-PS geopolymer. The paper subject is interesting and required minor revision as follows:

- 1. The research objectives should be clearly stated in the abstract, and it should be clear whether these objectives have been achieved.
- 2. In "Methods," just 2.5%, 5%, and 7.5% of silica fume were mentioned; however, 10% was used but not mentioned in Methods.
- 3. The quality of the diagram and figures needs to be updated with better quality.
- 4. The chemical composition of pumice stone powder used in the study should show that Si and Al content significantly affects a geopolymer's physical and mechanical properties with pumice.
- 5. Add the standard specifications of the absorption and density tests.
- 6. The result and discussion should be rewritten in some separate subsections and discussed separately. Density, compressive strength and flexural strength, for each sub-section, discuss each parameter arranged (first effect of %SF, then effect of Molarity and effect of age of sample).
- 7. There are no reasons or justifications for why %SF increases strength and why 12M has higher strength.
- 8. The findings of the study should be compared with previous studies about a combination of SF and pumice or GGBFS and Pumice.
- 9. English language editing is required.
- 10. It is very important to provide microstructure analysis for a better understanding of the

internal activation of the geopolymer paste.

Is the work clearly and accurately presented and does it cite the current literature? $\ensuremath{\ensuremath{\mathsf{Yes}}}$

Is the study design appropriate and is the work technically sound? $\ensuremath{\mathsf{Yes}}$

Are sufficient details of methods and analysis provided to allow replication by others? Partly

If applicable, is the statistical analysis and its interpretation appropriate? Partly

Are all the source data underlying the results available to ensure full reproducibility? Partly

Are the conclusions drawn adequately supported by the results? $\ensuremath{\mathsf{Yes}}$

Competing Interests: No competing interests were disclosed.

Reviewer Expertise: Geopolymer Concrete, waste-based Geopolymers, Alkaline activated Materials

I confirm that I have read this submission and believe that I have an appropriate level of expertise to confirm that it is of an acceptable scientific standard, however I have significant reservations, as outlined above.

Reviewer Report 21 January 2025

https://doi.org/10.5256/f1000research.169994.r359270

© **2025 Bashir M.** This is an open access peer review report distributed under the terms of the Creative Commons Attribution License, which permits unrestricted use, distribution, and reproduction in any medium, provided the original work is properly cited.

? Muhammad Tariq Bashir 匝

CECOS University of IT and Emerging Sciences, INTI International University, Peshawar, Pakistan

Comments and Suggestions for Authors

The manuscript entitled, "Use of pumice stone and silica fume as precursor material for the design of a geopolymer" investigates the use of pumice stone and silica fume in geopolymers, addressing sustainability by reducing reliance on traditional cement. The authors detail the characterization of materials, mix design, and testing procedures, ensuring reproducibility. However, I have few comments and suggestion to be addressed as follows:

• The discussion could delve deeper into the microstructural changes due to silica fume

- The extended curing time (120 hours) raises practical concerns for scalability and costs. Consider discussing alternative curing methods or shorter durations that might yield comparable results.
- The distinction between water in the alkaline solution and additional water is addressed, but further clarity in the methods section would be beneficial for replication.
- The authors conclude that the material is unsuitable for structural applications. Highlight potential non-structural applications.
- Strengths and limitations of the material should be more critically compared with similar research to contextualize findings. Authors can consider the following papers: [Ref 1], [Ref 2] and [Ref 3].
- Ensure consistent terminology (e.g., sodium silicate solution vs. sodium silicate) and review the text for minor grammatical issues.

Recommendation

The paper contributes to research on sustainable construction materials and can be accepted after minor revisions. Addressing the mentioned comments and suggestions will further strengthen the impact and clarity of the study.

References

1. Farid F, Rehman A, Bashir M, Khan M, et al.: Synergistic Effects of Corn Stalk Ash and Fly Ash on the Properties of Lightweight Concrete Using Pumice Aggregate. Iranian Journal of Science and Technology, Transactions of Civil Engineering. 2024. Publisher Full Text

2. Uddin M, Bashir M, Khan A, Alsharari F, et al.: Effect of Silica Fume on Compressive Strength and Water Absorption of the Portland Cement-Silica Fume Blended Mortar. Arabian Journal for Science and Engineering. 2024; 49 (4): 4803-4811 Publisher Full Text

3. Oti J, Adeleke BO, Anowie FX, Kinuthia JM, et al.: Mechanical Properties of a Sustainable Low-Carbon Geopolymer Concrete Using a Pumice-Derived Sodium Silicate Solution. Materials (Basel). 2024; 17 (8). PubMed Abstract | Publisher Full Text

Is the work clearly and accurately presented and does it cite the current literature? Partly

Is the study design appropriate and is the work technically sound?

Yes

Are sufficient details of methods and analysis provided to allow replication by others? Partly

If applicable, is the statistical analysis and its interpretation appropriate? Yes

Are all the source data underlying the results available to ensure full reproducibility? Partly

Are the conclusions drawn adequately supported by the results?

Yes

Competing Interests: No competing interests were disclosed.

Reviewer Expertise: Composite Materials, Water & Wastewater, Sustainability

I confirm that I have read this submission and believe that I have an appropriate level of expertise to confirm that it is of an acceptable scientific standard, however I have significant reservations, as outlined above.

Reviewer Report 29 August 2024

https://doi.org/10.5256/f1000research.169994.r313351

© **2024 K. H. Amen Bzeni D.** This is an open access peer review report distributed under the terms of the Creative Commons Attribution License, which permits unrestricted use, distribution, and reproduction in any medium, provided the original work is properly cited.



Dillshad K. H. Amen Bzeni 匝

Salahadin University, Erbil,, Kurdistan Region, Iraq

The author answered all the comments properly and deserves to be indexed in this Journal

Is the work clearly and accurately presented and does it cite the current literature? $\ensuremath{\mathsf{Yes}}$

Is the study design appropriate and is the work technically sound? Yes

Are sufficient details of methods and analysis provided to allow replication by others? $\ensuremath{\mathsf{Yes}}$

If applicable, is the statistical analysis and its interpretation appropriate? $\ensuremath{\mathsf{Yes}}$

Are all the source data underlying the results available to ensure full reproducibility? $\ensuremath{\mathsf{Yes}}$

Are the conclusions drawn adequately supported by the results? $\ensuremath{\mathsf{Yes}}$

Competing Interests: No competing interests were disclosed.

I confirm that I have read this submission and believe that I have an appropriate level of expertise to confirm that it is of an acceptable scientific standard.

Version 1

Reviewer Report 28 June 2024

https://doi.org/10.5256/f1000research.161926.r288184

© **2024 K. H. Amen Bzeni D.** This is an open access peer review report distributed under the terms of the Creative Commons Attribution License, which permits unrestricted use, distribution, and reproduction in any medium, provided the original work is properly cited.

? 🔹 Dillshad K. H. Amen Bzeni 匝

Salahadin University, Erbil,, Kurdistan Region, Iraq

The article describes using pumice combined with silica fume as a source material to make geopolymer concrete. This research is notable because it explores alternatives to normal cement by using pumice, a naturally occurring volcanic stone present in several regions. The study attempted to improve the qualities of geopolymer material by combining silica fume and pumice in varied ratios.

The paper is written in clear and accessible English, and the technique is consistent with established research methods for data collecting and presentation. The findings are reported properly. However, the text does not provide a scientific discussion or analysis of why pumice stone powder did not give adequate strength for structural concrete. Scanning Electron Microscope (SEM) investigations would have been useful for investigating the microstructure of the geopolymer material, demonstrating how silica fume increased strength, and revealing cavities and defects in the structure.

The specimens' 120-hour (5-day) cure duration raises questions regarding the costs and time of geopolymer manufacture. Furthermore, the article does not identify the water-to-binder ratio, whether the water stated is part of the alkali solution or additional pure water in the mixture.

The article might benefit from a more in-depth examination, including earlier data on pumice stone powder, to highlight variations in the geopolymer's characteristics compared to other research findings.

Is the work clearly and accurately presented and does it cite the current literature? $\ensuremath{\ensuremath{\mathsf{Yes}}}$

Is the study design appropriate and is the work technically sound?

Yes

Are sufficient details of methods and analysis provided to allow replication by others? $\ensuremath{\mathbb{No}}$

If applicable, is the statistical analysis and its interpretation appropriate?

Not applicable

Are all the source data underlying the results available to ensure full reproducibility? Yes

Are the conclusions drawn adequately supported by the results?

Partly

Competing Interests: No competing interests were disclosed.

Reviewer Expertise: Concrete technology, special types of concrete including geopolymer concrete , fiber reinforced concrete

I confirm that I have read this submission and believe that I have an appropriate level of expertise to confirm that it is of an acceptable scientific standard, however I have significant reservations, as outlined above.

Author Response 03 Aug 2024

Andrea Zarate

Dear Dillshad K. H. Amen Bzeni

Thank you for your comments, we have submitted an amended manuscript including your recommendations.

1- The text does not provide a scientific discussion or analysis of why pumice stone powder did not give adequate strength for structural concrete.

Discussion has been added to the amended manuscript, considering compressive strengths studied in other investigations.

2-Scanning Electron Microscope (SEM) investigations would have been useful for investigating the microstructure of the geopolymer material, demonstrating how silica fume increased strength, and revealing cavities and defects in the structure. Future research will include SEM of the geopolymer material as we are investigating the mechanical behavior of geopolymer using a variety of industrial residues

3-The specimens' 120-hour (5-day) cure duration raises questions regarding the costs and time of geopolymer manufacture.

The research has an exploratory-correlational scope, for this reason it was not intended to find the cost-benefit through its experimental implementation in the quality control laboratory of materials, in these early stages it is only intended to meet the minimum requirements to make the applicability of the geopolymer as a binder material reliable.

4-The article does not identify the water-to-binder ratio, whether the water stated is part of the alkali solution or additional pure water in the mixture.

This has been clarified in the article as the alkaline solution includes distilled water and the chemical compounds: Sodium hydroxide NaOH and Sodium silicate Na2SiO3

5-The article might benefit from a more in-depth examination, including earlier data on pumice stone powder, to highlight variations in the geopolymer's characteristics compared to other research findings.

More information has been added to the amended manuscript.

Kind Regards,

The authors

Competing Interests: No competing interests were disclosed.

Reviewer Report 24 June 2024

https://doi.org/10.5256/f1000research.161926.r288182

© **2024 Zeyad A.** This is an open access peer review report distributed under the terms of the Creative Commons Attribution License, which permits unrestricted use, distribution, and reproduction in any medium, provided the original work is properly cited.



Abdullah Zeyad

Jazan University,, Jazan, Saudi Arabia

Dear authors,

Please respond to the following comments.

1- The introduction requires expansion of previous relevant research.

2- List all references used in this study.

3- Provide the paper with failure models for the samples.

4- It should highlight what the research has achieved that is consistent with the purpose of the study.

Thanks

Is the work clearly and accurately presented and does it cite the current literature? $\ensuremath{\mathsf{Yes}}$

Is the study design appropriate and is the work technically sound?

Yes

Are sufficient details of methods and analysis provided to allow replication by others? Partly

If applicable, is the statistical analysis and its interpretation appropriate?

Not applicable

Are all the source data underlying the results available to ensure full reproducibility? Partly

Are the conclusions drawn adequately supported by the results?

Yes

Competing Interests: No competing interests were disclosed.

Reviewer Expertise: Use of pumice stone in production geopolymer concrete

I confirm that I have read this submission and believe that I have an appropriate level of expertise to confirm that it is of an acceptable scientific standard.

Author Response 03 Aug 2024

Andrea Zarate

Dear Abdullah Zeyad

Thank you for your comments, we have submitted an amended manuscript including your recommendations.

1- The introduction requires expansion of previous relevant research.
Additional information has been included in the introduction.
2- List all references used in this study
The reference list has been updated including the new references used.
3- Provide the paper with failure models for the samples
Figure 12 and 13 have been included showing the failure models for the samples.
4- It should highlight what the research has achieved that is consistent with the purpose of the study
The third conclusion highlights the importance of the use of SF in geopolymeric samples by improving the mechanical behavior.

The authors

Competing Interests: No competing interests were disclosed.

Page 23 of 24

The benefits of publishing with F1000Research:

- Your article is published within days, with no editorial bias
- You can publish traditional articles, null/negative results, case reports, data notes and more
- The peer review process is transparent and collaborative
- Your article is indexed in PubMed after passing peer review
- Dedicated customer support at every stage

For pre-submission enquiries, contact research@f1000.com

F1000 Research