

## Characterization of the properties of perlite geopolymer pastes

G.M. Tsaousi✉, I. Douni, D. Panias

National Technical University of Athens (NTUA) (Athens, Greece)  
✉[margitsaousi@metal.ntua.gr](mailto:margitsaousi@metal.ntua.gr)

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**ABSTRACT:** This paper deals with the characterization of perlite-based geopolymer pastes, using fine perlite as raw material. The present study examined the effects of the main synthesis parameters such as perlite to activator ratio, NaOH concentration, the addition of soluble silica to the activator, and curing temperature on the setting time, the stability in an aquatic environment, the viscosity of the paste, and the compressive strength of the solidified geopolymers. The results showed that these inorganic polymer pastes are non-Newtonian shear thinning fluids that achieve low viscosities at high shear stresses. The optimum synthesis conditions for the geopolymer pastes proved to be a) a low initial NaOH concentration in the alkaline phase (2–5 M) and b) a solid to liquid ratio of 1.2–1.4 g/mL. If very fast setting is necessary, the pastes should be prepared with a soluble silica-doped alkaline activating phase and cured at high temperatures around 90 °C.

**KEYWORDS:** Perlite; Geopolymer paste; Alkali activation; Characterization

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**RESUMEN:** *Caracterización de las propiedades de pastas geopoliméricas de perlita.* El presente trabajo trata de la caracterización de pastas geopoliméricas basadas en perlita utilizando perlita fina como materia prima. Se han examinado los efectos que tienen los principales parámetros de síntesis —tales como la relación perlita/activador, la concentración de NaOH, la adición de sílice soluble al activador y la temperatura de curado— sobre el tiempo de fraguado, la estabilidad en medio acuoso, la viscosidad de la pasta y la resistencia a la compresión de los geopolímeros solidificados. Los resultados han mostrado que estas pastas poliméricas inorgánicas son fluidos no-Newtonianos de viscosidad estructural que alcanzan bajos niveles de viscosidad a altas tensiones de cizalla. Las condiciones óptimas de síntesis para las pastas geopoliméricas resultaron ser a) baja concentración inicial de NaOH en la fase alcalina (2–5 M) y b) una relación sólido-líquido entre 1,2 y 1,4 g/mL. Si se requiere fraguado muy rápido, se tienen que preparar las pastas con un activador alcalino dopado con sílice soluble y curar a altas temperaturas cercanas a los 90 °C.

**PALABRAS CLAVE:** Perlita; Pasta geopolimérica; Activación alcalina; Caracterización

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### 1. INTRODUCTION

Geopolymerization is an innovative technology that can utilize solid aluminosilicate raw materials (powders) and alkali metal silicate solutions to produce new materials characterized by

three-dimensional polymeric structures called geopolymers or inorganic polymers (1). Extensive research in the field of geopolymeric synthesis in recent decades has proved that geopolymers possess excellent mechanical and physicochemical properties. They exhibit high surface hardness, high

mechanical strength, fire and chemical resistance, thermal stability, low density, and micro- or nanoporosity (2–9). As a result of these properties, inorganic polymers are considered ideal alternatives for many industrial applications, mainly in the construction sector.

The geopolymerization process is based on an exothermic heterogeneous chemical reaction between a solid aluminosilicate raw material and an alkali metal silicate solution under atmospheric conditions and at temperatures up to 100 °C. The materials synthesized by this reaction present mainly an amorphous or semi-crystalline Si-O-Al and/or Si-O-Si frame. A variety of solid industrial residues or wastes such as fly ash, metallurgical slags, and mine wastes, and industrial minerals such as kaolinite and feldspars (10–19), have been used for the synthesis of geopolymers. Perlite, however, has not been extensively studied as a potential aluminosilicate source for the synthesis of inorganic polymers through geopolymerization. The main reason for this is that perlite has long been considered as an inefficient raw material for geopolymerization that produces final materials lacking the desired mechanical properties. Perlite has been activated with alkaline solutions to produce geopolymer binders or mortars. After casting at 60 °C the materials produced were found to be porous and fragile (20). In the case of mortars, sodium silicate-activated mixtures slowly reach moderate strengths at room temperature but mixtures activated with sodium hydroxide do not develop the required strength. The mechanical properties have been found to improve with 100 °C dry curing (21).

Perlite is an amorphous aluminosilicate volcanic glass which, upon rapid controlled heating, expands into a foam material of low bulk density. Perlite deposits are normally exploited by means of low-cost open-pit mining methods followed by in-situ milling and screening. Greece and Turkey are the world's leading perlite producers, with an annual production in 2015 of 700,000 tons and 1,100,000 tons, respectively (22), while in Europe the annual production of perlite was estimated at about 1,935,000 tons in 2015 (22).

This paper deals with the characterization of perlite-based geopolymer pastes, using fine perlite as raw material. It studies the effects of the main synthesis parameters—such as perlite-to-activator ratio (S/L), NaOH and soluble silica concentrations in the activator, and curing temperature—on the setting time and rheological properties of the geopolymer pastes synthesized.

## 2. EXPERIMENTAL

### 2.1. Materials

The raw materials used for the synthesis of geopolymer paste were fine perlite, sodium hydroxide, and a sodium silicate solution. The fine perlite originated from Imerys deposits on the Greek island of Milos. Table 1 shows the chemical analysis of this perlite on a dry basis, determined using a Xepos X-ray fluorescence spectrometer and X-LAB software. The main chemical components of the perlite used were found to be silicon, aluminum, sodium, and potassium, while magnesium and calcium were minor constituents. Loss on ignition was determined as the amount of perlite remaining after 1 h at 1100 °C.

Figure 1 shows the mineralogical analysis of the perlite used, which was determined by X-ray diffraction (XRD) on a Siemens D5000 diffractometer using Cu K $\alpha$  radiation ( $\lambda = 1.5418$  Å) operating at 40kV, 30 mA, at  $2\theta$  range from 2° to 80° with a 0.02° sec<sup>-1</sup> step. Fine perlite is an amorphous volcanic aluminosilicate glass that contains some crystalline phases, such as illite, feldspars (albite and anorthite), and quartz (23). Raw perlite is extremely fine, with a mean particle size of  $d_{50} = 6.87$   $\mu\text{m}$  measured on a Malvern laser particle size analyzer. Its skeletal density, measured with a Quantachrome Stereopycnometer, is 2433 kg/m<sup>3</sup>. Table 2 shows the surface characteristics, such as BET specific surface area and pore volume, measured by a Quantachrome Nova-1200 Ver 5.01 porosimeter.

The alkaline activator used for the synthesis of the geopolymer pastes was an aqueous sodium hydroxide and sodium silicate solution that was prepared by dissolving sodium hydroxide pellets (Merck Chemicals, 99 % purity) in deionized water and mixing this solution with the appropriate volume of sodium silicate solution (Merck Chemicals, extra purity). The characteristics of the sodium silicate solution used to study the effect of the initial Si concentration in the activating phase were 7.5–8.5 % Na<sub>2</sub>O, 25.5–28.5 % SiO<sub>2</sub>, and 1296–1396 kg/m<sup>3</sup> at 20 °C.

### 2.2. Paste preparation

The viscous geopolymer paste was prepared by mixing perlite with the alkaline activating solution using a mechanical stirrer for 5 minutes until a homogeneous mixture was obtained. The resulting paste was molded in appropriate open Teflon

TABLE 1. Chemical analysis of perlite

Oxides (%) w/w								
Na <sub>2</sub> O	MgO	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	K <sub>2</sub> O	CaO	Fe <sub>2</sub> O <sub>3</sub>	LOI	TOTAL
3.70	1.45	13.56	67.21	4.85	1.11	2.27	5.54	99.69

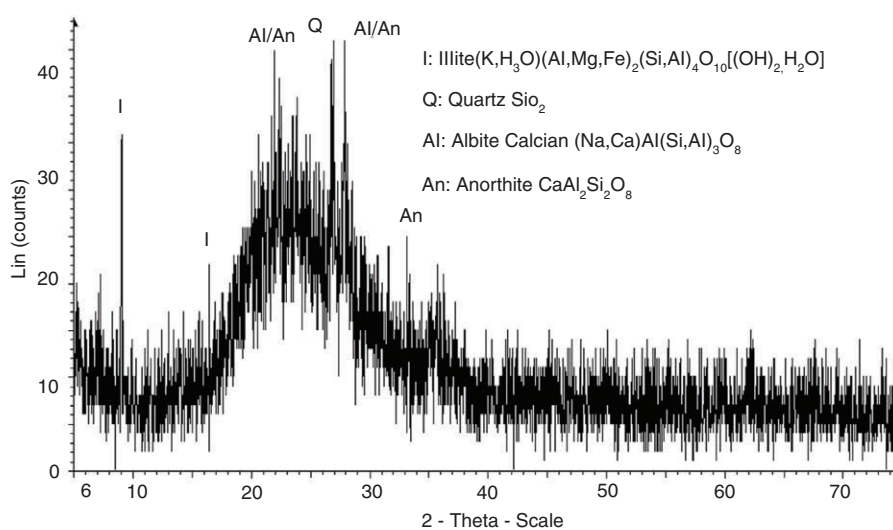


FIGURE 1. X-ray diffractogram of perlite.

TABLE 2. Surface properties of perlite

Surface characteristics	Value
Specific surface area (m <sup>2</sup> /g)	14.48
Total pore volume (cm <sup>3</sup> /g)	0.03
Average pore diameter (Å)	83.46
All pores diameter (Å)	< 1060.79

molds (5 cm cubes) and then cured at four different temperatures (50 °C, 70 °C, 90 °C, 100 °C). The maximum linear shrinkage observed in the final materials was 1 %. The NaOH concentration used in the alkaline activating solution ranged from 2 M to 12 M, and the solid to liquid ratio examined was 1.2–1.4 g/mL. The soluble silica concentration used in the activating phase ranged from 0.7 M to 2.7 M. After curing, the specimens were demolded and kept in a dry atmosphere.

### 2.3. Characterization tests

The setting time of the geopolymer pastes was measured using a MATEST Vicat apparatus in accordance with EN 196-3:2005.

The viscosity of the geopolymer pastes was measured immediately after their preparation at ambient temperature using a Brookfield Viscometer LV+.

The hydrolytic stability of the solidified geopolymer pastes was determined by means of a dissolution test in deionized water. An aliquot of 2 g of solidified sample was totally immersed in 100 mL of deionized water and left in contact for 24 h at ambient temperature. Then, the solution was filtered and analyzed for its silicon and aluminum content using an inductively coupled plasma mass spectrometer.

The remaining piece of the geopolymer was dried for 24 h at 100 °C and then weighed to determine the % weight loss (WL).

The dissolution of silicon from the perlite in the sodium hydroxide solutions was studied in a 500 mL mechanically stirred spherical glass reactor equipped with a thermostatically controlled heating mantle connected to a mercury contact thermometer, a mechanical stirrer with speed controller, and a glass condenser. The dissolution tests were performed at 30 °C with a pulp density of 16 g/L, NaOH concentrations of 1–10 M, and 24 h retention time. At the end of each test the suspension was filtered and the filtrate was further analyzed for its silicon content using an inductively coupled plasma mass spectrometer.

The compressive strength of the 5 cm cubic geopolymer specimens was measured using an Advantest 9 apparatus in accordance with EN772-1. After the preparation of the paste, the slurry was molded into appropriate molds and cured in two phases (48 h at 70 °C and 96 h at 55 °C). The solidified geopolymers remained at ambient temperature for 23 more days, after which their compressive strength was immediately measured.

## 3. RESULTS AND DISCUSSION

### 3.1. Effect of NaOH concentration on geopolymer paste properties

The effect of the initial NaOH concentration in the activating phase on the properties of the geopolymer pastes was studied for the range of 2–12 M. The activator consisted of a pure NaOH solution without the addition of soluble silica in the form of

a sodium silicate solution. The solid to liquid ratio was kept constant at 1.2 g/mL and the pastes were cured at 50 °C, 70 °C, 90 °C, and 100 °C. The results are shown in Figure 2, where the setting time of the geopolymer pastes synthesized at different temperatures is plotted against the initial concentration of the NaOH solution.

The results showed a strong trend towards increase in setting time as a function of NaOH concentration. At 50 °C, as the NaOH concentration rose from 2 M to 6 M, the setting time increased almost six-fold from 20 to 120 hours. For NaOH concentrations over 6 M, the setting time at this temperature was invariable. At 70 °C, the setting time was generally shorter than at 50 °C. It was invariable in the 2–5 M NaOH concentration range; then, when the NaOH concentration exceeded 5 M, the setting process slowed considerably, only to reach another plateau for NaOH concentrations over 8 M. The setting of the

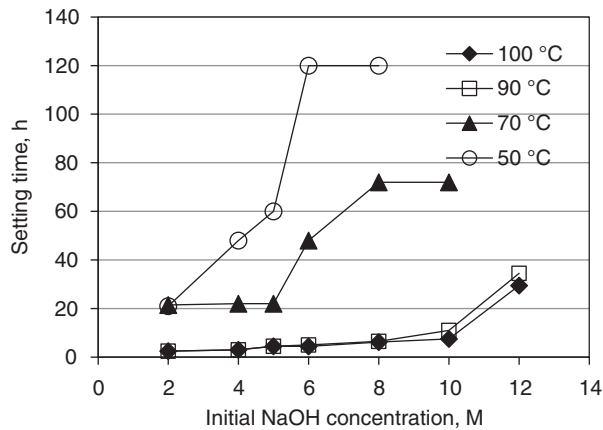


FIGURE 2. Setting time of geopolymer pastes as a function of initial NaOH concentration in the activator.

geopolymer pastes was much faster at 90 °C and 100 °C.

The results revealed that the perlite geopolymer pastes solidified faster under lower NaOH concentrations in the alkaline activating phase as well as at higher curing temperatures. The former of the above observations would seem to run counter to the common perception that geopolymerization technology is most effective at high NaOH concentrations (16, 24, 25). As a matter of fact, as seen in Figure 3a, Si dissolution from perlite after 24 hours retention time at 30 °C did indeed increase as the initial NaOH concentration increased, but a ten-fold increase in the NaOH concentration (from 1 M to 10 M) caused only a 2.5-fold increase in the silicon dissolution (from 10% to 25%) leaving, according to the dissolution reaction [eq.1], a substantial excess amount of free NaOH and establishing lower  $\text{SiO}_2/\text{Na}_2\text{O}$  mass ratios in the aqueous phase. The lower  $\text{SiO}_2/\text{Na}_2\text{O}$  mass ratios favor the formation of monomer and oligomer Si species (Fig. 3b) (26–28) and therefore inhibit the polycondensation phenomena, slowing the setting time of perlite geopolymer pastes (27). Besides, the lability of geopolymer gel synthesized with low  $\text{SiO}_2/\text{Na}_2\text{O}$  ratios in the activating solution will be much greater than that of specimens with higher  $\text{SiO}_2/\text{Na}_2\text{O}$  ratios (28).



The compressive strength of the cubic geopolymer specimens in relation to the initial sodium concentration in the activation solution is presented in Figure 4. In this experimental series, the concentration of NaOH varied between 2 M and 6 M, and the solid to liquid ratio was kept constant at 1.2 g/mL. The formulation using 4 M of activator

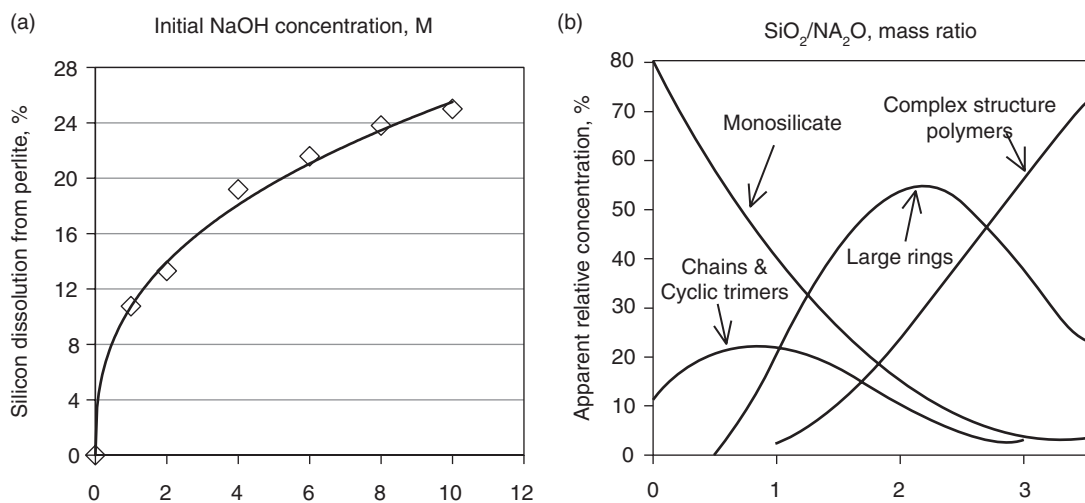


FIGURE 3. (a) Silicon dissolution from perlite in several NaOH solutions at 30 °C, 24 h and pulp density 16g/L (b) Qualitative interpretation of soluble silicate species equilibria in 1m aqueous silicate solution.

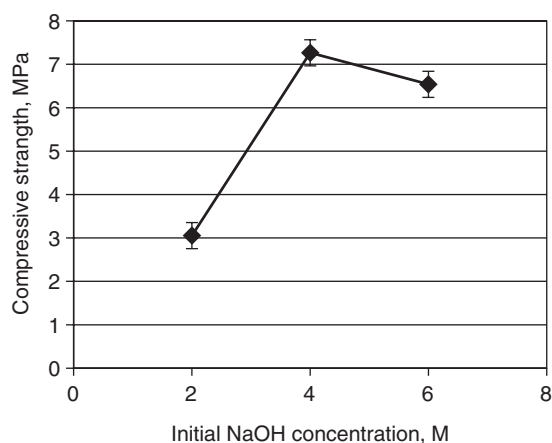


FIGURE 4. Perlite paste compressive strength versus initial NaOH concentration with standard deviation 0.32 MPa for 2 M, 0.25 MPa for 4 M, and 0.28 MPa for 6 M. Error bars indicate the average deviation from the mean over the three samples measured.

was the one that resulted in higher compressive strength. Further increases of NaOH reduced the compressive strength of the synthesized geopolymers: the number of free alkalis was so large that they inhibited the raw material's reaction, inducing a severe degradation of the geopolymeric matrix (29). In addition, the species equilibrium at high alkaline conditions shifts towards mononuclear species formation, slowing the process of polycondensation and reducing its effectiveness as measured by the compressive strength of the geopolymeric materials (16).

The inefficient geopolymerization of the perlite pastes at high NaOH concentrations in the activating phase led to structures with limited hydrolytic stability, as seen in Table 3. The results showed that the lowest % weight loss and Al and Si concentrations in the immersion solution of the hydrolytic test were observed for the material synthesized with 2 M NaOH solution; these specimens maintained their structural integrity after immersion in the deionized water (29). The specimens synthesized at 6 M and 10 M NaOH concentrations in the activating phase, on the other hand, were largely destroyed, losing 58.05 % and 64.14 % of their initial weight, respectively, and liberating significant amounts of Si and Al into the immersion solution. The hydrolytic stability of the geopolymers is reflected in the stability of the aluminosilicate gel in aquatic conditions. Contact with water causes hydrolysis of Si-O-Si bonds in the gel and release of Si followed by partial degradation of the structure (30). The presence of Al ions in the leachate indicates a certain degree of dealumination due to the degradation of Al-O-Si bonds (31).

Measurements of the paste viscosity offer further evidence of the efficient geopolymerization of perlite under the lowest studied initial NaOH concentration in the activating phase. This can be seen in Figure 5,

TABLE 3. Hydrolytic stability test results for geopolymeric specimens synthesized with different initial NaOH concentrations at a solid to liquid ratio of 1.2 g/mL and cured at 90 °C for 24 hours

[NaOH], M	% WL	Al (ppm)	Si (ppm)
2	9.14	< 1.3	42.6
6	58.05	4.1	799
10	64.14	7.6	1070

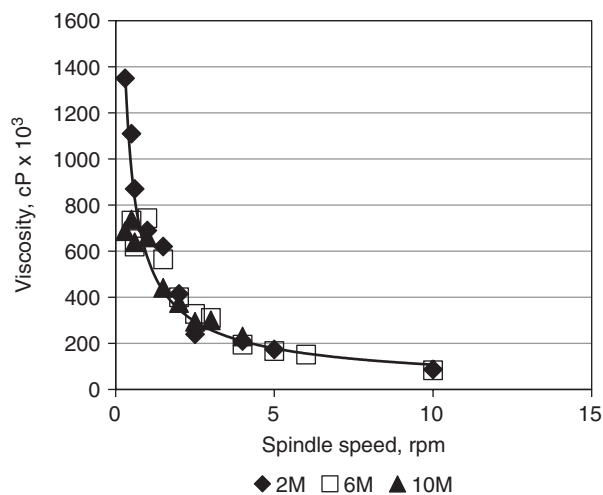


FIGURE 5. Perlite paste viscosity versus NaOH concentration and viscometer spindle speed at ambient temperature.

which shows the effect of NaOH concentration on the viscosity of the perlite pastes synthesized. Perlite pastes are non-Newtonian shear thinning fluids that achieve very low viscosities at high shear stresses. No significant effect of NaOH concentration on viscosity was revealed, indicating that the early viscosity is more related to the large amount of suspended perlite particles in the paste (increased solid to liquid ratio) than to the physical properties of the geopolymer paste. The viscosity measurements are not related to the geopolymer paste itself, because the geopolymerization process has not been completed. An exception can be found in the high viscosity values observed from 800,000 to 1,400,000 centipoise measured at spindle speeds lower than 1 rpm for the paste synthesized using a 2 M NaOH solution as activator. This observation proves once more that there is faster polymerization and therefore solidification at 2 M NaOH, indicating that the polycondensation phenomena in the perlite pastes are optimized under low NaOH concentration.

### 3.2. Effect of solid to liquid ratio

The effect on setting time of the solid to liquid (S/L) ratio used for the preparation of the perlite geopolymer pastes was investigated in the limited range of 1.2 to 1.4 g/mL, due to the very bad workability

of pastes at higher S/L values. The NaOH concentration in the activating phase was kept constant at 2 M, and the pastes were cured at temperatures ranging from 50 °C to 100 °C. The results are shown in Figure 6, where the setting time is plotted against the S/L ratio. The results showed that the S/L ratio did not affect the setting time of the paste at a constant curing temperature. Moreover, the setting of pastes in the temperature range of 50–70 °C was very slow, and was not completed until after 21–24 hours, even when the S/L ratio was at its highest value of 1.4 g/mL. On the other hand, at the highest studied curing temperatures of 90–100 °C, the setting time was almost seven to eight times faster, at only 2.5–3 hours.

Tests were also performed at a low S/L ratio of 1.2 g/mL and high S/L ratio of 1.4 g/mL using activating solutions with NaOH concentrations ranging from 2 M to 10 M. The results presented in Figure 7 showed that for both S/L ratios, the increase in NaOH concentration caused an increase in setting time, as was to be expected. The new observation was that the pastes with the higher S/L ratio solidified faster under the same NaOH concentration in the activating phase. This was attributed to the fact that an increased S/L ratio, which means there is a higher ratio of perlite per volume of the activating solution, can cause an increase in the concentration of silicon and aluminum precursors in the aqueous phase of geopolymerization (provided that perlite is the only source of soluble silicon and aluminum in the system), which in turn accelerates the polycondensation phenomena and therefore reduces the setting time of the pastes (32). Besides, when a low S/L ratio was employed, there was more fluid medium in the mix, limiting the possibility of contact between

the activating solution and the solid materials. In contrast, it is believed that when a higher S/L ratio was used, the contact between the reacting materials was improved and the geopolymerization mechanism was accelerated (33, 34).

### 3.3. Effect of initial SiO<sub>2</sub> content

The effect of the initial soluble silica concentration in the activating phase on the setting time of the perlite geopolymer pastes synthesized in this study was determined for the range of 0–2.8 M SiO<sub>2</sub>. Soluble silica was added in the form of sodium silicate solution while the NaOH concentration of the activator was kept constant at 2 M, which was the optimum value obtained from the previous experimental series.

As shown in Figure 8, for the curing temperatures studied, the setting time of the pastes decreased as the silicon content in the aqueous phase increased (35). Soluble silicate is an essential factor of geopolymerization process, as it provides the aqueous phase of the geopolymeric system with soluble silicate species, which are necessary for the initiation of oligomers formation and thus the polycondensation process (16, 36–39). At each curing temperature, an increase in the initial SiO<sub>2</sub> concentration in the aqueous phase under constant NaOH concentration resulted in an increase in the SiO<sub>2</sub>/Na<sub>2</sub>O mass ratio at the early stages of the geopolymerization process, therefore speeding up the setting process. As can also be seen in Figure 8, the setting time of the perlite geopolymer pastes converged towards the same value as the SiO<sub>2</sub> concentration in the aqueous phase increased, independently of the curing temperature. The convergence slope was substantially

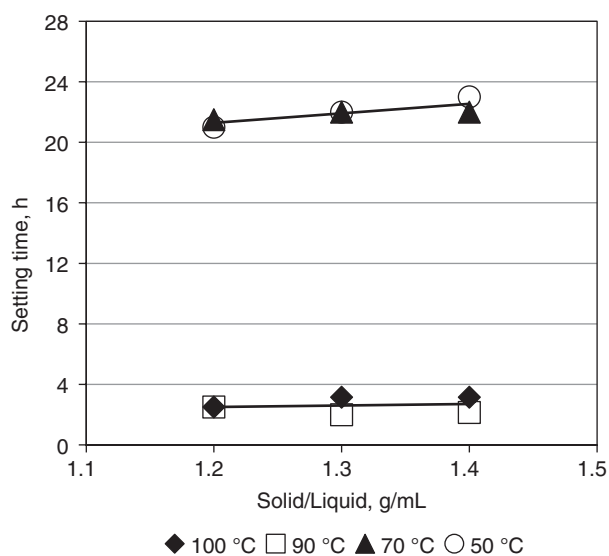


FIGURE 6. Setting time versus solid to liquid ratio at different curing temperatures. ([NaOH]:2 M)

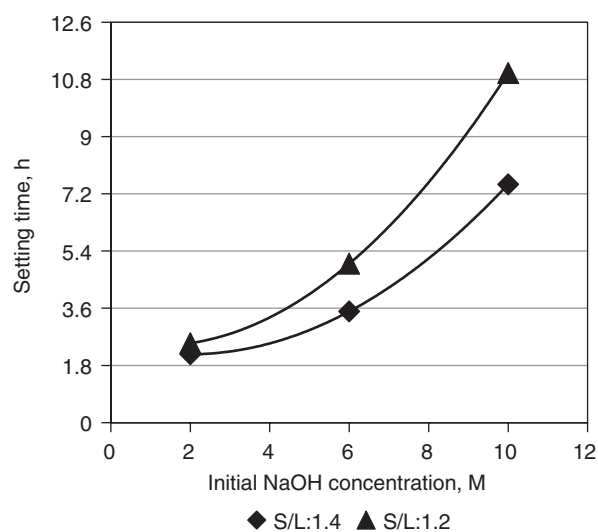


FIGURE 7. Setting time versus [NaOH] at solid to liquid ratio 1.2 and 1.4 g/mL. (Curing temperature 90 °C)

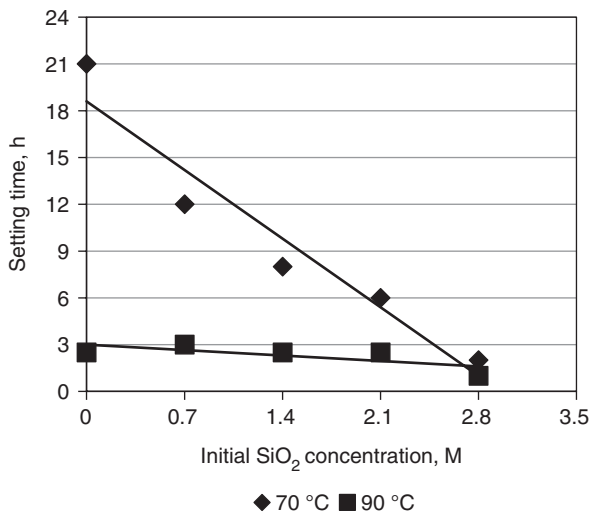


FIGURE 8. Setting time versus initial SiO<sub>2</sub> concentration in activating solution at different curing temperatures ([NaOH] = 2 M, S/L ratio = 1.2 g/mL).

steeper at lower temperatures, indicating that the effect of doping with soluble silica is more significant at lower curing temperatures. This is attributed to the slow and small rate of perlite dissolution in an alkaline environment, as seen in Figure 3a, which makes the SiO<sub>2</sub>/Na<sub>2</sub>O mass ratio in the aqueous phase of geopolymerization increase slowly, rendering doping with soluble silica a necessary procedure to speed up the setting of the paste. The temperature increase positively affects the dissolution rate, speeding the increase in the SiO<sub>2</sub>/Na<sub>2</sub>O mass ratio under the same conditions and thus smoothing the effect of soluble silica doping on setting time.

#### 4. CONCLUSIONS

- This work studied the geopolymerization of fine perlite. Due to the limited dissolution of perlite in sodium hydroxide solutions, perlite geopolymer pastes solidify faster when the sodium hydroxide concentration in the alkaline activating phase is around 2 M.
- An increase in NaOH concentration slows the setting time of the geopolymer pastes. An increase in curing temperature from 50 °C to 90 °C has a positive effect on setting time, speeding the solidification process.
- Doping the initial activating phase with soluble silica substantially shortens the setting time of perlite pastes, which converges towards the low value of 1–2 h as the initial SiO<sub>2</sub> concentration of the activating solution approaches 1.7 M. The S/L ratio has no significant effect on setting time.
- Perlite pastes are non-Newtonian shear thinning fluids that achieve very low viscosities at high shear stresses. The measured viscosities ranged

from 100,000 to 1,400,000 centipoises. There is no significant effect of the initial NaOH concentration in the activating phase on the paste viscosity.

- The formulation with 4 M of activator produced the geopolymer with the highest compressive strength.
- The optimum synthesis conditions for perlite geopolymer pastes have two typical characteristics: a) a low initial NaOH concentration in the alkaline activating phase (2–5 M) and b) an S/L ratio of 1.2–1.4 g/mL. In practical cases where very fast setting is necessary, the pastes should be prepared with a soluble silica-doped alkaline activating phase and cured at high temperatures around 90 °C.

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