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Use of the Algerian natural pozzolan for the production of a geopolymer as a complete cement replacement

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ABSTRACT

This paper investigates the use of the Algerian Beni Saf natural Pozzolan (BSP) to produce a geopolymer (GP) as a complete replacement material for the traditional binder of OPC. No such study has been carried out on using the Algerian BSP for GP synthesis, especially as the latter's properties have a high dependency on the precursor material characteristics. The research sets to establish the optimal combination of alkaline activators that corresponds to the BSP, sand content, and curing temperature. A series of experiments were carried out on BSP-based GP mortars in fresh and hardened states to investigate properties such workability, CS, and FS at 3, 7, 28, and 90 days. Selected GP samples were subjected to microstructure characterization experiments, including FTIR and SEM-EDS. The results have shown that workability is greatly affected by the studied parameters. The optimal values of CS and FS at 28 days were 38 and 12 MPa, respectively. These were obtained for sand/pozzolan ratio, Si/Al, Si/Na, and temperature equal to 2.5, 6.1, 2, and 80 °C respectively. The microstructure analyses also revealed a considerable change in the microstructure GP samples.

1. Introduction

Geopolymers are materials classed as inorganic polymers [1-3]. They are produced by the exothermic chemical reaction of dehydroxylated silica and alumina present in the source materials, with a high pH alkaline activator [3,4]. The source materials usually used are supplementary cementitious admixtures, either natural minerals such as calcined kaolin [5] and Natural Pozzolan (NP) [2], or industrial waste such as slag, fly ash, and glass waste in the form of ground powder [1]. Alkaline activators more often used for inducing the dissolution of aluminosilicates were either potassium or sodium silicates (K2SiO3 or Na₂SiO₃) mixed or not with a concentrated solution of sodium or potassium hydroxide (KOH and/or NaOH) [1-5]. The reaction between the reactive products is called "geopolymerization" [4]. In contrast to the hydration reaction found in conventional cementitious binders water is not involved in the geopolymerization process but is added to enhance the flowability of the mixture [1]. The mechanism of this reaction is not entirely understood [6,7], this is due in particular to the speed of the reaction and overlaps the latter's processes with each other [4,8]. In the sixties of the last century, Glukhovsky proposed a general model to

explain the mechanism of the reaction between aluminosilicates-based materials and alkaline activators [7,8]. Glukhovsky's model separates the geopolymerization process into three stages: firstly, the dissolution of amorphous aluminosilicates present in the raw materials under the effect of electrostatic reaction of the high alkalinity of the medium, then reorientation and coagulation of dissolved silicates and aluminates by hydrolysis, and finally the polycondensation and coagulation of the gel to form a rigid network [7–10]. Moreover, some researchers relied on the knowledge found in zeolite synthesis, and based on previous experimental results; they expanded Glukhovsky's theories to describe the main processes that occur during the formation of GPs [4–11]. This reaction is followed by a curing process at ambient temperature or slightly higher (lower than 100 °C) to develop a rigid 3-dimensional tetrahedral coordination network, which consists of a Si-O-Al succession bond in a semi-crystalline or amorphous structure [3,11,12].

This new generation of materials is the subject of a lot of research, especially in the last decades [13–15], owing to its wide-ranging usability in various fields such as fire and thermal protection, ceramics industry, encapsulation of radioactive and toxic waste and other applications, but also due to their growing technical, economic and

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Fig. 1. The results of the particle size distribution obtained from BSP powder.

 Table 1

 The main physical properties of Beni Saf's Pozzolan (BSP).

Particle si	ze characte	eristics	Absolute density	The Blaine's specific area		
D ₁₀ (µm)	D ₅₀ (μm)	D ₉₀ (μm)	(g/cm ³)	(cm²/g)		
1.99	14.43	42.30	2.76	6500		

environmental interest [15,16]. In civil engineering, these materials show great promise to be used as a reliable alternative binder to traditional cement, but with the additional benefit of considerably reducing greenhouse gas emissions [14-18]. Moreover, the GPs materials can sometimes be a relatively inexpensive option by comparison to conventional Portland cement [15]. The selection of raw materials for GPs synthesis depends on different technical and economic criteria such as chemical and mineralogical composition, availability, cost, reactivity and type of application [15,17]. Some research works, such as that of Bondar [9], Kani [19], Tchakoute [20], and others have proved the possibility of using NP for making GP cement, although they are few compared with the use of fly ash and metakaolin [2,21]. The mechanical, Physico-chemical, mineralogical, and microstructural properties of the manufactured GP cement differ according to several factors, including properties related to the nature of the precursor materials as the chemical and mineralogical compositions of the NP used [3,9]. Djobo et al., [21] studied volcanic ash and showed that the mechanical activation of the latter for up to 60 min improves its reactivity, which leads to a significant increase in mechanical strength (MS). Other factors associated with the alkaline activators used to formulate GPs, such as the type, nature, concentration, and further factors related to the mixing conditions and the processing system [21]. Irshida et al., [22] investigated the key parameters affecting GP concrete proprieties. They concluded that the compressive strength (CS) of the produced GP is affected by the type and concentration of the alkaline activator used, and the workability was improved when using KOH and increasing the water to NP ratio. In the recent research, González-García et al., [23] reported in their study on the effect of alkaline activator type on the evolution of geopolymerzation reaction that NP dissolved better in NaOH solution then KOH [9,23].

In Algeria, it's known that NP is more available than fly ash because power plants that use coal are considered the first source of fly ash [1,11]. However, the energy production in Algeria mainly depends on hydroelectric plants, and power plants operate on natural gas. Furthermore, coal-fired power plants are not environmentally friendly, which makes the GP made from fly ash indirectly harmful to the environment. Therefore, using an NP can provide a significant environmental advantage over fly ash-based GP. Economically, NP is less expensive than kaolin because its use is usually limited to the cement industry as an active addition to clinker with low percentages for the production of Portland cement. Nevertheless, kaolin has many applications in the manufacturing of several industrial products such as ceramics, paper, plastic, rubber. In addition, NP does not require heat treatment like kaolinite, which can give us a significant economic advantage over metakaolin-based GP.

The present research concerns the technical aspect because the physicochemical and mineralogical properties of NP vary according to the source [3], and they are controlled by many factors, such as the kind of volcanic eruption, the temperature and chemical content of the magma, the nature of the dissolved gases, the crystalline composition, and the climatic conditions of the volcano region [2,13]. Due to this diversity in the characteristics of raw materials of geological origin, such as NP, there is no systematic standard or document on the methodology or procedures for the preparation of alkaline activators with such precise parameters that allow these activators to be optimally matched to the precursor materials [13,24]. Because the quantities, molar ratios, and concentrations of activators depend on the properties and characteristics of the precursor materials themselves [3,9]. For this purpose, each precursor needs the right activator or a combination of several activators to be identified with its parameters [7,15]. In this context, and based on the previous research mentioned above, the current study selected the Beni Saf Pozzolan (BSP) from Algeria as a primary source of aluminosilicates for the production of GP. In order to establish the optimal combination of alkaline activators that corresponds to the BSP, sand content, and curing temperature, a series of experiments on GPs pastes and mortars were carried out, in both the fresh and cured state, which included workability, the CS and flexural strength (FS) of 3, 7, 28, and 90-days old test specimens. Microstructural studies were also conducted using Fourier Transform Infrared spectroscopy (FTIR) and scanning electron microscopy (SEM-EDS).

2. Materials and experimental methods

2.1. Materials used and their properties

2.1.1. Beni Saf's pozzolan (BSP) characterization

The NP used throughout this study as the base material of aluminosilicates was scoria-type. It is obtained from the Algerian company of pozzolan and building materials (SPMC spa Béni-Saf) located in the northwest of Algeria. The raw pozzolan has the form of a dark basaltic tuff. This latter was kept in a 105 °C oven for 24 h to make it more easily grindable by removing any moisture, which leads to a particle clogging phenomenon. Afterward, it was grounded in a ball mill over 75 min and screened to obtain particles with a maximum size of less than 80 µm. This treatment directly affects the particle size distribution of BSP powder, the reaction kinetics, dry and wet properties of the synthesized GP binder [25]. The BSP powder was dispersed in distilled water to perform particle size analysis using the "HORIBA LA-960" device, and the results are shown in Fig. 1. The fineness and specific gravity results of BSP powder are given in Table 1 and are measured using a Blaine automatic permeabilimeter and pycnometer, respectively.

The chemical and mineralogical composition are among the most fundamental factors in the choice of precursor materials for GP production [26]. For pozzolan, these characteristics vary from one deposit to another [13]. An X-ray fluorescence analysis (XRF) was carried out on BSP with a "Phillips MagiX-Pro PW–2440 Spectrometer" to characterize their chemical composition, and the results in the form percentages by weights of oxide compositions are reported in Table 2. As seen, the results indicate that BSP used contained 61.52% of SiO₂ and Al₂O₃ with a molar ratio of about 4.3 and contains relatively a high percentage of calcium oxide (10.35%) which can contribute to improved mechanical properties and give a higher compressive strength (CS) [9]. Nevertheless, it remained lower than 20%, so it was not considered cementitious on its own (they could not react and hydrate naturally with distilled

Table 2

Oxide composition of BSP obtained by XRF analyses (wt %).

Oxides	LOI*	SiO_2	Al_2O_3	Fe ₂ O ₃	CaO	MgO	SO_3	K ₂ O	Na ₂ O	P_2O_5	TiO ₂
BSP	8.07	44.10	17.42	8.97	10.35	3.58	0.02	1.47	3.20	0.72	2.12



Fig. 2. XRD Diffractogramme of precursor material (BSP).



Fig. 3. The morphology of BSP performed with SEM-EDS.

water) [6,24]. On the other hand, the high carbon content found in BSP, given by a high percentage of the loss on ignition (8.07%), may adversely affect the MS. The dark color on which the BSP appears is most probably due to the presence of iron oxide (Fe_2O_3), with a relatively high percentage (8.97%) by comparison with other pozzolans.

Fig. 2 shows the mineralogical analysis of the BSP, which was performed by X-ray diffraction using an Empyearn PANalytical machine, operating with a copper anode, K α radiation of wavelength λ = 1.540598 Å at 45 kV and 40 mA. The scanning of the sample was performed in continuous mode from $2\theta = 5^{\circ}$ to 75° with a step of 0.013°. The mineralogical identification of the principal and secondary crystalline phases was determined by HighScore Plus software with the ICSD database, and it includes Analcime H₂Al_{0.9}Na_{0.9}O₇Si₂ (PDF#015–9332); Anorthite Al₂Ca₁O₈Si₂, (PDF#000–0654); Diopside Ca₁Mg₁O₆Si₂, (PDF#005–2359); Hematite Fe₂O₃, (PDF#015–4191); Albite $Al_1Na_1O_8Si_3$, (PDF#010–0496). The degree of crystallinity of BSP is about 77%, and it was calculated from XRD patterns with the following formula [21]:

$$\% X_c = \frac{A_c}{A_a + A_c} \times 100 \tag{1}$$

where Ac is the crystalline phase that represents the surface of the crystalline peaks included in the interval 20 between 20 and 40° , and Aa is the amorphous phase that represents the area included between the diffractogram halo and the baseline in the same interval [21].

Images with a scanning electron microscope coupled with EDS are also performed on the pozzolan (Fig. 3) to identify its microstructure and compositions. This image shows that the pozzolan particles have irregular shapes with rather pointed tips of different sizes consisting mainly of oxygen, silicon, and aluminum atoms in a tetrahedral structure [13]. Table 3

The physical characteristics of the sand used.

parameters	values
absolute density	2.64 g/cm ³
modulus of fineness	2.65
absorption coefficient	0.2

Table 4

The mixture compositions used for the preparation of geopolymer mortar specimens.

Mixture	Sand/BSP	Si/Al molar	Si/Na molar	Water/SGP
M-1	2.50	6.1	2.00	0.37
M-2	2.75	6.1	2.00	0.37
M-3	3.00	6.1	2.00	0.37
M-4	3.25	6.1	2.00	0.37
M-5	3.00	5.2	2.00	0.40
M-6	3.00	5.5	2.00	0.40
M-7	3.00	5.8	2.00	0.40
M-8	3.00	6.1	2.00	0.40
M-9	3.00	6.1	1.25	0.37
M-10	3.00	6.1	1.50	0.37
M-11	3.00	6.1	1.75	0.37
M-12	3.00	6.1	2.00	0.37
M-13	3.00	6.1	2.25	0.37

Several research studies have suggested testing the CS of pozzolanic cement mortars to show the quality of the pozzolan. The CS tests were conducted on BSP with OPC-CEM I 42.5 to estimate its pozzolanic activity according to ASTM C311 and C618 specifications. The results indicated that the strength of the average of six samples is 96.3 and 82.79% for specimens aged 7 and 28 days, respectively. These results are well above 75%, showing great activity of BSP with Portland cement.

2.1.2. Alkalin solution

The alkaline solution used to initiate the geopolymerization reaction is constituted by a blend of dissolved sodium hydroxide commercialized with a purity of about 98% in the form of pellets supplied by the manufacturer "JSC KAUSTIK VOLOGOGRAD" and soluble sodium silicates. The latter is NASIL® 3.35 which is manufactured by the group "INDUSTRIAS QUÍMICAS DEL EBRO IQE", the proportions of their components (% by weight) are given as follows: SiO₂ = 26.4%, Na₂O = 8%, and H₂O = 65.5%. The purpose of adding sodium silicate is to improve the geopolymerization process and training of geopolymeric gel [3].

2.1.2.1. Fine aggregate and water. In this study, a sand standardized CEN EN 196–1 of the granular fraction 0/2 and siliceous nature as aggregates for the confection of mortar specimens were used. The physical characteristics are presented in Table 3. To ensure the purity of the mixture and to avoid additional effects resulting from pollution by the presence of mineral salts in the drinking water, distilled water was used either for the preparation of the alkaline solution or to adjust the workability of the mixture.

2.2. Synthesis of geopolymer mortars

2.2.1. Samples composition

The composition of 13 mixtures summarized in Table 4 is prepared to study the effect of three parameters with equal steps for each on the behavior of geopolymer mortars in both the fresh and hardened state. The compositions of the systems were modified by changing the mass ratio of Sand/BSP, the molar ratios of SiO₂/Al₂O₃, and SiO₂/Na₂O in the system, and the water to Solid of Geopolymer (SGP). Finally, to determine the most appropriate processing temperature for the mechanical strength, the optimal ratios obtained previously were used to prepare

four mixtures, each of which was maintained at a different temperature (20, 40, 60, 80 °C) until the test age. It should be noted that the mixtures of each parameter were made on the same day to keep the same synthesis conditions and that the ranges of each parameter were chosen based on previous research available in the literature.

2.2.1.1. Mixing. The specimens of geopolymer mortars were prepared using the European standard EN 196–1 [27]. At first, a low-speed mixing of sand standardized with BSP powder in a dry state for 1 min followed by the gradual addition of all the alkaline solution prepared previously and additional water if necessary for 30 sec that following, and continue mixing for 90 sec before stopping the mixer. Then the mixing bowl is removed, followed by removing the dough adhering to the bottom and sidewalls of the mixer. Then mixing the dough is resumed for an additional 90 secs at a higher speed. Adequate workability has been obtained with water to solid of GP ratio equal to 0.37, and it is increased to 0.4 if necessary.

2.2.1.2. Casting and hardening of samples. The mixture obtained is transferred to metal moulds to make prismatic samples of $40 \times 40 \times 160$ mm³ according to the EN 196-1 standard. Firstly, each interior compartment of the mould was lightly oiled before assembling them to facilitate the release of the specimens. Next, the mould, which must be securely assembled, was placed on the shock table and filled evenly with mortar up to halfway through the mould. Then, the first layer of mortar was compacted chronologically for 60 s (one shock/sec), and introduced the second layer of mortar, then continued to compacter again with 60 shocks to evacuate the air bubbles trapped inside the mould during the casting. Finally, the samples prepared were maintained in special plastic oven bags that were tightly sealed to avoid water evaporation during curing. Then, stored in an oven at 80 °C and atmospheric pressure for 24 h before demolding and returned to the oven until strength testing. The elapsed time from completion mixing to loading into the oven was approximately 4-5 min.

2.3. Testing methods

2.3.1. Workability

The workability of the mortar is one of the most important properties that needs be considered when preparing the samples.

In this research, the flowability of the GP mortar studied was evaluated by measuring the slump and spread flow test using a mini-cone with the following dimensions: $D_1 = 50 \text{ mm}$, $D_2 = 100 \text{ mm}$, and H = 150 mm; where D_1 , D_2 and H are the upper diameter, the lower diameter and the height of the cone, respectively. The test board and the inside walls of the cone were slightly moistened to minimize the friction effect. The cone was then filled with mortar in three layers of equal thickness, and then each one was packed with a metal (25 S per layer). Finally, after filling the cone and removing and over-flowing mortar from the top, the cone was lifted gradually, and the mixture was let until it settles then measurements are taken. The experiment was performed twice for each mixture, the average of two measurements was taken in two opposite directions for spread flow, and a single measurement was taken from the middle of the surface for slump.

2.3.1.1. Mechanical strength. The MS was measured using a ControLab 250/15 kN automatic compression machine, type (E0161/1), and necessary equipment following the recommendations of EN 196–1. The samples were tested at different ages of 3, 7, 28, and 90 days with a constant loading speed of about 50 \pm 10 N/sec for the FS and 2400 \pm 200 N/sec for the CS. Three samples of 4×4×16 mm³ for each mixture were averaged to estimate the FS, and then each half of the resulting prismatic sample was retained to evaluate the CS on the lateral surface of the 40×40 mm² section (average of six samples).

Table 5

The results of the CS of the GP mortar at 3, 7, and 28 days cured under different conditions.

Curing condition	processing mode (a)			processing mode (b)			processing mode (c)		
Age	3 d	7 d	28 d	3 d	7 d	28 d	3 d	7 d	28 d
CS MPa	7.8	24.7	29.5	10.1	27.7	35.2	22.4	27.6	25.3



Fig. 4. Casting of GP mortar samples $40 \times 40 \times 160$ mm.



Fig. 5. Measuring the slump and flow of the fresh GP mortar.

2.3.1.2. The microstructure analysis. To investigate the impact of various parameters on the microstructural properties of BSP-based GP, four samples were selected from the 13 previous compositions (Table 4)

for microstructural characterization with SEM-EDS and FTIR. First, a small portion of about 1 cm³ was placed in a vacuum chamber and covered with a thin layer of gold. Then the sample was transferred to the cell of an SEM "ZEISS EVO 15" coupled with EDS to take several micrographic images. Secondly, to identify the different types of bonds existing before and after the geopolymerization reaction. FTIR analyses are performed on both the powdered BSP and the selected samples of reacted geopolymer. IR spectra were taken between 400 and 4000 cm⁻¹ using an "Agilent Cary 630 FTIR" device.

3. Results and discussion

3.1. Effect of the hardening condition

At first, preliminary setting time and CS studies were performed on alkali-activated BSP under laboratory conditions. The results obtained showed that the mixture took a long time to set (initial and final setting time was 1485 and 1947 min) and has a low CS (Fig. 12). These results can be explained by the low amount of reactive aluminosilicates species (amorphous phases) having participated in the geopolymerization reaction [2,3,9], which is clearly evidenced by the mineralogical analysis (XRD) of the precursor material (see Fig. 3). Similar observations have been reported in previous research [14,20,28]. To reducing setting time and increasing mechanical properties of the synthesized GP to make it usable in structural applications, heat treatment of the latter is necessary to promote the geopolymerization reaction [29]. For this reason, the specimens were hardened under three different conditions and then examined for CS after 3, 7, and 28 days of curing in the oven at a temperature of 80 °C till to test age: a) sealed curing at room temperature for 24 h and then heat-treated in the oven at 80 °C, b) sealed curing at 80 °C, and c) curing exposed to the oven air at 80 °C. The results presented in Table 5 show that the hardening mode (b) of the specimens is most suitable to their long-term CS. See (Figs. 4-6).

3.2. Workability

3.2.1. Effect of Sand/BSP mass ratio

The appropriate amount of sand and precursor material must be taken into consideration when synthesizing the GP mortar based on NP. Fig. 7 presents the evolution of mortar workability as a function of sand to BSP mass ratio. As can be seen, both the slump and spread flow of mortars decreased gradually as the sand to BSP ratio increased. The reduction in the slump was approximately 15, 9, and 20 % when sand to BSP raised from 2.5 to 2.75, 2.75 to 3, and 3 to 3.25, respectively. On the other hand, the spread flow reduction was nearly 30, 14, and 16% when the mass ratio sand/BSP increased from 2.5 to 2.75, from 2.75 to 3, and from 3 to 3.25, respectively. Kotwal et al [30] have found similar results. The amount of sand in the mixture may explain this trend, as the sand grains absorbed a large amount of water (capillary water), which increased the water demands when the sand to PSB ratio was higher. This is due to the increased surface area of solid grains with higher amounts of sand that have a spherical shape, which means less free water in the mix. Therefore, the greater sand to precursor ratio, the more water was consumed by the sand aggregates, that impeded the flow of the GP mortar and resulted in lower workability [31]. This trend was similar to the behavior of PC mortars. These results are consistent with these reported by Bhowmich and Ghosh in their work on fly ash-based GP mortars [25].



Fig. 6. The initial and final setting time measured under laboratory conditions.



Fig. 7. The variation of the slump and the spread flow according to the mass ratio sand/BSP.

3.2.1.1. Effect of Si/Al molar ratio. The Si/Al molar ratio plays an important role in determining the rheological behavior of geopolymers [2,5,13,24]. The test results of the influence Si/Al molar ratio on the evolution of the workability of the evaluated mortars are reported in Fig.8. The latter shows that the slump increased from 97.5 to 118 mm and 118 to 124.5 mm when Si/Al molar ratio increased from 5.2 to 5.5 and 5.5 to 5.8, respectively. Similar behavior was observed with spread flow, which increased from 168 to 222 mm and 222 to 257 mm when the Si/Al molar ratio increased from 5.2 to 5.5 and from 5.5 to 5.8, respectively. This trend can be explained by the increases in the quantity of free water present in the Na₂SiO₃ solution, which increased with the Si/Al molar ratio from 5.2 to 5.8. Consequently, the higher amount of Na₂SiO₃ solution in the mixture will make it softer and more manageable [3]. Although there is a noticeable decrease in workability when the Si/Al molar ratio was increased from 5.8 to 6.1, it can be seen that the slump decreases from 124.5 to 119 mm while spread flow decreases



Fig. 8. The variation of the slump and spread the flow as a function of the Si/Al molar ratio.

from 257 to 225.1 mm. This decrease in the workability of mortar can be translated by the speed of the condensation stage and promotes the rapid formation of a three-dimensional geopolymer structure when the Si/Al molar ratio increases and reaches the threshold value, which was equal to 5.8 for adequate workability under these conditions. In other words, the increasing amount of Si(OH⁻) species in the mixture promotes the condensation step rapidly compared to the other geopolymerization steps, causing the mixture to become firmer, losing its workability [32]. Firdous et al., [2] stated that heat emitted during hydration contributes to gel condensation and revs the setting of the mixture.

3.2.1.2. Effect of Si/Na molar ratio. Fig. 9 represents the effect of the Si/Na molar ratio of alkaline solution on the workability of geopolymer mortars. In general, the variables appear to be in a direct relationship with each other. The flow and slump values increase with a higher Si/Na molar ratio. The increase in a slump was estimated to be about 10, 12, 5,



Fig. 9. The variation of the slump and spread the flow as a function of the Si/Na molar ratio.

and 11%. Whereas the increase in flow was estimated to be about 31, 12, 6, and 12% as Si/Na molar ratio increased from 1.25 to 2.25. This increase is mainly due to the decrease in the NaOH concentration, and the availability of more free water, which reduces the viscosity of the alkaline solution when the Si/Na molar ratio increases. Kotwal et al., [30] reported in their studies conducted on fly ash-based GP that the

mortar flow decreased when the amount of NaOH increased because the presence of OH⁻ ions with higher amounts leads to the acceleration of the precursor dissolution, which results in the rapid formation of the geopolymer gel. This makes the mortar more and more workable. Similar results have been reported by Bhowmich and Ghosh [25].

3.3. Mechanical strength

3.3.1. Effect of Sand/BSP mass ratio

Fig. 10 shows the results obtained by investigating the effect of precursor material (BSP) content on MS. Generally, the results reveal that CS increases by an average of 17.25% and 60.57% after 3 to 7 and 7 to 28 days of processing, respectively.

This increase can be attributed to the continuous process of geopolymerization, which occurs at a low rate during the first few days, and gradually improves until 28 days. This contributes to the forming of more geopolymeric bonds, which enhances the CS. Nevertheless, there is a little reduction in CS from 28 to 90 days of curing, estimated to be 3.55% on average. The most likely reason for this decrease is the prolonged duration of the heat treatment in the oven. The same behavior was observed for FS evolution.

In addition, an inverse relationship can be seen between MS and the sand/BSP ratio. Both CS and FS in various curing ages decrease as the Sand/BSP ratio increases. The decrease in CS can be estimated to average 3.5, 5 and 7%, while the reduction in FS is estimated to average 9, 14, and 16%, when the Sand/BSP ratio increases by 2.75, 3, and 3.25



Fig. 10. The effect of the Sand/BSP ratio on compressive strength (left) and flexural strength (right).



Fig. 11. The effect of the SiO_2/Al_2O_3 molar ratio on the mechanical strength of BSP-based geopolymer mortar, compressive strength (left) and flexural strength (right).



Fig. 12. The effect of the SiO₂/Na₂O molar ratio on the mechanical strength of BSP-based geopolymer mortar, compressive strength (left) and flexural strength (right).



Fig. 13. The effect of the curing temperature on the mechanical strength of BSP-based geopolymer mortar, compressive strength (left) and flexural strength (right).

respectively. These results explain that a low ratio between sand and BSP implies a greater presence of reagents species provided through BSP dissolution, which contributes to the formation of more bonds that will improve the MS. In other words, the MS of the samples decreases when more sand is added because the amount of gal available by polymerization may not be sufficient to surround all the sand grains to form geopolymeric bonds [22]. Although the optimal sand/BSP ratio is 2.5 for better mechanical performance, a value equal to 3 was chosen to complete the study. Firstly, for economic reasons to use lower quantities of raw materials, and secondly for technical factors that permit comparing the strength results obtained in GP-based BSP mortars with those of OPC according to the recommendations of the European standard EN196-1 [27].

3.3.2. Effect of Si/Al molar ratio

Previous research has shown that the Si/Al molar ratio strongly affects the MS of GP products [7,9]. The mixtures composition (M5 to M8) mentioned in Table 4 studies the impact of the SiO₂/Al₂O₃ molar ratio on the MS of GP mortar. This ratio was determined by dividing the number of moles of SiO₂ by that of Al₂O₃, where the amount of SiO₂ presents in both the BSP and the Na₂SiO₃ used in the alkaline solution, while the amount of Al₂O₃ presents only in the BSP. In the set of results mentioned in Fig.11, it appears that the MS increases proportionally with the SiO₂/Al₂O₃ molar ratio ranging from 5.2 to 6.1.This development in the MS can be attributed to the formation of a higher amount of Sodium Alumino-Silicate Hydrated gel phase (N-A-S-H) with each

subsequent increase in the SiO2/Al2O3 molar ratio. Considering the amount of BSP, which remains constant in all mixtures M5 to M8, any increase in the SiO₂/Al₂O₃ molar ratio means a higher sodium silicate content in the mixt. This Na2SiO3 promotes the polycondensation of dissolved species $Si(OH)_4$ and $Al(OH)_4^-$ from the surface of BSP particles, and enables the geopolymerization reaction to occur at a higher rate, [9,33]. Furthermore, the increased sodium silicate content in the mixture results in higher amounts of dissolved SiO₂ [9]. This latter actively contributes to the formation of the long-chain (N-A-S-H) gel phases, which in turn enhances the mechanical strength [3,5]. These obtained findings align with the observations from microstructural analysis using SEM-EDX (Fig.15a), where the highest Si/Al molar ratio (Si/Al = 6.1) exhibits a denser and more cohesive morphology [5,7]. On the other hand, the lowest Si/Al ratio (Si/Al = 5.2) displays a heterogeneous and less condensed microstructure (Fig. 15b). In the same context, Bondar et al. [9] suggested that using a combination of Na₂SiO₃ with NaOH as an activator will lead to the forming of long-chain oligomers compared to the use of alkali hydroxide alone because the siliceous part in the Na₂SiO₃ can react with cations, which generates a geopolymer with higher MS. Moreover, the increase of Na₂SiO₃ in the mixture stimulates the polycondensation process during a reaction. Furthermore, this makes it possible for the formation of new bonds and produces a GP with a higher MS [32].

3.3.3. Effect of Si/Na molar ratio

Sodium silicate solution is defined by the modulus of silica, which



Fig. 14. IR spectra of initial BSP and GP samples corresponding to mixtures M-1, M-5, samples cured at 20 °C, and M-9.



Fig. 15a. SEM micrographs with EDS analysis of GP mortar corresponding to M-12 mix after 90 days of curing at 80 °C.

varies between 1.6 and 3.85 [9]. The Na₂SiO₃ used in this study has a modulus of 3.35, which was modified by adding sodium hydroxide in different amounts. Fig.12 reveals the MS results of GP mortar made from BSP with different SiO₂/Na₂O molar ratios. Generally, the result showed that the MS still increases from 3 to 90 days of curing when using low molar ratios of Si/Na (1.25-1.75), while it slightly decreases after 90 days of curing when higher molar ratios are used (2 and 2.25). These results may be explained by the fact that high NaOH concentrations when using low Si/Na molar ratios, which provide a higher amount of dissolved BSP aluminosilicates and long-chain geopolymers produced even in the long term. While the use of higher molar ratios means the presence of lower NaOH concentrations, these lead to less dissolution of the aluminosilicate present in the BSP as well as the presence of a small amount of the amorphous phase, allowing to achieve the optimal MS of the produced GP in a shorter time (28 days) [9,12]. A notable decrease in CS at 28 days (about 14%) was observed when the SiO2/Na2O molar ratio was increased from 1.25 to 1.5. A similar trend was also recorded in the FS. The reason for this reduction is not fully understood yet, but may be due to the presence of a constant amount of Na₂SiO₃ in the mixture with a higher SiO₂/Na₂O ratio, which means that the alkaline activation effect is weaker, and the dissolution of the BSP particles is lower. Therefore, this reduces the MS of the produced GP [9]. There is no significant change in CS when the SiO2/Na2O molar ratio raise from 1.5 to 1.75. Although, there is a slight improvement in the FS. The stability of the MS in this range may be due to the equivalency between the dissolution and condensation rate of aluminosilicates. There is also a noticeable increase in the CS by an average of 26% when the molar ratio

SiO₂/Na₂O increases from 1.75 to 2, while it decreases to return to its initial values when the ratio rises from 2.0 to 2.25. The same behavior was observed for the FS. This was explained by the decrease in the concentration of NaOH as the molar ratio of SiO₂/Na₂O increased from 1.75 to 2. The high concentrations of NaOH in the medium lead to an increase in the alkaline solution viscosity, which hinders the mobility of the dissolved species for their condensation to form a homogeneous geopolymer gel. Moreover, the presence of higher quantities of free Na⁺ ions leads to the efflorescence phenomena (see fig. 15c), which results in the fragility of the microstructure of the GP matrix. For this reason, it is not advisable to use high concentrations of NaOH to improve MS [9]. While a ratio higher than 2, leads to partial dissolution of the aluminosilicates species of BSP due to the low concentration of NaOH used in the activation solution [9]. Therefore, the optimal silica modulus for BSP activation should be close to 2 for the best performance.

3.3.4. Effect of curing temperature

The curing temperature has a crucial role in the determination of the mechanical properties of GP, especially for the geopolymerization reaction of NP [24]. Fig. 13 indicates the results of the MS of BSP-based GP cured at different temperatures (20, 40, 60, and 80 °C). In general, the increases in the curing temperature from 20 to 80 °C show a significant improvement in MS. The higher processing temperature leads to an increase in the mobility of (OH⁻) ions, which contributes to the dissolution of higher amounts of alumina and silica from the BSP. The increased mobility of these dissolved species in the presence of elevated temperatures (80 °C) leads to an increase in the possibility that they collide



Fig. 15b. SEM micrographs with EDS analysis of GP mortar corresponding to M-5 mix after 90 days of curing at 80 °C.

with each other and help them form a strong hydrate network (N-A-S-H), which contributes to the development of MS. It is worth noting also that the MS of the samples cured at 20 °C for three days was very low to be measured by the machine because they did not have enough time to react and form a gel network (NASH) at an early age. A similar result was reported in a previous study conducted by Djobo et al., [14] on volcanic ash-based GP mortars, suggesting that the curing of this later at hightemperature plays an efficient role in the formation and condensation of the gel, resulting in a good connection between the GP gel and the aggregates and leading to a better MS. This explanation is clearly apparent in the SEM images of the sample cured at 20 °C (Fig. 15d), where a poor association between the aggregates and the GP matrix. Similarly, Ghafoori et al., [24] suggested in their study on the effect of curing conditions on the mechanical properties of NP-based geopolymers that the use of elevated temperature catalyzes the geopolymerization reaction, which significantly increases the MS. However, the use of curing temperatures above 90 °C can have adverse effects such as rapid evaporation of structural water, which induces micro cracking.

3.4. The microstructure study

3.4.1. FTIR

Fig.14 shows the FTIR spectra of the BSP and geopolymer corresponding to the compositions M-1, M-5, and M-9 of Table 3 and the samples cured at 20 °C. The first observation drawn from Fig. 13 is the presence of large bands observed in the regions of 3456 and 1636 cm⁻¹ in all spectra, and the bond observed at 2360 cm⁻¹ in spectra M-1, M-5,

and M-9 which represent stretching and bending vibrations of the O-H bonds of water. The latter comes from the absorption by BSP or geopolymers samples of atmospheric moisture [2,21]. It is also possible to observe the appearance of two peaks around 460 and 1040 cm⁻¹ in the IR spectra of BSP and the samples M-5 and M-9. These bands are due to the asymmetric expansion vibrations of Si-O-Al, and Si-O-Si of the original aluminosilicates structure [2,20], which disappear in the spectra of the sample M-1 and that cured at 20 °C. This indicates the degradation of these covalent bonds at a high rate under the pH influence of the activator. The bond localized at 1490 cm⁻¹ in the IR spectrum of BSP corresponds to the asymmetric stretching of the CO₃²⁻ group presented in carbonate minerals [9]. This is due to carbonization either by diopside absorption of atmospheric CO2 during the grinding of BSP or by the natural dissolution of CO_2 as the magma cools [21,34]. While the bond presented at 1427 cm⁻¹ in the spectrum of the samples M-5 and M-9 are attributed to the vibration of the O-C-O bonds which are formed by the phenomenon of atmospheric carbonation of unreacted Na⁺ during the geopolymerization process [2,21]. The appearance of this phenomenon mainly in these samples and not in the others (M-1 and samples cured at 20 °C) is either due to the low degree of geopolymerization (M-9) or due to the low content of reactive silicate (M-5) which plays an important role in Na⁺ fixation [21]. Another change can be observed between the IR spectra around the 778 cm^{-1} bond. This last one is attributed to the Al stretching of 6 coordination Al(VI)-OH and Al(VI)-O where it appears with relatively high intensity in the specter of BSP and sample M-9 but it is decreasing in the specters of the samples cured at 20 °C and M-5. However, this bond disappears in the IR specter of



Fig. 15c. SEM micrographs with EDS analysis of GP mortar corresponding to M-9 mix after 90 days of curing at 80 °C.

sample M-1. This change was explained by the rupture of these bonds under the effect of an electrostatic reaction of the alkaline medium [9].

3.4.2. Sem-eds

SEM coupled with EDX analysis was used to study the microstructure of the selected GP mortar after 90 days of curing. Fig. 15(a-d) represents the morphology with two enlargements and chemical compounds of the GP matrix corresponding to the mixtures M-12, M-5, M-9, and M-1 hardened at 20 °C, respectively. The samples studied by SEM-EDS show notable changes in the development of microstructures. Fig. 15a shows a homogeneous and well-packed microstructure with a few unreacted BSP particles and voids. The cracks are likely to be the result of excessive evaporation of water under the effect of curing at a relatively high temperature or due to the CS tests previously performed on the sample. Sodium crystals in the form of needles can be observed in small amounts, which is probably due to the curing of the samples at relatively high temperatures for a prolonged period [13,35]. On the other hand, Si, Na, and Al are the predominant chemical compounds that were detected according to the EDS results (Fig. 15a). These elements participate in the geopolymerization reaction to form strong Si-O-Al and Si-O-Si bonds that represent the essential components for the formation of gel sodium aluminosilicates hydrate (N-A-S-H), where Na⁺ is introduced to balance the negative charge of Al [2,21]. This gel is defined as the bonding phase in the matrix that gives it a dense and continued microstructure after condensation. These findings can explain the increase in the MS of this mixture.

Fig. 15b represents the typical microstructure of the geopolymer matrix obtained with the M-5 composition, where the lowest SiO₂/Al₂O₃ molar ratio was used in this study. A heterogeneous and poorly condensed microstructure can be observed, with a higher amount of unreacted BSP, which causes a reduction in mechanical strength. A NASH-type gel was formed but at a shorter distance and dispersed manner compared to the M12 mixture, where a higher SiO_2/Al_2O_3 molar ratio was used. These observations confirm the previous results of the mechanical behavior where the strength improves as the Si/Al molar ratio increases. These findings can be explained by the deficiency of reactive Si species provided by the sodium silicate, which would contribute to the catalysis and the formation of long chains, which in turn ensure higher coagulation of the gel (NASH). The results of the EDS analysis show a great amount of Si, which belongs to the pozzolan, but it contributes a small percentage to the formation of the gel (NASH), which is due to its non-reactive nature (chemically stable).

Fig. 15c shows the typical geopolymer microstructure corresponding to the M-9 composition, where a lower SiO₂/Na₂O molar ratio was used (1.25). The microstructure of this sample appears looser and sparser than M-12, with the appearance of more micro-cracks and voids. These observations can be explained by the evaporation of water before the end of the reaction or by the incomplete condensation of the gel (N-A-S-H) due to the saturation of the medium by high amounts of Na₂O, which prevents its achievement. Another change can be observed in the SEM micrographs of this sample compared to that of M-12. Where micro-crystals in the form of needles appeared. That is a result of excess



Fig. 15d. SEM micrographs with EDS analysis of GP mortar corresponding to M-1 mix after 90 days of curing at 20 °C.

NaOH, which does not fully contribute to the formation of the N-A-S-H gel during the geopolymerization process [13]. The results of the EDS analyses are consistent with the observations made above, where a small amount of Si and Al formed for gel (NASH) compared to the amount of Na in the medium. Yadollahi et al., [35]. have reported similar findings in their study on pumice-based geopolymers.

The curing temperature generally has a significant effect on determining the mechanical strength properties of geopolymers as well as their microstructure. Fig. 15d shows the surface of the geopolymer mortar sample corresponding to the M-1 mixture, curing at room temperature (20 °C). As can be seen, the microstructure of this sample appears loose and less adherent to each other compared to sample M-12. A higher amount of unreacted BSP appeared as particles of different shapes and sizes with a light grey color. In addition, the absence of cohesion between the geopolymer matrix and the sand particles explains the low MS results recorded in the preliminary study. The results of the elemental analysis carried out by EDS in the regions located in the GP matrix also showed the presence of the elements Si and Al, but with lower quantities, especially for Na, which indicates that it prevents the formation of alkali aluminosilicates gel through BSP geopolymerization reaction at low temperatures.

4. Conclusion

This work investigates the feasibility of using Algerian natural pozzolan (BSP) as a precursor material with a mixture of NaOH and Na₂SiO₃ as an alkaline activator to produce geopolymer mortar. The

effects of the key factors such as hardening conditions, sand content, SiO₂/Al₂O₃, and SiO₂/Na₂O molar ratios in the alkali solution, as well as curing temperature, on the basic BSP-based geopolymer proprieties including the compressive and flexural strength, workability, and microstructural configuration. Based on the results obtained through this study, the following can be concluded:

- XRF analysis shows that the BSP powder has a significant amount of SiO₂ and Al₂O₃ (>61%), which are necessary for geopolymers synthesis. However, XRD analysis reveals that BSP contains over 77% crystalline phases, which are unsuitable for the geopolymerization reaction.
- Due to the low reactivity and mineralogical composition of the BSP, the ambient conditions are not sufficient for the synthesis of geopolymers with adequate MS especially at early age, and increasing the curing temperature up to 80 °C was necessary.
- The sand/BSP mass ratio of 2.5 provided better workability and higher mechanical strength. Its increase up to 3.25 reduced significantly the workability and slightly the MS.
- The Si/Al molar ratio of 5.8 gives optimum workability, and incrementally increasing it from 5.2 to 6.1 enhances the MS, this latter was confirmed by the observation of a more cohesive microstructure at a higher ratio.
- Despite increasing the Si/Na molar ratio from 1.25 to 2.25 gradually improves the workability of GP mortars, the optimum MS is achieved with a ratio of 2.

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- The microstructure analysis reveals that a lower ratio of 1.25 leads to the formation of needle-shaped crystals, which can be attributed to Na₂CO₃.
- Increasing the curing temperature up to 80 °C improves the reactivity of the BSP and enhances the N-A-S-H gel production through the geopolymerization process, which results in a geopolymer with higher MS.
- The microstructure analysis shows that increasing the curing temperature up to 80 °C enhances the reactivity of the BSP and promotes the formation of N-A-S-H gel through the geopolymerization process, leading to the production of a geopolymer with a higher MS.

Finally, this study represents the first stage in using the Algerian natural pozzolan for GP synthesis. It can be concluded that the use of BSP with Na_2SiO_3 and NaOH as chemical activators enables the production of GP binders with rheological and mechanical properties comparable to those of OPC. Indeed, additional work is required including studying reaction kinetics; improve the BSP reactivity through the production of one-part GP binders, BSP calcination, or the addition of other reactive Al_2O_3 -rich substances such as metakaolin.

CRediT authorship contribution statement

Oualid Mahieddine Hamdi: Methodology, Formal analysis, Conceptualization, Investigation, Writing – original draft, Writing – review & editing. **Ali Ahmed-Chaouch:** Testing, Supervision, Visualisation, Writing – review & editing. **Messaoud Saïdani:** Writing – review & editing, Validation, Formal analysis, Investigation. **Hichem Alioui:** Review & editing, Validation, Investigation.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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