



# Feasibility of producing sustainable geopolymer composites made of locally available natural pozzolan

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## Abstract

The feasibility of using locally available natural pozzolanic materials as complete replacement of cement binder to produce sustainable geopolymer concrete was investigated. Compressive strength, flowability, and microstructure of geopolymer mortar were measured and compared to highlight the effect of various parameters such as sand/pozzolan ratio, water/pozzolan ratio, type and concentration of activators, presence of soluble silicates, and curing age on the behavior of natural pozzolan-based geopolymer mortar. Two types of activators, namely sodium and potassium hydroxides were used as an activating solution for geopolymerization. Experimental results revealed that the compressive strength of natural pozzolan-based geopolymer mortars was affected by the type and concentration of activators. The strength was enhanced with curing ages and presence of soluble silicates but reduced with increasing water/pozzolan ratio. The flowability of geopolymer mortar increased with increasing the water/pozzolan ratio and presence of soluble silicates but decreased with increasing the sand/pozzolan ratio. SEM images showed a well-packed and homogenous microstructure of geopolymer mortar.

**Keywords** Geopolymer · Compressive strength · Flowability · Microstructure · Natural pozzolan

## Introduction

Concrete is the most commonly used material for construction in the world. In the manufacture of concrete, ordinary Portland cement (OPC) represents the traditional used binder material. However, the process of OPC production is a serious contributor to the atmospheric pollution. Production of one ton of cement releases about one ton of carbon dioxide (CO<sub>2</sub>) into the atmosphere which represents 5–7% of the global man-made CO<sub>2</sub> emissions [1]. In fact, the major sources of CO<sub>2</sub> are calcination of calcium carbonates under high temperatures during the manufacturing process and fuel combustion needed to generate the required high temperature. Therefore, development of more sustainable alternative binders is necessary to reduce the carbon footprint of concrete production. Recently, a new environmental friendly binder has come to attention. This binder is an alkaline-activated material called geopolymer. Contrary to OPC, geopolymer cement does not depend on calcining calcium

carbonate; hence it can reduce the CO<sub>2</sub> emissions by up to 90% [2, 3]. Moreover, geopolymer concrete can provide comparable strength workability and durability properties as compared with conventional OPC concrete.

Geopolymers usually are manufactured by combining source materials with high composition of silica and alumina with strong alkali activators (potassium or sodium hydroxides) in addition to sodium/potassium silicates. The source materials are either naturally available (like volcanic tuffs and pozzolan) or as by-product (like slag and fly ash). The properties of geopolymer concrete are very sensitive to wide range of parameters such as source materials composition, curing conditions, aggregate–binder ratio, and activator concentrations and dosage [4–23]. Many studies had been conducted recently to explore the behavior of geopolymer mortar. Nadoushan et al. [8] studied the effect of activator type and concentration on the behavior of geopolymer made of natural pozzolan and slag. They found that the geopolymer composite prepared with KOH has higher flowability and compressive strength compared to specimens containing NaOH solution. Guades [14] investigated the effect of sand/fly ash ratio on the compressive and tensile strengths of fly ash-based geopolymer mortar. The results showed that both strengths were decreased with increasing sand/fly ash

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ratio at a ratio between 0 and 1.5. Moon et al. [18] investigated the feasibility of producing pozzolan-based geopolymers prepared with sodium hydroxide and sodium silicate. They found that partial substitution of sodium hydroxide with sodium silicate solution leads to the enhancement of compressive strength of the specimens. Djobo et al. [11] investigated the durability and compressive strength of volcanic ash-based geopolymer mortars. They found a maximum strength of 37.9 MPa for specimens cured at 80 °C for 90 days. Chindaprasirt et al. [5] investigated the properties of class C fly ash-based geopolymer mortar prepared with different sand to fly ash ratios. They found that the compressive strength of specimen prepared with a ratio of 2.75 and cured at 70 °C for 3 days was 52 MPa. Extended the curing process at elevated temperatures reduced the compressive strength. Temuujin et al. [24] reported that optimizing the dosage of activator can attain the compressive strength of geopolymers made of fly ash with high level of aggregate. Other studies used the bottom ash to produce geopolymer mortar with comparable properties [4, 25]. Moreover, Brough et al. [26] prepared slag-based geopolymer mortars. The mortar gained strength of almost 40 MPa at water to binder ratio of 0.42. They also found that mortars activated with sodium silicate owned higher compressive strength compared to KOH-activated mortars. Yang et al. [27] reported that the flowability of geopolymers increased with the decrease of aggregate to binder ratio and increase of water to binder ratio.

The above literatures reveal that the behavior of geopolymers made of fly ash or slag has been widely investigated, but relatively fewer studies were focused on natural pozzolan-based geopolymers. This study explores the feasibility of using locally available natural pozzolan to produce sustainable geopolymer concrete. The effect of different parameters such as sand/pozzolan ratio, water/pozzolan ratio, type and concentration of activators, presence of soluble silicates, and curing age on the compressive strength and flowability of natural pozzolan-based geopolymer mortar is experimentally investigated. The microstructure of the geopolymer composites was also investigated using scanning electron microscopy (SEM) approach.

## Materials and experimental procedures

### Material properties

The natural pozzolanic material used in this work was collected from the Jordanian Desert located at the northern–eastern region of Jordan. The natural pozzolanic material was then ground in a ball mill at the Jordan University of Science and Technology Laboratory to have finer powder. To identify the microstructure (Fig. 1) and the particle size distribution (Fig. 2) of the natural pozzolanic

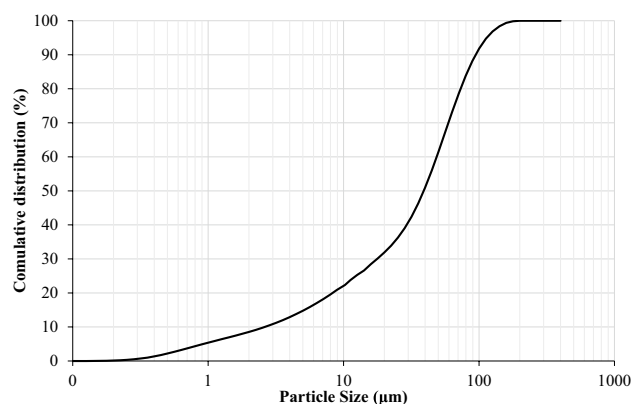


Fig. 1 Particle size distribution of the ground natural pozzolan

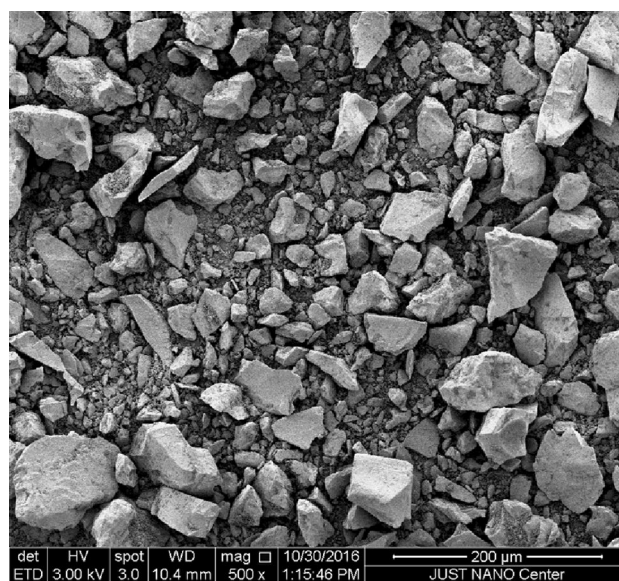
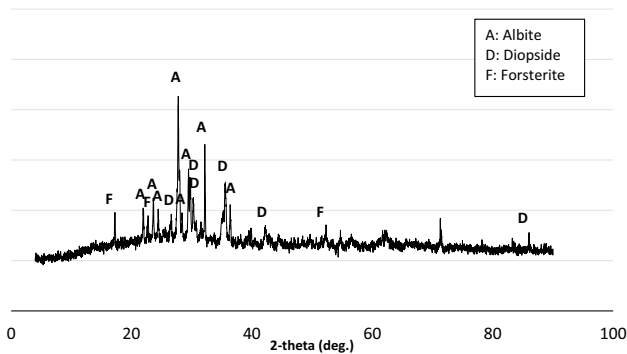


Fig. 2 Scanning electron microscopy (SEM) image of natural pozzolan used in this study

materials, scanning electron microscopy (SEM) imaging and sieve analysis test were performed. In addition, the chemical composition of the natural pozzolan was determined using MagiX X-ray fluorescence (XRF) spectrometer and summarized in Table 1. According to the ASTM C618 standards, it can be classified as class N natural pozzolan. The mineralogical composition of the pozzolan was also determined using Ultima IV X-ray diffractometer (XRD) and shown in Fig. 3. Commercially available sodium hydroxide and potassium hydroxide in solid flakes formed in addition to sodium silicates in soluble form were used as an activating solution for geopolymerization. Locally available silica sand with specific gravity of 2.59 was used to prepare the mortar specimens.

**Table 1** Chemical composition of natural pozzolan

Chemical component	(%)
SiO <sub>2</sub>	39.74
Al <sub>2</sub> O <sub>3</sub>	13.31
CaO	10.53
Fe <sub>2</sub> O <sub>3</sub>	12.68
MgO	4.92
Na <sub>2</sub> O	3.04
TiO <sub>2</sub>	2.43
K <sub>2</sub> O	1.17
P <sub>2</sub> O <sub>5</sub>	0.31
MnO	0.16
Other	11.71

**Fig. 3** XRD patterns of natural pozzolan used in this study

### Mixture design proportions

A total of 80 mixtures were made to investigate the effect of five factors related to the flowability and compressive strength of geopolymer mortar, namely sand to pozzolan ratio ( $s/p$ ), water to pozzolan ratio ( $w/p$ ), type and concentration of activators, soluble silica to pozzolan ratio ( $ss/p$ ), and curing age. The  $s/p$  ratio and  $w/p$  ratio were chosen in the range of 1.75–3.00 and 0.35–0.50, respectively. Two types of activators were used: sodium hydroxides and potassium hydroxides. The hydroxide content was calculated as weight ratio of pozzolan and was ranged as 0.08–0.016. In the case of using soluble silicate, the content was calculated as a weight ratio of pozzolan (0.08–0.14). Table 2 shows the mixing proportions of all mixtures.

### Mixing procedure and specimen preparation

The alkali hydroxides in addition to sodium silicates were dissolved in water in specific amounts. The solution was then heated to allow faster dissolving of silicates and then let cool in room temperatures for 2 h prior to use. After that, the geopolymer mortars were prepared by adding the alkaline solution to the pozzolan and blending for 2 min. Then, the

silica sand was added and the whole mixture was mixed for 3 min. After mixing, the flow of the fresh geopolymer was measured according to the ASTM C1437. The geopolymer mortar was then casted into 50 mm cubic molds to measure the compressive strength. All specimens were cured at room temperature for 24 h then in the oven at 80 °C for additional 24 h. After that the cured specimens were demolded and insulated in plastic bags and kept at room temperature until the test was conducted.

### Testing procedures

Compressive strength test was conducted on specimens cured for 3, 7, and 28 days using automatic testing machine (ELE International) with a capacity of 250 kN. Three identical samples for each mixture were tested according to ASTM C109. In addition, SEM images were captured for various specimens using QUANTA FEG 450, FEI Machine to investigate the microstructure of the geopolymer mortar. The selected samples were cut into 10-mm squares and then coated with gold prior testing.

## Results and discussion

### Effect of sand to pozzolan ratio ( $s/p$ )

#### Compressive strength

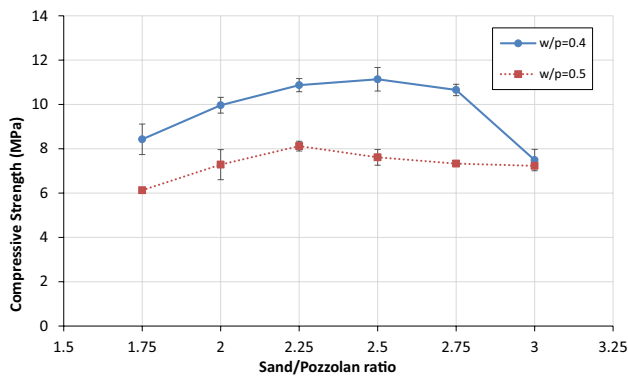
Figure 4 shows the 28-day compressive strength of sodium-activated specimens prepared with different  $s/p$  ratios and  $w/p$  ratios. The figure reveals that the compressive strength increases with increasing the  $s/p$  ratio up to a certain value then decreased. The maximum strength was measured at sand/pozzolan ratio of 2.5. This behavior can be explained as follows: adding sand to the mix increases its strength because the sand particles have higher strength than the binder itself. With adding extra amount of sand, the amount of gel formed due to the geopolymerization process may not be enough to coat and glue all sand particles resulting in strength reduction [14]. On the other hand, Fig. 5 shows the 28-day compressive strength of potassium-activated specimens prepared with different  $s/p$  and  $w/p$  ratios. The figure shows that the compressive strength decreases with increasing the  $s/p$  ratio regardless of the  $w/p$  ratio.

#### Flowability

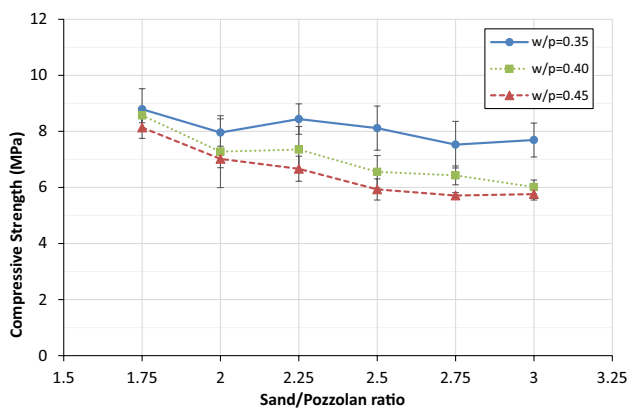
The effect of  $s/p$  ratio on the flow of geopolymer mortar activated with either sodium or potassium hydroxides is presented in Fig. 6. It is clear that the flowability of the

**Table 2** Mixing proportions of geopolymer mortar

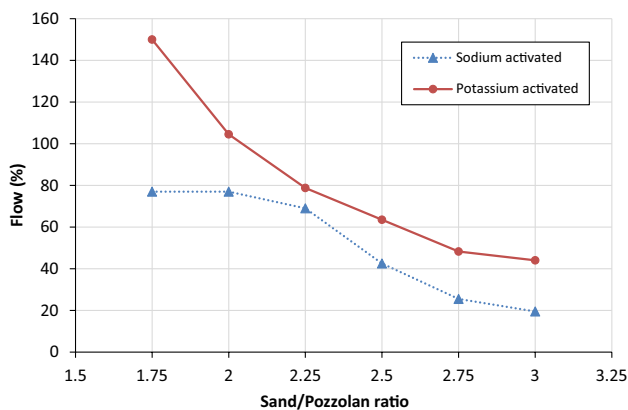
Mix #	s/p	w/p	NaOH/p	KOH/p	ss/p	Mix #	s/p	w/p	NaOH/p	KOH/p	ss/p
M1	1.75	0.40	0.10	–	0.10	M50	2.50	0.45	–	0.10	0.10
M2	2.00	0.40	0.10	–	0.10	M51	2.75	0.45	–	0.10	0.10
M3	2.25	0.40	0.10	–	0.10	M52	3.00	0.45	–	0.10	0.10
M4	2.50	0.40	0.10	–	0.10	M53	1.75	0.35	–	0.08	0.10
M5	2.75	0.40	0.10	–	0.10	M54	1.75	0.35	–	0.10	0.10
M6	3.00	0.40	0.10	–	0.10	M55	1.75	0.35	–	0.12	0.10
M7	1.75	0.45	0.10	–	0.10	M56	1.75	0.35	–	0.14	0.10
M8	2.00	0.45	0.10	–	0.10	M57	1.75	0.35	–	0.16	0.10
M9	2.25	0.45	0.10	–	0.10	M58	1.75	0.35	–	0.12	0.08
M10	2.50	0.45	0.10	–	0.10	M59	1.75	0.35	–	0.12	0.10
M11	2.75	0.45	0.10	–	0.10	M60	1.75	0.35	–	0.12	0.12
M12	3.00	0.45	0.10	–	0.10	M61	1.75	0.35	–	0.12	0.14
M13	1.75	0.50	0.10	–	0.10	M62	1.75	0.35	–	0.12	0.16
M14	2.00	0.50	0.10	–	0.10	M63	2.50	0.40	0.10	–	0.10
M15	2.25	0.50	0.10	–	0.10	M64	2.50	0.40	0.10	–	0.10
M16	2.50	0.50	0.10	–	0.10	M65	2.50	0.40	0.10	–	0.10
M17	2.75	0.50	0.10	–	0.10	M66	2.50	0.40	0.10	–	0.10
M18	3.00	0.50	0.10	–	0.10	M67	2.50	0.40	0.10	–	0.10
M19	2.25	0.45	0.08	–	0.10	M68	2.50	0.35	0.10	–	0.10
M20	2.25	0.45	0.10	–	0.10	M69	2.50	0.35	0.10	–	0.10
M21	2.25	0.45	0.12	–	0.10	M70	2.50	0.35	0.10	–	0.10
M22	2.25	0.45	0.14	–	0.10	M71	2.50	0.35	0.10	–	0.10
M23	2.25	0.45	0.16	–	0.10	M72	2.50	0.35	0.10	–	0.10
M24	2.25	0.45	0.10	–	0.08	M73	1.75	0.35	–	0.16	0.14
M25	2.25	0.45	0.10	–	0.10	M74	1.75	0.35	–	0.16	0.14
M26	2.25	0.45	0.10	–	0.12	M75	1.75	0.35	–	0.16	0.14
M27	2.25	0.45	0.10	–	0.14	M76	1.75	0.35	–	0.16	0.14
M28	2.25	0.45	0.10	–	0.16	M77	1.75	0.35	–	0.16	0.14
M29	1.75	0.40	–	0.17	0.10	M78	1.75	0.30	–	0.16	0.14
M30	2.00	0.40	–	0.17	0.10	M79	1.75	0.30	–	0.16	0.14
M31	2.25	0.40	–	0.17	0.10	M80	1.75	0.30	–	0.16	0.14
M32	2.50	0.40	–	0.17	0.10						
M33	2.75	0.40	–	0.17	0.10						
M34	3.00	0.40	–	0.17	0.10						
M35	1.75	0.40	–	0.10	0.10						
M36	2.00	0.40	–	0.10	0.10						
M37	2.25	0.40	–	0.10	0.10						
M38	2.50	0.40	–	0.10	0.10						
M39	2.75	0.40	–	0.10	0.10						
M40	3.00	0.40	–	0.10	0.10						
M41	1.75	0.35	–	0.10	0.10						
M42	2.00	0.35	–	0.10	0.10						
M43	2.25	0.35	–	0.10	0.10						
M44	2.50	0.35	–	0.10	0.10						
M45	2.75	0.35	–	0.10	0.10						
M46	3.00	0.35	–	0.10	0.10						
M47	1.75	0.45	–	0.10	0.10						
M48	2.00	0.45	–	0.10	0.10						
M49	2.25	0.45	–	0.10	0.10						



**Fig. 4** 28-day compressive strength of sodium-activated specimens prepared with different *s/p* ratios and *w/p* ratios

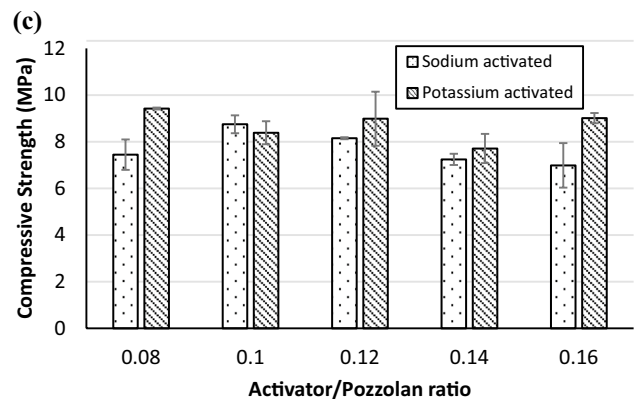
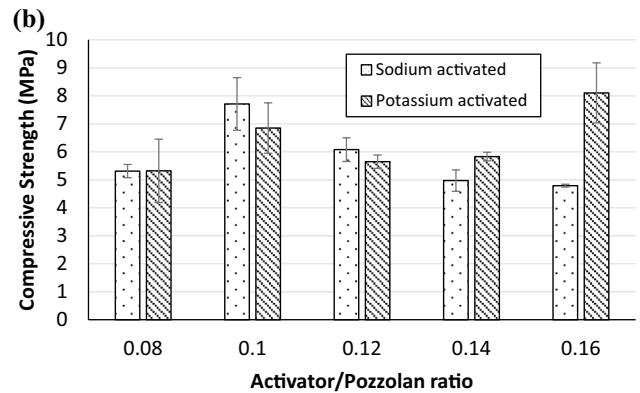
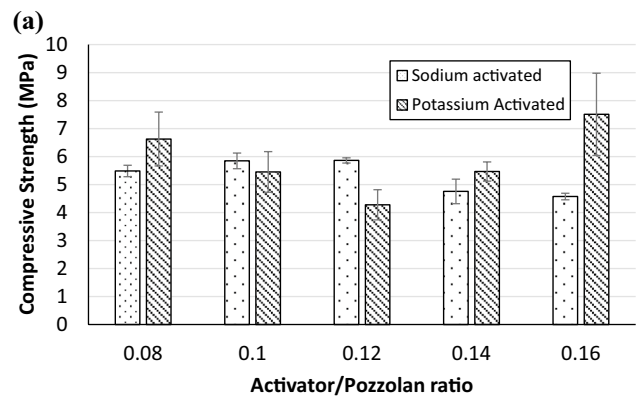


**Fig. 5** 28-day compressive strength of potassium-activated specimens prepared with different *s/p* ratios and *w/p* ratios



**Fig. 6** Flow of geopolymer mortars prepared with *w/p* ratio of 0.4 and different *s/p* ratios

geopolymer mortar decreases with increasing the *s/p* ratio regardless the type of activator. This finding agrees with results reported in [4, 5].



**Fig. 7** Compressive strength of geopolymer mortars prepared with different activators type and concentrations cured at **a** 3 days, **b** 7 days, **c** 28 days

### Effect of alkali solution type and concentration

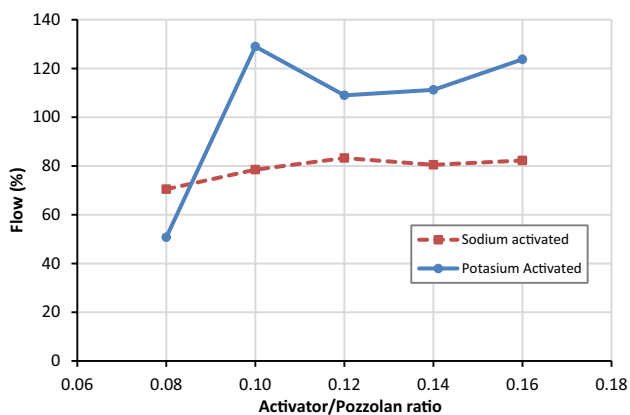
#### Compressive strength

In general, alkali solutions play a major role in the geopolymerization process and thus have great influence on the mechanical properties of geopolymer mortar. Figure 7 shows the compressive strength of geopolymer mortars activated using different amount of either sodium or potassium hydroxides. Five different concentrations of NaOH or KOH were used throughout the experimental work. It is noted that, using low concentration of NaOH was not sufficient

to produce strong chemical reaction ended in lower strength mortars. By increasing the amount of sodium hydroxide, the compressive strength of mortar specimens was increased. This improvement may be accredited to the higher degree of Si and Al leaching. By adding extra amount of sodium hydroxides, the compressive strength was decreased. This reduction could be accredited to the extra hydroxide ions which caused the precipitation of aluminosilicate gel at very early ages [7, 9]. Same behavior was observed for specimens cured at 3, 7, and 28 days, and mentioned in the literatures [7–9, 28]. Figure 7 also indicates that, for high concentration of hydroxides activators ( $A/P$  equals 0.14 and 0.16), the compressive strength of potassium-activated specimens is higher than that of sodium-activated specimens. The difference in the activation potential between NaOH and KOH may be due to the difference in ionic diameter between potassium and sodium.

### Flowability

Figure 8 shows the flow of geopolymer mortars activated using different amount of either sodium or potassium hydroxides. Five different concentrations of NaOH or KOH were used throughout the experimental work. It is clear that the flowability of natural pozzolan based geopolymer mortars activated with KOH is higher than that of specimens activated with NaOH. This finding may be accredited to many reasons such as: the KOH activating solution is less viscous than the NaOH solution [8], the difference in ionic diameter between sodium and potassium [29], and the difference in the surface hydrolysis of the aluminosilicate particles present in the raw material in the case of using KOH compared to NaOH [30, 31]. Moreover, for sodium-activated specimens, the figure reveals that the flow of the mortars increases with increasing the NaOH content.

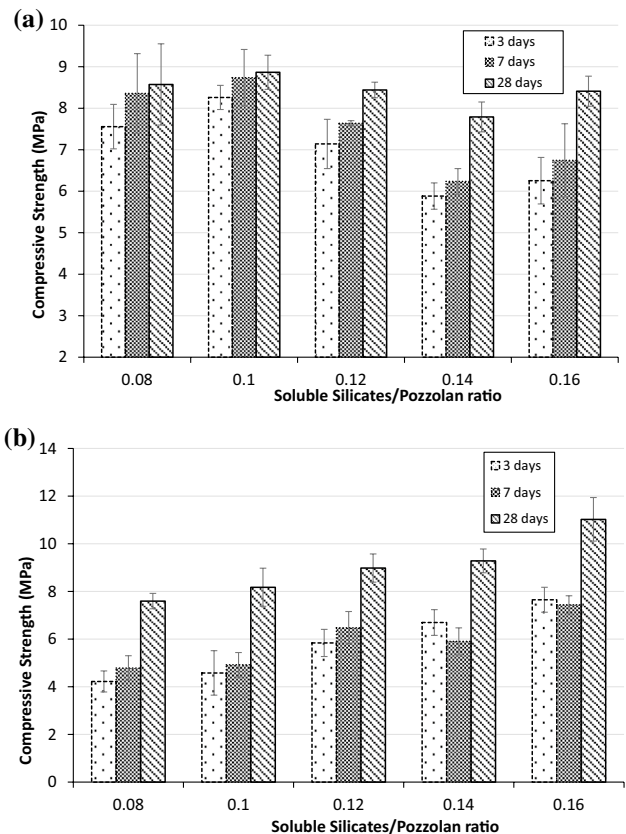


**Fig. 8** Flow of geopolymer mortars prepared with different types and concentrations of activators

## Effect of using soluble silicates

### Compressive strength

The alkali activating solution could have an additional source of silica such as silica gel or sodium silicate. In this work, different amount of soluble silicates were added to the alkali solution. It is clear that the compressive strength of sodium-activated geopolymer mortar was increased to a certain limit then decreased with adding extra soluble silicates to the activating solution as shown in Fig. 9a. This finding agrees with the literatures [6, 17]. The addition of low content of soluble silicates resulted in dense geopolymeric gel thus enhance the strength [6]. Moreover, using high content of soluble silicates resulted in a reduced skeletal density of the geopolymer gel [6] and increased the quantity of unreacted particles providing defect locations [17]. On the other hand, the presence of soluble silicates in the potassium-activated geopolymer mortar increased their compressive strength as shown in Fig. 9b. This enhancement could be attributed to the fact that the extra amount of silicates improves the dissolution rate of Si and Al thus



**Fig. 9** Effect of using soluble silicates on the compressive strength of geopolymer mortars, **a** sodium activated, **b** potassium activated

increase the degree of geopolymerization and enhance the strength [8].

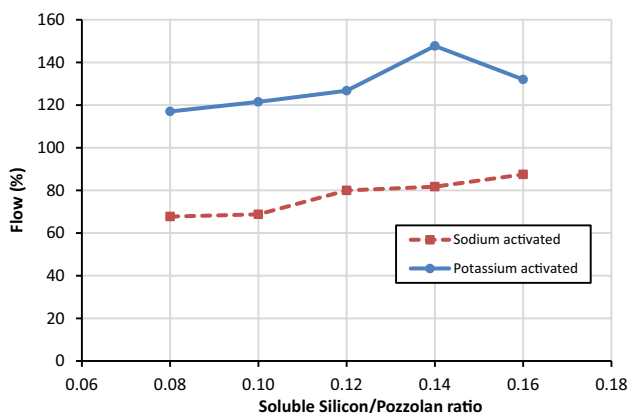
### Flowability

Figure 10 shows the flow of geopolymer mortars activated with sodium or potassium hydroxides and containing different contents of soluble silicates. It is clear that adding soluble silicates enhances the workability of the geopolymer mortars regardless of the alkali activator type. The flow of the mortars increased with increasing the added amount of silicates. This may be attributed to fact that the silicate solution enhances the dissolution of raw material in alkaline environment, thus improving the workability [8].

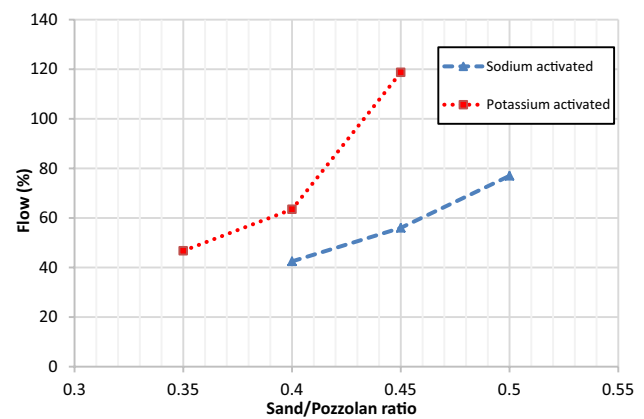
### Effect of water to pozzolan ratio ( $w/p$ )

#### Compressive strength

In geopolymerization process, water does not contribute directly to the chemical reactions. The role of water is mainly to provide the required workability to the geopolymer mix and to act as transport medium between the aluminate ions and dissolved silicates [5]. However, it was noticed that the compressive strength of geopolymer mortar activated by either sodium or potassium oxides decreases with increasing the  $w/p$  ratio as shown in Figs. 4 and 5, respectively. The effect of the  $w/p$  ratio on the compressive strength was more significant in the case of using sodium hydroxides rather than potassium hydroxides. These findings agree with the results reported in [12].



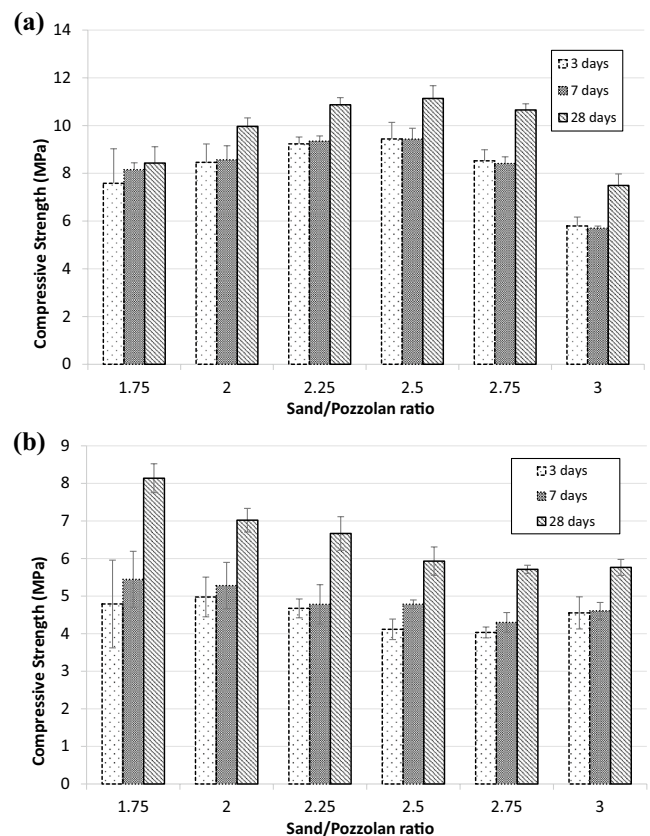
**Fig. 10** Effect of using soluble silicates on the flow of geopolymer mortars



**Fig. 11** Flow of geopolymer mortars prepared with  $s/p$  ratio of 2.5 and different  $w/p$  ratios

### Flowability

Figure 11 reveals that the flowability of geopolymer mortar activated by either sodium or potassium oxides increases with increasing the  $w/p$  ratio. This enhancement



**Fig. 12** Effect of curing age on compressive strength of geopolymer mortar, **a** sodium activated, **b** potassium activated

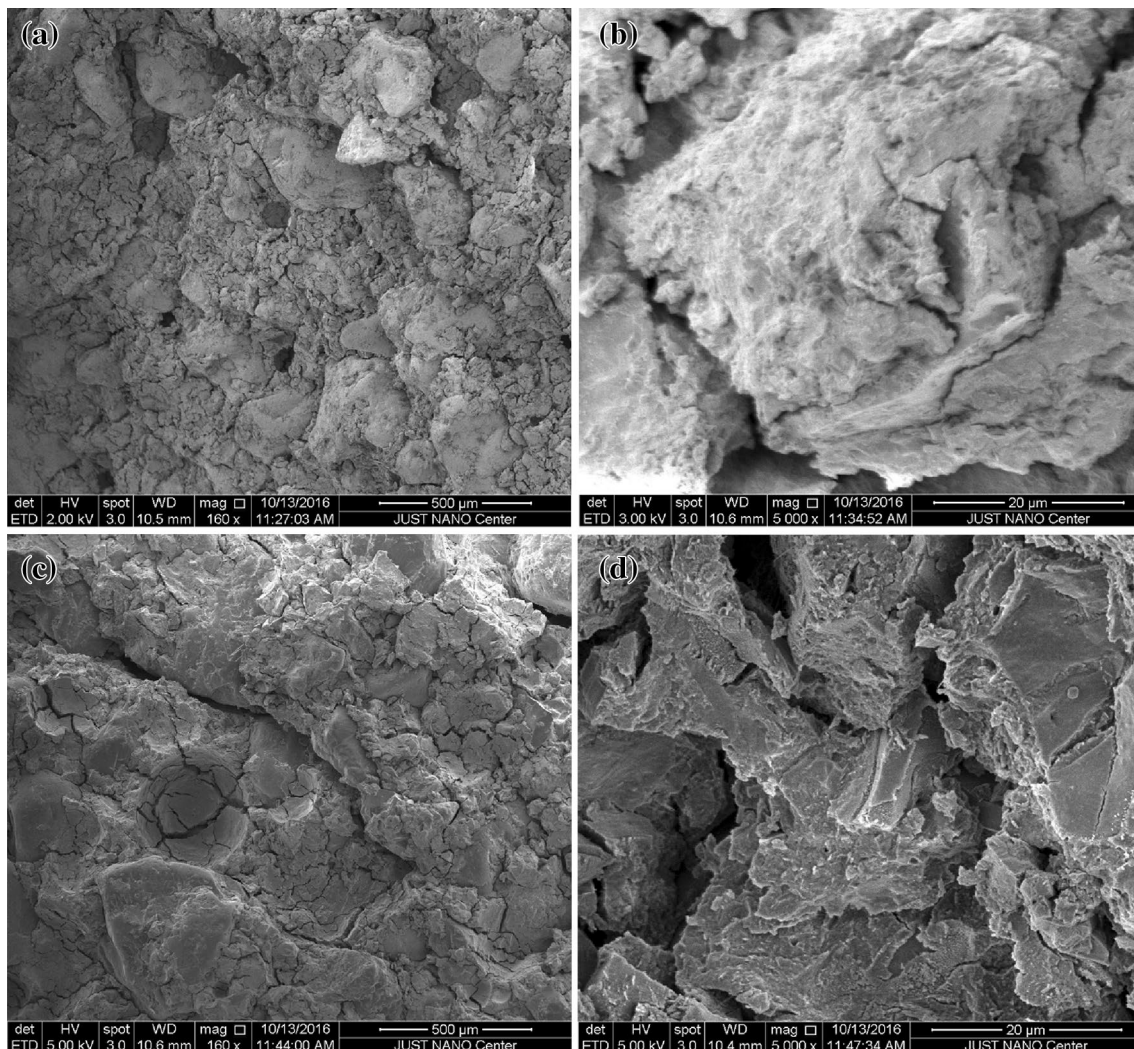
in flowability may be attributed to the increase in the free water which has no role in the chemical reactions [15].

### Effect of curing age

Figure 12 highlights the effect of curing age on the compressive strength of sodium- and potassium-activated geopolymer mortar prepared with 0.4  $w/p$  ratio. The figure reveals that the compressive strength of natural pozzolan-based geopolymer mortar developed with curing time [8, 14, 18]. This development is more considerable in the case of potassium-activated than sodium-activated specimens. Moreover, the results indicate that the  $s/p$  ratio insignificantly affected the strength increasing pattern with curing ages. The limited strength development with time could be attributed to the rapid strength gain rate of geopolymer mortar [8].

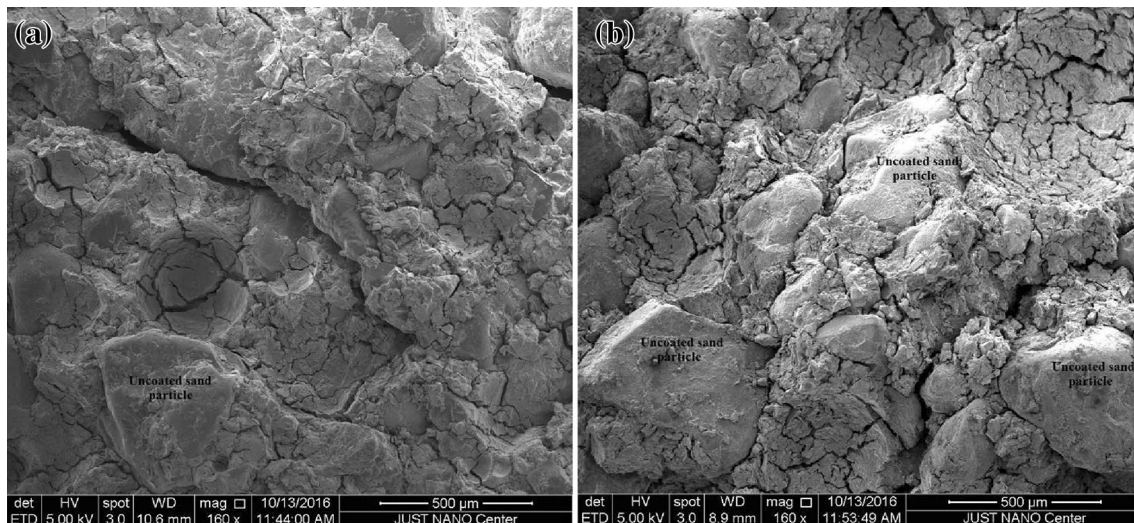
### Scanning electron microscopy (SEM) imaging

The effect of water content and concentration of sodium hydroxides on the microstructure of natural pozzolan-based geopolymer mortar were investigated using SEM imaging. Figure 13 shows SEM micrographs of selected sodium-activated geopolymer mortar specimens prepared with different water content. A well-packed and dense microstructure was observed. The microcracks' intensity and the presence of the voids were noticed to be increased with increasing water content. These observations may explain the reduction in the compressive strength. Figure 14 shows SEM micrographs of selected sodium-activated geopolymer mortar specimens prepared with two different NaOH concentrations. More homogenous and denser microstructure was observed when increasing the NaOH content. In addition, more uncoated sand particles were observed within specimens prepared



**Fig. 13** SEM micrographs of natural pozzolan-based geopolymer mortar at different magnification factors prepared with  $w/p$  ratio of 0.45 (a) and (b) and 0.5 (c, d)





**Fig. 14** SEM micrographs of natural pozzolan-based geopolymer mortar prepared with NaOH/ $p$  ratio of **a** 8%, **b** 10%

with lower NaOH concentration. These findings explain the enhancement in compressive strength with increasing the sodium oxide content. Finally, it is important to mention that the SEM imaging process clearly explains the effect of the studied parameters on the microstructure of the specimens. However, the images had to be taken at a specific (very small) area; thus they may not perfectly show the attitude but at least give an indication about it.

## Conclusions

An experimental study was conducted herein to investigate the behavior of natural pozzolan-based geopolymer composites. The effect of various factors on its compressive strength, flowability, and microstructure was studied. Based on the results, the following conclusions are drawn:

1. The compressive strength of natural pozzolan-based geopolymer mortar containing sodium hydroxide increased with increasing the  $s/p$  ratio up to a certain value and then decreased, whereas the compressive strength of potassium-activated specimens decreased with increasing the  $s/p$  ratio.
2. The flowability of the pozzolan-based geopolymer mortar decreased with increasing the  $s/p$  ratio regardless the type of activator.
3. Increasing  $w/p$  ratio decreased the compressive strength, but increased the flowability of pozzolan-based geopolymer mortar activated by either sodium or potassium oxides. The influence was more significant in the case of using sodium oxides rather than potassium oxides.

4. The compressive strength of natural pozzolan-based geopolymer mortar developed with curing time. This development is more considerable in the case of potassium-activated than sodium-activated specimens.
5. For low concentration of oxide activators, the compressive strength of sodium-activated specimens is higher than that of potassium-activated specimens. On the contrary, opposite conclusion was observed in the case of using high concentration of oxide activators.
6. The flowability of natural pozzolan-based geopolymer mortar containing KOH is higher than identical NaOH.
7. The presence of soluble silicate in the pozzolan-based geopolymer mortar enhances its flowability and thus increases its compressive strength.

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