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Strength development of ground perlite-based geopolymer mortars

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Abstract. Raw perlite is a volcanic alumino-silicate and is used as aggregate in the construction industry. The high silica and alumina contained in the raw perlite allows the production of geopolymer mortar with the help of alkaline solutions. In this study, different geopolymer mortars are obtained by mixing ground perlite (GP), sodium hydroxide (NaOH), water and CEN standard sand and the strength and microstructure of these mortars are investigated. Mortar specimens are placed in the oven 24 hours after casting and kept at different temperatures and times, then the specimens are cured under laboratory conditions until the day of strength tests. After curing, unit weight, ultrasound pulse velocity, flexural and compressive strengths are determined. Experimental results indicate that the mechanical properties of the mortars enhance with increasing oven-curing period and temperatures as well as increasing NaOH molarity. In addition, SEM/EDS and XRD analyses are performed on the mortar specimens and the results are interpreted.

Keywords: geopolymer; ground perlite; oven curing; mortar; strength; microstructure

1. Introduction

Economic and environmental problems arise as a result of Portland cement production, which has led to an efficient investigation of more economic and environmentally harmless binders (Çelikten *et al.* 2019, Duxson *et al.* 2007, Hossain *et al.* 2015). Geopolymers, as the alternatives for these binders, are generally obtained by the formation of alumina silicate as a result of dissolution-precipitation reactions using solutions (Provis 2014, Çelikten *et al.* 2019, Duxson *et al.* 2007, Hossain *et al.* 2015). Today, researchers are working on the production of cement-free binders by mixing pozzolans with different solutions (Hojati and Radlinska 2017, Provis and Bernal 2014, Sarıdemir and Çelikten 2017). The solutions principally used for the manufacturing of geopolymers are sodium hydroxide (NaOH), sodium metasilicate (Na_2SiO_3), sodium carbonate (Na_2CO_3) and potassium hydroxide (KOH) (Wang *et al.* 1995, Hardjito *et al.* 2004, Pacheco-Torgal *et al.* 2008, Yang and Song 2009). These solutions have positive and negative influences on the mechanical properties of the mortars (Fernández-Jiménez *et al.* 1999, Bakharev *et al.* 1999, Krizan and Zivanovic 2002, Bernal *et al.* 2011). These positive aspects are porous structure formation, low hydration temperature, resistance to chemical attack, freeze-thaw resistance, strong aggregate-binding interface and low permeability (Shi *et al.* 2003, Roy *et al.* 2000, Bakharev *et al.* 2002, Bakharev *et al.* 2003, Puertas *et al.* 2003, Shi and Xie 1998). The negative effects are rapid setting, low workability, micro crack formation, high shrinkage and efflorescence (Živica 2007, Collins and Sanjayan 1999, Aydın 2013).

Many studies have been carried out to activate natural or artificial materials with an alkaline activator. The raw materials employed for the production of geopolymers or alkali-activated materials are generally amorphous materials such as natural pozzolans (Nourredine *et al.* 2019, Bondar *et al.* 2011), blast furnace slag (Bilgiç *et al.* 2018, Aziz *et al.* 2019, Al Safi 2019), fly ash (Rattanasak and Chindaprasirt 2009, Patil *et al.* 2014, Gunasekera *et al.* 2017) and metakaolin (Alanazi *et al.* 2017, Lizcano *et al.* 2012). The geopolymerization process is directly associated with the chemical composition and structure of these raw materials and solubility of Si and Al (Erdoğan 2014, Davidovits 2008). Due to the depletion of resources, researchers are in search of alternative precursor materials for geopolymer synthesis (Hassan *et al.* 2018, Moraes *et al.* 2018, Tchakoute *et al.* 2013, Saxena *et al.* 2017).

Perlite is an acidic volcanic glass, and the color of raw perlite can vary from bright black to transparent light gray. Perlite ore is usually obtained by blasting with open mining methods. The ground perlite (GP) is obtained by crushing, grinding and grading of perlite ore. GP has a potential to be used for the production of geopolymers due to its appropriate content of alumina (Al_2O_3) and silicon dioxide (SiO_2) (Topçu and Işıkdağ 2007, Demirboğa and Gül 2003, Işıkdağ 2015). Due to its natural structure and suitable chemical composition, it is also used as aggregate or pozzolan in building elements such as plaster and brick (Işıkdağ 2015, Çelik *et al.* 2013). The utilization of expanded perlite as aggregate (Topçu and Işıkdağ 2008, Şengül *et al.* 2011), calcined perlite (Ramezanianpour *et al.* 2014) and GP as pozzolan (Yu *et al.* 2003) in concrete are also investigated. High perlite reserves in some countries

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Table 1 Properties of ground perlite

Chemical Composition							
%							
SiO_2	Al_2O_3	K_2O	Na_2O	MgO	CaO	Fe_2O_3	TiO_2
71.36	13.08	5.42	3.21	0.12	0.96	0.78	0.11
other properties							
pH		Loss of Ignition			Bulk Density, kg/m^3		
7.71		2.12			1152		

Table 2 Gradation of CEN standard sand

Sieve diameter, mm	2.0	1.6	1.0	0.50	0.16	0.08
Cumulative percent retained	0.0	7.3	32.7	67.1	87.2	99.1

encourages researchers to investigate the possible use of GP as a geopolymer raw material (Erdoğan 2014). According to several researches, GP can be activated by using NaOH, $NaAlO_2$ and H_2O_2 solutions and it is possible to reach high strengths by adding artificial pozzolan to the mixtures (Vance *et al.* 2009, Vaou and Panias 2010). The influence of NaOH and KOH solutions, GP-based geopolymers have been produced that achieve a compressive strength (f_c) of 30-35 MPa at oven curing temperatures above 65-70°C or at room temperatures (Erdoğan 2011, Taxiarchou *et al.* 2012).

The unique contribution of this study is the detailed investigation of the influences of Na concentration and oven curing conditions on strength development of GP-based geopolymers. For this purpose, GP-based geopolymer mortars are produced with four different NaOH molarities and the mortars are cured in four different conditions. Besides, microstructural properties of the geopolymer mortars are investigated.

2. Experimental study

2.1 Materials

Mortar mixtures were produced in different mixing ratios and curing conditions using GP, CEN standard sand, NaOH and tap water.

The GP used in the study was obtained from a manufacturer. The manufacturer obtains perlite ore from Kütahya region in Turkey by blasting with open mining methods. Then ground perlite (GP) is produced after crushing, grinding and grading of perlite ore by the manufacturer. Table 1 shows the physical and chemical properties of the GP.

CEN standard sand employed in GP-based geopolymer mortars was obtained from Cement Plant of Trakya in Turkey. The gradation of the sand is illustrated in the Table 2.

NaOH was provided in ~97% purity in solid form from the local sources and used in the mixtures as solution by dissoving in the mix water. The geopolymer mortars were produced using 4, 8, 12, 16 molar (M) NaOH solutions, separately.

2.2 Production method and curing details

Forty-eight prism-shaped geopolymer specimens, 4×4×16 cm in size, were produced for use in the experiments and codes were given to these specimens as indicated in Table 3. Three specimens casting were made for each specimen code and each code was explained by the NaOH molarity, oven curing temperature and oven residence time, respectively. The specimen production method was designed to make the GP chemically and thermally binding to form geopolymer. The sand/GP mass ratio was determined as 3.0 in the mortar production and mixing was performed under laboratory conditions. To obtain the solution, water and NaOH were mixed to achieve the molarity values determined in the work, then sand and GP were poured in the mortar mixer. The stirrer was run slowly (136 rpm) for 30 seconds and the solution was added during stirring. The mixtures were then immobilized in the mixer for 90 seconds. Lastly, the mixer was run rapidly (281 rpm) for 60 s and the mixtures were allowed to stand for 15 s, then poured into 4×4×16 cm molds. After molding, the specimens were allowed to be compressed on the vibrator table and allowed to cure (80 or 120°C, 24 or 48 hours) in the oven 24 hours after casting, so that the GP became binding. After 72 hours, all of the specimens were taken from the mold and kept dry for curing in laboratory conditions (25±3°C) until the day of strength tests (7, 14 and 28 days) as shown in Fig. 1.

Table 3 Mixture codes and ratios for 3 prism specimens

Series Code	Specimen Code	Molarity	GP (g)	NaOH (g)	Sand (g)	Water (g)	Oven curing condition
1	GP4/80/24	4	450	48	1350	300	80ºC 24 h
	GP8/80/24	8	450	96	1350	300	
	GP12/80/24	12	450	144	1350	300	
	GP16/80/24	16	450	192	1350	300	
2	GP4/80/48	4	450	48	1350	300	80ºC 48 h
	GP8/80/48	8	450	96	1350	300	
	GP12/80/48	12	450	144	1350	300	
	GP16/80/48	16	450	192	1350	300	
3	GP4/120/24	4	450	48	1350	300	120ºC 24 h
	GP8/120/24	8	450	96	1350	300	
	GP12/120/24	12	450	144	1350	300	
	GP16/120/24	16	450	192	1350	300	
4	GP4/120/48	4	450	48	1350	300	120ºC 48 h
	GP8/120/48	8	450	96	1350	300	
	GP12/120/48	12	450	144	1350	300	
	GP16/120/48	16	450	192	1350	300	

Fig. 1 The views of the GP-based geopolymer mortars cured at 120ºC for 48 hours

(a) Compressive strength test

(b) Flexural strength test

Fig. 2 Strength tests on mortars

2.3 Tests

In this study, changes in unit weight, ultrasonic pulse velocity (U_{pv}), compressive strength (f_c) and flexural strength (f_{fs}) values of GP-based geopolymer mortars with NaOH molarity and oven-curing conditions were investigated.

The unit weights of the 28-day-old specimens were determined before strength tests and the f_c and f_{fs} values of 7, 14 and 28 days were found as shown in Fig. 2. Unit weight, U_{pv}, f_{fs} and f_c values for each mixture were determined by means of the three specimens.

Unit weight of mortars were obtained on the 28 days aged 4x4x16 cm prism specimens. Specimens were saturated and tested in surface dry condition. Initially, the weight of each specimen was measured. Then the means of the weights of three specimens were calculated to use in the study.

Ultrasound pulse velocity (U_{pv}) values are calculated in specimens at the age of 7, 14 and 28 days. The U_{pv} tests were conducted through the instrument of an ultrasonic non-destructive digital tester on the 4×4×16 cm specimens in respect of ASTM C 597-09. The frequency of the tester was 54 kHz and its accuracy was 0.11 s. The U_{pv} values were calculated by measuring the transition duration of the ultrasonic pulse in the mortar specimens.

The flexural strength (f_{fs}) tests were conducted on the 4×4×16 cm prismatic specimens to obtain f_{fs} values of the mortars at the end of 7th, 14th and 28th days as shown in Fig. 2(b). (TS EN 196-1). As seen on the Fig. 2(b), the f_{fs} test of the mortar specimens at 7, 14 and 28 days was performed under three-point loading condition. The averages of the results of three mortar specimens were determined as the f_{fs} value of each mortar mixture.

The 7,14 and 28 days' f_c values of mortars were determined as seen on the Fig. 2(a) with respect to the TS EN 196-1 standard. The measurements were made by placing 4×4 cm plates to top and bottom of the mortars which were broken into semi-prisms during the f_{fs} test. The averages of the results of six semi-prisms were determined as the f_c value of each mortar mixture.

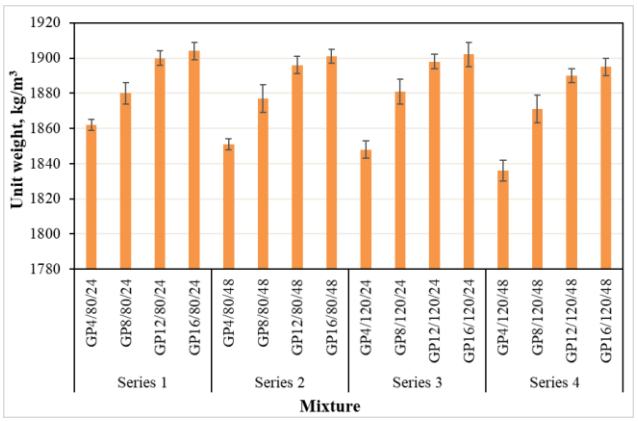

Fig. 3 Unit weights of mortars

GP16/120/48 coded geopolymer mortar was used for the characterization tests due to its high strength property. For the purpose, powdered samples were taken from the GP16/120/48 coded mortars for *X*-ray diffraction analysis (XRD, Panalytical Empyrean). Besides, small particles were used in electron microscopy and energy dispersive *X*-ray spectroscopy analyses (SEM/EDS, Zeiss-Supra 40VP).

3. Results and discussion

3.1 Unit weight

The unit weights of the mortars increase with increasing NaOH molarity as seen in Fig. 3. This can be attributed to the fact that the NaOH mass added to the mixture increases the total mass amount. The unit weights of the high temperature oven cured specimens are lesser than those of the oven cured at low temperature, and the unit weights of the oven cured specimens for 24 hours are higher than those of the oven cured for 48 hours.

This can be explained by the fact that mortar mixtures with higher temperature and longer curing time lose more water. The minimum unit weight is obtained as 1836 kg/m^3 at 28 days of GP4/120/48, and the unit weights of the mortars increase by ~3.2% due to the increase in NaOH molarity in the 28-day mixture series 4.

3.2 Ultrasound pulse velocity

The U_{pv} values of the mortars increase as the increase of NaOH molarity for each batch as shown in Fig. 4. The U_{pv} values of the mortars exposed to high temperature oven curing are found to be higher than those exposed to low temperature oven curing. U_{pv} values of the mortars exposed to oven curing for 24 hours are lower than those exposed to oven curing for 48 hours. This is due to the better activation of the GP at higher oven curing temperature and longer curing periods of mortar

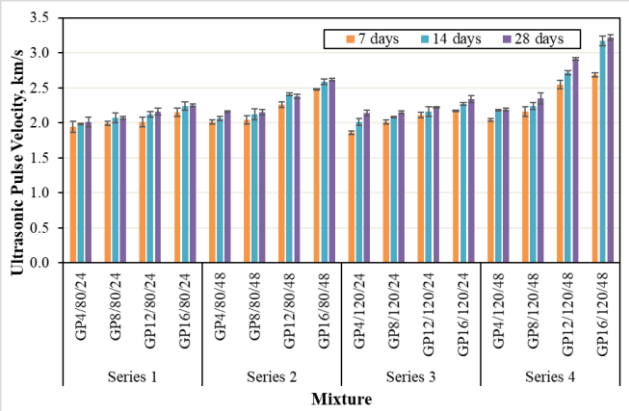

Fig. 4 Ultrasonic pulse velocities of mortars

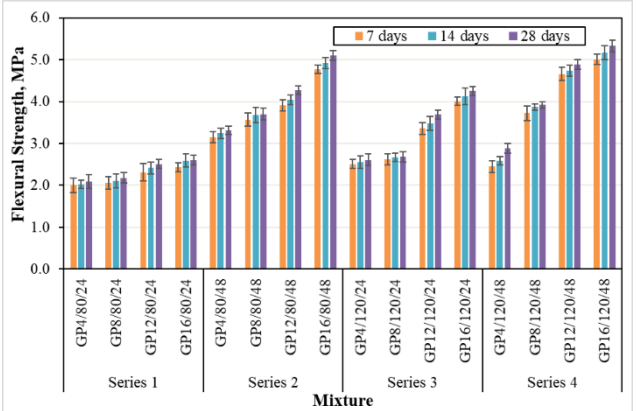

Fig. 5 Flexural strengths of mortars

mixtures. This resulted in a less porous structure in the mortars. The maximum U_{pv} of 3.22 km/s was obtained on GP16/120/48 mortars at the age of 28 days. Besides, it is observed that the U_{pv} of the mortars increased by ~47.1% due to the increase in NaOH molarity in mixture series 4 of the same age. The increment in the U_{pv} of mortars with NaOH molarity can be explained by the increased polymerization rate resulting in a more compact internal structure. Similar trend in the U_{pv} of and fly ash-based geopolymers is stated in a previous work (Wang *et al.* 2015).

3.3 Flexural strength

The f_{fs} test results are given in the Fig. 5. It can be observed on the Fig. 5 that the lowest f_{fs} value of 2.00 MPa and the highest f_{fs} value of 5.33 MPa were obtained from the 7 days old GP4/80/24 and 28 days old GP16/120/48 mortars, respectively. The f_{fs} values of the mortars increase with increasing NaOH molarity for each series with exceptions. The enhancement in the f_{fs} values of the mortars from 4M NaOH to 16M was in the range of 21-28%, 51-54%, 59-63% and 85-105% for the series 1, 2, 3 and 4, respectively. The higher the molarity of the NaOH solution, the easier the dissolution of Al and Si species from the GP. Additionally, the mechanical properties of geopolymers is relevant to the polymerization order, which is mainly influenced by the soluble aluminate and silicate of the geopolymeric system. The higher the degree of polymerization in the geopolymeric systems, the higher the acquired mechanical properties, generally (Panias *et al.* 2007). Similar results reported by El Hafid *et al.* (Hafid *et al.* 2017) for calcined clay-based geopolymers. Researchers state that the f_{fs} of calcined clay-based geopolymers enhanced with increasing NaOH concentration from 4M to 11.5M. The increment is attributed to excessive development of the zeolitic-fibery microstructure of the calcined clay-based geopolymers. The precursor materials for geopolymerization of the previous study (calcined clay) is different from the present study (GP), but strength development of geopolymers with NaOH content are similar with each other. Besides, Celikten and Sarıdemir (2018) reported a significant f_{fs} increase as the increasing alkaline content from 4% Na to 8% Na for fly ash-based geopolymer mortars. In addition, the f_{fs} of the mortars exposed to high temperature oven curing are found to be higher than those exposed to low temperature oven curing with exceptions. The f_{fs} of the mortars exposed to oven curing for 24 hours is found to be lower than those exposed to oven curing for 48 hours. This is due to the better activation of the GP as a result of the curing of the mortar mixtures at higher temperatures and longer oven curing period. These results in higher strength in mortars.

3.4 Compressive strength

The results of the f_c tests are illustrated in the Fig. 6. The lowest f_c value of 10.18 MPa and the highest f_c value of 41.59 MPa were achieved on the GP4/80/24 mortars at the age of 7 days and GP16/120/48 mortars at the age of 28 days, respectively. It can be seen on the Fig. 6 that the f_c values of the mortars increase with increment of NaOH molarity for each batch with exceptions. The average f_c increase with increasing NaOH molarity from 4M to 16M was 32.6%, 63.4%, 57.1% and 87.4% for the mortar series 1, 2, 3 and 4, respectively. The amount of alkaline activator is a very important factor for starting of the geopolymerization process since a high alkaline medium is required for increasing the surface hydrolysis of the alumino-silicate particles available in the raw material such as GP (Part *et al.* 2015). Besides, Nagaraj and Venkatesh Babu (2018) report that molarity of the alkaline activator plays a vital role in developing f_c of geopolymers. The researchers also report that the f_c of self-compacting geopolymer concretes produced with fly ash and blast furnace slag increase with molarity of NaOH. The results of the researchers are in harmony with the results of GP-based geopolymer mortars produced in this study. In the work of Somna *et al.* (2011), fly ash-based geopolymer pastes were with 4.5 to 16.5 M NaOH produced by curing at ambient temperature. They reported that the f_c values of the pastes increased with the molarity of NaOH from 4.5 to 14 M, significantly. They attributed this increment to higher degree of silica and alumina leaching at high NaOH molarities. However, they reported that the f_c of the pastes started to decrease with more NaOH content than 14M due to early precipitation of alumino-silicate products. In another work, Erdoğan (2014) reported a higher rate of f_c development with higher NaOH molarities for GP-based

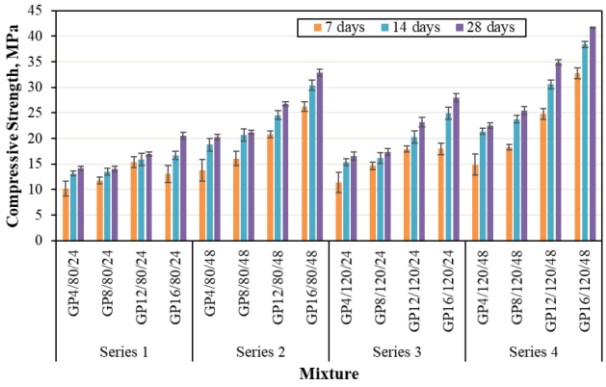

Fig. 6 Compressive strengths of mortars

geopolymers cured at 100ºC for 1 day. The results of Erdoğan (2014) is in harmony with this present work. While these papers reported increment in f_c with the increase in the alkali content, some other researches indicate a total contradiction in f_c development. Taxiarchou *et al.* (2013) observed higher f_c on the GP-based geopolymer concretes produced with lower alkaline content. He *et al.* (2013) studied on rice husk ash and red mud-based geopolymer composites and they stated that higher molarity of NaOH had resulted in a decrease in f_c of the composites. These contrasting trends can be explained by restricted the leaching of Si and Al ions by the high viscosity of NaOH solution, premature precipitation of geopolymeric gels due to the too much OH^- ions and also the availability of partially reacted or unreacted precursor particles because of the imperfect dissolution of Al and Si species (Part *et al.* 2015).

The f_c of the GP-based geopolymer mortars increased with increasing of oven curing temperature and the period of the oven curing. The increase in the f_c values of the mortars by increasing oven curing temperature from 80ºC to 120ºC was between 6% and 49%. Additionally, the f_c values of the mortars were increased between 25% and 82% by increasing oven curing period from 24h to 48h. The increase in the f_c of the geopolymer binders with increasing oven-curing temperature and the curing period were also reported by Aliabdo *et al.* (2016) for fly ash-based geopolymer concretes, and by Mo *et al.* (2014) for metakaolin-based geopolymers (from 20°C to 60°C). The enhancement in the f_c of the geopolymer binders with increasing curing temperature and period can be attributed to extent of dissolution of precursors (especially Si and Al) from the amorphous phases in the precursors (Mo *et al.* 2014).

3.5 SEM/EDS analysis

SEM/EDS results of the GP16/120/48 sample are illustrated in Figs. 7-9. GP is found to have a round structure and light color. A GP particle surrounded by N–A–S–H and the other geopolymeric gel products are identified on the Figs. 7-9. In addition, needle-like structures are detected on Fig. 8. These type of structures are similar to zeolitic-fibrous phases (Hafid *et al.* 2017). Besides, micro-crack formations are seen on the SEM images. The micro

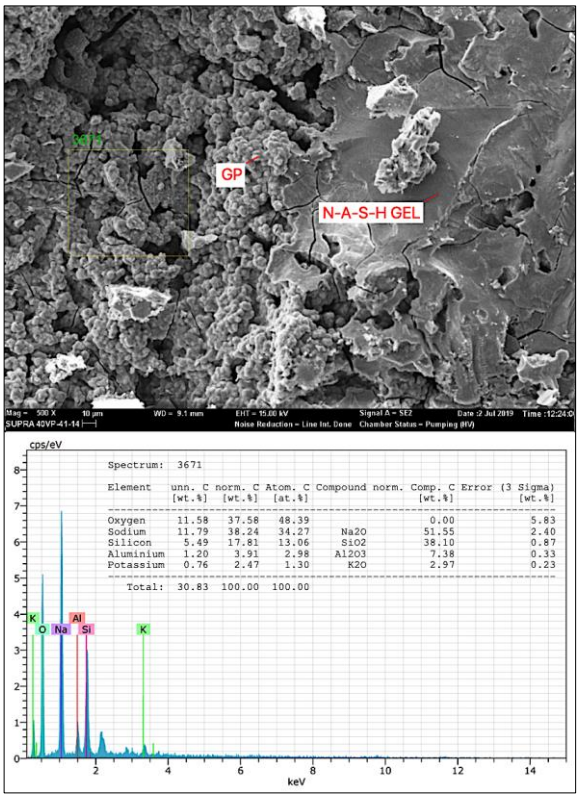

Fig. 7 SEM/EDS analysis of GP16/120/48 (X500)

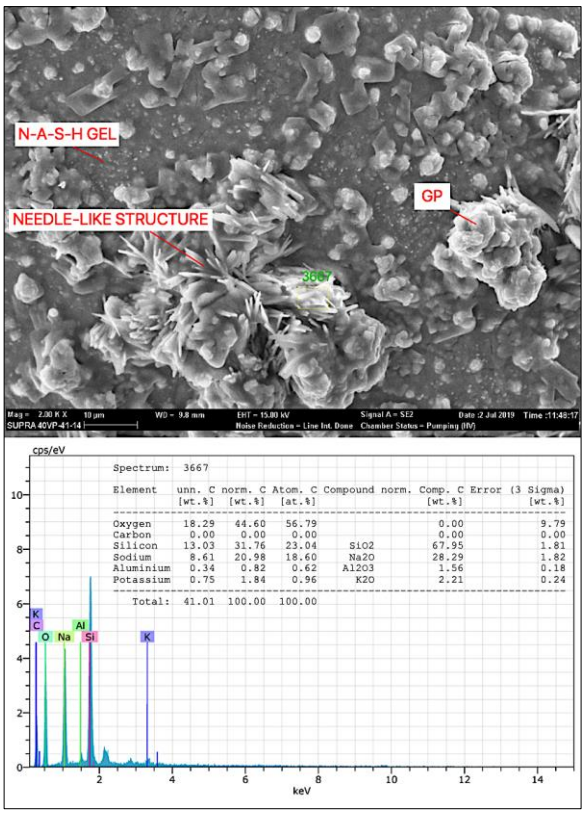

Fig. 8 SEM/EDS analysis of GP16/120/48 (X2000)

crack formations can be attributed to the oven-curing process. According to SEM images, stains on the surfaces of the GP granules prove the formation of

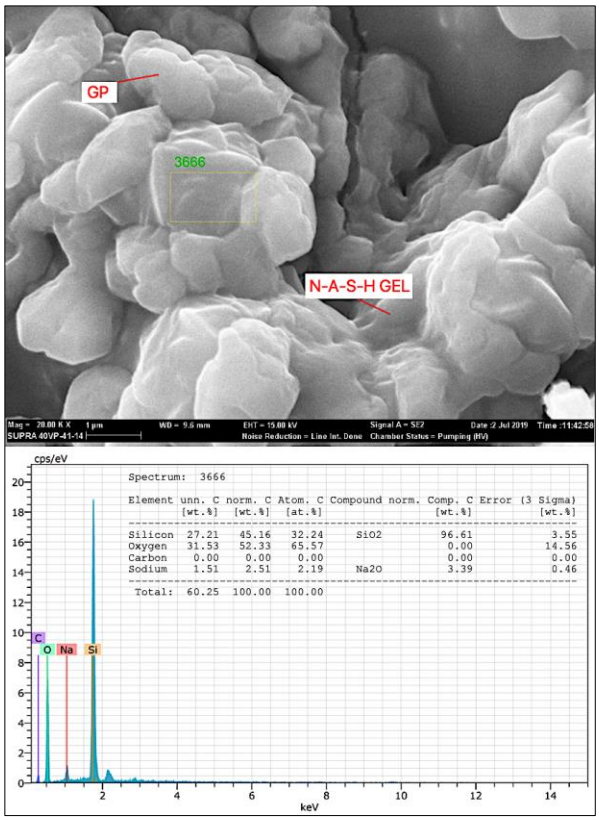

Fig. 9 SEM/EDS analysis of GP16/120/48 (X20000)

geopolymerization (Papa *et al.* 2018). Besides, it is observed that the geopolymerization of GP is heterogeneous. Well reacted GP, poorly activated GP and also un-reacted GP particles are seen on the SEM images. Additionally, sufficient bond strength is observed between N–A–S–H and other geopolymeric gel products and sand on the SEM images. This bond strength is found to be effective on the f_c, f_{fs} and impermeability of the mortars produced using NaOH solution. EDS analysis is performed in three different areas on the same sample. Na_2O, SiO_2, Al_2O_3 and K_2O compounds and their ratios are determined. The gel occurred as a result of geopolymerization of GP can be determined as mainly the N-A-S-H.

3.6 XRD analysis

The XRD analysis result of the powder sample of GP16/120/48 is given in Fig. 10. In addition to amorphous phases seen between 2θ of 25° and 33°, semi-crystaline and crystalline phases were also detected on the XRD spectrum of the GP-based geopolymer mortar. Compounds such as albite, leucite and quartz are detected from the spectrum. The peaks of albite and leucite minerals indicate the presence of GP and geopolymeric gel products. The quartz mineral seen in the XRD spectrum is determined the presence of SiO_2 in GP and sand. Provis and Bernal (2014) stated that hydroxide activation causes to increase the ratio of crystalline phases to disordered products in the geopolymers with respect to silicate activation. Therefore, the crystalline phases seen in the GP-based geopolymers in this study can be attributed to the NaOH activation.

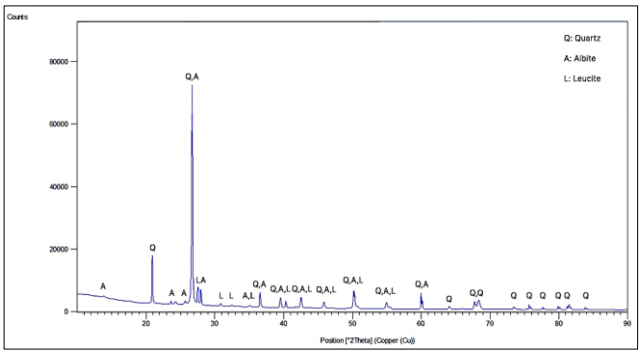

Fig. 10 XRD spectrum of GP16/120/48

4. Conclusions

The influences of NaOH molarity, oven-curing temperature and oven-curing duration on the strength development of the GP-based geopolymer mortars are investigated in this work. According to the results obtained in the study, the following results can be summarized:

- The investigated properties of the GP-based geopolymer mortars are significantly influenced by NaOH molarity and oven-curing conditions.
- The unit weights of the mortars increase with increasing NaOH molarity and decrease with increasing oven curing temperature and duration.
- Ultrasonic pulse velocity, flexural and compressive strengths of the mortars increase with increment in NaOH molarity from 4M to 16M gradually with exceptions
- Strength properties and ultrasonic pulse velocities of GP-based geopolymer mortars enhance with oven curing temperature and duration and dry curing period with exceptions.
- The maximum 28 days' ultrasonic pulse velocity of 3.22 km/sec., flexural strength of 5.33 MPa and compressive strength of 41.59 MPa are achieved on GP16/120/48 geopolymer mortars.
- The use of NaOH solution provides sufficient bond strength at the interface region between sand and geopolymeric gel matrix.
- Micro crack formations, un-reacted GP particles, poorly and well reacted GP are observed on the SEM analyses of the GP16/120/48 geopolymer mortars.
- Peaks for quartz albite and leucite minerals are seen on the XRD analysis of the GP16/120/48 geopolymer mortars.

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