Fabrication of sustainable geopolymer mortar incorporating granite waste

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Abstract. The objective of the present paper is to valorize granite powder wastes generated from granite mining and processing industry which cause vast environmental pollutions, in production of valuable building materials using of geopolymer technology by alkaline activating. The current work focuses on the effect of incorporation of granite waste from 0 up to 15% on the characterization of the formed geopolymer and track the formed hardened materials using Fourier transform infra-red (FTIR) and X-ray diffraction technique (XRD), whereas the compressive strength and water absorption were used to estimate the optimum ratio of granite waste that can be used without a negative effect on the hardened mortar. Also, the effect of various compaction loads at 12.5 and 25 MPa on the physico-mechanical properties of the hardened mortar using low liquid to solid ratio. The results showed clear enhancement in the structure and performance of the produced geopolymer mortar up to 7.5% granite waste addition giving compressive strength values more than 46 MPa, while the compaction positively enhanced the compressive strength of the formed mortar with increasing of pressure loads with about 20-25% and decreasing the water absorption values by about 70% which can be related to better compaction of the matrix.

Keywords: granite waste; geopolymer; compact; composite

1. Introduction

Sustainability is a several definitions concept (Rao 2000), the common one declares that today's generation should not compromise the future generations' ability to meet their requires. The three leaders of sustainable development are economic and environmental protection as well as social development. It is known though that the Earth's capacity to support people is determined by natural constraints and human priorities (Egger 2006, Cohen 1995). Three-quarters of the world's energy consumed today by cities which also responsible for global pollution. Furthermore, United Nations predict that 60% of the world's population will live in cities by the year 2030 (United Nations Centre for Human Settlements 2001).

Geopolymers are amorphous three dimensional aluminosilicate materials with ceramic-like properties that are produced and hardened at ambient temperature and widely used in building sectors due to their wide sustainable applications. Up on alkaline activation using alkali hydroxide and silicate solution, polymerization takes place when reactive aluminosilicate are rapidly dissolved

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and free [SiO$_4$] and [AlO$_4$] tetrahedral units are released in solution. The tetrahedral units are alternatively linked to polymeric precursors by sharing oxygen atoms forming thus amorphous geopolymer. Positive ions such as K+ or Na+ that are present in framework cavities, balance the negative charge (Davidovits 1994, Duxson *et al.* 2007).

Recently, due to the increased demand on the granite applications in construction sector, an increased amount of granite wastes generated from this industrial process. This vast amount of the wastes causes many environmental pollution and need researchers' attention. Many researchers have studied the granite waste utilization. Granite waste generated by the stone crushing industry has accumulated over years and the reduction in waste generation by manufacturing value-added products from the granite stone waste will boost up the economy of the granite stone industry (Kala 2013).

Another type of granite waste is granite powder which is waste product obtained during the process of sawing of granite rocks in granite industries. As this granite dust is creating many environmental hazards, its disposal is considered a great problem. Production of self-compacting concrete consumes huge amount of powder materials to maintain sufficient yield value of the fresh mix and then bleeding reduced, segregation and settlement. Hence, it is worthwhile to investigate the influence of granite powder in self-compacting concrete SCC as filler (Paralada 2016).

Granite powder waste produced can be partially replace sand with beneficial effect on the mechanical properties such as compressive strength, split tensile strength, modulus of elasticity. The test results obtained indicate that the test results indicated that the values of both plastic and drying shrinkage of concrete in the granite powder concrete specimens were nominal than those of ordinary concrete specimens (Kala and Partheeban 2010).

Xavier *et al.* (2012) presented paper describes the impact of weathering on clay bricks containing from 0 to 10% granite powder, an industrial by-product, where the specimens were fired at 500, 700 or 900ºC and then exposed to natural environmental conditions or accelerated laboratory weathering. Their physico-mechanical properties were evaluated to determine composition effect of raw materials on durability of fired clay.

Gonzalez *et al.* (2019) utilized granite waste powder along with red mud (bauxite residue) for production of ceramics with high mechanical properties; where granite used as a source of fluxing oxides for the ceramic industry, where a set of ceramic pieces made of red mud and granite waste were prepared and resulted in high mechanical properties while leaching results are below critical levels established by regulations.

Granite powder waste, when properly sieved into an appropriate size distribution, can be used as a suitable reinforcement for geopolymer. It creates a sustainable, cost-effective and reliable structural geopolymer composite utilizing resources that are easily attained worldwide (Roper *et al.* 2015), where granite powder selected mainly due to its cost, availability and environmental impact.

Khater and Ezzat (2018) studied the effect of combination between the low liquid/solid (l/s) ratio and pressure compaction on the properties of the hardened engineered stone alkaline hybrid composites of water cooled slag/metakaolin using granite as filler and metakaolin using fine sand as filler in the ratio of 1:3, while the used activator was 5% sodium hydroxide with low liquid to solid.

The current work focuses on the effect of incorporation of granite waste from 0 up to 15% on the characterization of the formed geopolymer and follow the formed hardened materials using Fourier transform infra-red (FTIR) and X-ray diffraction technique (XRD). On the other hand, compressive strength and water absorption were used to estimate the optimum ratio of granite waste that can be used without negative effect on the hardened mortar. Also, the effect of various compaction loads at 12.5 and 25 MPa on the physico-mechanical properties of the hardened mortar using low l/s ratio.

Table 1 Chemical composition of starting materials (Mass, %)

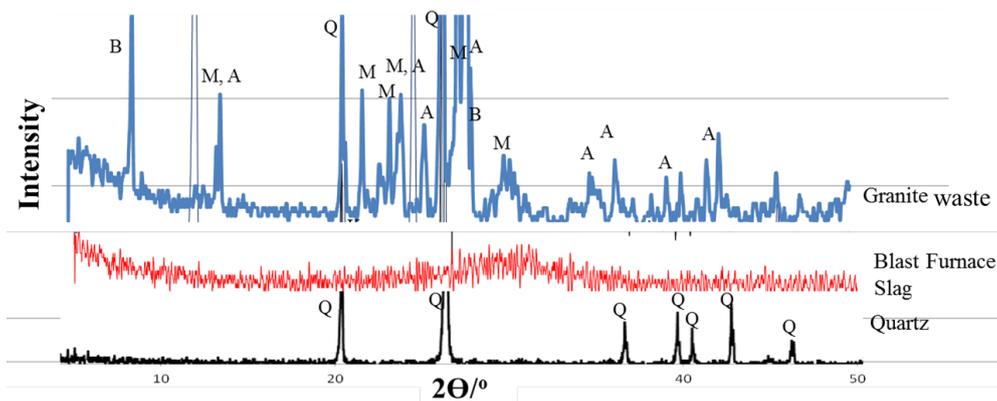| Oxide content (%)	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	K_2O	Na_2O	TiO_2	MnO	P_2O_5	Cl-	L.O.I.	BaO	SrO	Tatal
Water-Cooled Slag (GGBFS)	36.67	10.31	0.50	38.82	1.70	2.17	1.03	0.48	0.57	4.04	0.04	0.050	0.12	3.28	0.18	99.96
Granite waste powder	71.84	18.95	1.33	0.92	0.39	-	0.69	4.82	0.25	0.02	0.02	-	0.41	-	-	99.63
Fine Sand (Sand dunes)	89.91	2.00	1.45	1.56	1.91	0.87	0.37	0.06	0.03	0.04	0.03	0.12	1.65	-	-	99.98

Fig. 1 Xray diffraction pattern of the starting raw materials (Q: Quartz, M-Microcline, B=Biotite, A-Albite)

2. Experimental procedures

2.1 Materials

The materials used in this investigation were ground granulated blast furnace slag (GGBFS), supplied by the Iron and Steel Factory- Helwan, Egypt and granite waste sourced from the left over of Aswan granites industrial processing process. Fine sand (<1 mm) was used in production of the geopolymer mortar. Sodium hydroxide (NaOH) in the form of pellets was used as an alkali activator, and calcium hydroxide [99%, Sigma Aldrich]

The chemical compositions of the primary raw materials are given in Table 1, whilst the mineralogical characterization of the raw materials was done using X-ray diffraction analysis as represented in (Fig. 1). GGBFS which is a rich aluminosilicate material composed of dominant content of SiO_2, CaO, Al_2O_3 Fe_2O_3, and MnO as illustrated in Table 1, while it is mineralogically classified as an amorphous material as shown from Fig. 1. Granite waste composed mainly of SiO_2 and Al_2O_3 however its mineralogical composition revealed that it composed mainly of quartz, microcline and biotite.

Table 2 Composition of the geoplymer mixesw (Mass, %)

Mix No.	Water cooled slag (WS), %	Sand dune (<1 mm), %	Granite powder, %	Hydrated lime, %	Pressing, MPa^2	NaOH, %	Water/binder, %
g1	45.0	50	0.0	5	-	8	0.40
g2	42.5	50	2.5	5	-	8	0.40
g3	40.0	50	5.0	5	-	8	0.40
g4	37.5	50	7.5	5	-	8	0.40
g5	35.0	50	10.0	5	-	8	0.40
g6	30.0	50	15.0	5	-	8	0.40
g4-1	37.5	50	7.5	5	12.5	8	0.10
g4-2	37.5	50	7.5	5	25.0	8	0.10
g6-1	30.0	50	15.0	5	12.5	8	0.10
g7	45.0	50	5.0	-	-	8	0.28
g8	40.0	50	10.0	-	-	8	0.28
g9	35.0	50	15.0	-	-	8	0.28

2.2 Processing, molding and curing

Alkaline activated mortar mixes were made by hand-mixing of raw materials, passing a sieve of 90 μm with an alkaline activator (8% sodium hydroxide calculated from the total weight) for about 10 min and then further for about 5 min with an electronic mixer as represented in Table 2. Mixes were cast into 2.5 cm cubic steel moulds, vibrated for compaction, sealed with a lid to minimize any loss of evaporable water, left to cure undisturbed under ambient temperature for 24 hrs, demolded and then subjected to curing at 40° C with 100% relative humidity (R.H.) up to the end of testing time.

In case of pressed samples l/s ratio was about 10% from the total weight of the mixture as shown in the table. The mixture was cast into 50 mm lubricated cubic steel mold, vibrated for compaction, and pressed under a pressing pressure of 12.5 and 25 MPa, and then sealed with a plastic sheet as to minimize loss of evaporable water.

Samples exposed then to compressive strength measurements, and the resulted specimens fragments were subjected for stopping of the hydration process by immersion in acetone for 24 hr before being oven dried at 60° C, and pulverized for analysis (Ke *et al.* 2014).

2.3 Methods of investigation

Chemical analysis was carried out using Axios-wave length dispersion X-ray Fluorescence (PW4400/WD-XRF) Sequential Spectrometer (PAnalytical, Netherland, 2009). The X ray diffraction -XRD analysis was carried out using a Philips PW3050/60 Diffractometer. The data were identified according to the XRD software. Perkin Elmer FTIR Spectrum RX1 Spectrometer (Fourier Transformation Infra-Red) was used to evaluate the functional groups in the sample. Small amount of potassium bromide (KBr) and geopolymer powder were mixed and placed in the sample holder then the mix was pressed at 295 MPa for 2 min to produce specimen for examination, The wave number was ranging from 400 to 4000 cm^{-1} (Bakarev 2006, Panias *et al*. 2007).
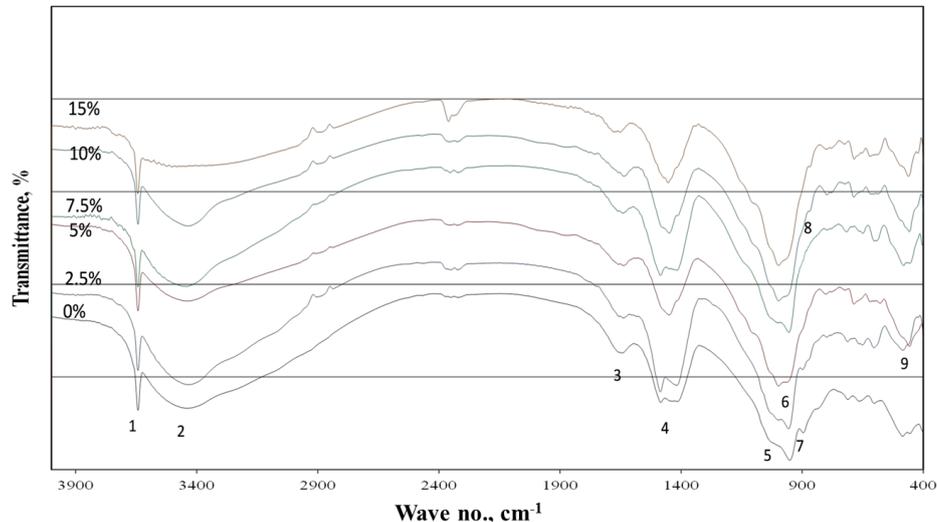
Fig. 2 FTIR spectra of 28 days alkali activated mortars having various ratios of granite powder (1,2: Stretching vibration of O-H bond, 3: Bending vibration of (HOH), 4: Stretching vibration of CO_2, 5: Asymmetric stretching vibration (Si-O-Si), 6: Asymmetric stretching vibration (T-O-Si), 7: Si-O Stretching, OH bending (Si-OH), 8: out of plane bending vibration of CO2, 9: Bending vibration (Si-O-Si and O-Si-O))

Water absorption measurements of the bricks were carried out according to ASTM C140 (2016). The percentage absorption was calculated using the equation:

Absorption (%)=[(W2-W1)/ W1]×100

whereW1=weight of specimen after complete drying at 105°C, W2=final weight of surface dry sample after immersion in water for at least 24 hr.

Compressive strength tests were carried out using five tones German Brüf pressing machine with a loading rate of 100 MPa/s determined according to ASTM-C109 (2016).

3. Results and discussion

3.1 FTIR and XRD spectroscopy

FTIR spectra of 28 days cured alkali activated mortar incorporating various granite waste ratios are shown in Fig. 2. The characteristics bands for the present geopolymer structure are: hydration groups and combined water allocated for stretching vibration of O-H bond for free hydrated lime at about 3640 cm^{-1} in addition to stretching vibration of O-H bond at about 3410 cm^{-1} and bending vibration for H-O-H at about 1620 cm^{-1}, stretching vibration of CO_2 located at about1430-1450 cm^{-1}, asymmetric stretching vibration (Si-O-Si) at about 960-990 cm^{-1} for non-solubilized silica, asymmetric stretching vibration (Ti-O-Si) at about 945 cm^{-1} where T=Si or Al, Si-O stretching-OH bending of Si-OH at about 880 cm^{-1}, out of plane bending vibration of CO_2 at about 865 cm^{-1} and bending vibration (Si-O-Si and O-Si-O) in the region 450-470 cm^{-1}.

The pattern revealed the growth of the main asymmetric band of T-O-Si with increasing of granite waste content in the mix up to 7.5% with the decrease of the intensity of shoulder band at about 1000 cm^{-1} for non-solubilized silica. This reflect increase in dissolution and geopolymerization
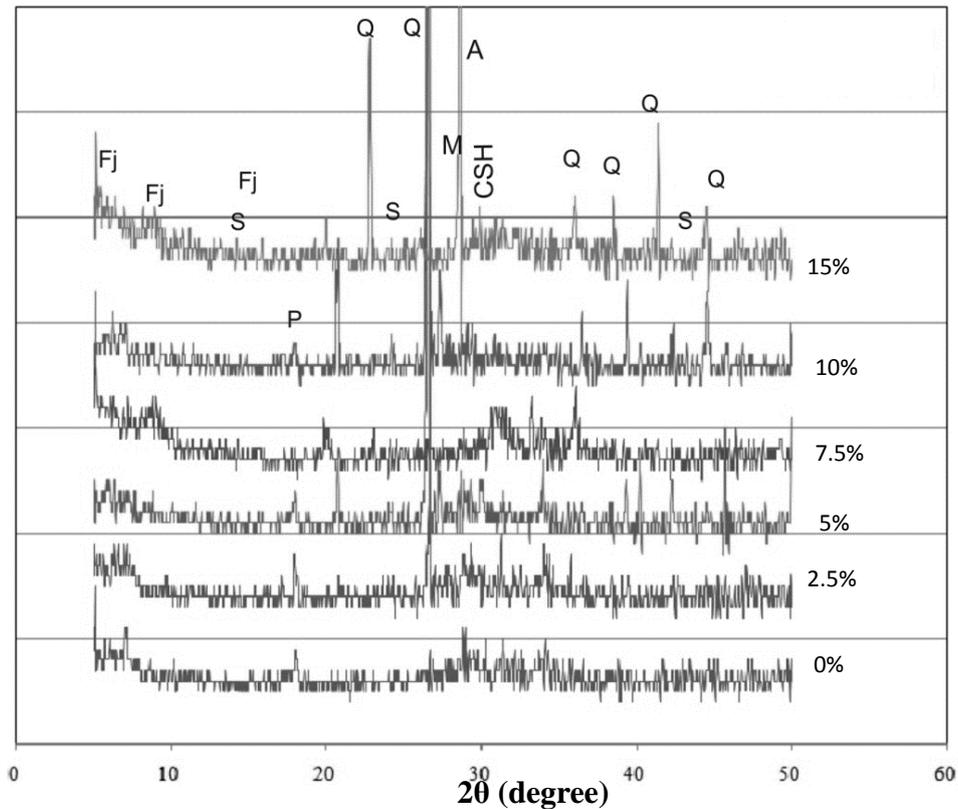
Fig. 3 XRD pattern of 28 days alkali activated mortars enhanced with various ratios of granite powder (Q: Quartz, Fj: Fanjasite, A: Albite, C: Calcite, S: Sodalite, P: Portlandite, CSH: Calcium silicate hydrate, M: Microcline)

of unreacted materials with granite waste addition, where the added waste participate in increasing of alumina and silica persecutors and so leads to increase the geopolymerization reaction. This is confirmed also by shifting of the amorphous asymmetric band at about 945 cm^{-1} (CSH and NASH) to the right, indicating an increase in vitreous geopolymer content, where the added hydrated lime interacts with dissolved silica forming CSH that acts as nucleation sites for geopolymer formation and accumulation (Khater 2013, Jiang *et al*. 2005, Temunjin *et al*. 2009).

Another major notice was the decreased intensity of Si-O stretching-OH bending of Si-OH where the free terminal hydroxide incorporated in the geopolymer formation. While further increase in granite waste accompanied by reincrease of the previous band as well as the non-solubilized silica band up to 15%, where the increased silica content in granite waste alter Si/Al ratio leading to formation of less stable geopolymer chains. Moreover, the filling effect of granite waste overcome the geopolymeric precursor effect and so hinders the propagation of the geopolymer network. Another elucidation from the pattern is the splitting of carbonate bands at about 1430-1450 cm^{-1} (v C-O) into two peaks, which indicates the distorted nature of CO_3 mineral (Kalinkin *et al*. 2002, Kalinkin *et al*. 2004), which could be attributed to partial carbonation of C-S-H gel in air atmosphere. However, it can be noticed the diminishing of out of plane bending vibrational band for carbonate at about 870 cm^{-1} with increase of granite waste up to 15%.

XRD pattern of alkali activated mortar mixes having various granite waste ratios are presented in Fig. 3, where a significant difference observed between the crystalline phases and geopolymeric matrix from the control mix compared to the other mixes. All the patterns show the typical amorphous structure of Geopolymer with a wide diffraction hump in the range of 20 to 35 (2-Theta) as assumed by many researchers (Provis *et al*. 2005, Yip *et al.* 2005), this amorphous hump is attributed to the amorphous aluminosilicate. Moreover the formation of CSH along with geopolymer enhances the geopolymerization reaction (Khater 2013, Jiang *et al*. 2005, Temuujin *et al.* 2009). It was noticed that this amorphous peak charachteristic to geopolymerization appear enhanced in geopolymer mortar with 7.5% granite waste, beside to the complete consumption of hydrated lime and formation of extra CSH binding phase. While further increase in granite waste in the mix leads to the increase in formation of crystalline zeolite phases (Faujasite and sodalite) in accordance of FTIR illustrations where there was a decrease in asymmetric T-O-Si and an increase in intensity of non-solubilized silica band. However, it is clear that the diffraction peak of quartz was not affected by geopolymerization reaction, which suggests that it doesn't contribute in the geopolymerization reaction. Calcite on the other hand with CSH gel at 29.4° 2θ, where this carbonates may be from the source materials or from atmospheric weathering during sample preparations (Bernal *et al*. 2010, Bernal *et al*. 2011).

3.2 Physicomechanical properties

The Compressive strength results of alkali activated mortar mixes with different granite waste percentage and with 5% hydrated lime cured for 7, 14, 28 and 60 days are presented in Fig. 4. The compressive strength of geopolymer specimens cured for 28 days with 7.5% granite waste increased by about 30%, and declined by about 12% with increasing of granite waste up to 15%. However the Geopolymer specimens cured for 60 days with 7.5% granite waste increased by about 60%
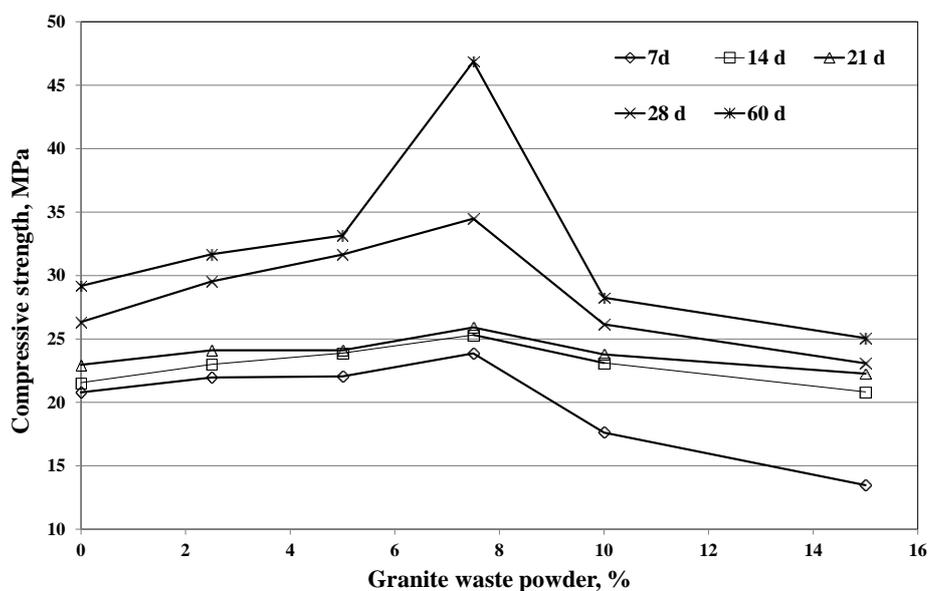

Fig. 4 Compressive strength of alkali activated mortars enhanced with various ratios of granite powder and having 5% calcium hydroxide
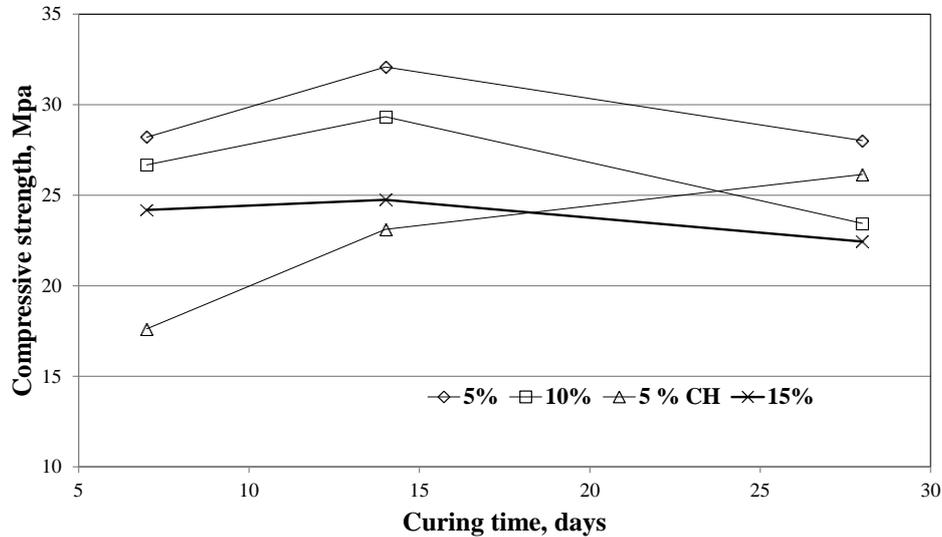
Fig. 5 Compressive strength of alkali activated mortars enhanced with various ratios of granite powder without clcium hydroxide and compared with mix having 5% calcium hydroxide

compared to the control geopolymer specimen and declined by about 14% with increase in granite waste up to 15%.

The increase in strength of mortar with 7.5% granite wastes, optimum ratio, can be attributed to increase in dissolution by the action of activator and release of reactive silica and alumina persecutors, which takes part in the geopolymer formation as confirmed from FTIR, and XRD, where the amorphous geopolymer content as well as CSH increased greatly by accommodation of granite in mortar up to the optimum ratio, while further increase in granite wastes over the ratio increase the silica and alumina which will not completely polymerized as the activator is consumed and in turn will form crystalline sodalite as well as Faujasite that will results in strength decline. Also, the decline in compressive strength of specimens with granite waste over 7.5% cured at 28 and 60 days can be explained further by the presence of excess granite waste in the mix that don't participate in the geopolymerization reaction, thus acting as a filler.

The compressive strength of alkali activated mortar cured for 28 days with difference ratios of granite waste in the absence of hydrated lime compared to mortar with 5% hydrated lime is expressed in Fig. 5. The results showed that there is enhanced in strength of mortar cured for 14 days for mortar with 5, 10 and 15 % granite waste as compared to 7 days values. This can be related to the strengthening effect of CSH formed by the interaction of hydrated lime formed from calcium and dissolved silica supplied from active slag interaction with alkaline activator, beside to the positive effect on strength presented by geopolymerization reaction. However a sharp decline occurs in strength occurs after 28 days of curing, due to the release of excess of silica and alumina in the mixture which doesn't contribute in geopolymerization reaction. On the other hand, the presence of hydrated lime in the mortar mix and absence of granite waste showed the same trend of increase in strength with curing up to 14 days. However the decline in strength of mortar with hydrated lime after curing for 28 days is still lower than mix with 5% and 10% granite waste with no hydrated lime but still higher that mix with 15% granite waste.

Compressive strength as well as water absorption of geopolymer mortar having 7.5 and 15%
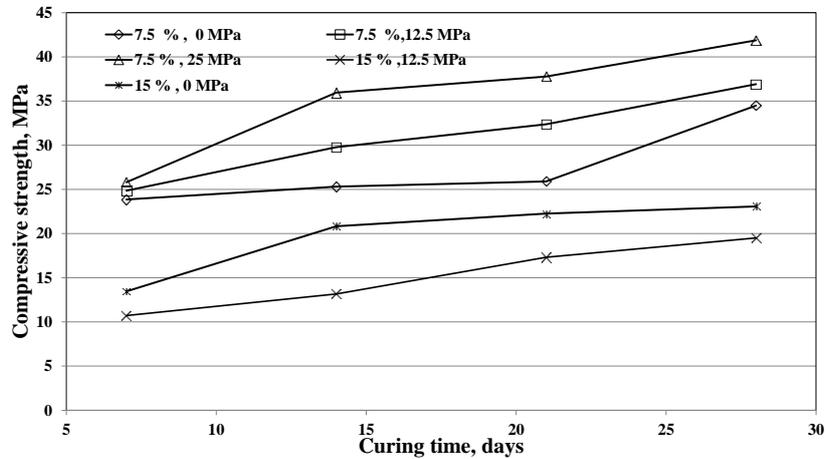
Fig. 6 Compressive strength of alkali activated mortars having various ratios of granite powder, pressed at various pressing pressure
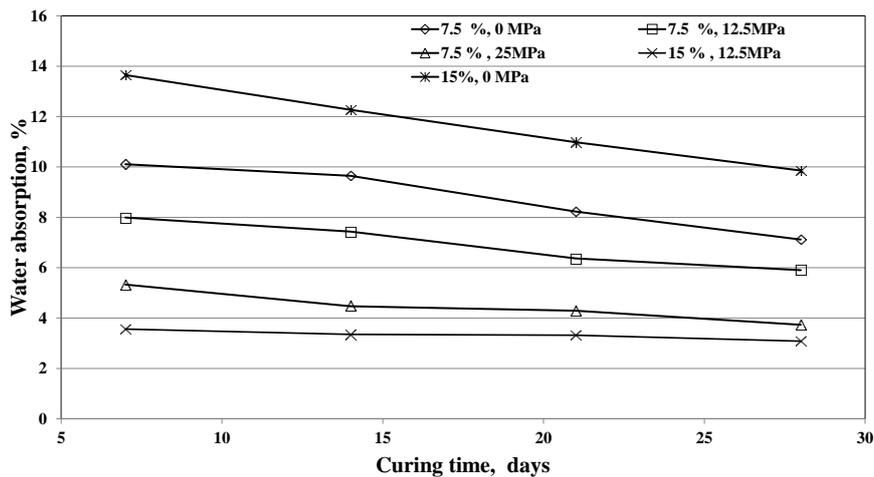

Fig. 7 Water absorption of alkali activated mortars having various ratios of granite powder, pressed at various pressing pressure

granite waste and pressed at 12.5 and 25 MPa and compared with control mortar without pressing are represented in Figs. 6, 7. The results show the increase of strength in all pressed mortar mixes along with hydration age up to 28 days, whereas mix having 7.5% granite waste, pressed at 25 MPa possess the maximum strength.

Results indicate that the combination between low l/s ratio and high pressure compaction results in the formation of green composite with high quality engineering properties (Khater and Ezzat 2018, Feng and Meyer 2009, Freidin 2007). There is an increased strength for 28 days mortar composite with increasing load up to 25 MPa for mix incorporating 7.5% granite waste powder giving values of 34.5, 37 and 42 MPa for pressure load of 0, 12.5 and 25 MPa, respectively, while decreased to 19.5 MPa when pressing to 12.5 MPa for 15% granite waste. It can be recognized that, increasing pressure for geopolymer mortar having 7.5% granite waste exposed to better structure

modification and rearrangement giving compacted structure with lower porosity and high compressive strength. It can be noticed also the strength gain with load increase from 12.5 to 25 MPa was 13.5% as compared with lower pressure load. On the other hand the geopolymer specimens without pressing give lower value strength because of decrease the physical and mechanical interconnection between particles in the geopolymer matrix. Also, results show a sharp decrease of strength with increasing of granite powder up to 15%. The hardening and consolidation of geopolymer rely on the reactivity of the starting materials. However, granite powder achieves low degree of reaction due to their low reactivity and mineralogical composition which generally consists of highly crystallized minerals.

On the other hand, the water absorption of pressed geopolymer mortar composites having 7.5 and 15% granite waste and pressed at 12.5 and 25 MPa decreased with increasing pressure loads as represented in Fig. 7, where they giving 7.12, 5.90 and 3.73% for 0, 12.5 MPa and 25 MPa for 7.5% granite, while attains 9.86 and 3.08% for granite waste of 15% at 0 and 25 MPa after 28 days of curing. From the pattern, water absorption decreased with load increase up to 25 MPa confirming the homogeneity and compatibility of the matrix at pressures up to 25 MPa.

4. Conclusions

The main concluded remarks of this paper are:

1. The valorization of granite waste powder which is low valuable material in producing durable dense composite with the aid of geopolymer chemistry.

2. Granite waste powder can be positively used up to 7.5% from the total weight with an enhancement in performance and compressive strength of geopolymer mortar activated by sodium hydroxide and calcium hydroxide.

3. Further increase in granite waste leads to strength decrease as well as deactivation in the formed geopolymer structure.

4. Formed geopolymer mortar with various granite waste powder without calcium hydroxide exhibit strength increase up to 14 days then loss in strength occurs due the absence of CSH binding materials formed in mortars incorporating hydrated lime in which their strength increases with time, in spite the early strength of mixes without lime is higher than of those having hydrated lime.

5. Results indicated also, better modification and reorganization of the composites with increasing pressure load leading to better enhancement on physicomechanical giving values of 34.5, 37 and 42 MPa for pressure load of 0, 12.5 and 25 MPa, respectively, while decreased to 19.5 MPa when pressing to 12.5 MPa for 15% granite waste.

6. The costs of the produced geopolymer product assumed to be reduce the cost by about 20% as compared to their comparable building products.

Data availability: Authors can provide information about the data presented in an article and provide a reason if data is not available to access.

Conflict of interest: Authors declare that there is no conflict of interest in the article.

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