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The effects of nanoparticles on early age compressive strength of ash-based geopolymers

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Abstract

In the present work, compressive strength of ash based geopolymers containing 97 wt% rice husk ash + fly ash and 3 wt% nano alumina + nano silica has been designed by Taguchi method at 2 and 7 days of water curing. 4 factors including the percentage of nano silica (at 3 levels of 1, 2 and 3 wt%), oven curing temperature (at 3 levels of 25, 70 and 90 °C), oven curing time (at 3 levels of 2, 4 and 8 h) and NaOH concentration (at 3 levels of 5, 8 and 12 M) were considered. By utilizing L9 Taguchi array, 9 series of experiments were conducted on the prepared specimens. The alkali activating was done by a mixture of sodium hydroxide (NaOH) and sodium silicate (Na_2SiO_3) solutions. The obtained results were evaluated by analysis of variance (ANOVA) method to determine the optimum level of each factor. In all produced specimens, the optimum level of nano silica, oven curing temperature and oven curing time were 3 wt%, 90 °C and 8 h, respectively. The oven curing time was not an important factor for determining the compressive strength. To validate the accuracy of the optimum conditions suggested by ANOVA, compressive specimens were made and tested in accordance to the optimum conditions for each of 2 and 7 days water curing regimes. The obtained compressive strength acquired from this situation was higher than those of proposed in 9 series experiments for each of 2 and 7 days water curing regimes.

Keywords: Geopolymer; Compressive strength; Ashes mixture; SiO_2 nanoparticles; Al_2O_3 nanoparticles; Taguchi experiment design

1. Introduction

Inorganic polymeric binders (geopolymers) are a group of alkali-activated materials exhibiting superior engineering properties compared to Portland cement. Synthesis of geopolymers is based on the activation of suitable aluminosilicate materials by an alkali metal hydroxide and an alkali metal salt. A time period of curing at temperatures in the range between 25 and 90 °C completes the process necessary for geopolymerization reactions. Geopolymers have been illustrated to exhibit superior mechanical properties with respect to those of traditional concrete specimens. However, compared to other material processing technologies, research focused on understanding the relationships between composition, processing, microstructure, and the mechanical properties of geopolymers needs more completion [1].

The compressive strength of an inorganic polymer depends on both the ratio of Si/Al and the types of the utilized raw material. Fly ash (FA) is recently used as a source material to produce geopolymer because of its suitable chemical composition along with favorable size and shape. Fly ash is a by-product of coal-fired electric power stations. Literature survey specifies that fly ash is primarily composed of SiO_2, Al_2O_3 and Fe_2O_3. Since the quality of fly ash depends on the type and the quality of coal along with the performance of the power plant, difficulties sometimes remain to control its chemical composition. In order to achieve a suitable chemical composition to produce geopolymers, the preferred method is to blend fly ash with another high silica source [2].

Rice husk ash (RHA) is a by-product from the burning of rice husk. The husk of the rice is removed in the farming process before it is sold and consumed. It has been found beneficial to burn this rice husk in kilns to make various things. The rice husk ash is then used as a substitute or admixture in cement. Therefore the entire rice product is used in an efficient and environmentally friendly approach. Utilization of RHA to produce high strength concrete has been addressed in several

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works [3–5]. RHA cannot be used alone to produce geopolymers because of its unsuitable SiO_2/Al_2O_3 ratio. Hence, it should be used by another alumina-rich source like pozzolanic fly ashes.

The main factors affecting the compressive strength of ash based geopolymers include the particle size of the utilized ash, curing temperature, curing time, NaOH concentration, etc. In the previous works [6–8], investigations on the effects of ash particle size on compressive strength of geopolymers showed that the finer the particle size, the higher the compressive strength. The authority of fine ashes to produce high strength geopolymers has been established in several works [1,9,10]. The effects of other main factors on compressive strength of geopolymers have not been completely approved. While some of researchers showed the increased strength with high NaOH concentration [2,6–8,11], the others [12,13] showed the negative impact of high concentration on this property. The effect of oven curing time and temperature on compressive strength is still unclear. Pan et al. [14] showed the increased oven curing time (up to 96 h) and temperature (up to 80 °C) results in higher compressive strength of different fly ash-based geopolymers. Rangan and his co-workers [15–18] have carried out extensive research on fly ash-based geopolymer concrete. They [17] reported that concretes could be manufactured by using fly ash in combination with sodium silicate and sodium hydroxide solution. After heat curing at 60 °C for 24 h, geopolymer concretes showed optimum engineering properties [18].

One of the additive materials to concrete specimens to acquire higher strengths is nanoparticle such as SiO_2 [19] and Al_2O_3 [20]. Nanoparticles can act as heterogeneous nuclei for cement pastes, further accelerating cement hydration because of their high reactivity, as nano-reinforcement, and as nano-filler, densifying the microstructure, thereby, leading to a reduced porosity. The most significant issue for all nanoparticles is that of effective dispersion [19,20]. As authors' literature survey, there is no works on utilizing nanoparticles in geopolymers to evaluate their mechanical properties.

Because of the variety of main factors deal with geopolymer properties, there are no works incorporating all factors effects on compressive strength of these materials. To overcome this discrepancy, a suitable experiment design can help to investigate the effects of more parameters simultaneously by the minimum required experiments. In this work, Taguchi experiment design has been conducted to evaluate the effects of oven curing time and temperature, nano silica content and NaOH concentration on compressive strength of fine ash based geopolymers. In the other words, 4 factors each at 3 levels were considered. Taguchi method suggested 9 series of experiments for each of 2 and 7 days of water curing regimes. Afterward, the obtained results were analyzed by ANOVA method to determine the optimum levels of the considered factors.

2. Experimental procedure

Table 1
Chemical composition of FA, RHBA and WG (wt%).

Material	SiO_2	Al_2O_3	Fe_2O_3	CaO	SO_3	Na_2O	Loss on ignition
FA	38.71	32.19	10.11	10.15	2.11	0.42	0.24
RHBA	95.23	0.04	0.08	0.24	0.03	–	1.22
WG	34.21	–	–	–	–	13.11	–

The used cementitious materials in this work were FA and RHA. Their chemical composition has been illustrated in Table 1 which was determined by a Philips PW 1480 X-ray fluorescence (XRF) apparatus. In addition, Fig. 1a and b shows XRD pattern of the RHA and FA, respectively which were conducted on a Philips PW 1800 apparatus. The as-received ashes were sieved and the particles passing the finesses of 33 μm were grinded using Los Angeles mill for 180 min which yielded different samples for FA and RHA. The average particle size obtained for FA was 4 μm with the BET specific surface of 35.4 m^2/g. The average particle size obtained for RHA was 6 μm with the BET specific surface of 29.8 m^2/g. The mixture of two produced samples was used in the experiment. Fig. 2 shows the particle size distribution of the two produced samples which was determined by Blain method.

3 wt% of the mixed ash was replaced by a mixture of SiO_2 and Al_2O_3 nanoparticles which were prepared from Nano Pars Lima Co. The average particle size of both SiO_2 and Al_2O_3 nanoparticles was approximately 80 nm. The properties of SiO_2 and Al_2O_3 nanoparticles have been given in Table 2. TEM micrographs of SiO_2 and Al_2O_3 nanoparticles has been illustrated in Fig. 3a and b, respectively and their XRD pattern in Fig. 4a and b, respectively.

Sodium silicate solution or water glass (WG) and sodium hydroxide (NaOH) were used as the solution part of the mixture. WG was used without following modification, but the

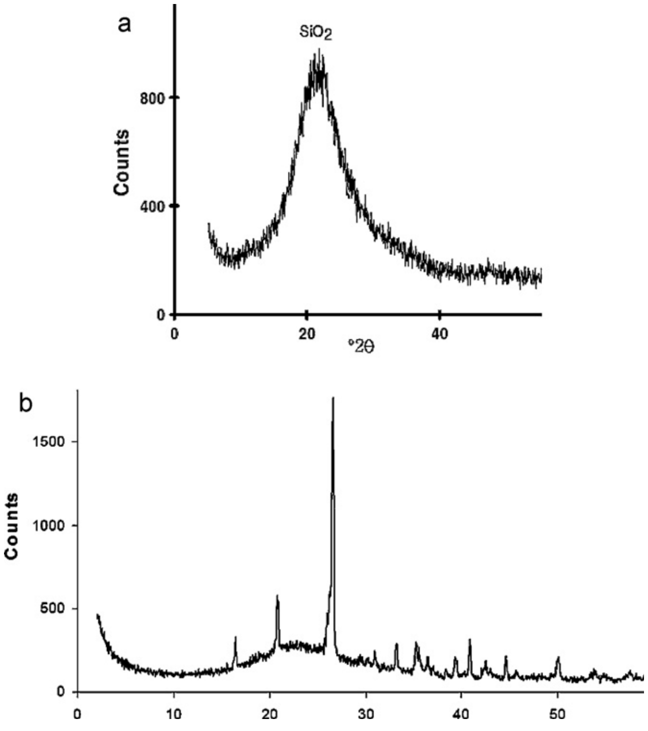

Fig. 1. XRD pattern of (a) RHA and (b) FA used in this study.

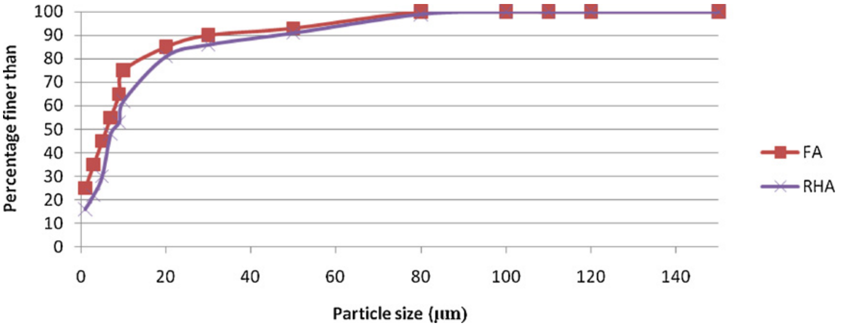

Fig. 2. Particle size distribution pattern of the different ashes used in this study.

sodium hydroxide was diluted to different concentrations before using. The chemical composition of the utilized WG is also given in Table 1.

Four main considered factors for Taguchi experiment design were nano silica weight percent, oven curing temperature, oven curing time and NaOH concentration each at 3 levels. The 3 levels of each factor have been illustrated in Table 3. Taguchi experiment design was performed by Qualitek 4 software. The suggested design by Taguchi method for 4 factors at 3 levels is L9 array in accordance to Table 4.

Totally 9 series of geopolymer specimens with the mixture proportions illustrated in Table 5 were prepared for compressive strength tests. The mixed alkali activator of sodium silicate solution and sodium hydroxide was used. Sodium hydroxide was diluted by tap water to have concentrations of 5, 8 and 12 M. The solution was left under ambient conditions until the excess heat had completely dissipated to avoid accelerating the setting of the geopolymeric specimens. The sodium silicate solution without preparation was mixed with the sodium hydroxide solution. The ratio of the sodium silicate solution to sodium hydroxide solution was 2.5 by weight for all mixtures because this ratio demonstrated the best properties for fly ash-based geopolymer [21,22]. For all samples, the mass ratio of alkali activator to FA-RHA mixture was 0.4. To ensure the complete dispersion of nanoparticles in the paste, they were mixed by alkali activator solution and then the mixture was added to the base ash. Pastes were mixed by shaking for 5–10 min to give complete homogenization. The mixtures were cast in 50 mm edge polypropylene cubic molds. The mixing was done in an air-conditioned room at approximately 25 °C. The molds were half-filled, vibrated for 45 s, filled to the top, again vibrated for 45 s, and sealed with the lid. The mixtures were then precured for 24 h at room temperature because this precuring time has been found to be beneficial to strength development [23]. Precuring time before application of heat induces significant dissolution of silica and alumina from fly ash and formation of a continuous matrix phase, increasing, therefore, the homogeneity of the geopolymeric materials [23,24]. After the precuring process, the samples and molds were placed in a water bath to prevent moisture loss and the carbonation of the surface. The curing regimes (water and oven curing) for each series of specimens were in accordance to Table 4. For the specimens cured at elevated temperature (70 and 90 °C), the time of oven-curing were also considered in water curing regime.

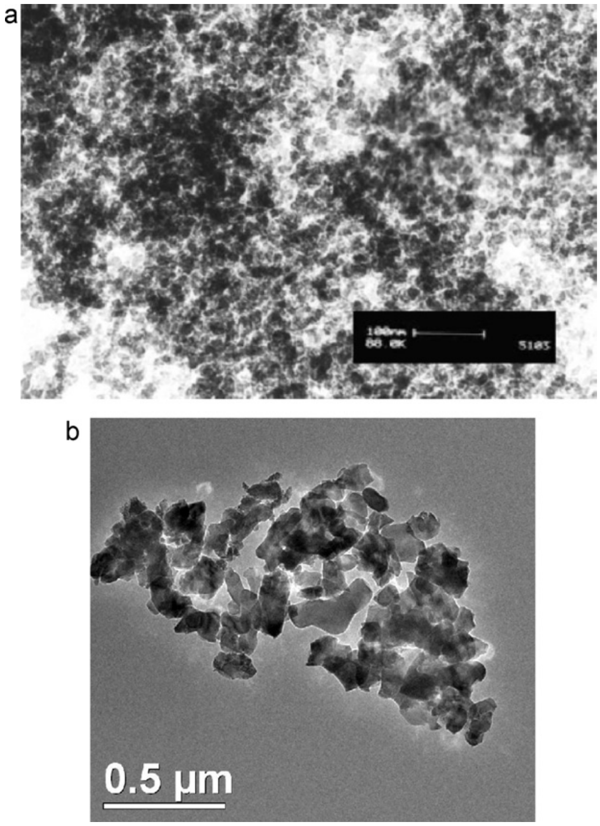

Fig. 3. TEM micrographs of (a) SiO_2 and (b) Al_2O_3 nanoparticles.

Table 2
Properties of SiO_2 and Al_2O_3 nanoparticles.

Nanoparticle type	Size (nm)	Specific surface (m^2/g)	Purity (%)	Appearance	Bulk density (g/cm^3)
SiO_2	80	>800	99.9	White powder	0.3
Al_2O_3 (α)	80	>10	99.9	White powder	1.6

Table 3
The introduced levels for each factor in Taguchi experiment design.

Factor	Level 1	Level 2	Level 3
SiO_2 content (wt%)	1	2	3
Oven curing temperature (°C)	25	70	90
Oven curing time (h)	2	4	8
NaOH concentration (M)	5	8	12

The compressive strength results of the produced specimens were measured on the cubic samples. The tests were carried out 3 times on each series and the average strength values were reported.

The microstructural characteristic of geopolymeric specimens was analyzed by using SEM which was conducted on a 7538 Oxford apparatus. Secondary electron (SE) imaging was used to study the samples which were prepared under conditions that ensured their subsequent viability for analytical purposes.

3. Results and discussion

Fig. 5 compares the compressive strength of geopolymers at 2 and 7 days of water curing regimes. After 7 days, the compressive strength has been increased showing the more completed geopolymerization reactions. This is in accordance to the previous works [6].

Table 6 shows the percentage of enhancement in the compressive strength of the specimens due to increase in water curing regime from 2 to 7 days. According to Table 6, the

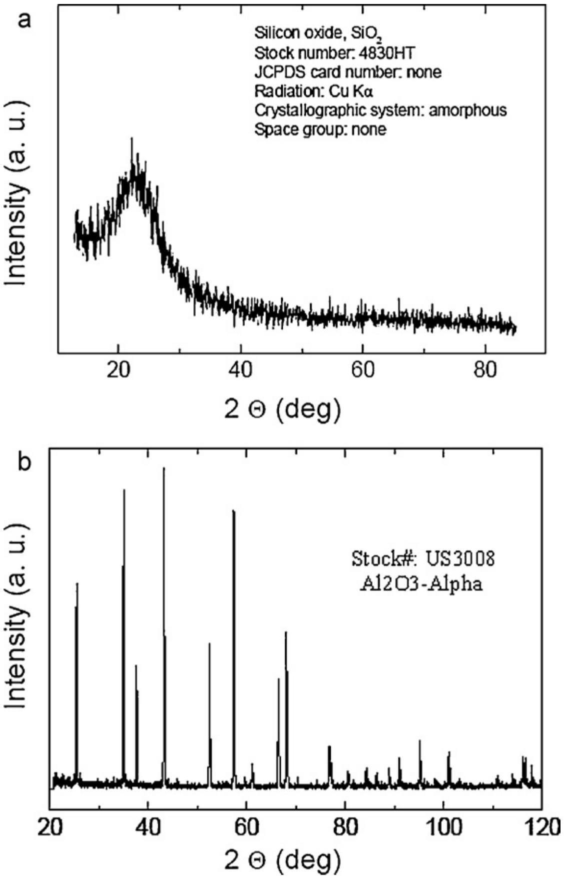

Fig. 4. XRD patterns of (a) SiO_2 and (b) Al_2O_3 nanoparticles.

Table 4
Suggested experiment series by Taguchi method for 3 factors in 3 levels in this work.

Experiment series	SiO_2 content (wt%)	Oven curing temperature (°C)	Oven curing time (h)	NaOH concentration (M)
1	1	25	2[a]	5
2	1	70	4	8
3	1	90	8	12
4	2	25	4[a]	12
5	2	70	8	5
6	2	90	2	8
7	3	25	8[a]	8
8	3	70	2	12
9	3	90	4	5

[a] Notification: at 25 °C, there was no oven curing and therefore, the suggested times should be eliminated.

Table 5
Mixture proportion for the specimens (the quantities are in kg/m^3).

Sample designation	RHA	FA	WG	NaOH	Nano SiO_2	Nano Al_2O_3
N1	720	2880	860	344	36	72
N2	720	2880	860	344	36	72
N3	720	2880	860	344	36	72
N4	720	2880	860	344	72	36
N5	720	2880	860	344	72	36
N6	720	2880	860	344	72	36
N7	720	2880	860	344	108	–
N8	720	2880	860	344	108	–
N9	720	2880	860	344	108	–

Alkali activator (WG + NaOH) to FA–RHBA-nanoparticles mixture ratio is 0.4.

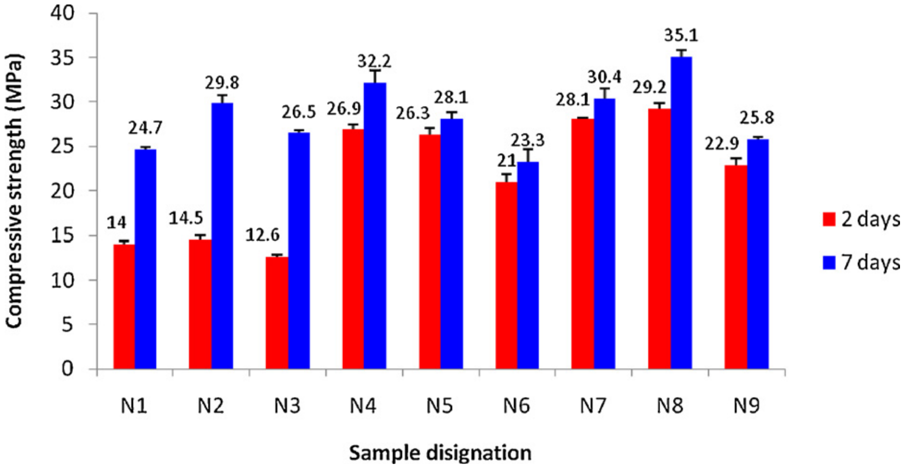

Fig. 5. Compressive strength of geopolymers at 2 and 7 days water curing regime.

Table 6
The percentage of enhancement in the compressive strength of the specimens due to increase in water curing regime from 2 to 7 days.

Experiment series	Sample designation	Percentage of strength enhancement
1	N1	43.32
2	N2	51.34
3	N3	52.45
4	N4	16.46
5	N5	6.41
6	N6	9.87
7	N7	7.57
8	N8	16.81
9	N9	11.24

highest increase is related to the specimens cured at 25 °C (N1–N3 specimens). This shows that completion of geopolymerization at 70 and 90 °C at 2 days of water curing is more than that of 25 °C. The highest increase in strength at 25 °C (N1–N3 specimens), 70 °C (N4–N7 specimens) and 90 °C (N7–N9 specimens) is related to N3, N6 and N8 specimens, respectively all with NaOH concentration of 12 M. On the other hand, the lowest increase in strength at 25 °C (N1–N3 specimens), 70 °C (N4–N7 specimens) and 90 °C (N7–N9 specimens) is related to N1 (with NaOH concentration of 5 M), N5 (with NaOH concentration of 8 M) and N7 (with NaOH concentration of 8 M) specimens, respectively. As seen, the highest increase at each curing temperature has been achieved at NaOH concentration of 12 M while the lowest increase at 5 and 8 M. These results may indicate that NaOH concentration has not evident effect on increasing the compressive strength as important as oven curing temperature. Same situation is observed about oven curing time. As indicated, the highest strength in each group was related to N3, N6 and N8 specimens with 8, 2 and 2 h oven curing while the lowest strength obtained for N1, N5 and N7 specimens 2, 8 and 8 h oven curing. These conditions are completely complicated and required to be considered by a more accurate analysis like ANOVA as discussed in the following.

The significance of the main effects, the percentage of participation and the optimum condition of the considered factors on the compressive strength of the specimens at 2 days of water curing which was obtained by ANOVA, has been presented in Fig. 6, Tables 7 and 8, respectively.

Fig. 6 shows that the oven curing temperature is the main factor affects the compressive strength by a participation percentage of 86.616 (Table 7) and the temperature of 90 °C (Table 8). While the concentration of NaOH solution has its effect, the changes in curing time and nano silica weight percent have no considerable effect on strength at 2 days of curing. This

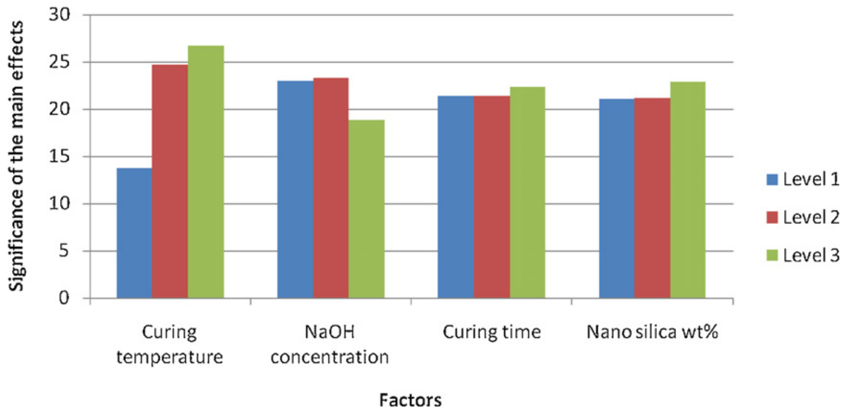

Fig. 6. The significance of the main effects of the considered factors on the compressive strength at 2 days of water curing.

Table 7
The participation percentage of the considered factors on the compressive strength at 2 days of water curing.

Factors	Oven curing temperature	NaOH concentration	Oven curing time	SiO_2 (wt%)
Percentage of participation	86.616	11.053	0.492	1.836

has been shown in Table 7 where the participation percentage of oven curing time and nano silica weight percent are 0.492 and 1.836, respectively. Table 7 shows that the percentage of participation for NaOH concentration is 11.53 and Fig. 6 shows no significant difference between NaOH concentrations of 5 and 8 M. The selection of 8 M by ANOVA in Table 8 is due to a slightly more participation of this concentration.

The obtained results on the effects of oven curing temperature on compressive strength of geopolymers show that the increased temperature consequences the increased strength [25]. However, in some cases, increasing the curing temperature to more than 75 °C has been shown to decrease the strength [6–8]. It seems that the chemical composition of the base ash has a key role to determine the optimum temperature. In this study, utilization of a mixture of RHA and FA resulted in the optimum temperature of 90 °C.

Although in approximately all geopolymers, the increase in oven curing temperature up to an optimum level has been reported as an effective factor on increasing the strength [6–8,23–25], the role of NaOH concentration has not been approved. While in our previous works [6–8] the NaOH concentration of 12 M has been illustrated the most effective concentration on compressive strength, some researchers [24] have shown the negative impact of NaOH concentration more than 10 M. It seems that there is a strong relationship between the base utilized ash and NaOH concentration. If the extra concentration of NaOH could not be used by the ash, the strength will be decreased due to the incomplete geopolymerization reactions. Our experimental evident also showed that NaOH concentration of 12 M has an inverse effect on compressive strength. Fig. 7a shows a specimen made by NaOH with a concentration of 12 M. The incomplete reaction at the middle of the specimen is clearly observed. This is while for 5 and 8 M concentrations as it has been shown in Fig. 7b and c, the reaction has been completed. However, this completion is more evident in the specimen with NaOH concentration of 8 M.

According to Table 7, the participation of curing time in determining the compressive strength is very low. However, Table 8 shows that the optimum curing time of 8 h results in increasing the compressive strength of geopolymers [25].

Table 8
The optimum condition of the considered factors on the compressive strength at 2 days of water curing.

Factors	Oven curing temperature (°C)	NaOH concentration (M)	Oven curing time (h)	SiO_2 (wt%)
Percentage of participation	90	8	8	3

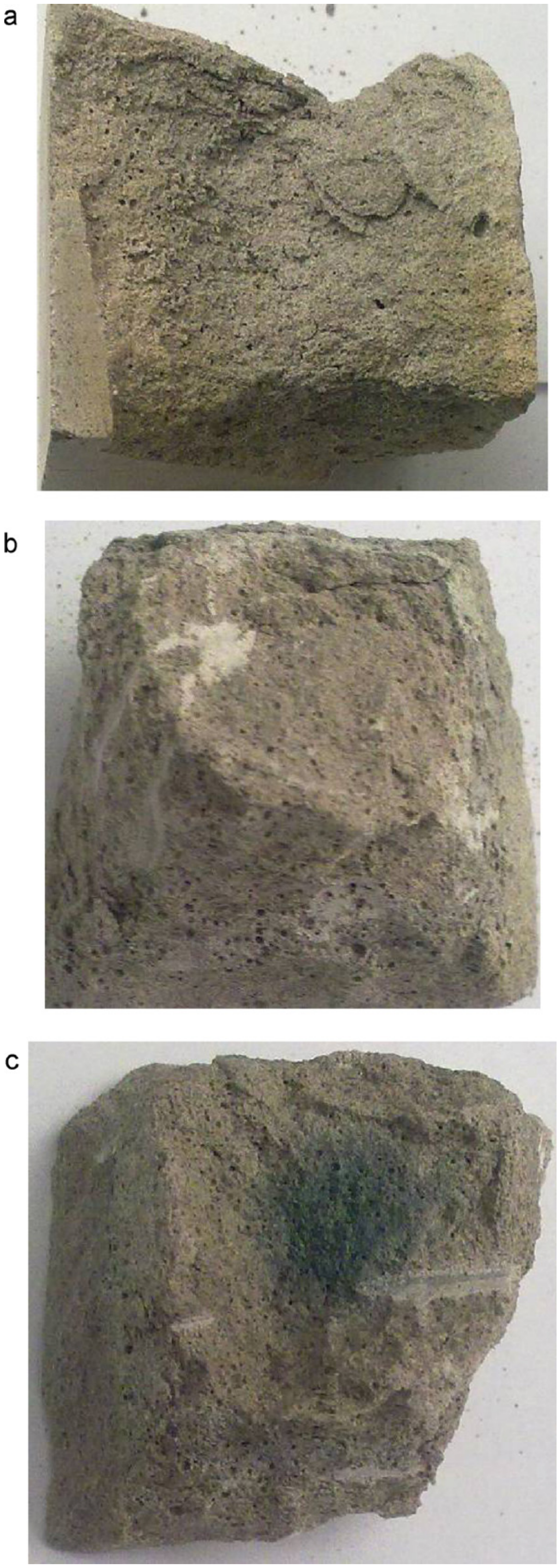

Fig. 7. The fractured geopolymers specimen after compressive strength tests with NaOH concentrations of (a) 5, (b) 8 and (c)12 M.

The participation of nano SiO_2 on compressive strength of the geopolymers is also low, but according to Table 8, the optimum nano SiO_2 content is 3 wt%. In the other words, the

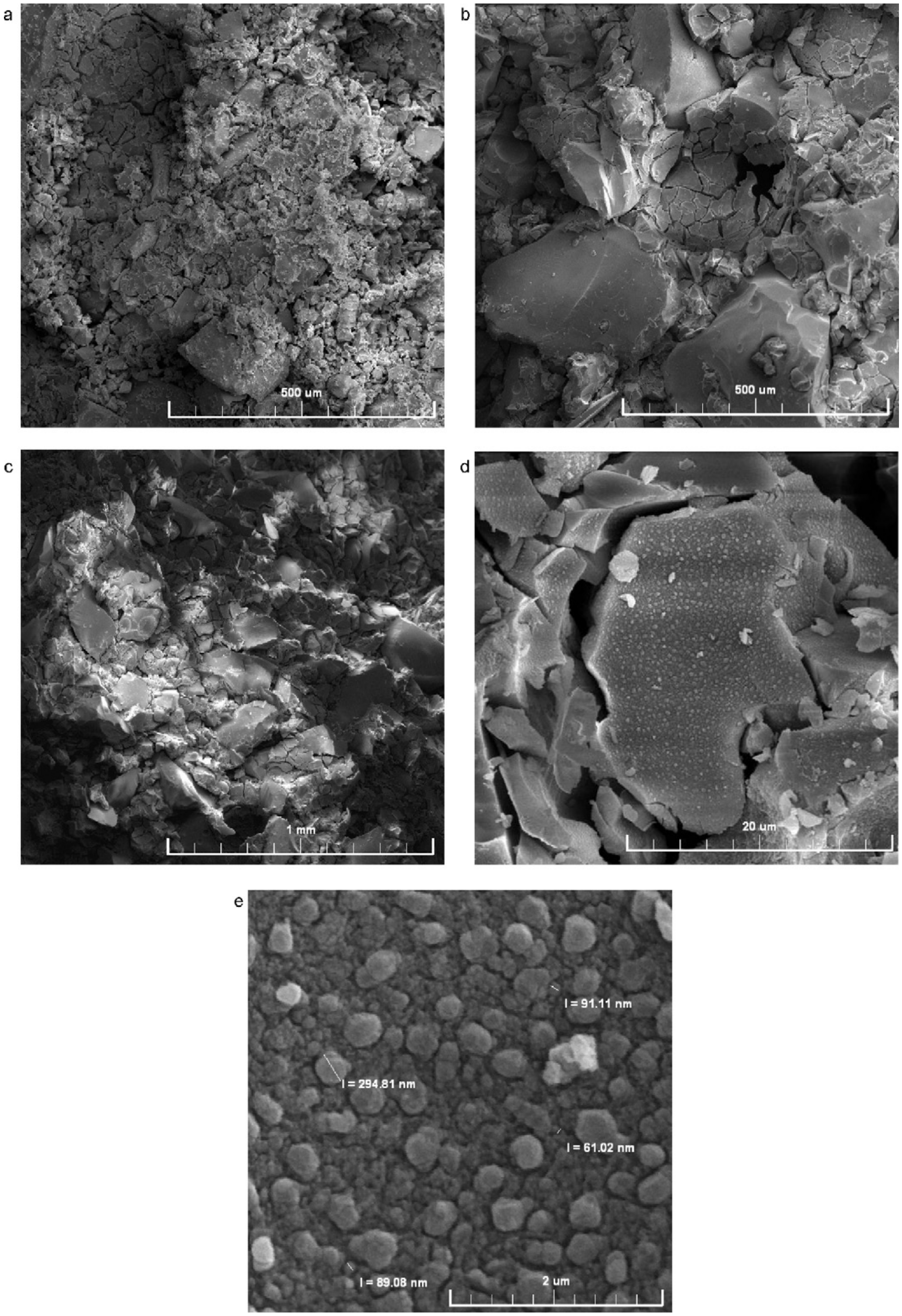

Fig. 8. SEM micrographs of the specimens with NaOH concentration of (a) 5, (b) 8 and (c) 12 M, (d) nanoparticles distribution in an specimen and (e) part d with higher magnification.

optimum content of nano Al_2O_3 is zero. Several works have been conducted on the effects of SiO_2/Al_2O_3 ratio on compressive strength of geopolymers and it has been shown that there is an optimum ratio for the highest strength. For instance, in our previous works [6–8], this ratio was obtained equals 2.99. In the present work, this ratio has been considered equals 2.54. Disaffiliation of nano Al_2O_3 in determining the compressive strength could be justified by Fig. 1. According to Fig. 1a, the consumed nano SiO_2 is amorphous while Fig. 1b shows that the utilized Al_2O_3 is crystalline. Several works conducted on concrete specimens [3] shows that only an amorphous material could effectively participate on increasing

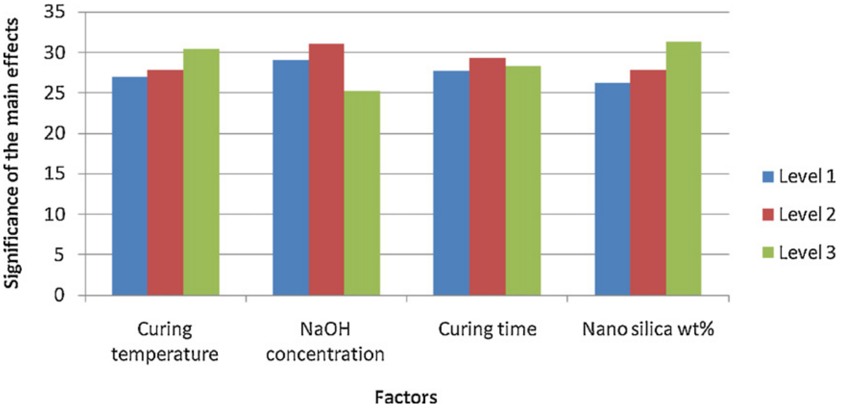

Fig. 9. The significance of the main effects of the considered factors on the compressive strength at 7 days of water curing.

Table 9
The participation percentage of the considered factors on the compressive strength at 7 days of water curing.

Factors	Oven curing temperature	NaOH concentration	Oven curing time	SiO_2 (wt%)
Percentage of participation	16.568	45.442	3.228	34.759

the strength. Separation of an atom from the crystalline structure for generating fresh bands is a hard, time-consuming phenomenon. So, the utilized Al_2O_3 probably not participated in geopolymerization and hence its percentage of participation is zero.

SEM micrographs of the fractured surface of the specimens with NaOH concentrations of 5, 8 and 12 M have been shown in Fig. 8a, b and c, respectively. Fig. 8b, the specimen with NaOH concentration of 8 M, shows the generation of fewer cracks with respect to the specimens with NaOH concentrations of 5 M (Fig. 8a) and 12 M (Fig. 8c). As indicated previously, one of the affective parameters in increasing the strength is utilizing NaOH concentration of 8 M for the studied ash.

Fig. 8d and e shows the distribution of nanoparticles in a studied specimen in different magnifications. It is obvious that the nanoparticles have been finely dispersed and hence can help the specimen to have an increased strength. One of the suggestions that can help to produce more strengthened specimen may be using lower percentage of nanoparticles. According to Fig. 8d, in several cases, the nanoparticle sizes are more than 80 nm (the primary size of nanoparticles). This may be due to agglomeration of the nanoparticles. When a uniform distribution of nanoparticles is achieved and nanoparticles have not enough space for supplying the paste, they are agglomerated and the additional nanoparticles could not participate on increasing the strength [26].

On the whole, increasing amorphous SiO_2 nanoparticles (decreasing crystalline Al_2O_3 nanoparticles), increasing curing time and temperature and utilizing low and medium NaOH concentration could result in the highest strength for the studied ash at 2 days of water curing.

For verification experiment, 3 specimens were made according to the optimum condition of Table 8 and the average compressive strength of 31.0 ± 1.4 MPa was achieved higher than those examined in the previous 9 series experiments.

The significance of the main effects, the percentage of participation and the optimum condition of the considered factors on the compressive strength of the specimens at 7 days of water curing which was obtained by ANOVA, has been presented in Fig. 9, Tables 9 and 10, respectively.

Table 10 shows that the main percentage of participation on determining the compressive strength is related to the NaOH concentration and SiO_2 nanoparticles content. It seems that by increasing the water curing regime, nanoparticles can act as a heterogeneous nuclei [19] and accelerator and promoter of the reaction [26]. In this condition, the participation percentage of curing temperature is decreased. It seems that curing temperature has its effect at 2 days of curing and after that, the reaction between nanoparticles, NaOH and the utilized ash can control the following reaction. Since nanoparticles can contribute in this stage, NaOH participation will increase and operates as the main factor. However, similar to the 2 days water curing regime, the optimum conditions for curing temperature and NaOH concentration are 90 °C and 8 M, respectively. According to Tables 9 and 10, the participation percentage of curing time and its optimum level are 3.228 and 4 h. This shows that this parameter has an unimportant effect on compressive strength similar to 2 days water curing regime.

Table 10
The optimum condition of the considered factors on the compressive strength at 7 days of water curing.

Factors	Oven curing temperature (°C)	NaOH concentration (M)	Oven curing time (h)	SiO_2 (wt%)
Percentage of participation	90	8	4	3

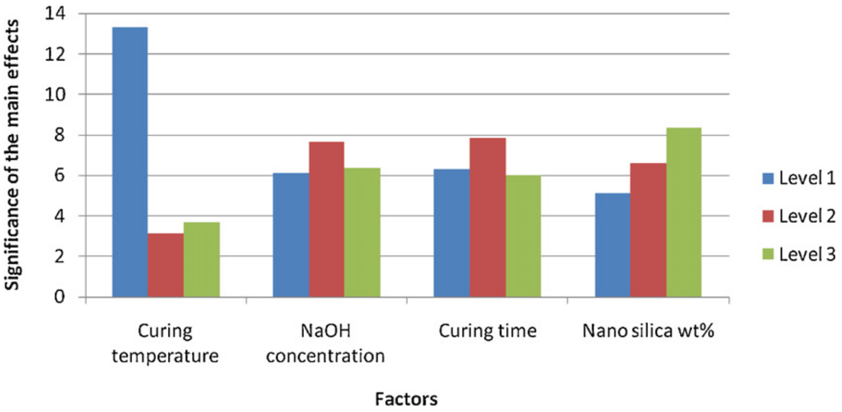

Fig. 10. The significance of the main effects of the considered factors on the compressive strength between 2 and 7 days of water curing.

Table 11
The participation percentage of the considered factors on the compressive strength between 2 and 7 days of water curing.

Factors	Oven curing temperature (°C)	NaOH concentration (M)	Oven curing time (h)	SiO_2 (wt%)
Percentage of participation	88.388	1.902	2.618	7.089

Table 12
The optimum condition of the considered factors on the compressive strength between 2 and 7 days of water curing.

Factors	Oven curing temperature (°C)	NaOH concentration (M)	Oven curing time (h)	SiO_2 (wt%)
Percentage of participation	25	8	4	3

On the whole, increasing SiO_2 nanoparticle content, increasing the curing time and utilizing of medium NaOH concentration with 4 h oven curing could produce a specimen with the highest strength.

For verifying the results, 3 specimens were made and tested in accordance to the optimum condition of Table 10 and an average compressive strength of 38.5 ± 1.9 was achieved more than those examined in the 9 previous experiments at 7 days of water curing regime.

The significance of the main effects, the percentage of participation and the optimum condition of the considered factors on the compressive strength of the specimens for increasing the water curing regime from 2 to 7 days which was obtained by ANOVA, has been presented in Fig. 10, Tables 11 and 12, respectively.

From Table 12 it is evident that the curing temperature of 25 °C has the highest effect on increasing the compressive strength after 2 days until reaching to 7 days of water curing. When the temperature is 90 °C, the geopolymerization reaction will be roughly completed and after that the increase in strength is not sensible. But, when curing is occurred at 25 °C, the geopolymerization will promote slower and after that by increasing the water curing time to 7 days, the reaction will be completed more. Therefore, geopolymerization in the absence of oven curing will more complete between the 2 and 7 days water curing regime. In this condition, again NaOH concentration of 12 M and utilizing of nano Al_2O_3 has not been suggested and this confirms that in high concentration of NaOH and utilizing of crystalline Al_2O_3, the reaction will promote slowly.

4. Conclusions

1. The obtained results showed that one can produce high strength geopolymers with RHA and FA together with nanoparticles.
2. The optimum curing temperature in both 2 and 7 days water curing regime was acquired 90 °C. The best compressive strengths in this temperature were 31.0 ± 1.4 and 38.5 ± 1.9 for 2 and 7 days of water curing, respectively.
3. The optimum NaOH concentration of 8 M has been shown to have the most effect on compressive strength of geopolymers.
4. Utilizing of 3 wt% SiO_2 nanoparticles resulted in obtaining the highest strength. Al_2O_3 nanoparticles had no effect on the compressive strength. This may be due to the crystalline nature of Al_2O_3 nanoparticles which could not effectively contribute on increasing the strength.
5. The oven curing time was not a significant factor in determining the compressive strength.

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