Received: 19 November 2019 | Revised: 19 February 2020 | Accepted: 6 April 2020
DOI: 10.1002/suco.201900479

TECHNICAL PAPER

fib CEB-FIP WILEY

Influence of nano SiO_2 and nano $CaCO_3$ particles on strength, workability, and microstructural properties of fly ash-based geopolymer

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Funding information
Erciyes Üniversitesi, Grant/Award Number: FDK-2018-7912

Abstract

The influence of nano SiO_2(NS) and $CaCO_3$(NC) particles on the properties of class F fly ash based geopolymer mortar activated with different sodium ion concentrations have been investigated. Mortar mixture proportions were 1:3:0.3 for binder, sand, and water, respectively. Nano SiO_2 and $CaCO_3$ particles were replaced with a binder by weight basis at the ratios of 1, 2, and 3% in the mixtures. Sodium concentrations amount used were 8, 10, and 12% Na^+ of binder content. Geopolymer mortar samples were cured at 60, 75, and 90°C in a furnace for 24, 48, and 72 hr. After the heat curing process, flexural, and compressive strength tests were performed. The changes in the microstructure of geopolymer due to influence of nanoparticles were examined by utilizing isothermal calorimetric studies on geopolymer paste, and field-emission scanning electron microscopy (FESEM). Based on laboratory work results, it was concluded that for all sodium ion concentrations, the addition of nano SiO_2 and $CaCO_3$ particles improved the flexural and compressive strengths after 24 hr heat curing. However, the favorable effects of nanoparticles on strength properties tend to disappear after 48 and 72 hr heat curing. The results of isothermal calorimetric studies showed that nano SiO_2 and $CaCO_3$ particles accelerated the geopolymeric reactions at an early age. FESEM results showed that additions of nanoparticles made the microstructure of geopolymer products more intense and compact.

KEYWORDS

Fly ash, geopolymer, morphology, nano $CaCO_3$, nano SiO_2, reaction kinetics

1 | INTRODUCTION

Currently, studies on producing geopolymer are taking more concern as an alternative binder to normal Portland cement.[1–4] Geopolymer is known as a novel inorganic polymer binding material obtained from the reaction of a solid aluminosilicate source (as being generally class F fly

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ash) with alkaline solutions. It is reported that geopolymeric reaction of class F fly ash depends on the acceleration of the chemical reaction with alkali solutions and heat curing at 40–75°C.[5–8] Geopolymeric binder ensures strength, durability, and environmental sustainability. Therefore, it is accepted as a greener alternative to plain Portland cement in the construction field over the last two decades due to a large amount of greenhouse gas emission of plain Portland cement production. One tone of Portland cement production results in approximately one ton of carbon dioxide emissions into the atmosphere. Cement industry contributes about 8% of global CO_2 emission in the world.[8–16]

Advancement in new technology made possible to produce nano sized solid materials called nanoparticles. Utilizing and incorporation of nanoparticles in cement or cementitious binder is a novel concept among the researchers.[17–20] In the following sections, a literature review on introducing nanoparticles in cementitious materials was presented.

Deb et al. worked on the influence of nano SiO_2 addition on the development of properties of fly ash-based geopolymer mortar mixtures at room temperature curing. Strength, results showed that the addition of nano SiO_2 improved strength development favorably. For instance, while control samples developed 29 MPa, samples produced with 2% nano SiO_2 addition developed 67 MPa compressive strength. The authors attributed this behavior to that inclusion of soluble silicates accelerated the geopolymeric reaction process and helped build the long-chain silicate oligomers in the geopolymer matrix and thus improved the strength of this behavior was also detected by other researchers.[21–24] Furthermore, Fernandez and Palomo[25] reported that the extent of the soluble silicon concentration is an important parameter that plays a significant role in controlling the geopolymerization process. The authors concluded that the optimal amount of nano SiO_2 was about 2% replacement with fly ash in the mixture. EDX and scanning electron microscopy (SEM) analysis were carried out to evaluate microstructural changes due to the addition of nano SiO_2. They claimed that inclusion of nano SiO_2 improved microstructure, and made microstructure denser and more compact, thus, contributing and resulting with higher strength.[26]

Nath et al., studied on morphology and microstructure of fly ash-based geopolymers. They used an isothermal calorimetry measurement results to identify and observe the influence of the mixture parameter on the formation of geopolymerization. They identify the morphological property by SEM pictures. Based on laboratory study, it was reported that the morphology of geopolymer changed by the increase in alkali-concentration as well as curing temperature. They concluded that optimum results were obtained from the mixture made with 8 M NaOH concentration.[27]

Ibrahim et al., aimed to improve pozzolanic reaction kinetics and mechanical properties of the mixture made with natural pozzolana, to achieve this they added nano SiO_2 at the replacement ratios of 1, 2.5, 5, and 7.5% of binder amount into the mixture. They reported that the highest compressive strength was obtained from samples of the mixture that made with 5% nano SiO_2. Similarly, the evaluation of SEM pictures showed that mixtures made with 5% nano SiO_2 exhibited more homogenous and denser microstructure as well as it contained a lesser amount of unreactive SiO_2 particles in comparison to other mixtures.[28]

Gao et al., studied the influence of incorporation of nano-silica into eco-friendly alkali activated slag-fly ash blends on the properties of the mixture including the fresh behaviors, reaction kinetics, gel structure, porosity, and strength for different nano-silica contents and slag/fly ash ratios. Based on laboratory research, they reported that the inclusion of nano-silica in alkali activated slag-fly ash mixture reduced workability significantly with the amount of nano-silica, setting times increased, and reaction kinetics slowed down. They concluded that nano-silica addition up to 2% replacement ratio result with an increase in compressive strength while it reduced the porosity value of samples. They claimed that the favorable influence of nano-silica addition was a result of filler influence of nanomaterials as well as the amorphous nature of nano-silica added to the mixture. They also reported that the slag content showed a dominant influence on setting times, early age reaction, compressive strength, and porosity in the blended alkali system.[29]

Naskara and Chakraborty, produced nano-particles added low calcium fly ash-based geopolymer concrete activated with sodium hydroxide and sodium silicate to investigate the influence of adding different kind nano-particles on the properties of geopolymer concrete mixtures. They reported that the addition of nano-silica did not significantly contribute to 7 and 28 days compressive strength of fly ash based geopolymer.[30]

Adak et al., investigated the properties of fly ash-based alkali geopolymer produced by alkali activation. They used 8, 10, and 12 M alkali solution in preparation of a geopolymer mixture. Also, nano-silica was added to a geopolymer mixture with the amount of 0, 4, 6, 8, and 10% of the weight of fly ash. They cured a geopolymer mixture at laboratory ambient temperature for 3, 7, and 28 days; then tested. Maximal compressive strength (in the order of 40 MPa) was obtained from 6% nano-silica added mixture that made with a 12 M alkali solution. They carried out further analysis on the same mixture containing 6% nano-silica, X-ray diffraction (XRD), and field-emission scanning electron microscopy (FESEM) analysis. Based on XRD and FESEM analysis that they observed more crystalline phases existed in the sample containing nanoparticles in comparison to a sample containing no nanoparticles. They attributed the improvement in abovementioned properties to the transformation of an amorphous compound to crystalline compound as supported by XRD and FESEM analysis. Also, they reported that the microstructure of the sample containing nano-silica was denser than that of the sample made without nano-silica. Furthermore, they observed unreacted fly ash particles on a sample made without nano-silica.[17]

Rong et al., carried out a laboratory investigation to find the influence of nanoparticles addition to the cementitious composites system. In the context of the study, mechanical properties, hydration kinetics, and microstructural analysis were carried out. As a result of the study, they reported that up to 3% addition of nanoparticles inclusion increased flexural and compressive strength, however, utilizing more than 3% addition result with a decrease in strength due to agglomeration of nano-SiO_2 particles. They determined that nano-silica addition fasten the hydration process. Porosity and average pore size were reduced by the presence of nano-silica in the mixture, an increase in the amount of nano-silica caused more reduction in porosity and pore size. They observed that nano-silica added mixture had more homogenous and denser microstructure which was attributed to filler effect and additional pozzolanic property of nano-silica particles.[31]

Assadi et al., carried out a laboratory study to evaluate and determine the difference caused by mixing methods of adding nano-silica on microstructural and mechanical properties of flax fabric reinforced geopolymer composites mixtures. As a result of the study, adding nano-silica into mixture in the dry state gave favorable results than that of nano-silica added in the wet state. In general, nano-silica addition results with lower water absorption and porosity values in comparison to reference mixture made without nano-silica. They reported that dry-mixed geopolymer nanocomposite with 1.0 wt% nano-silica reduced the porosity (by 27%) and water absorption (by 35%) and increased the density (by 15%). It also results with an increase in flexural tensile and compressive strength, the increase was in the order of 28% for both strengths. They stated that these increases and improvements in the properties of geopolymer were due to the filler effect of nano-silica as well as being and providing extra amorphous silicate content to the mixture system. However, it was concluded that the addition of nano-silica beyond 1.0 for dry-mix and 2.0 wt% for wet-mix, respectively, influenced the engineering properties oppositely.[32]

Yesilmen et al., studied on nano-modification for improvement of ductility properties of cementitious mixtures made with the addition of nanoparticles to high volume fly ash and cement system. They compared flexural tensile and compressive strength development of mixtures. Microstructural surface characteristics were analyzed using SEM. They concluded that the addition of nanoparticles to high volume fly ash cementitious systems improved the engineering properties of mixtures.[33]

Phoo-ngernkham et al., added SiO_2 and Al_2O_3 nanoparticles to high calcium fly ash-based geopolymer paste and investigated the engineering properties of mixtures produced at laboratory ambient conditions. Nanoparticles were added to mixtures as a mass replacement in ratios of 0, 1, 2, and 3% of fly ash mass. Alkali activator was blended with sodium hydroxide and sodium silicate. According to testing results, it was determined that nano SiO_2 reduced setting times of geopolymer more than Al_2O_3 did. Adding nanoparticles up to 2% replacement, regardless of being silica or alumina to mixture increased flexural tensile and compressive strength and elastic modulus. In addition, XRD and SEM studies carried out showed that nanoparticles added geopolymer mixture owned denser microstructure and increased rate of reaction product.[34]

A literature survey has shown that the utilization of nanoparticles in geopolymeric mixtures needs more study since published materials on this subject are limited. Particularly, there is no literature on the reaction kinetics of neither on fly ash-based geopolymer mixture nor geopolymer mixtures made with nanoparticles addition. The aim of this study is to provide comprehensive data on this subject. For this purpose, a systematic, and parametric experimental work was undertaken. In the study, experimental variables were selected as the amount of NaOH or molarity, activation temperature, heat curing period as well as the type and amount of nanoparticle.

2 | METHODOLOGY OF RESEARCH

2.1 | Materials

2.1.1 | Fly ash

Fly ash was obtained from Iskenderun Thermal Power Plant located in Sugozu village of Adana located in southern Turkey. Chemical compositions of class F fly ash and XRD results were presented in Table 1 and Figure 1, respectively. As can be seen from the chemical compositions total amount of $SiO_2 + Al_2O_3 + Fe_2O_3$, is more than 70% and CaO amount is less than 10%. Therefore, fly ash can be classified as class F type and low lime fly ash according to ASTM C618[35] specification. The specific gravity of fly ash was 2.36 and it's remaining on 45 μm sieve was 11%.

TABLE 1 Oxide composition of fly ash (%)

Oxide	SiO_2	Al_2O_3	Fe_2O_3	CaO	SO_3	Na_2O	K_2O	MgO	P_2O_5	TiO_2
Fly ash	57.34	22.05	7.92	2.71	0.57	1.70	2.72	1.79	0.66	1.05

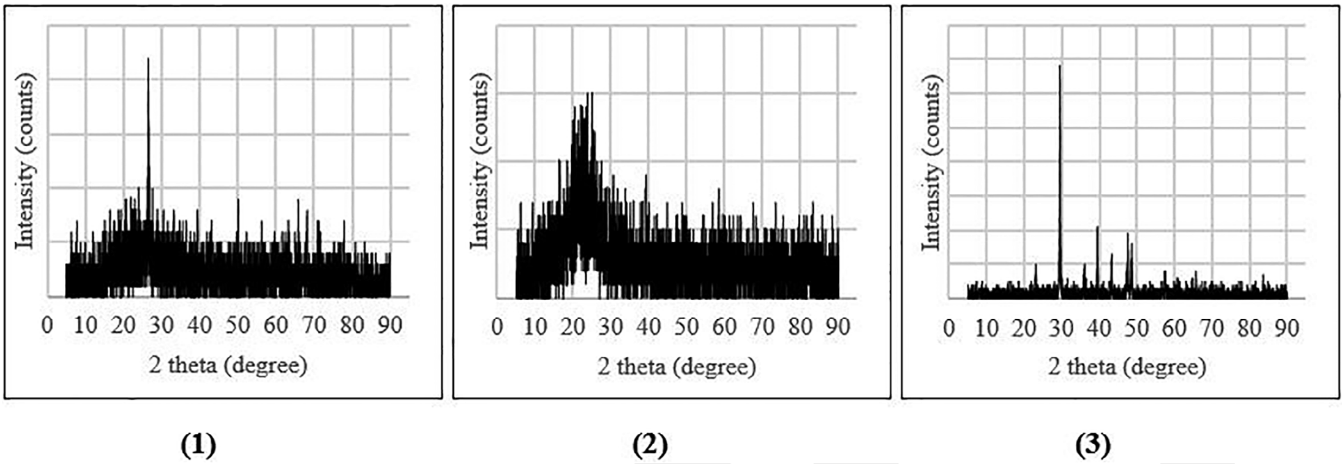


FIGURE 1 X-ray diffraction (XRD) of fly ash (1) nano SiO_2 (2) and nano $CaCO_3$ (3)

TABLE 2 Chemical oxide compositions of nano SiO_2 and $CaCO_3$

Oxide	SiO_2 (%)	CaO (%)	Na_2O (%)	P_2O_5 (%)	Al_2O_3 (%)	SO_3 (%)	MgO	SrO
Nano SiO_2	98.61	—	0.34	0.30	0.38	0.37	—	—
Nano $CaCO_3$	0.57	99.00	—	—	0.09	—	0.28	0.05

2.1.2 | Nanoparticles

The nano SiO_2 (NS) and nano $CaCO_3$ (NC) particles were provided from Nanografi Co. Ltd., Turkey. The chemical compositions of nano-silica and calcium carbonate particles and XRD results are given in Table 2 and Figure 1, respectively. According to Table 2, NS particles have 98.61% SiO_2 and NC particles have 99% $CaCO_3$. XRD patterns showed that nano SiO_2 is completely amorphous indicated by a raised background whereas nano $CaCO_3$ composed of well-crystallized calcite phase. The average sizes of NS and NC are 12–22 nm, and 50–70 nm, respectively, as stated by the manufacturer.

2.1.3 | Sand

Standard Rilem sand complied with TS EN 196–1[36] was supplied from Limak Cement Factory, and used in this investigation during mortar production. Sieve analysis of standard sand and limits of standard specifications were presented in Table 3. As to sieve analysis results, sand is suitable for usage in mortar.

2.1.4 | Activator and water

As an alkali activator, sodium hydroxide was chosen to be used in this study. Its chemical compositions were presented in Table 4. Clean and drinkable tap water was used for the production of geopolymer mortar.

2.2 | Sample preparation

All the samples were produced at a sand/binder of 3 and a water/binder of 0.3. Nano SiO_2 and nano $CaCO_3$ particles replacement ratios of 0, 1, 2, and 3% by mass of fly ash were used for mixture production. Activator dosages as Na^+ amount were 8, 10, and 12% of fly ash amount in mass basis. NaOH and water solution were prepared 1 day before mixing. In the production of mortar containing nanoparticles, the ultrasonic method, wet mix method, or dry mix method are generally preferred. In this research, dry mix method was adopted, since, dry mix method mostly gave better results than others.[32,37]

In the production of mortar first, nano SiO_2 and $CaCO_3$ particles were mixed with fly ash for 5 min by mixer on dry condition. (The) The sequence of mixing procedure presented

TABLE 3 Grading of sand used and standard specification

Sieve size (mm)	0.08	0.16	0.50	1.0	1.6	2.0
Remaining on sieve (%)	99	86	72	35	8	0
Standard limits (%)	99 ± 1	87 ± 5	67 ± 5	33 ± 5	7 ± 5	0

TABLE 4 Chemical ingredient of alkali activator

NaOH (%)	Na_2CO_3 (%)	CI (%)	Al (%)	Fe (%)	SO_4 (%)
98.49	1.42	0.03	0.02	0.01	0.02

in Figure 2. Three cell prism samples having dimensions of 40 × 40 × 160 mm (depth, height, length), were used to produce the prismatic specimens. Mix proportions of the mortars were given in Table 5 for three-cell mold. Notation of mixtures' names was explained in the following. For example, 8 N-NS2 means, mixture contain 8% Na^+(8 N) and 2% nanosilica particles (NS). The produced samples were cured at 60, 75, and 90°C, for 24, 48, and 72 hr in a laboratory oven. After heat curing process, the specimens were taken out of the oven and demolded and allowed to cool down to the laboratory environment 23 ± 2°C temperature, before testing.

In addition, paste samples for FESEM imaging were prepared with similar compositions to mortars except for the absence of sand. The hardened samples were then broken to pieces of 5 mm in size and finally coated before FESEM examinations.

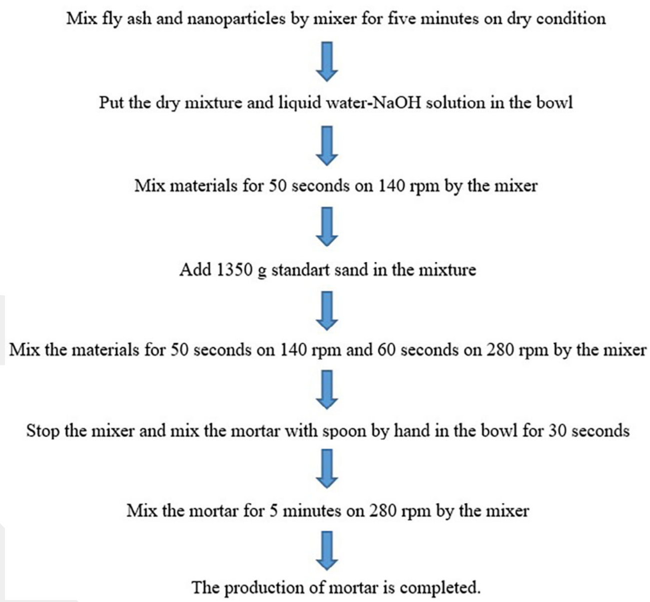


FIGURE 2 Mix steps

2.3 | Experimental program

2.3.1 | Flow tests for workability, unit weights, and ultrasonic pulse velocities

The flow test was performed according to TS EN 1015–3 specification.[38] For each mortar mixture, the flow diameter was measured in two directions and workability results were noted an average of two values. After the heat curing process, the samples taken out from the laboratory furnace were considered dry and their weights were measured, and the unit weights of the samples were determined. Before flexural and compressive strength testing, the ultrasonic pulse velocities of samples were measured after 24, 48, and 72 hr heat curing process according to ASTM C597-09[39] for each mixture.

2.3.2 | Compressive and flexural strength

Flexural tensile strength measurements were conducted on three prismatic specimens with 40 × 40 × 160 mm according to TS EN 1015–11[40] standard. Three-point loading test set up was used for flexural tensile strength. After the flexural tensile test, compressive strength was measured using six broken pieces of the specimen according to TS EN 1015–11[40] standard. Flexural tensile strength results were noted as an average of three specimens for each mixture. The compressive strength results were obtained from an average of six specimens for each mixture.

2.3.3 | FESEM analysis

Microstructural examinations were carried out the specimens containing nanoparticles in 2% NS, NC, and reference mix without nanoparticle after 60, 75, and 90°C heat curing process. The examinations were conducted on the fractured surfaces of hardened pastes by using a Zeiss GeminiSEM 500 FESEM in a vacuumed environment.

2.3.4 | Reaction kinetics

The reaction kinetics of geopolymer binder compositions with and without nanoparticles were examined by isothermal calorimetry (TAM Air, TA Instruments). In order to determine the reaction kinetics of the geopolymer pastes, the

TABLE 5 Mix proportion

Name of specimens	Fly ash g	Nano particle g	Sand g	Na^+ ratio %	NaOH g	Water g	Heat curing condition °C	Heat curing time Hr
8 N-NP0	450.0	0	1,350	8	62.6	135	60-75-90	24-48-72
8 N-NS1	445.5	4.5	1,350	8	62.6	135	60-75-90	24-48-72
8 N-NS2	441.0	9.0	1,350	8	62.6	135	60-75-90	24-48-72
8 N-NS3	436.5	13.5	1,350	8	62.6	135	60-75-90	24-48-72
8 N-NC1	445.5	4.5	1,350	8	62.6	135	60-75-90	24-48-72
8 N-NC2	441.0	9.0	1,350	8	62.6	135	60-75-90	24-48-72
8 N-NC3	436.5	13.5	1,350	8	62.6	135	60-75-90	24-48-72
10 N-NP0	450.0	0	1,350	10	78.3	135	60-75-90	24-48-72
10 N-NS1	445.5	4.5	1,350	10	78.3	135	60-75-90	24-48-72
10 N-NS2	441.0	9.0	1,350	10	78.3	135	60-75-90	24-48-72
10 N-NS3	436.5	13.5	1,350	10	78.3	135	60-75-90	24-48-72
10 N-NC1	445.5	4.5	1,350	10	78.3	135	60-75-90	24-48-72
10 N-NC2	441.0	9.0	1,350	10	78.3	135	60-75-90	24-48-72
10 N-NC3	436.5	13.5	1,350	10	78.3	135	60-75-90	24-48-72
12 N-NP0	450.0	0	1,350	12	93.9	135	60-75-90	24-48-72
12 N-NS1	445.5	4.5	1,350	12	93.9	135	60-75-90	24-48-72
12 N-NS2	441.0	9.0	1,350	12	93.9	135	60-75-90	24-48-72
12 N-NS3	436.5	13.5	1,350	12	93.9	135	60-75-90	24-48-72
12 N-NC1	445.5	4.5	1,350	12	93.9	135	60-75-90	24-48-72
12 N-NC2	441.0	9.0	1,350	12	93.9	135	60-75-90	24-48-72
12 N-NC3	436.5	13.5	1,350	12	93.9	135	60-75-90	24-48-72

samples were produced with 2% nanoparticle replacement for fly ash and also without nanoparticle, at various temperatures of 60, 75, and 90°C for 72 hr. The same geopolymer paste preparation procedure as applied for FESEM samples was also used. Samples were then immediately placed into the calorimeter. The calorimetry data at initial 15 min after mixing were excluded in order to ignore the data before the equilibration of samples within the instrument.

3 | RESULT AND DISCUSSION

3.1 | Flow tests, unit weights, and ultrasonic pulse velocities

3.1.1 | Flow test

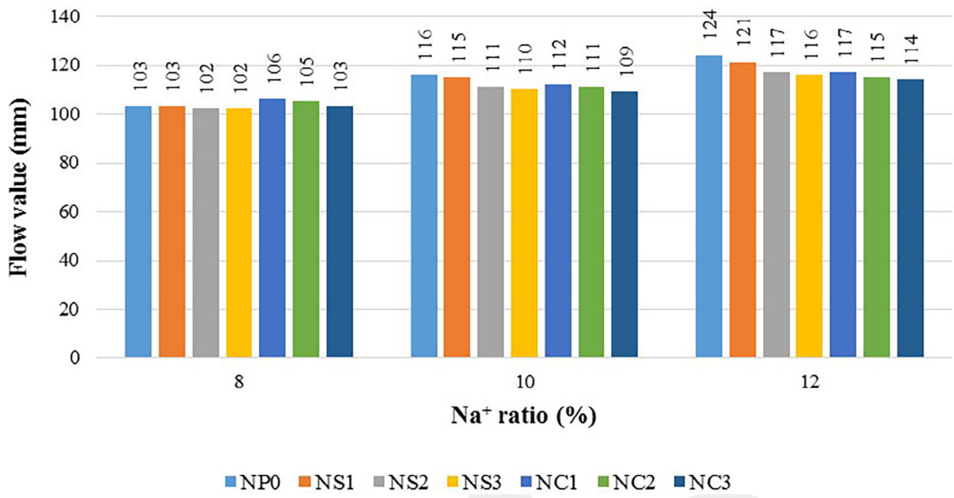
Flow workability testing results presented in Figure 3. Figure 3 shows that an increase in sodium concentrations improves the workability of mortar mixtures. While sodium concentration increased in the mixture from 8 to 10% and 12%; workability improvements were observed as approximately 12% and 20% in comparison to 8% sodium concentration, respectively. Higher sodium ion concentration result with higher workability. This finding found to be valid for all mixtures regardless of containing nanoparticles.

Addition of nano SiO_2 and $CaCO_3$ did not significantly influence the workability of the mixture at 8% sodium ion concentration. However, they tend to reduce the workability of the mixture at high sodium ion concentration (10 and 12%). It is known that the addition of nanoparticles in the mixture increases the specific surface area of powder material and causes more water absorption and reduces workability.[41]

3.1.2 | Unit weights and ultrasonic pulse velocities

Weight of prismatic specimens and ultrasonic pulse velocities were measured. Their unit weight was determined using the volume of specimens. Unit weight and ultrasonic pulse velocity measurement results were presented in Figures 4 and 5, respectively, for all mixtures, including control and nanoparticles added mixture. Addition of nanoparticles did not significantly

FIGURE 3 Flow values, (mm)



influence unit weight of specimens. Measured unit weights were found to various within 2.05–2.25 g/cm.[3] It was observed that increasing heat curing time tends to reduce unit weight of mortar specimens regardless of nanoparticles addition. This is explained by the evaporation of water within the sample, more curing time result with more evaporation of water.

A close observation of Figure 4 shows that increasing sodium ion concentration in the mixture increased the unit weight of the mortar mixture. It is accepted that the boiling point of NaOH solution increases with the increase in NaOH concentration, hence, boiling points temperature and evaporation are directly related.[12] Hence, higher Na^+ concentrations suppress the evaporation of water and result with higher unit weight.

Ultrasonic pulse velocity (UPV) of mixtures containing nanoparticles particularly for those mixtures cured for 24 hr were found to be higher than that of UPV of reference mixtures. This proves that the addition of nanoparticles improves and causes denser microstructure mostly for all mortar samples. It was observed that as the heat curing time increased, the UPV values tend to increase due to continuing geopolymeric reactions that fill the voids of sample and forming denser microstructure.[17,32] However, UPV values tend to reduce for the samples cured at 90°C for 72 hr. This was thought to be due to fine cracks occurred in samples for longer heat curing at high temperatures. Moreover, UPV values reduce as sodium concentrations increased for the samples that cured at 60°C temperature for 24 hr. This could be attributed to the slow development of geopolymeric reactions that take place at 60°C for 24 hr of heat curing.

3.2 | Compressive and flexural strength

Compressive and flexural tensile strengths of all geopolymer mortar mixtures made for all mixture parameters and curing parameters including reference and nanoparticles added mixtures were given in Table 6.

3.2.1 | Effect of nanoparticles

Laboratory results presented in Table 6 illustrate that the addition of nanoparticles either nano NS or nano NC in mixtures influenced the strength properties favorably at all heat curing temperature particularly for 24 hr heat curing. This positive influence was observed for all sodium ion concentration levels. NS addition increased the compressive strength up to 62% for 24 hr (see mixture coded 8 N-NS3-75°C). Similarly, NC addition increased the compressive strength up to 64% for 24 hr (see mixture coded 8 N-NC3-90°C). In the same manner, NS addition increased flexural strength up to 57% for 24 hr (see mixture coded 8 N-NS3-75°C). Similarly, nano NC addition increased flexural strength up to 54% for 24 hr (see mixture coded 8 N-NC2-60°C). The significant influence of nanoparticles on strength properties was observed at 8% Na concentration for 24 hr heat curing time. The favorable influence of nanoparticles on strength properties tends to disappear for 48 and 72 hr heat curing. This was explained in the following that high specific surface area of nanoparticle increases geopolymeric reaction kinetics due to seeding effect for 24 hr curing.[37] Findings of this study for 24 hr heat curing time was found to be in line with other published materials. Some researchers reported that the addition of nano SiO_2 and nano $CaCO_3$ forms extra CSH, CASH, and NASH, thus contribute to the enhancement of strength properties.[34,42]

Other researchers stated that in general NS particles provide extra amorphous SiO_2 in the mixture and due to their very high specific surface, NS accelerates the reaction

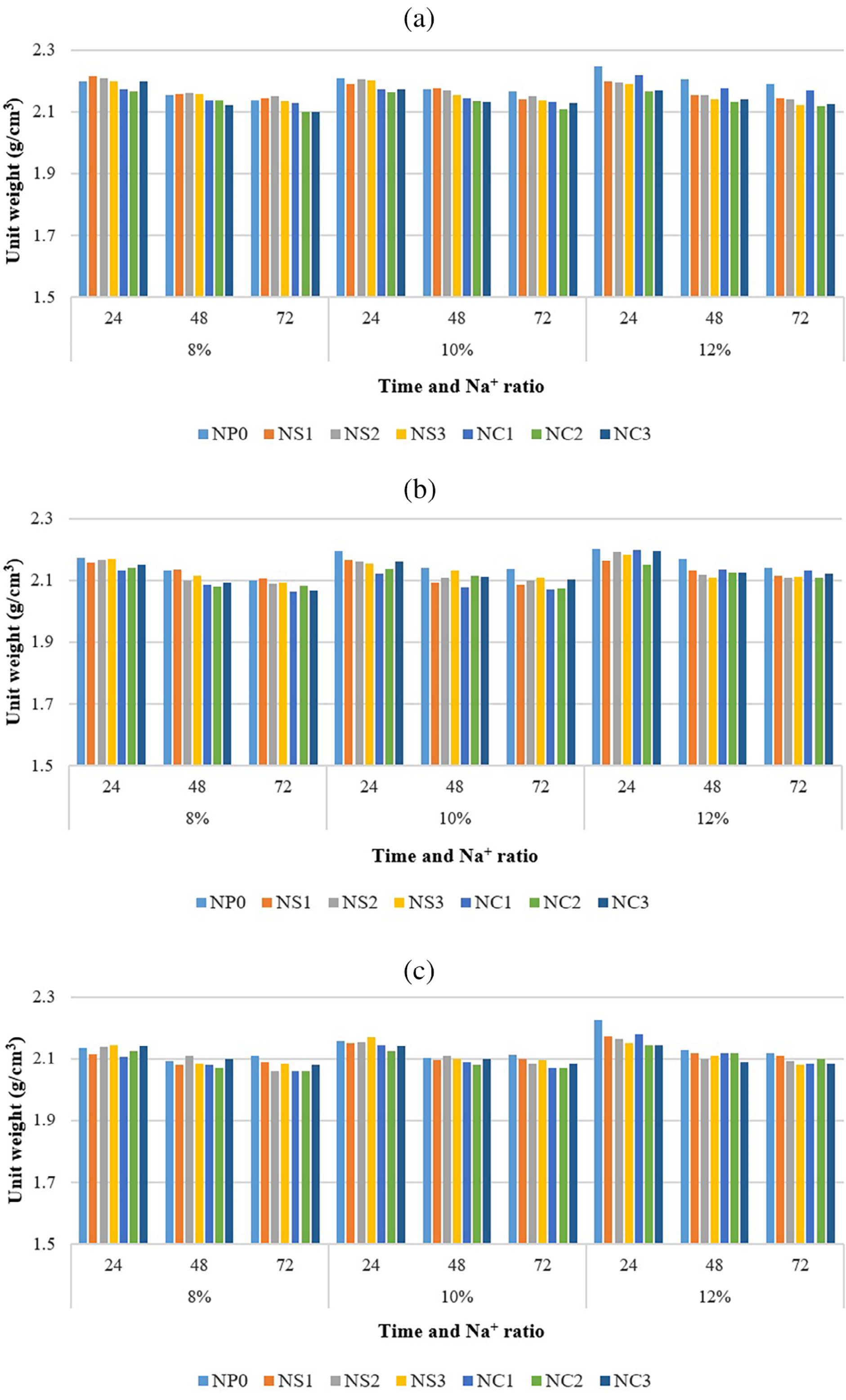


FIGURE 4 Unit weights after heat curing, (a) 60, (b) 75, and (c) 90°C (g/cm^3)

kinetics. Also, NS provides the filler effect and this effect improves strength properties significantly.[31,32,34,43] However, for longer, heat curing time (i.e., 48 and 72 hr) dominate the reaction kinetics and influence of nanoparticles (NS and NC) disappear. Contribution of nano NC to strength properties was influenced more than that of the contribution of NS.

3.2.2 | Effect of Na^+ ratio

In this study, sodium concentrations were selected as % 8, %10, and %12 of binder amount. The influence of changing sodium concentrations in the mixture showed that an increase in sodium concentrations results with higher compressive and flexural tensile

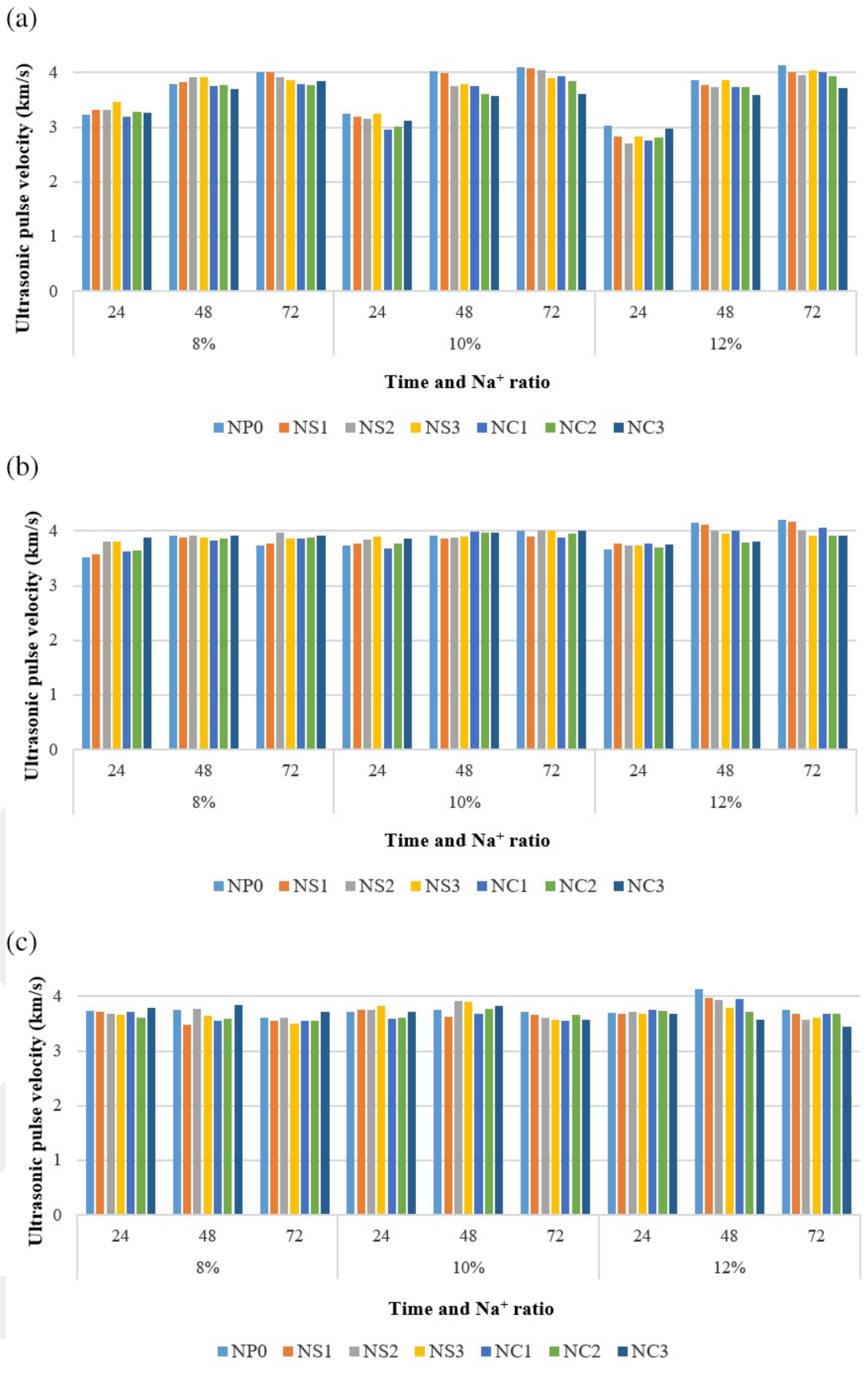


FIGURE 5 Ultrasonic pulse velocities after heat curing, (a) 60, (b) 75, and (c) 90°C (km/s)

strength. This is attributed to the reaction kinetics of geopolymer. This findings are in line with published literature.[12,44,45]

However, contradicting finding was also observed at 60°C temperature for only 24 hr curing. For example, 8% sodium concentrations provided higher strength than that of 10 and 12% sodium concentration. Usage more than optimal Na concentration caused the coagulation of SiO_2 and this results in a decrease in strengths.[7,44] These findings are also valid for all the mixtures produced.

3.2.3 | Effect of heat curing time and temperature

As mentioned before that all mixtures produced in the study were subjected to heat curing of 60, 75, and 90°C

TABLE 6 Compressive strength results (flexural strength results), MPa

	Na^+ ratio	8%			10%			12%		
Heat curing temperature	**Heat curing time (hr)**	**24**	**48**	**72**	**24**	**48**	**72**	**24**	**48**	**72**
60°C	NP0	12.3 (2.4)	29.9 (4.5)	42.2 (6.7)	8.8 (2.4)	32.7 (6.9)	41.0 (7.5)	4.9 (1.8)	26.3 (6.5)	42.9 (9.2)
	NS1	16.5 (2.7)	33.8 (4.7)	45.2 (6.1)	9.1 (2.9)	34.6 (6.7)	42.4 (7.8)	4.6 (1.8)	26.1 (6.0)	41.1 (9.2)
	NS2	18.6 (3.3)	38.6 (6.3)	39.9 (5.9)	9.0 (3.0)	30.5 (5.6)	41.8 (7.0)	3.9 (1.6)	23.1 (6.5)	42.3 (8.6)
	NS3	19.4 (3.3)	41.4 (6.7)	42.2 (5.6)	11.1 (3.3)	33.9 (6.4)	43.3 (6.6)	5.5 (2.5)	31.0 (7.6)	43.4 (8.9)
	NC1	15.2 (3.1)	28.1 (4.9)	36.9 (6.5)	8.5 (2.5)	29.1 (5.5)	37.9 (6.9)	4.0 (1.3)	25.4 (5.6)	39.7 (8.4)
	NC2	18.1 (3.7)	27.9 (5.5)	36.8 (5.8)	12.1 (2.9)	30.5 (5.7)	37.7 (5.8)	5.6 (2.3)	30.1 (6.6)	40.4 (8.2)
	NC3	14.9 (3.4)	29.1 (6.5)	33.0 (6.5)	12.5 (2.5)	29.7 (5.1)	39.3 (5.2)	6.6 (2.5)	31.2 (5.8)	43.3 (7.8)
75°C	NP0	22.6 (4.6)	39.2 (6.7)	42.4 (7.6)	30.2 (6.2)	45.6 (6.5)	54.0 (8.6)	28.4 (6.0)	50.1 (9.3)	71.9 (11.6)
	NS1	27.0 (5.1)	40.9 (6.9)	46.7 (8.3)	33.1 (6.4)	50.8 (6.9)	56.3 (9.1)	31.6 (6.6)	56.4 (9.4)	72.1 (10.1)
	NS2	34.2 (6.7)	50.2 (6.6)	54.2 (8.8)	39.5 (7.6)	50.1 (6.8)	59.6 (8.4)	34.9 (7.2)	56.8 (8.6)	68.0 (12.2)
	NS3	36.6 (7.2)	53.7 (6.6)	54.9 (9.2)	46.5 (7.8)	55.4 (6.2)	65.7 (8.0)	34.9 (7.2)	59.7 (8.5)	66.0 (11.1)
	NC1	27.7 (5.6)	39.6 (7.3)	41.2 (7.9)	36.4 (6.0)	53.7 (7.0)	56.6 (9.3)	40.0 (7.5)	57.6 (9.1)	63.2 (9.8)
	NC2	32.5 (6.2)	44.7 (5.5)	46.1 (8.7)	40.9 (7.1)	55.0 (7.2)	59.5 (9.7)	42.1 (6.7)	54.5 (9.7)	57.9 (11.7)
	NC3	34.3 (6.6)	45.5 (6.8)	47.0 (8.9)	46.4 (7.5)	55.6 (6.8)	61.5 (8.1)	45.1 (7.7)	53.5 (9.8)	55.1 (10.7)
90°C	NP0	27.4 (5.0)	44.5 (9.3)	41.5 (8.6)	37.7 (6.5)	58.0 (9.1)	53.9 (8.7)	47.1 (6.1)	73.8 (10.3)	69.7 (10.7)
	NS1	30.0 (5.0)	46.4 (4.9)	43.8 (7.9)	41.7 (6.5)	58.6 (9.2)	57.6 (8.8)	51.1 (6.2)	72.1 (10.0)	65.2 (10.3)
	NS2	39.8 (6.2)	58.8 (9.9)	53.7 (8.7)	48.2 (6.4)	68.3 (9.4)	62.5 (9.0)	57.3 (6.6)	73.0 (9.7)	67.0 (9.0)
	NS3	44.5 (6.3)	59.0 (9.6)	53.7 (9.2)	51.0 (7.2)	69.7 (9.1)	62.9 (9.1)	63.7 (6.4)	67.4 (9.4)	70.2 (9.4)
	NC1	37.5 (5.8)	46.1 (8.6)	48.3 (7.9)	43.4 (6.0)	59.1 (10.1)	52.7 (9.1)	54.1 (6.9)	61.1 (10.0)	63.4 (9.1)
	NC2	41.7 (5.7)	48.4 (9.3)	51.0 (8.3)	49.3 (7.0)	61.6 (10.1)	60.8 (9.6)	59.5 (7.1)	63.4 (10.5)	62.3 (10.2)
	NC3	45.0 (6.4)	55.8 (10.6)	55.1 (10.1)	52.6 (7.1)	68.0 (9.4)	64.4 (9.5)	51.9 (6.5)	62.7 (10.1)	60.2 (8.9)

for 24, 48, and 72 hr separately. When strength properties of mixtures were closely examined. Increase in curing time results in a significant increase in strength. A similar conclusions were also made by other researchers.[12,46] In general, increasing curing temperature (from 60 to 90°C) increases strength value.[44,45,47–49]

However, longer curing time process than the need of geopolymeric reaction at high curing (i.e., 90°C) temperature caused a reduction in compressive and flexural strength. For example, compressive and flexural strength values of samples cured at 90°C for 72 hr decreased in comparison to 48 hr heat curing at 90°C. Compressive strength was influenced more than flexural strength did. Similar influence was observed on UPV values. UPV values of samples cured at 90°C for 72 hr decreased in comparison to 48 hr heat curing at 90°C.

3.3 | FESEM analysis results

The morphology of the geopolymer specimens made with nanoparticles addition and reference paste (without nanoparticle) were examined by FESEM. Dosage of nanoparticles within paste mixtures was 2% replacement for fly ash by weight. FESEM images were taken from geopolymer paste samples cured at 60°C in an oven was activated with 8% Na; 75°C oven cured sample was activated with 10% Na; 90°C oven cured sample was activated with 12% Na concentration. These Na^+ concentration ratios were selected as they provided optimal compressive strength results with their curing temperature. Their curing duration in the oven was selected as 24 hr.

The FESEM micrographs were shown in Figures 6-8. . In micrographs, the formation of sodium aluminosilicate gel and unreacted fly ash particles were clearly observed which is in line with some previous reports in published literature.[26,50,51] According to FESEM micrographs, the main differences between reference mixture (a) and the mixture containing nanoparticles (b and c) that the matrices of geopolymer pastes with nanoparticles had denser microstructures and higher amount of crystalline compound when compared to the reference paste without nanoparticle.

Also, geopolymer paste without nanoparticle contains more unreacted and/or partially reacted fly ash particles than that of geopolymer pastes containing nanoparticles. The addition of NS and NC results in a higher amount of geopolymeric reaction products in comparison to reference paste. This finding supports the explanations made for strength development by extra gel formation, seeding effects, and filler effects. Similar results have also been reported for fly ash based geopolymer containing NS in the literature.[17,26,31,34]

In addition, it can be seen from Figure 6-8 that while curing temperature increases from 60 to 75°C and 90°C. The microstructure of geopolymer gets more and more dense as well as compact to a higher degree. This finding is valid for each geopolymer paste, separately. The difference observed between microstructures of samples reflects itself in the development of compressive and flexural strength of samples. For example, the highest strength was obtained from geopolymer sample cured at 90°C which seemed to have the densest and compact microstructure (see Figure 8b,c).

3.4 | Reaction kinetic results

Reaction kinetics studies were conducted for the selected pastes exhibited the highest strength performance with certain temperature and alkali concentration combinations, for example, 8% Na^+ concentration at 60°C, 10% Na^+ concentration at 75°C, and 12% Na^+ concentration at 90°C. The rate of reaction and corresponding cumulative heat of reaction curves are shown in Figure 9-11 for the pastes with 2% NC and NS as well as the reference paste without nanoparticle. The data are shown in Figure 9-11 generally suggests that the heights of rate peaks were increased and the reaction was accelerated with the addition of NC and NS at all curing temperatures. Comparing NC and NS in terms of their influence on the rate of reaction of geopolymer pastes, nano-SiO_2 addition increased the hydration rate further. For instance, for curing temperature of 60°C, NC and NS addition increased the height of rate peak 11 and 44%, respectively (Figure 9a). For curing temperature of 75°C, these corresponding increments were approximately 20 and 50% (Figure 10a). In addition, it is clearly observed that curing temperature increased the reaction rates of geopolymer pastes as expected when compared to the rate peaks at different curing temperatures.[27,52] (Figures 9, 10 and 11).

Comparing the rate peaks in terms of the occurrence time of the peak, NC, and NS addition accelerated, or shifted to the left, by approximately 2 hr for geopolymer pastes cured at 60°C concerning paste without nanoparticle as shown in Figure 9a. The acceleration of the time at which peaks occurred in the case of nanoparticle addition was slight for the curing temperatures of 75 and 90°C.

The effect of nanoparticle addition on the degree of reaction can be interpreted from the cumulative heat of reaction curves given in Figures 9b, 10b, and 11b. The curves indicate that nanoparticle additions increase the degree of geoploymerization reaction more or less. The increases were more pronounced at 75 and 90°C when compared to 60°C. Comparing the effects of NC and NS

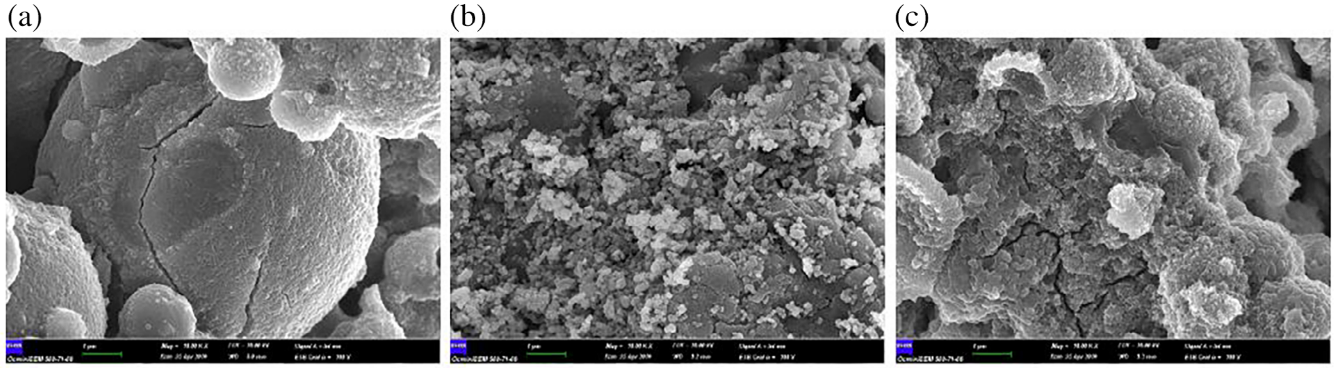


FIGURE 6 Field-emission scanning electron microscopy (FESEM) images of geopolymer samples after 24 hr 60°C heat curing (a) without nanoparticle (b) containing 2% NS (c) containing 2% NC

FIGURE 7 Field-emission scanning electron microscopy (FESEM) images of geopolymer samples after 24 hr 75°C heat curing (a) without nanoparticle (b) containing 2% NS (c) containing 2% NC

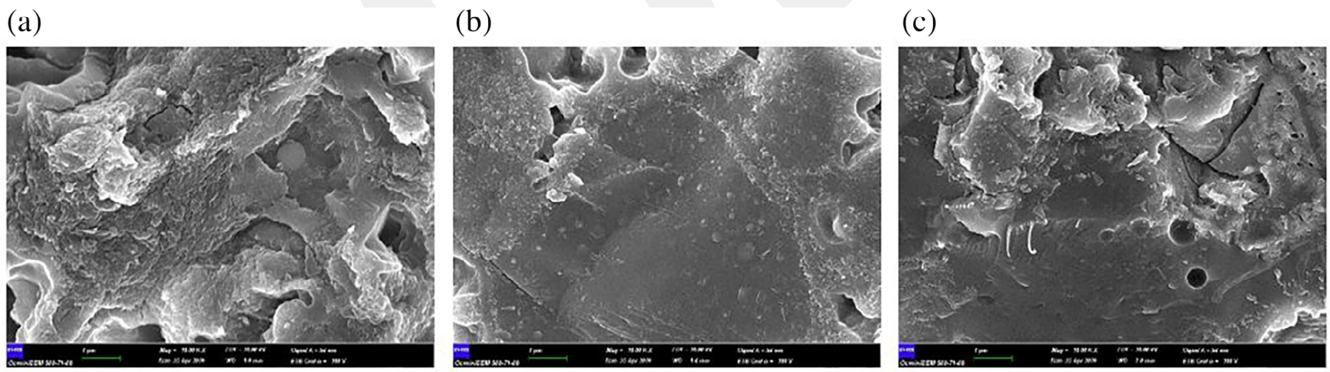


FIGURE 8 Field-emission scanning electron microscopy (FESEM) images of geopolymer samples after 24 hr 90°C heat curing (a) without nanoparticle (b) containing 2% NS (c) containing 2% NC

on the degree of reaction as indicated by cumulative heat, NC addition resulted in a higher cumulative heat for curing temperature of 75°C, however, the reverse was true for 60 and 90°C at the end of 80 hr of measurements.

Specifically, for curing temperature of 90°C, nano-SiO_2 addition caused a lower cumulative heat than NC addition before 40 hr of reaction, however, it showed a higher heat of reaction at the end of 80 hr (Figure 11b). The relative increase in cumulative heat of reaction for NS addition is the result of the secondary minor peak shown in the corresponding rate of reaction curve between 35 and 45 hr (Figure 11a). This minor peak is probably attributed to delayed geopolymerization of larger agglomerated particles of NS at a relatively higher temperature. Contributions of nanoparticle additions to the rate and the degree of reactions discussed above were in line with compressive and

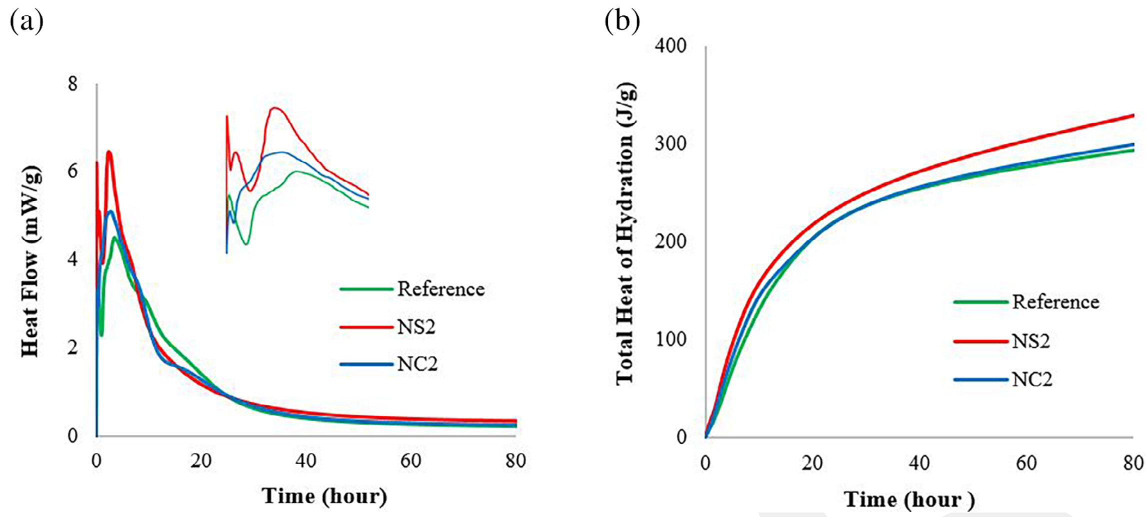


FIGURE 9 (a) Rate of hydration curves of geopolymer pastes at 60°C, (b) Cumulative heat of hydration of geopolymer pastes at 60°C

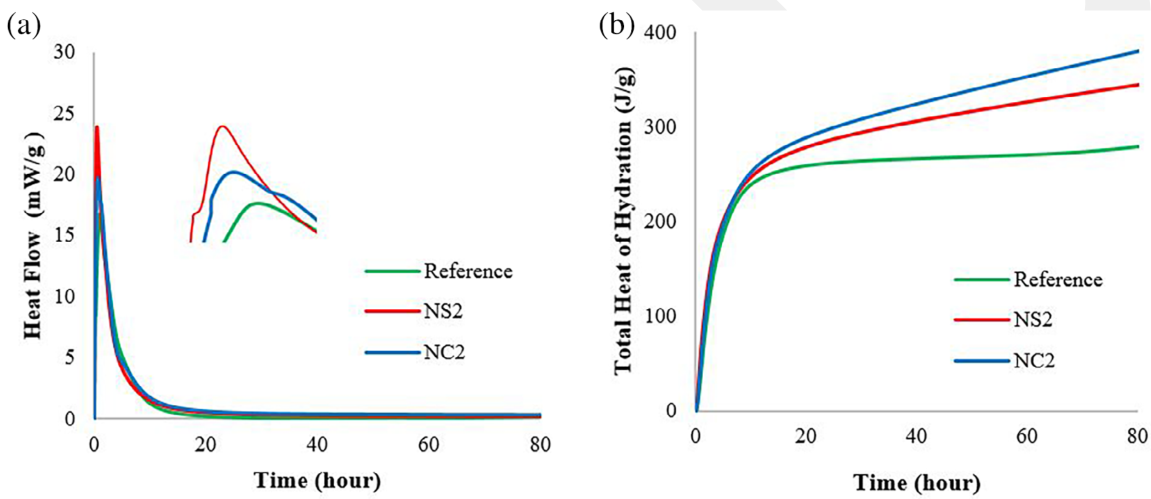


FIGURE 10 (a) Rate of hydration curves of geopolymer pastes at 75°C, (b) Cumulative heat of hydration of geopolymer pastes at 75°C

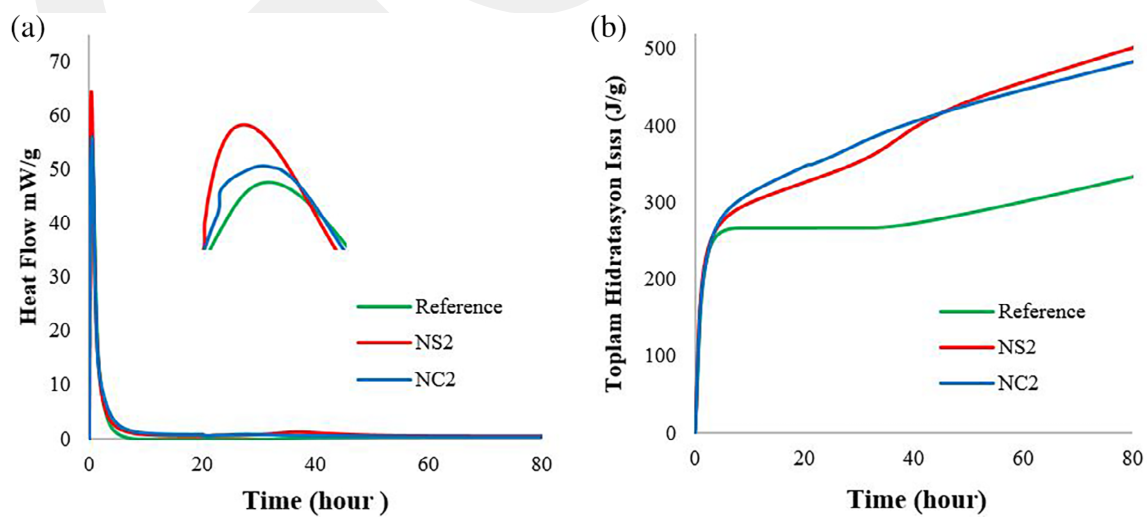


FIGURE 11 (a) Rate of hydration curves of geopolymer pastes at 90°C, (b) Cumulative heat of hydration of geopolymer pastes at 90°C

flexural strength development of corresponding hardened mortars shown in Table 6. The maximum contribution of nanoparticle additions to the strength development of geopolymer mortars was obtained for the first 24 hr of curing, which is in line with the pronounced effects of nanoparticles on the rate of reaction curves at initial hours.

4 | CONCLUSIONS

Based on the experimental results in this study, the following conclusions can be drawn:

1 An increase in alkali activator content (NaOH) resulted in an improvement in the workability of geopolymer mortar. However, the inclusion of nanoparticles in the mixture behaved contrary and reduced the workability of mixture due to their higher specific surface area.
2 Inclusion of nanoparticles in mortar mixtures and increase in curing time of mixtures caused higher UPV values due to the accelerating effect of nanoparticles in a chemical reaction as well as continuing geopolymeric reaction thus causing denser microstructure.
3 Nanoparticles addition significantly increased compressive and flexural strength of mortars for 24 hr heat curing duration, the increases were up to 65 and 45%, respectively, in comparison to control strength values.
4 Favorable influence of nanoparticles on strength values diminished with the increase in heat curing duration (from 24 to 72 hr). Therefore, it can be concluded that nanoparticles addition can be helpful to increase the early age strength of geopolymer mortar.
5 According to laboratory work results, it can be said that 2% NS and NC usage are optimum for geopolymer mortar.
6 FESEM micrographs represented that NS and NC additions improved the microstructure of samples as being denser in comparison to samples produced without nanoparticles.
7 Based on reaction kinetics measurement, the addition of nanoparticles into mixtures caused an acceleration in geopolymeric reactions at all curing temperatures as well as for all alkali activator amounts. In particular, NS exhibited a better performance when compared to NC in terms of increases in the rate of reaction.

ACKNOWLEDGMENTS

This work with project code FDK-2018-7912 is supported by Erciyes Üniversitesi Scientific Research Project Coordination Unit.

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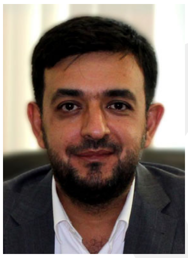

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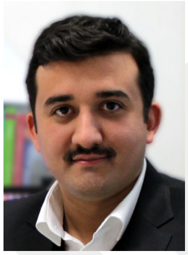

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How to cite this article: Durak U, Karahan O, Uzal B, İlkentapar S, Atiş CD. Influence of nano SiO_2 and nano $CaCO_3$ particles on strength, workability, and microstructural properties of fly ash-based geopolymer. *Structural Concrete*. 2020;1–16. https://doi.org/10.1002/suco.201900479