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EFFECT OF INCORPORATING NANO ZEOLITE ON THE STRENGTH OF NATURAL POZZOLAN-BASED GEOPOLYMER CONCRETE

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Natural pozzolan is a green and sustainable material, which has a strong potential for utilization in the geopolymer concrete. At room temperature, the development of strength in natural pozzolan based geopolymer concrete is fair, however, it is desirable, to have higher strength and to enhance the early age strength development. The effect of nano zeolite (NZ) as an additive to the mix by incorporating it at 3% and 5% by weight of natural pozzolan on the strength development and microstructural properties of natural pozzolan based geopolymer concrete mixes are presented. Scanning electron microscopy (SEM) was utilized to determine the morphology of the paste. The results indicated that 5% nanozeolite replacement resulted in superior strength and denser microstructure compared to other mixes. The addition of nanozeolite improved dissolution of source material in the alkaline activators and enhanced transformation of it to form polymeric compounds.

Keywords: Natural pozzolan (NP), Geopolymer concrete, Nano zeolite, Compressive strength, Morphology.

1 INTRODUCTION

The utilization of nano materials in the building material research is growing due to their ability to enhance the engineering properties of binders (Jayapalan *et al.* 2013). The hardened properties of these materials are improved due to the development of compact microstructure as a result of high reactivity to form compounds (Balaguru and Chong 2006). Also, there is a particle packing effect (Sanchez and Sobolev 2010). Currently, alkali activated binders are being extensively studied due to their multiple benefits including potential alternative to ordinary Portland cement (Rovnaník 2010). These benefits are: lower environmental impact, high early strength, and better durability performance (Yusuf *et al.* 2014, Brough and Atkinson 2002). However, strength development of these binders depends on the chemical composition of precursor materials as well as curing conditions. Thus far, industrial byproduct, such as fly ash, has been extensively used as source material in synthesizing AABs and cured at temperatures between 40 to 80 °C (Hardjito *et al.* 2005). The strength development is rapid when these binders are cured at elevated temperature because of the fact that heat accelerates the reactivity. On the contrary to improve the reaction kinetics, finer materials can be added to the primary precursor materials to improve the reactivity. Most significantly, the fineness of the precursor material has a profound effect on the rate of reactivity (Wang *et al.* 2005). The finer materials dissolve easily in the alkaline solution leading to a higher conversion of the source materials to polymeric compounds which enhances the strength of these binders (Phoo-ngernkham *et al.* 2014). It is estimated that the dissolution of source materials having particle size of more than 20 µm is rather slow, whereas, materials with particle size of less than 2 µm react rapidly (Wang *et al.* 2005). In this regard nano materials with enormous specific surface area are believed to be potentially useful in improving the strength development of these binders. Earlier studies have used nanosilica and nano alumina to improve the properties of these binders. Phoo-ngernkham *et al.* (2014) studied the influence of incorporating NS and nanoalumina (NA) on the strength and microstructure of high calcium fly ash-based AAB cured under ambient conditions at a given alkaline activator concentration and ratio. It was reported that 2% nano material was sufficient to obtain enhanced strength due to the formation of calcium silicate hydrate (C-S-H) or calcium alumina silicate hydrate (C-A-S-H) gel along with sodium alumina silicate hydrate (N-A-S-H) gel (Phoo-ngernkham *et al.* 2014). In another study (Adak *et al.* 2014), fly ash was replaced with up to 10% NS in developing alkali activated mortar even though the source material was rich in silica and alumina. It was reported that the strength increased by 15% due to 6% replacement of fly ash with NS (Adak *et al.* 2014). In this study, natural pozzolan based binder was developed and to improve its properties nano zeolite (NZ) addition was proposed. The developed binder was evaluated by conducting mechanical tests and studying its microstructure.

2 MATERIALS AND METHODS

2.1 Materials

Natural pozzolan (NP), the main precursor material used in the study, was in a powdered form of volcanic rock. The chemical composition of NP is given in Table 1. The chemical composition of NZ used in the study is shown in Table 2. 14M NaOH solution and Na_2SiO_3 of 3.3 silica modulus were used as alkaline activators. The composition of sodium silicate includes: H_2O: 62.5%, SiO_2: 28.75% and Na_2O: 8.75%. Crushed limestone, having specific gravity of 2.56, was used as coarse aggregate while dune sand having specific gravity of 2.62 was utilized as fine aggregate in the concrete mixtures.

Table 1. Chemical composition of NP.

Oxides	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	Na_2O	P_2O_5	TiO_2	LOI
Weight, %	40.48	12.90	17.62	11.83	8.33	1.67	3.60	1.37	0.60	1.6

Table 2. Properties of nano zeolite.

Property	Solid content, %	Avg. Particle size, nm	Bulk density, g/cm^3	Specific surface area, m^2/g	Na_2O content, %	Viscosity cps	pH
Value	50	35	1.4	80	0.2	15	9.5

2.2 Methods

Table 3 shows the quantities of constituent materials of mixes prepared by incorporating NZ. All the AAC mixtures were prepared with a constant Na_2SiO_3/NaOH weight ratio of 2.50. The concrete specimens were de-molded after 1 day of casting, divided into two groups and placed in plastic bags to avoid evaporation of moisture. For curing, one group of concrete specimens were kept in the laboratory which was maintained at $23\pm2\ ^{\circ}C$, while, the other group was cured at elevated temperature in the oven that was maintained at $60\pm2\ ^{\circ}C$. The compressive strength of concrete was measured after 1/2, 1, 3, 7, 14, and 28 days of elevated temperature curing, while, it was measured after 3, 7, 14, 28 and 90 days in case of room temperature curing (23±2 °C) on 50 mm cube specimens according to ASTM C109-10 (2010). Alkali activated paste was prepared and cured at elevated temperature for 28 days after which morphology was determined using a JEOL model 5800 LV instrument.

Table 3. Quantities of AAC mix constituents.

Mix #	Legend	NP, kg/m^3	NZ, kg/m^3	Na$_2$SiO$_3$, kg/m^3	NaOH, kg/m^3	FA, kg/m^3	CA, kg/m^3
M0	0%-NZ	400	0	150	60	650	1206
M1	3%-NS	388	12	150	60	646	1200
M2	5%-NS	380	20	150	60	640	1188

3 RESULTS AND DISCUSSION

3.1 Compressive Strength

The compressive strength development in the AAC cured in the oven prepared with 0%, 3%, and 5% NZ is depicted in Figure 1. Figure 2 gives the strength development in the AAC cured at room temperature. The compressive strength in both the curing regimes increased with period of curing. However, the strength development was faster in case of specimens cured in the oven compared to the ones at room temperature. 7 days of heat curing was sufficient to gain maximum strength in the AAC prepared with various NZ contents, while, the strength development was continued in the AAC cured at room temperature even after 28 days. The maximum compressive strength measured in case of AAC cured in the oven was 37.52 MPa, 41.54 MPa and 43.62 MPa synthesized with 0%, 3% and 5% NZ, respectively, while, it was 27.93 MPa, 32.56 MPa and 33.97 MPa in the room temperature cured batch of concrete specimens. Based on these results, the strength gain in the concrete prepared with 3% and 5% NZ addition cured at elevated temperature over control samples was 10% and 16%, respectively; however, this value was 16% and 21% in case of specimens cured at room temperature. It is to be noted that maximum compressive strength was gained in case of concrete specimens cured at room conditions was at 90 days. The improvement in the performance of AAC containing NZ, in terms of compressive strength, particularly with 5%, could be attributed to the enhanced transformation of source materials to the polymeric gel in the presence of highly reactive NZ as well as due to the possible particle packing effect of nanoparticles in the binder structure (Yip *et al.* 2005).

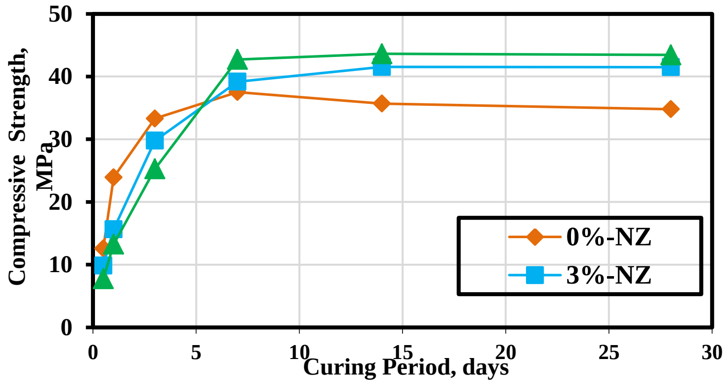

Figure 1. Compressive strength development in the concrete cured at elevated temperature.

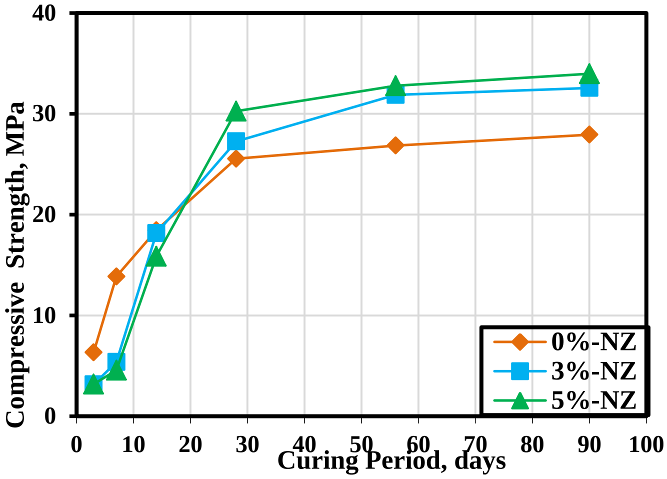

Figure 2. Compressive strength development in the concrete cured at room temperature.

3.2 Morphology

Figure 3 presents the SEM images of geopolymer paste incorporating NZ cured at elevated temperature for 7 days. Morphology of alkali activated paste was not carried out for the specimens cured at room temperature. Microstructure of the specimen prepared without NZ was very porous in nature with widespread cracks. When NZ was added to the mixture, microstructure started to densify with reduction in cracks and voids. In the mixtures prepared by incorporating 3% and 5% NZ, micrographs showed denser microstructure with homogenous gel-like matrix. The microstructure of AAP modified with 3% and 5% NZ was more or less similar. The more homogenous gel in case of AAP modified with 3% and 5% NZ with undefined boundaries most probably consisted of polymeric compounds. Based on the several micrographs observed while taking the images, there was partial filling of voids in the specimens prepared with 3% and 5% NZ which resulted in higher compressive strength. On the contrary, in the specimens prepared without NZ, the solid area was less, which did not beneficially helped in increasing the strength of developed concrete. These outcomes are consistent with the compressive strength results wherein 3% and 5% replacement of natural pozzolan with NZ lead to superior strength. However, the initial sluggishness in the development of strength in the concrete mixes containing higher NZ was due to the increase in the silica content for a given amount of alkaline solution which may have contributed in delayed polymerization, particularly in the specimens cured at room temperature (Panias *et al.* 2007, Oh *et al.* 2010). However, there was considerable strength gain in these mixes when cured at elevated temperature. Nonetheless, as the curing continued due to the availability of additional soluble silica accelerated the polymerization process, which resulted in enhanced transformation of source materials to the polymeric gel (Nath and Sarker 2017). These results are indicative of the fact that in the process of polymerization due to the availability of soluble silica, particularly in the mixes incorporating micro silica, resulted in the formation of CSH or CASH gels along with the NASH products (Juhyuk *et al.* 2014).

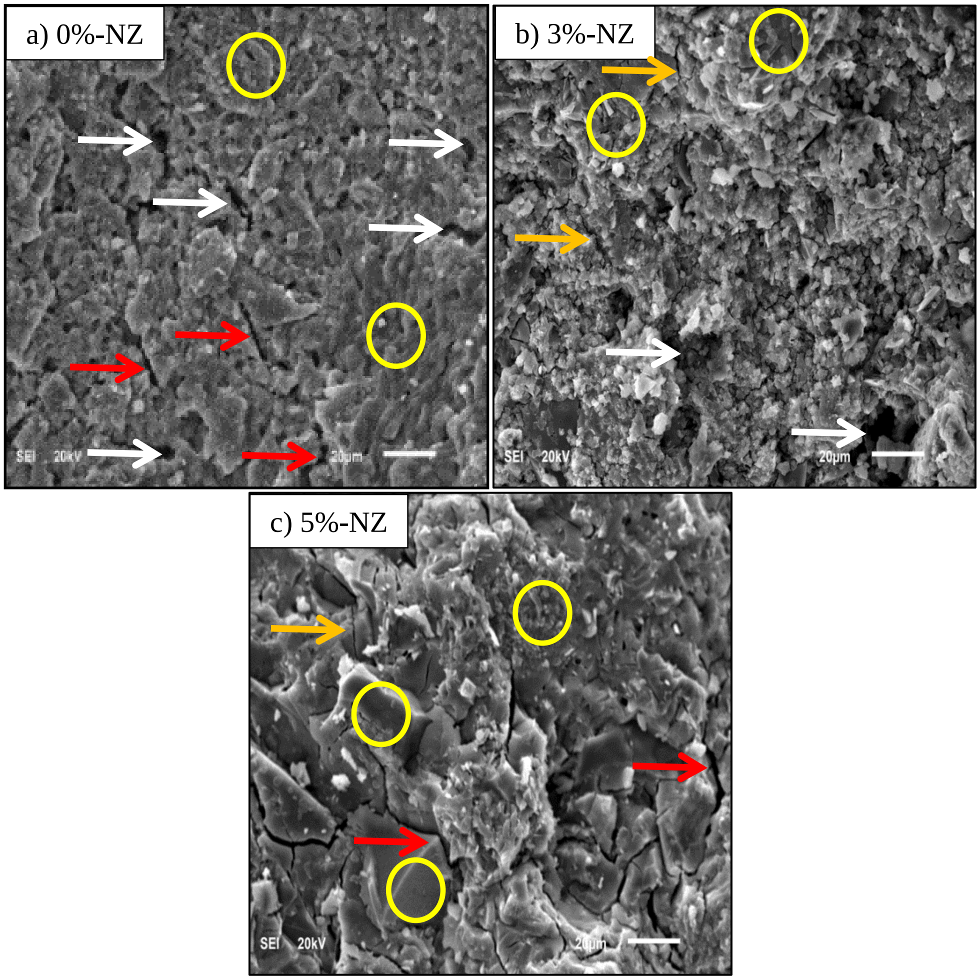

Figure 3. SEM images of alkali activated paste modified with NZ. (white arrows: voids, red arrows: wide cracks, yellow arrows: fine cracks, yellow circles: uniform gel).

4 CONCLUSIONS

The maximum compressive strength of thermally cured AAC prepared with and without NZ was higher than room temperature cured specimens. The strength development was faster when concrete was cured at elevated temperature, while it was slower in room conditions. However, the AAC prepared with 3% and 5% exhibited enhancement in strength compared to control mixture when cured at both the regimes. According to these outcomes, the concrete developed in this study has potential to be used for structural purposes, as the strength was more than 40 MPa and 30 MPa when cured in the oven and room temperature, respectively. The microstructure of AAC modified with 3% and 5% NZ showed denser matrix and compact probably due to enhanced transformation of source material in to polymeric compounds. On the other hand, the microstructure of AAC prepared without NZ was relatively discrete and non-uniform.

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References

Adak D., Sarkar S., and Mandal S., Effect of Nano Silica on Strength and Durability of Fly Ash Based Geopolymer Mortar, *Construction and Building Materials*, 70, 453–459, 2014.

ASTM C109-10, Standard Test Method for Compressive Strength of Hydraulic Cement Mortars (Using 2-in. or (50-mm) Cube Specimens), *ASTM International*, 2010.

Balaguru P., and Chong K., *Nanotechnology and Concrete: Research Opportunities*, Proceedings of ACI Session on Nanotechnology of Concrete: Recent Developments and Future Perspectives, Denver, USA, November 7, 2006.

Brough A.R., and Atkinson A., Sodium Silicate-Based Alkali-Activated Slag Mortars: Part I. Strength, Hydration and Microstructure, *Cement and Concrete Composites*, 32, 865–879, 2002.

Hardjito, D., Wallah, S. E., and Sumajouw, D. M. J., and Rangan, B. V., Fly Ash-Based Geopolymer Concrete, *Australian Journal of Structural Engineering*, 6(1), 77-85, 2005.

Jayapalan, A. R., Lee B. Y., and Kurtis K. E. Can Nanotechnology Be 'Green'? Comparing Efficacy of Nano and Microparticles in Cementitious Materials, *Cement and Concrete Composites*, 36, 16-24, 2013.

Juhyuk, M. A., Sungchul, B. B., Celik, K. B., Yoon, S. B., Ki-Hyun, K. B., Kang, S. K. C., Paulo, J. M., and Monteiro, B., Characterization of Natural Pozzolan-Based Geopolymeric Binders, *Cement and Concrete Composites*, 53, 97-104, 2014.

Nath P., Sarker P.K., Flexural strength and elastic modulus of ambient-cured blended low-calcium fly ash geopolymer concrete, *Construction and Building Materials*, 130 (2017) 22–31.

Oh, J. E., Monteiro, P. J., Jun, S. S., Choi, S, and Clark, S. M., The Evolution of Strength and Crystalline Phases for Alkali-Activated Ground Blast Furnace Slag and Fly Ash Based Geopolymers, *Cement and Concrete Research*, 40(2), 189–196, 2010.

Panias, D., Giannopoulou, I. P., and Perraki, T., Effect of Synthesis Parameters on The Mechanical Properties of Fly Ash-Based Geopolymers, *Colloids and Surfaces, A*, 301(1), 246–254, 2007.

Phoo-ngernkham, T., Chindraprasirt, P., Sata, V., Hanjitsuwan, S., and Hatanaka, S., The Effect of Adding Nano-SiO_2 and Nano-Al_2O_3 on Properties of High Calcium Fly Ash Geopolymer Cured at Ambient Temperature, *Materials and Design*, 55; 58–65, 2014.

Rovnaník, P., Effect of Curing Temperature on The Development of Hard Structure of Metakaolin-Based Geopolymer, *Construction and Building Materials*, 24(7), 1176–1183, 2010.

Sanchez F., and Sobolev, K., Nanotechnology in Concrete – A Review, *Construction and Building Materials*, 24, 2060–2071, 2010.

Wang, P. Z., Trettin, R., and Rudert, V., Effect of Fineness and Particle Size Distribution of Granulated Blast Furnace-Slag on The Hydraulic Reactivity in Cement Systems, *Advanced Cement Research*, 17, 160-166, 2005.

Yip, C. K., Lukey, G. C., Van Deventer, J. S. J., Coexistence of Geopolymeric Gel and Calcium Silicate Hydrate at The Early Stage of Alkali Activation, *Cement and Concrete Research*, 35, 1688-1697, 2005.

Yusuf, M. O., Megat Johari, M. A., Ahmad, Z. A., and Maslehuddin, M., Strength and Microstructure of Alkali-Activated Binary Blended Binder Containing Palm Oil Fuel Ash and Ground Blast-Furnace Slag, *Construction and Building Materials*, 52, 504–510, 2014.