

EFFECT OF SODIUM HYDROXIDE CONCENTRATION AND SODIUM SILICATE TO SODIUM HYDROXIDE RATIO ON PROPERTIES OF CALCINED KAOLIN-WHITE PORTLAND CEMENT GEOPOLYMER

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ABSTRACT: This study presents the effects of sodium hydroxide concentration (NH) and sodium silicate to sodium hydroxide ratio (NS/NH) on the mechanical properties and microstructure of geopolymer pastes prepared from calcined kaolin (CK) and white Portland cement (WPC). Different curing conditions viz., ambient curing, temperature curing, and moist curing were used. The results indicated the increasing NH concentration and NS/NH ratio increased the compressive strength of CK-WPC geopolymers and gave the highest compressive strength at NH concentration of 15 molar and NS/NH ratio of 1.0. Furthermore, the use of 50% WPC increased the compressive strength of geopolymer paste. The samples with ambient temperature curing showed high early compressive strength equal to that with temperature curing. For moist curing, the compressive strengths of WPC samples at the later age were continuously developed and were higher than those with ambient and temperature curings. The microstructures of geopolymer paste were tested using SEM images and pattern of XRD (X-ray diffractometer) which showed the denser paste and higher calcium compound in the CK-WPC samples than those in CK samples.

Keywords: calcined kaolin, geopolymer, alkali-activated, calcium hydroxide, sodium hydroxide

1. INTRODUCTION

Geopolymer is a well-known cement as now commercialized as construction material. A three-dimensional amorphous structure of polycondensation of $\text{OSi}(\text{OH})_3$ and $\text{Al}(\text{OH})_4$ dimmers [1] makes geopolymer good in compressive strength. A binder used in the synthesis process is from Si and Al-rich compounds viz., fly ash, metakaolin and rice husk ash. Geopolymerization reaction required high energy to produce a rate of reaction at 40-90°C of curing [2], [3]. This high-temperature use is not compatible with in-situ working as real construction practice. Although this problem, many researchers report a method of the enhancement of compressive strength of geopolymer at low temperature or ambient condition. Those methods mostly suggested the use of calcium to accelerate the geopolymerization [4]. Calcium ions react with leached Si and Al in the matrix after attached to alkali medium and accelerate the polycondensation

of geopolymer gel by replacing Na ion [5]. Calcium compound such as CaO [6], $\text{Ca}(\text{OH})_2$ [7], [8] and even Portland cement (composed of 60% of calcium) can be added into the system by replacement method [9], [10]. A previous work reported the white Portland cement enhanced the compressive strength of calcined kaolin geopolymer. Calcium provides nucleation site for polycondensation and then makes geopolymer set more rapidly. Calcium reacts as CSH which fills micro-pore and relatively enhances compressive strength [11]. It should be noted that the presence of calcium ions in kaolin-based geopolymer increased material compressive strength [8], [12]

In geopolymer synthesis process, parameters affecting the mechanical properties and microstructure are as follows: the property of activator, portion of liquid activator and curing conditions [3], [13], [14]. A kaolinite-based geopolymer (Ca-free binders) has varied ranges of those parameters depending on the source of the binder. The suitable values of parameter were

presented as; NH concentration of 8 to 15 molar gave high compressive strength, a high ratio of NS to NH resulted in faster setting and higher compressive strength. For curing method, high-temperature curing of 40 to 90°C was reported as the improvement of compressive strength.

It is a beneficial way to use the commercial binder like Portland cement to improve the geopolymer properties to lower the production energy and suitably supports practical working. To obtain the data of proper mix proportion, the effects of various synthesis parameters should be tested and reported. This article studies the effects of alkali liquid viz., NH concentration and NS/NH ratio on the mechanical property and microstructure of CK geopolymer pastes with the addition of WPC. Three curing methods; ambient curing, high-temperature curing, and moist curing were tested. The microstructures of pastes were studied using SEM images (scanning electron microscopy) and the pattern of XRD (X-ray diffractometer).

2. MATERIALS AND METHODS

2.1 Materials and sample preparation

Calcined kaolin (CK) and white Portland cement (WPC) were used as source materials. The main chemical compositions are shown in Table 1. WPC contains a high amount of CaO (73.5%) and calcined kaolin contained 59.7% SiO₂ and 34.1% Al₂O₃. A synthesis process used liquid/binder ratio of 1.0. The sodium hydroxide solution prepared from solid sodium hydroxide dissolved with DI water and sodium silicate (NS, 15.32 % Na₂O, 32.87% SiO₂ and 51.81 % H₂O) were used.

Paste samples were prepared by mixing binders with NH and NS for 5 minutes in each circumstance. Fresh geopolymer pastes were tested

for setting time and cast in 25 cubic mm acyclic molds for the compressive strength test. Samples were wrapped with plastic film to protect moisture loss and were left for 1 hour in 23°C room. They were then demolded and put into different curing conditions as follows.

(1) Ambient curing (AC). The demolded samples were wrapped with a plastic to prevent moisture loss and kept in a control 23 °C room.

(2) Temperature curing (TC). The samples were wrapped with a plastic to prevent moisture loss and then oven-cured at 60 °C for 24 hours. After that, the samples were demolded and kept in a 23 °C room with plastic wrapped.

(3) Moisture curing (MC). The demolded samples were immersed in water in a 23 °C room until the testing age.

The mix portion has presented in Table. 2.

2.2 Test method

2.2.1 Time of setting and compressive strength

The setting times of fresh geopolymer pastes were tested using Vicat apparatus in accordance with ASTM C191 [15]. The compressive strengths of pastes at the age of 7 and 36 days were tested using an average of three 25x25x25 mm samples in accordance with ASTM C109 [16].

2.2.2 Microstructure

The microstructures images were studied using JEOL JEM-5910LV scanning electron microscopy (SEM). The chemical compositions of the products were analyzed using energy dispersive spectroscopy (EDS). The crystallography illustration using x-ray diffraction (XRD) method by the 2θ of 15° to 59.9916° and step using of 0.0229 ° with a step time of 240.5 seconds.

Table1 Chemical composition of binders

Materials	Chemical composition (%)										
	Na ₂ O	MgO	Al ₂ O ₃	SiO ₂	CaO	Fe ₂ O ₃	K ₂ O	SO ₃	P ₂ O ₅	LOI	other
Calcined kaolin	0.17	0.22	34.12	59.66	0.05	0.89	3.26	0.08	0.33	1.2	0.02
White Portland cement	0.01	0.88	1.94	15.86	73.48	0.23	0.13	4.94	0.02	2.3	0.21

3. RESULTS AND DISCUSSION

3.1 Time of setting

The setting time of WPC geopolymer pastes with a variation of NH concentration and NS/NH ratios were presented in Fig. 1. The increasing of NH concentration from 5M to 15M retarded the

time of setting of geopolymer pastes. Both of initial and final sets were longer from 8 to 24 min for the initial set and 36 to 51 min for the final set. But the increasing of NS/NH ratios from 0.33 to 1.5 shortened the initial set from 24 to 10 min and 53 to 30 min for final setting.

Time of setting of WPC geopolymer presented faster setting than CK geopolymer while the ordinary CK samples (10M of NH and MS/NH of 1.00) were previously reported the initial and final setting time of 47 and 72 minutes [17]. The presence of WPC in CK geopolymer decreased the initial and final setting time to 13 and 41 minutes respectively due to the Ca ion from WPC reacted as nucleation site which accelerated the hardening of geopolymer products [18]. The effect of NH concentration and NS/NH portion to setting time is reported similarly to other pure metakaolin or fly ash based geopolymer. A retarding of setting time when using higher NH concentration was due to two reasons; first is the higher leaching ability of binder element from high alkalinity increased the time of polycondensation and the second is the higher free Na⁺ ions in the system which also reported as geopolymerization retarder [18].

Besides, the ratio of NH to NS significantly affected the time of setting with high NH (low NS/NH ratio) resulted in a longer time of setting. The increase in NS/NH ratio decreased the portion of NH and thus decreased the time of setting. The reduction of hardening time was not only from the decreasing of NH but also from the increasing of reactive silicate from NS which readily condensed in geopolymerization reaction and then caused rapid hardening [19].

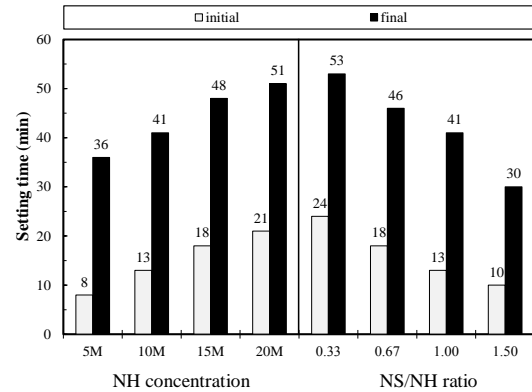


Fig.1 Time of setting of WPC mix pastes

Table 2 Mix portion and compressive strength of geopolymer pastes

Mix	CK (%binder)	WPC (%binder)	NS/NH ratio	NH concentration (molar)	water/solid ratio (w/s)	Compressive strength (MPa)					
						7 days			36 days		
						AC	TC	MC	AC	TC	MC
CK	100	0	0.33	10	0.483	28.9	36.0	32.6	-	-	-
CK	100	0	0.67	10	0.454	43.6	45.7	45.0	-	-	-
CK	100	0	1.00	10	0.436	45.4	50.0	53.4	58.7	54.7	55.3
CK	100	0	1.50	10	0.418	56.9	59.7	60.0	-	-	-
CK	100	0	1.00	5	0.535	16.3	17.1	15.8	-	-	-
CK	100	0	1.00	10	0.454	43.6	45.7	45.0	-	-	-
CK	100	0	1.00	15	0.436	56.3	59.6	61.9	-	-	-
CK	100	0	1.00	20	0.418	64.4	69.8	70.9	-	-	-
WPC	50	50	0.33	10	0.483	47.7	34.0	48.3	-	-	-
WPC	50	50	0.67	10	0.454	40.3	33.9	50.4	-	-	-
WPC	50	50	1.00	10	0.436	63.9	55.0	56.9	81.6	57.4	71.8
WPC	50	50	1.50	10	0.418	58.9	57.2	59.3	-	-	-
WPC	50	50	1.00	5	0.535	39.7	33.6	38.7	-	-	-
WPC	50	50	1.00	10	0.454	40.3	33.9	50.4	-	-	-
WPC	50	50	1.00	15	0.436	53.2	46.1	71.3	-	-	-
WPC	50	50	1.00	20	0.418	39.1	27.1	51.0	-	-	-

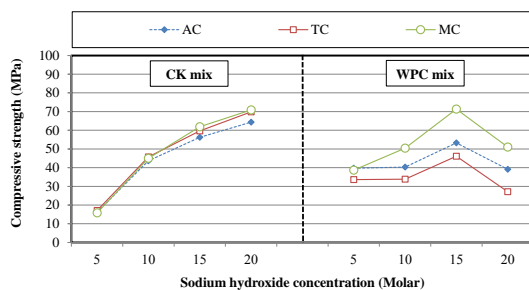
3.2 Compressive strength

The results of compressive strength of geopolymer pastes are presented in Table 2 and Fig.2. For CK mix, the increased NH concentration affected the increasing of compressive strength as

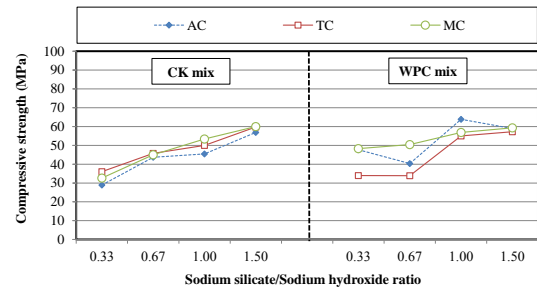
presented in Fig. 2(a). Using of 5M NH resulted in very low compressive strength due to the poor leaching ability of reactive element from binder [20]. It was previously reported that NH lower than 8M has mostly reacted as hydration reaction [12]. Since CK mix was lack of Ca element which

performs the hydration reaction, the CK mix thus showed low compressive strength. The high concentration of NH performed development of compressive strength, due to the higher precursor leaching from binder to complete the geopolymerization process than the lower NH concentration. For WPC mix, higher NH concentration presented the increasing of compressive strength similar to CK mixes, but the compressive strength dropped when NH concentration was very high at 20M. It was probably due to the excess alkali ions which were responsible for cation balance which can be described following the Ca^{2+} better in cation balance than NH. The high NH in WPC-added sample present in the excess Na which was previously reported as lowering of compressive strength [18].

Fig. 2(b) showed that the increasing NS/NH ratio increased the compressive strength of both CK and WPC mixes. Using of constant 10M NH with the increase in NS/NH ratio proportionally increased the reactive Si ions into the geopolymerization system and helpfully completed the polycondensation and also improved the compressive strength [21]. A higher degree of geopolymerization reaction was reported as the course of increasing of compressive strength [22]. But high NS/NH ratio increased the viscosity of fresh geopolymer, with the presence of additional Ca, WPC mix exhibited a reduction of setting time as presented in Fig.1 and also decreased compressive strength at very high NS/NH of 1.50. For AC curing, the compressive strength of WPC mix with NS/NH ratio of 0.67 was slightly lower than that with NS/NH ratio of 0.33. This was probably due to the higher degree of hydration reaction with Portland cement which can promote the early compressive strength of mix with high NH (low NS/NH).



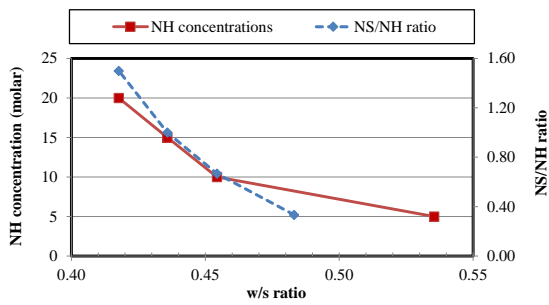
(a) Effect of sodium hydroxide concentration



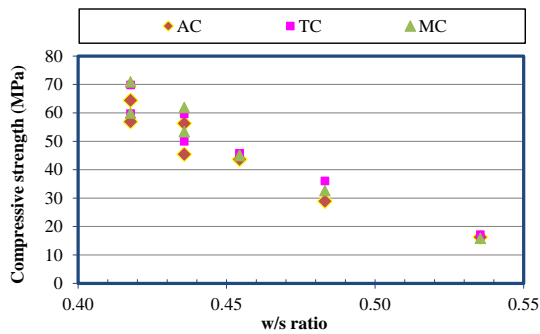
(b) Effect of sodium silicate/sodium hydroxide ratio

Fig.2 Effect of sodium hydroxide concentration and sodium silicate/sodium hydroxide ratio on 7 days compressive strength

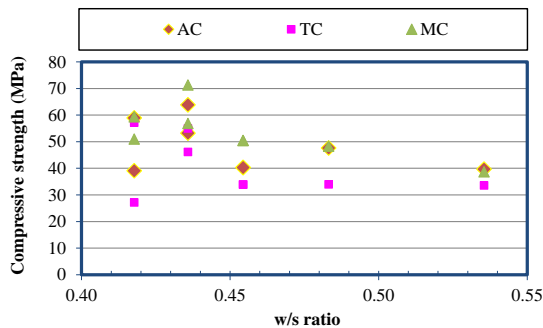
Considering the water to solid ratio (w/s), the increasing of NH concentration and NS/NH ratio decreased the w/s ratio as shown in Fig.3. NH concentrations of 5 to 20 molarity and NS/NH of 0.33 to 1.50 gave decreases in w/s ratio in the ranges of 0.535-0.418 and 0.483-0.418, respectively as presented in Table 2 and Fig. 3(a). The increase in the mass of NaOH flake (solid) portion increased the NH solution concentration thus reduced the volume of water and resulted in decreasing of water/solid ratio. At high NS/NH ratio, the NH content was low, and the water/solid ratio was thus decreased. The decreasing of w/s significantly increased the compressive strengths of CK and WPC geopolymers as displayed in Figs. 3(b) and 3(c). The lowest w/s of 0.418 gave the CK mix with highest compressive strength. But for WPC mix, the compressive strength was highest at w/s of 0.436 and lowered when w/s was lower than 0.436. The decreasing of compressive strength of WPC mix when the w/s is lower than 0.436 is probably due to the very low workability and fast setting of the mix with 20M NH and 1.50 NS/NH ratio with the rapid setting as presented in Fig.1. High strength was obtained with very low water content and with good compaction conditions. Without good compactions, compressive strength dropped due to high porosity [23].



(a) Relation of w/s to NH concentration and NS/NH ratio



(b) Relation of w/s to compressive strength of CK mix



(c) Relation of w/s to compressive strength of WPC mix

Fig.3 Relation of w/s ratio to compressive strength

The curing method also significantly affected the compressive strength of geopolymer pastes. For CK mix, slightly different compressive strengths of each curing method were obtained as shown in Fig.2 and Fig. 3(b). The temperature curing (TC) resulted in a slightly higher compressive strength than those with ambient (AC) and moist (MC) curings.

Temperature curing accelerates the geopolymerization reaction and promotes the development of compressive strength [13]. When the NH concentration and ratio of NS/NH were high in the ranges of 15-20M and 1.00-1.50,

respectively, the compressive strength of mix with MC curing was slightly higher than those with TC and AC curings. It was reported previously that the moist curing of geopolymer product positively affects the compressive strength [10]. Moreover, higher NH concentration and NS/NH ratio exhibited low workability and compaction degree which affected more moisture lost than those of lower NH and NS/NH values. Moist curing thus maintained the moisture content with a positive effect on compressive strength.

The compressive strength at 7 days of CK and WPC geopolymers were nearly equal. But WPC mix has clearly difference in the development of compressive strength of using of Portland cement (WPC) on geopolymer at the longer age of 36 days as presented in Fig. 4. The WPC mixes with AC and MC curings had higher compressive strength at later age than that of CK mix. It should be noted that using of WPC in geopolymer product can beneficially improve the compressive strength of geopolymer with low energy consumption in the production by the using of moist curing or moisture loss protection process.

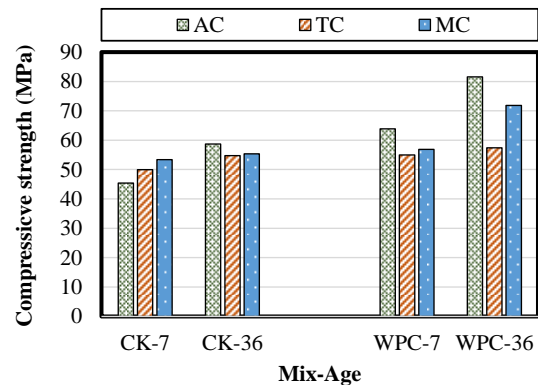


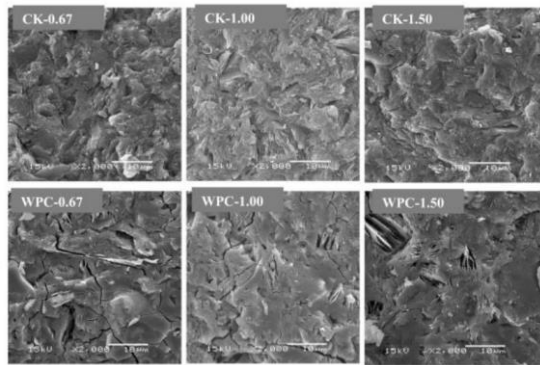
Fig.4 Compressive strength of geopolymer pastes at 7 and 36 days

3.3 Microstructure

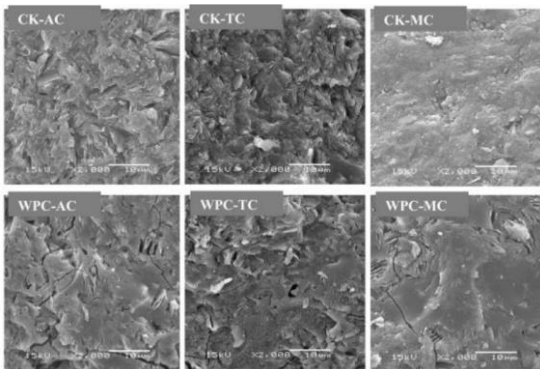
The SEM images of geopolymer pastes at 7 days with varied NS/NH ratio and curing method were illustrated in Fig.5. The increasing of NS/NH values of 0.67, 1.00 and 1.50 did not clearly present the different morphology in both CK and WPC mixed as shown in Fig. 5(a). The microstructure of CK paste presented a large platy form of kaolinite plate [24] from the unreacted calcined kaolin. The WPC showed the denser microstructure of paste matrix, less of the platy plate and denser smooth gel which probably was the co-existed hydration products from the addition of WPC in the system; that made the pastes denser and increased the

compressive strength [25].

The SEM images of geopolymer pastes with different curing methods are presented in Fig. 5(b). The samples of AC and TC curings had a similar microstructure, CK mix consisted of platy form of unreacted kaolinite and WPC mix showed the smooth and denser surface. The MC curing presented the smooth surface in both CK and WPC mixes. The smooth surface represented a dense and compacted paste gel with was related to high compressive strength compared to the other curing methods at 7-day age as shown in Fig. 4. The moisture curing protected water losses in geopolymer gel that could reduce the compressive strength in the absence of water in gel [2] and additionally increased the rate of hydration reaction in WPC mix that enhanced the strength development.



(a) Mix-NS/NH ratio

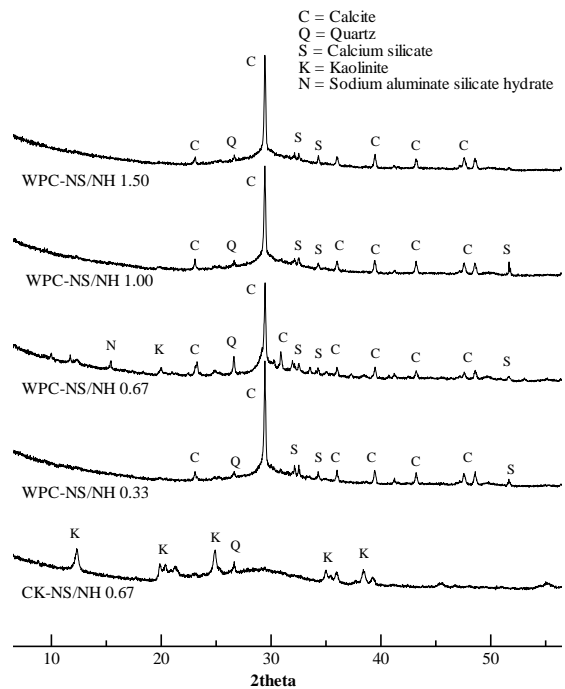


(b) Mix-Curing method

Fig.5 SEM images of CK-WPC geopolymer pastes

The XRD pattern of CK and WPC geopolymer paste samples are shown in Fig. 6. The CK paste had a broad hump at the region of 25°-37° 2θ which represented the major amorphous phase. CK mix has the remaining of kaolinite (K) and quartz (Q) crystalline phases which probably caused by unreacted of starting material. The adding of

Portland (WPC mix) reduced the amorphous phase of geopolymer pastes and showed the large crystalline peak of calcite (C), calcium silicate (S) and sodium aluminate silicate hydrate (N). Those crystalline products were from the hydration reaction that co-existed with the geopolymer gel [25]. The increasing of NS/NH ratio from 0.33 to 1.50 of WPC mix did not significantly affect the XRD pattern, by showing the same main peaks of calcite and calcium silicate.



Where; C (CaCO_3), Q (SiO_2), S (Ca_3SiO_5), K ($\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$), N ($\text{Na}_2\text{Al}_2\text{Si}_2.5\text{O}_9 \cdot 6.2\text{H}_2\text{O}$)

Fig.6 XRD pattern of CK-WPC geopolymer pastes

Different alkali activator properties viz., NH concentration and NS/NH ratio did not noticeably affect the SEM structure and XRD pattern. Due to its products were from the same geopolymerization reaction and the co-existing of products from hydration reaction. But it was showing the significantly affecting on hardening time and compressive strength. Moreover, the curing process was also the main factor affecting the compressive strength of CK-WPC geopolymer which usefully increased the development of strength at the later age.

4. CONCLUSION

The proper use of WPC in CK geopolymer can improve the compressive strength of geopolymer

pastes at ambient temperature production and positively help the development of compressive strength at the later age. The use of moist curing can protect the reduction of compressive strength due to losses of water in CK geopolymer and additionally increased the hydration rate in WPC mix and could be represented as a strengthening method. Especially, the variation of NH concentrations and NS/NH ratios also were significant factors affecting the CK-WPC geopolymer properties. The increasing of NH concentration increased the time of setting and compressive strength with the highest compressive strength at 10M. The increase in NS/NH ratio decreased the time of setting but increased the compressive strength with the highest value at NS/NH ratio of 1.00.

5. ACKNOWLEDGEMENTS

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