



EFFECT OF ALKALINE SOLUTIONS ON ENGINEERING PROPERTIES OF ALKALI-ACTIVATED GGBFS PASTE

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EFFECT OF ALKALINE SOLUTIONS ON ENGINEERING PROPERTIES OF ALKALI-ACTIVATED GGBFS PASTE

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and Wei-Lun Lin¹

Key words: alkaline solution, thermal conductivity, volumetric heat capacity, alkali-activated, slag.

ABSTRACT

In this study, two kinds of alkaline solutions, sodium hydroxide (NaOH) and sodium silicate (Na_2SiO_3), with three dosages of 0.5, 1.0 and 1.5% by weight were used to produce the alkali-activated ground granulated blast furnace slag (GGBFS) paste at three different liquid-solid ratios by weight of 0.45, 0.5 and 0.55. Most engineering properties of the paste were examined at age of 3, 7, 14, 28 and 56 days. Experimental results show that a mixed alkaline solution of sodium hydroxide and sodium silicate is necessary to produce an alkali-activated GGBFS paste with adequate workability and strength. Both the liquid-solid ratio and the amount of the alkaline solution affect the compressive strength in the range of 30.38 to 166.22 MPa at age of 28 days. After curing 28 days, alkali-activated GGBFS paste has the coefficient of thermal conductivity and volumetric heat capacity in range of 0.585-0.791 W/m²·K and 1.583-1.8 J/m³·K, respectively, which are lower than/close to those of normal weight concrete and thus can be used as a proper material for heat insulation. The microstructural porosity of alkali-activated GGBFS paste is closely related to its engineering and thermal properties.

I. INTRODUCTION

In 1992, the common consensus of suitable environment was established in "Agenda 21." Four major waste-related program areas were also suggested in order to provide a comprehensive and environmentally responsive framework for managing municipal solid wastes: (1) Minimizing wastes; (2)

Maximizing environmentally sound waste reuse and recycling; (3) Promoting environmentally sound waste disposal and treatment and (4) Extending waste service coverage [18]. Therefore, many industry wastes are thus required to be reused in construction engineering for environment sustainable consideration. The ground granulated blast furnace slag (GGBFS) with major components of SiO_2 , CaO, Al_2O_3 and the other oxides is a by-product from the iron and steel industry. A large quantity of GGBFS is produced every year in Taiwan and needs to be properly treated to avoid potential environmental problems. In general, GGBFS is commonly reused to replace portion of cement in making concrete, especially the high strength concrete (HSC) and the high performance concrete (HPC), on account for alleviating environmental impact, increasing workability and durability, and saving cost [1, 3, 15, 20]. In recently years, the alkali-activated GGBFS paste by mixing of GGBFS and alkaline solutions has been found to be a novel material with excellent compressive strength and fire-resistance capability [10, 14, 16, 17, 23]. The GGBFS particle is dissolved in the alkaline solution to form Si and Al gel on the surface of particle. Then, a polycondensation process for polysialate is occurred and forms the networked structure as the empirical form $\text{M}_n[-(\text{SiO}_2)_z - \text{AlO}_2]_n \cdot w\text{H}_2\text{O}$ [6, 7], where M indicates the positive ion of metal, n indicates the degree of polymerization, z indicates the ratio of silica to aluminum and normally equals to the value between 1 to 3.

Previous studies investigated the ratio of liquid to solid and the amount of alkaline solution are the significant factors to affect the mechanical properties of alumino-silicate based/ash-based geopolymers [19, 21]. The ratio of liquid to solid is defined as the weight ratio of alkaline solutions to waste alumino-silicate materials. The volume shrinkage of geopolymer paste during harden processing of dehydration is related to the ratio of liquid to solid. Lower ratio of liquid to solid may reduce the shrinkage of geopolymer paste. Xu *et al.* indicated that the ratio of liquid to solid usually affects the reaction of cement polymerization and its engineering properties. The relationship between the ratio of liquid to solid and the precipitation of aluminum and silica ions is linear. As the ratio of liquid to solid is increasing, the initial and final setting times of

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Table 1. Physic properties of GGBFS.

Bulk density (g/cm ³)	Fineness (cm ² /g)	Activity index	
		7 days	28 days
2.8	6000	89.5	119.6

Table 2. Chemical compositions of GGBFS.

SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	All other oxides	L.O.I.
33.47	14.79	0.40	41.61	9.15	0.58

geopolymer paste are also increased. The addition of alkali-activated concentration is an important control factor to affect other Si gel generation. Komnitsas *et al.* [9] indicate the hydroxide concentration is a control vital factor of strength. Their results show that a high alkali hydroxide concentration could obtain a high compressive strength, but when excess concentration is used, a decrease of the final strength occurs. Brough *et al.* [5] research results also show that although an optimal high alkali hydroxide concentration can significantly increase the strength and shorten the setting time, but it is liable to let the specimen becoming brittle and vulnerable to weathering, although this fault can be improved by adding other mineral additive or curing in water. Thus, an optimal high concentration of alkaline could accelerate the polymerization reaction and increase mechanical properties, but excessive concentration beyond optimal value also decreases the strength and durability.

On the other hand, both the thermal conductivity and volumetric heat capacity of geopolymer paste are the crucial factors to access its ability for heat transfer and suitability for heat insulation [2, 4, 8, 13, 22]. A material with lower thermal conductivity can reduce heat losses, save energy and serve as a good potential candidate for thermal isolation and/or fire resistant materials of structures. A material with high volumetric heat capacity indicates that a high heat energy from outer resource is needed to increase the inner temperature of material.

In this study, effects of the two kinds and three amounts of dosage of alkaline solutions, sodium hydroxide (NaOH) and sodium silicate (Na₂SiO₃), on the engineering and thermal properties of alkali-activated ground granulated blast furnace slag (GGBFS) paste were investigated. Detailed of experimental program and test results are described in the following sections.

II. EXPERIMENT WORK

1. Raw Materials

The characteristics of raw materials of alkali-activated GGBFS paste used in this study are described as follows:

- (1) Ground granulated blast furnace slag (GGBFS): A domestic product from CHC Resources Corporation, Taiwan, has the physic properties and chemical compositions as

Table 3. Chemical compositions of alkaline solutions.

	Sodium hydroxide (NaOH)	Sodium silicate (Na ₂ SiO ₃)
NaOH	98.2	---
NaCO ₃	0.165	---
NaCl	0.0135	---
Fe	0.0004	< 0.02
SiO ₂	---	29.2
Na ₂ O	---	8.19

shown in Tables 1 and 2, respectively.

- (2) Sodium hydroxide (NaOH) solution: The NaOH solution with 5M concentration was prepared by mixing the solid sodium hydroxide flake with deionized water in the laboratory with its chemical compositions shown in Table 3.
- (3) Sodium silicate solution (water glass) (Na₂SiO₃): A commercial sodium silicate solution produced in Taiwan has the chemical composition as shown in Table 3.

2. Mixture Proportions

Previous study [11] has indicated that an alkaline solution with only sodium hydroxide (NaOH) results in quick setting and high strength of alkali-activated GGBFS paste. On the other hand, an alkaline solution with only sodium silicate solution (Na₂SiO₃) renders the alkali-activated GGBFS paste into a good workability with hardly setting state and low strength. Thus, a mixed alkaline solution of sodium hydroxide and sodium silicate is necessary to produce an alkali-activated GGBFS paste with adequate workability and strength. By adjusting merits of both solutions to obtain an applicable workability and strengths of alkali-activated GGBFS paste from quite a few preliminary trials, this study finally adopts two parameters, the weight ratio of liquid to solid (GGBFS) (R_{LS}) and weight percentage of sodium oxide (Na₂O) to solid (GGBFS) (N), to conduct the investigation. Nine mixtures of alkali-activated GGBFS paste based on three values of R_{LS} , 0.45, 0.50 and 0.55, and three percentages of N , 0.5, 1.0 and 1.5%, were used. All the ingredients of nine mixtures, as shown in Table 4, were calculated from the following three equations:

$$\frac{W_{NO}}{W_{sl}} = N \quad (1)$$

$$\frac{W_{SH} + W_{SS}}{W_{sl}} = R_{LS} \quad (2)$$

$$\frac{W_{SH}}{\gamma_{SH}} + \frac{W_{SS}}{\gamma_{SS}} + \frac{W_{sl}}{\gamma_{sl}} = 1 \quad (3)$$

where W_{NO} , W_{SH} , W_{SS} and W_{sl} are weights, sodium oxide solution, sodium hydroxide solution, sodium silicate solution

Table 4. Mix proportion of alkali-activated GGBFS paste.

Designation	R_{LS}	N (%)	Solid		
			W_{sl} (GGBFS) (kg/m ³)	W_{SS} (Water glass) (kg/m ³)	W_{SH} (5M NaOH) (kg/m ³)
LS45N05	0.45	0.5	1743	719	65
LS45N10	0.45	1.0	1730	649	130
LS45N15	0.45	1.5	1718	580	193
LS50N05	0.50	0.5	1656	766	62
LS50N10	0.50	1.0	1644	699	123
LS50N15	0.50	1.5	1633	633	183
LS55N05	0.55	0.5	1577	808	59
LS55N10	0.55	1.0	1567	744	117
LS55N15	0.55	1.5	1556	681	175

and GGBFS in units of kg in sodium hydroxide solution, respectively; γ_{SH} , γ_{SS} and γ_{sl} are bulk density of sodium hydroxide solution, sodium silicate solution and GGBFS in units of kg/m^3 , respectively.

3. Specimen Preparation and Testing Program

After the GGBFS and 5M NaOH solution had been firstly mixed together in a mixer for about 5 minutes, the sodium silicate solution then was gradually added into the mixer and mixed for another 5 minutes. The flow test and setting time test of fresh alkali-activated GGBFS paste were determined afterwards according to ASTM C230M and ASTM C807 test methods, respectively. After the flow test, the cubic specimens ($50 \times 50 \times 50$ mm) and cylinder specimens ($\phi 50 \times 100$ mm) with each mix proportion were cast. After one day, these specimens were demoulded and cured at room temperature until the ages of 3, 7, 14, 28 and 56 days for uniaxial compressive strength test and dynamic modulus of elasticity and the age of 28 days for testing coefficient of thermal conductivity and volumetric heat capacity. In total, 135 cubic specimens and 27 cylindrical specimens were used.

The uniaxial compressive strength test and dynamic modulus of elasticity test were conducted in accord with ASTM C39 and ASTM C215 Standards, respectively. The equipment of ISOMET 2104 with surface probe was used to measure the coefficient of thermal conductivity and volumetric heat capacity of alkali-activated GGBFS paste. The ISOMET 2104, manufactured by Applied Precision Ltd., Slovakia, is a portable measuring instrument for direct measurement of heat transfer properties of a wide range of isotropic materials including cellular insulating materials, plastics, glasses and minerals with a dynamic measurement method that reduces the time of thermal conductivity measurements to 10-16 minutes. Finally, the microstructures of each specimen with different mix proportions were also investigated by using scanning electron microscope (SEM) and used to correlate the thermal and engineering properties of alkali-activated GGBFS paste.

Table 5. Flow value and setting of each mix proportion of alkali-activated GGBFS paste.

Designation	Flow value (%)	Initial setting time (min)	Final setting time (min)
LS45N05	70	380	440
LS45N10	95	270	335
LS45N15	105	20	35
LS50N05	95	540	605
LS50N10	140	370	455
LS50N15	150	65	80
LS55N05	115	650	735
LS55N10	*	510	650
LS55N15	*	170	185

*The fresh mix overflows the circumferential margin of round flow test table.

III. EXPERIMENT RESULTS AND DISCUSSION

1. Workability

The flow test and setting time test were used to evaluate workability of alkali-activated GGBFS paste. Their results are shown in Table 5, Figs. 1 and 2. The smallest values of flow test of 70~105% occur at the lowest R_{LS} of 0.45. With the increase of R_{LS} from 0.45 to 0.55, a substantial increase of flow test value is observed. Increasing the N values from 0.5 to 1.5% tends to increase the flow test value for all three sets of mixtures ($R_{LS} = 0.45, 0.50$ and 0.55). The smallest values of initial setting times of 20~380 minutes and final setting times of 35~440 minutes also occur at the lowest R_{LS} of 0.45. With the increase of R_{LS} from 0.45 to 0.55, a substantial increase of flow test value up to 170~650 minutes for initial setting time and 185~735 minutes for final setting time is observed. However, on the contrary, increasing the N values from 0.5 to 1.5% tends to substantially decrease the both setting times for all three sets of mixtures ($R_{LS} = 0.45, 0.50$ and 0.55).

Table 6. Compressive strengths of each mix proportion of alkali-activated GGBFS paste.

Designation	Compressive strength (Unit: MPa) (average of three specimens)				
	3 days	7 days	14 days	28 days	56 days
LS45N05	2.96	3.08	8.16	46.96	78.47
LS45N10	40.40	57.36	73.93	74.06	78.89
LS45N15	61.18	74.77	97.28	104.05	110.36
LS50N05	1.73	3.03	10.48	49.14	84.37
LS50N10	24.65	39.09	82.57	108.70	112.06
LS50N15	53.39	64.48	91.58	166.22	166.32
LS55N05	1.20	3.16	5.76	30.38	61.36
LS55N10	2.54	25.73	30.86	57.68	76.75
LS55N15	51.95	65.61	79.93	135.63	146.33



Fig. 1. Typical flow test photo of alkali-activated GGBFS paste with different ratios of liquid to solid ($N = 0.5\%$).

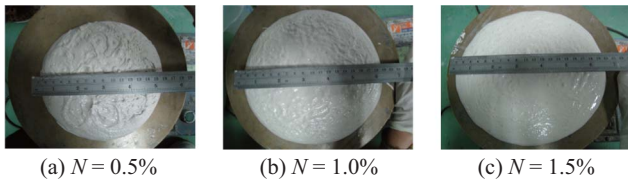


Fig. 2. Typical flow test photo of alkali-activated GGBFS paste with R_{LS} of 0.45 and different additions of alkaline solution.

The reason for such opposite variation of these two workability indices could be attributed to the fact that the increase of R_{LS} means an increase of water in the fresh paste such that the flow test values apparently increase too. But too much water tends to dilute the concentration of alkali-activated solution and, subsequently, postpone the alkali-activating speed and increase setting time. On the other hand, increase N values from 0.5 to 1.5% will thicken the concentration of alkaline solution to expedite alkali-activating reaction and reduce both setting times.

2. Compressive Strength

The results of uniaxial compressive strengths for all mixtures at ages of 3, 7, 14, 28 and 56 days are summarized in Table 6 and shown in Figs. 3 and 4. The highest values at age of 28 and 56 days occur at three mixture sets of R_{LS} of 0.50, rather than 0.45 or 0.55, in which a substantial increase of strength from 49.14 to 166.22 MPa at 28 days and from 84.37 to 166.32 MPa at 56 days with the increase of N values from 0.5 to 1.5%. The increase ratios are in the range from 97.1%

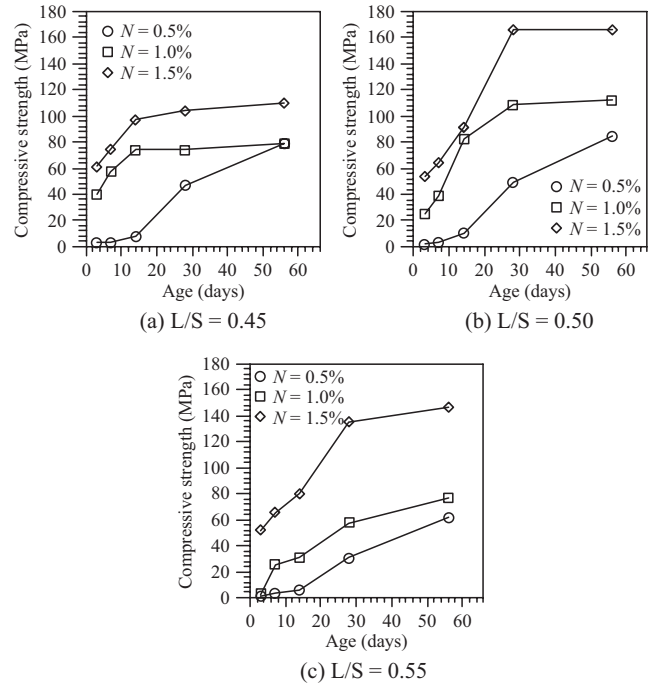


Fig. 3. Compressive strength of each mix group with different R_{LS} (L/S).

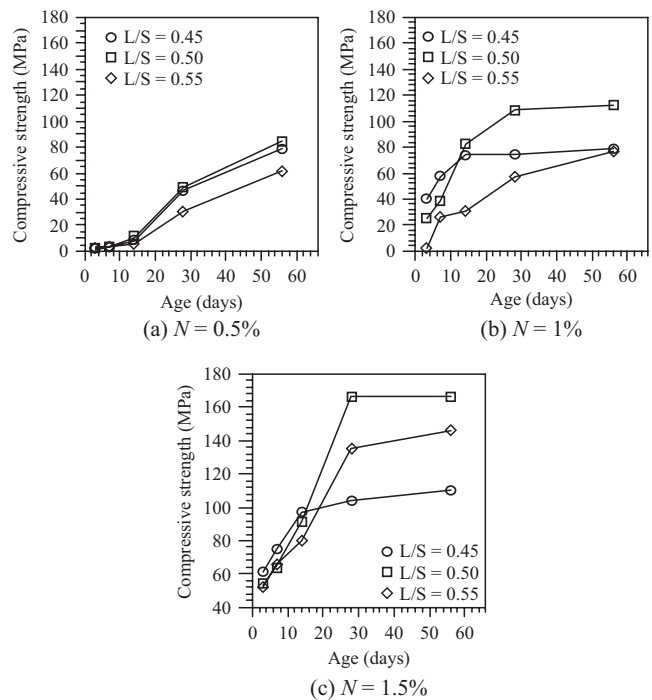


Fig. 4. Compressive strength of each mix group with different additions.

to 238.3%. At a fixed value of N , it seems that the index of R_{LS} is the key parameter to control the strength. Thus there exists an optimal value of R_{LS} of 0.50 and a significant strength increases when the N value increases to 1.5% for the mixture set of LS50N15. Unfortunately, Table 5 shows that the

Table 7. Dynamic elasticity modulus of each mix proportion of alkali-activated GGBFS paste.

Designation	Dynamic Modulus of Elasticity (Unit: GPa) (average of three specimens)				
	3 days	7 days	14 days	28 days	56 days
LS45N05	0.81	3.65	8.65	18.24	26.04
LS45N10	18.05	19.68	24.94	26.40	27.95
LS45N15	17.79	21.21	23.40	24.72	26.37
LS50N05	1.58	3.99	4.14	17.57	21.30
LS50N10	6.25	20.04	23.09	23.52	26.33
LS50N15	17.32	21.17	22.96	24.21	25.70
LS55N05	1.12	2.47	2.82	5.54	20.81
LS55N10	3.05	12.36	16.76	17.34	22.07
LS55N15	17.05	17.75	17.93	20.19	24.70

mixture set of LS50N15 also has a shortest setting time of 65 and 80 minutes. In practice, definitely, this mixture set is not the best choice for the practical application. Table 6 also shows that the mixture sets of N of 1.5% have very high early strengths at 3 days of 51.95 to 61.18 MPa and a longer setting times of 170 and 185 minutes, respectively, for mixture set of LS55N15. This mixture is apparently suitable for repair work. The compressive strengths for six mixtures with R_{LS} of 0.50 and 0.55 have increased substantially from 3 to 28 days. After 28 days, the compressive strengths for mixtures with $N = 1.0$ and 1.5% remain roughly constant. It is interesting to note that this phenomenon of polycondensation mechanism of alkali-activated GGBFS paste is quite similar to the hydration mechanism of Portland cement paste.

3. Dynamic Modulus of Elasticity

The results of dynamic modulus of elasticity tests on various mixes at ages of 3, 7, 14, 28 and 56 days are summarized in Table 7 and shown in Figs. 5 and 6. Among three mixture sets of $N = 0.5, 1.0$ and 1.5%, the highest values of dynamic moduli of elasticity of 27.95, 26.33 and 22.07 GPa roughly occur at $N = 1.0\%$. On the other hand, among three mixture sets of $R_{LS} = 0.45, 0.50$ and 0.55, the highest values of dynamic moduli of elasticity of 26.04, 27.95 and 26.37 GPa occur at $R_{LS} = 0.45$. Thus there exists an optimal combination of R_{LS} and N values to have a highest dynamic modulus of elasticity. But, unlike the considerable increase ratio of compressive strength, the increase ratios here are only between 7.3 and 26.7%. The dynamic moduli of elasticity of alkali-activated GGBFS paste are not very sensitive to the variations of R_{LS} and N . Furthermore, the relation between compressive strengths and dynamic modulus of elasticity for alkali-activated GGBFS paste is not closely correlated.

4. Coefficient of Thermal Conductivity and Volumetric Heat Capacity

Both test results of the coefficient of thermal conductivity and volumetric heat capacity for alkali-activated GGBFS paste at age of 28 days are shown in Tables 8, 9 and Fig. 7(a)

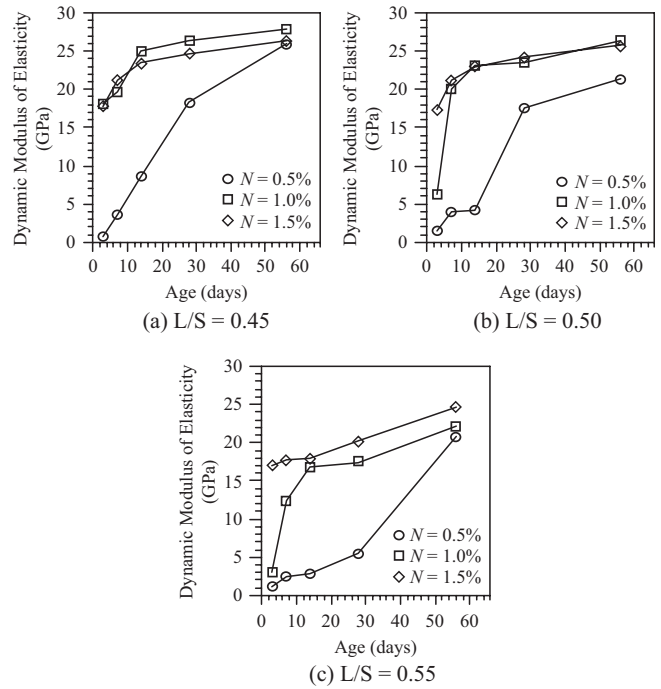


Fig. 5. Dynamic modulus of elasticity of each mix group with different R_{LS} (L/S).

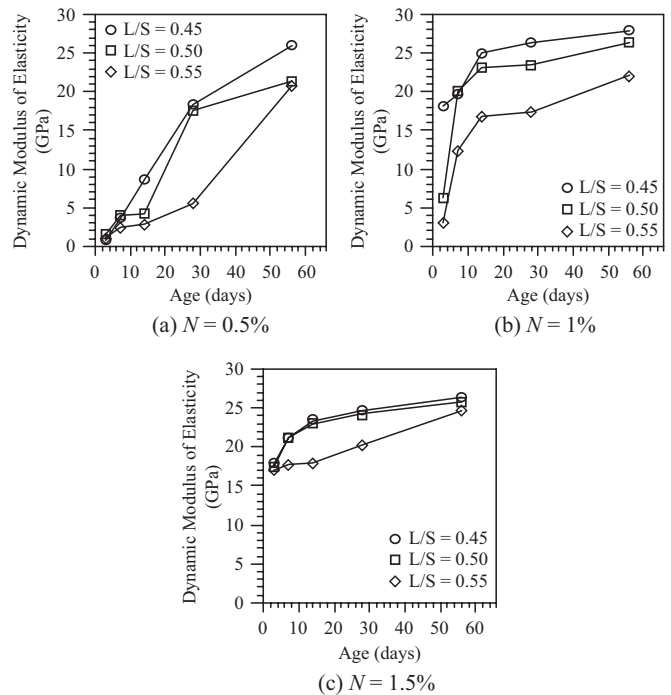


Fig. 6. Dynamic modulus of elasticity of each mix group with different additions.

and 7(b). When the added amount of sodium oxide is increased from 0.5 to 1.5% and the liquid to solid ratio (R_{LS}) from 0.45 to 0.55, the maximum increase ratio of coefficients of thermal conductivity is 35.2% (from 0.585 to 0.791

Table 8. Coefficient of thermal conductivity of alkali-activated GGBFS paste with different R_{LS} and N .

(Unit: $W/m \cdot K$)

	$R_{LS} = 0.45$		$R_{LS} = 0.50$		$R_{LS} = 0.55$	
	avg*	s**	avg*	s**	avg*	s**
$N = 0.5$	0.691	0.021	0.608	0.005	0.585	0.010
$N = 1.0$	0.787	0.004	0.764	0.003	0.762	0.013
$N = 1.5$	0.791	0.001	0.781	0.003	0.773	0.004

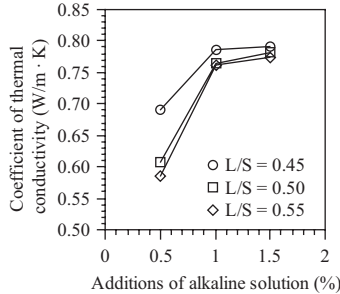
Note: * avg indicates the average value of observations.
 ** s indicates the standard deviation of observations.

Table 9. Volumetric heat capacity of alkali-activated GGBFS paste with different R_{LS} and N .

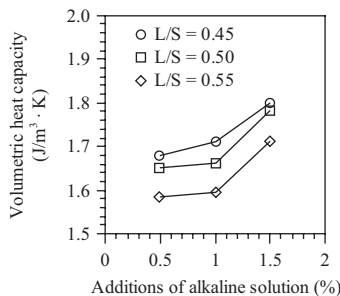
(Unit: $J/m^3 \cdot K$)

	$R_{LS} = 0.45$		$R_{LS} = 0.50$		$R_{LS} = 0.55$	
	avg*	s**	avg*	s**	avg*	s**
$N = 0.5$	1.678	0.016	1.653	0.006	1.583	0.006
$N = 1.0$	1.712	0.028	1.663	0.006	1.595	0.009
$N = 1.5$	1.800	0.010	1.782	0.004	1.710	0.027

Note: * avg indicates the average value of observations.
 ** s indicates the standard deviation of observations.



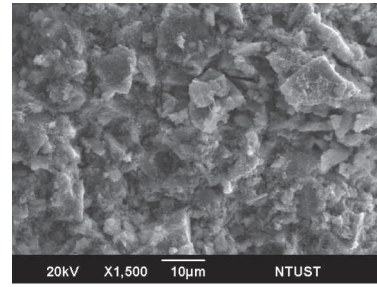
(a) Coefficient of thermal conductivity



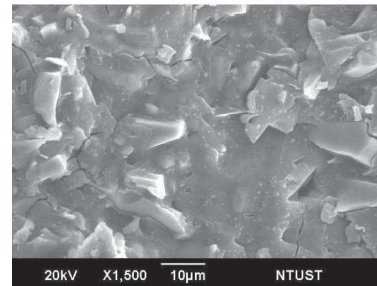
(b) Volumetric heat capacity

Fig. 7. Thermal properties of alkali-activated GGBFS paste with different R_{LS} values and additions of alkaline solution: (a) Coefficient of thermal conductivity, (b) Volumetric heat capacity.

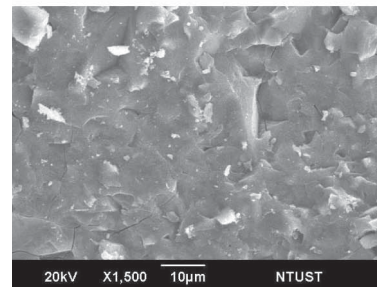
$W/m \cdot K$), and the maximum increase ratio of volumetric heat capacity is 13.7% (from 1.583 to 1.800 $W/m \cdot K$). The highest coefficients of thermal conductivity of 0.791 $W/m \cdot K$ is lower than that of 2.5 $W/m \cdot K$ and the highest volumetric heat



(a) $N = 0.5\%$



(b) $N = 1\%$



(c) $N = 1.5\%$

Fig. 8. Typical microstructure of alkali-activated GGBFS paste with R_{LS} of 0.45 and different additions of alkaline solution.

capacity of 1.8 $J/m^3 \cdot K$ is close to that of 2.06 $J/m^3 \cdot K$ for normal weight concrete [12]. Thus alkali-activated GGBFS paste can be used as a proper material for heat insulation and fire resistance of building construction.

5. Microstructure of Alkali-Activated GGBFS Paste

Typical photos of microstructures of alkali-activated GGBFS paste with R_{LS} of 0.45 and different amounts of added sodium oxide are shown in Fig. 8. The porosity of microstructure is gradually reduced, as shown in Fig. 8(a), (b) and (c), when the amount of added sodium oxide is increased from 0.5 to 1.5%. The characteristics of these microstructural variations of porosity structure could explain the beneficial improvement on the compressive strengths and dynamic elasticity moduli of alkali-activated GGBFS paste with the increased amount of added sodium oxide in the alkaline solution. Accordingly, the coefficient of thermal conductivity and volumetric heat capacity are increased as well.

Previous experimental results have shown that the smallest coefficients of thermal conductivity occurred as $N = 0.5\%$.

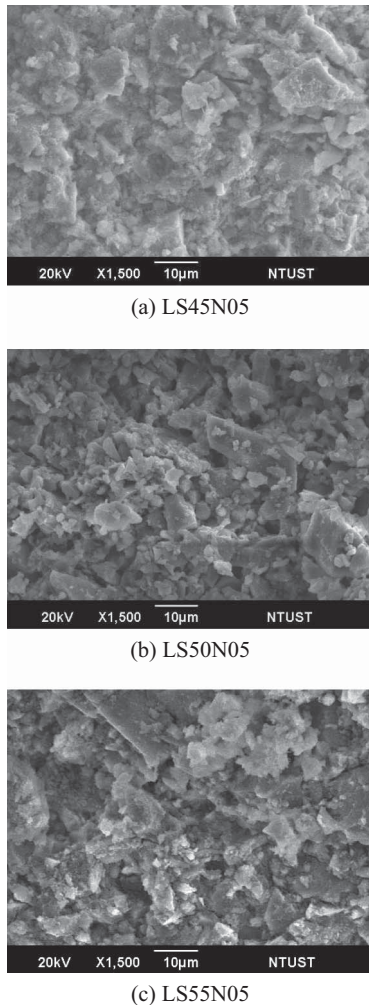


Fig. 9. Typical microstructure of alkali-activated GGBFS paste with different ratios of liquid to solid ($N = 0.5\%$).

Fig. 9(a), (b) and (c) show the microstructure of alkali-activated GGBFS paste with N of 0.5% and R_{LS} of 0.45, 0.50 and 0.55, respectively. These micrographs clearly show that a large portion of voids has formed in the microstructure of alkali-activated GGBFS paste and could also explain the cause of its low compressive strength, dynamic modulus of elasticity, coefficient of thermal conductivity and volumetric heat capacity as shown in the Fig. 4(a), 6(a), 7(a) and 7(b).

IV. CONCLUSIONS

Based on the experimental results and microstructural analyses of alkali-activated GGBFS paste presented in this study, the following conclusions can be drawn:

1. A combined alkaline solution of sodium hydroxide and sodium silicate solution is necessary in the mixture to produce alkali-activated GGBFS paste with adequate workability and other engineering properties, for example, a compressive strength of 166.32 MPa and a dynamic elas-

ticity modulus of 27.95 GPa at age of 56 days. Thus, a mixture methodology for such purpose is proposed (about 7~22% by weight of sodium hydroxide in the liquid as calculated from Table 4) and verified experimentally to be effective.

2. The polycondensation mechanism of alkali-activated GGBFS paste is quite similar to the hydration mechanism of Portland cement paste, in that, most of the engineering properties for both pastes are well developed at age of 28 days, and keep a steady slow growth until 56 days.
3. The flow values of fresh alkali-activated GGBFS paste monotonically increase with the increases of both the added amount of equivalent sodium oxide (Na_2O) and the liquid to solid ratio. However, the initial and final setting times also monotonically increase with the increase of the liquid to solid ratio due to the dilation effects, but substantially decrease with the increase of the added amount of equivalent sodium oxide (Na_2O).
4. The compressive strengths of alkali-activated GGBFS paste at all five ages will monotonically increase with the increase of added amount of equivalent sodium oxide (Na_2O) of alkaline solution in the mixture where a highest compressive strength of 166.32 MPa was found. However, increase of Na_2O in the mixture also notably expedites both the initial and final setting times of the paste to almost a same short 10 minutes such that its practicability may be thus limited.
5. An optimal value of liquid to solid ratio of about 50% of the mixture was found to have the higher compressive strength achieve to 166.32 MPa, that indicates that the effect of interaction between the amount of sodium oxide and the liquid to solid ratio on the chemical mechanism of strength development is worthy of further investigation in the future.
6. The dynamic moduli of elasticity of alkali-activated GGBFS paste are not very sensitive to the variations of the amount of equivalent sodium oxide and the liquid to solid ratio. Furthermore, the relation between compressive strengths and dynamic modulus of elasticity for alkali-activated GGBFS paste is not closely correlated.
7. The alkali-activated GGBFS paste has better thermal properties compared with those of normal weight concrete and thus can be used as a proper material for heat insulation and fire resistance of building construction.
8. The denser the microstructure of alkali-activated GGBFS paste is, the higher its compressive strength, dynamic modulus of elasticity, coefficient of thermal conductivity and volumetric heat capacity will be.

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