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Study on Compressive Strength Characteristics and Aging of Flow Value for Geopolymer Mortar Using of Fly Ash as Active Filler

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Abstract: *In this study, compressive strength characteristics and aging of flow value of geopolymer mortar using fly ash were experimentally examined. As a result, following knowledge was obtained. (1) Compressive strength increases using finely ground fly ash and high concentration aqueous sodium metasilicate solution. (2) Aging of flow value and compressive strength tend to be different depending on Concentration of sodium metasilicate aqueous solution, presence or absence of fine grinding of fly ash, and addition ratio of setting retarder. Moreover, it was hypothesized that state of floc formation is different depending on presence or absence of fine grinding of fly ash. Then, it was shown that it is necessary to verify influence on unit quantity of sodium metasilicate aqueous solution, aging of flow value, and compressive strength characteristics.*

Keywords: *fly ash, geopolymer, sodium metasilicate, anhydrous citric acid, compressive strength, flow value*

1. Introduction

Concrete is a very important construction material, and Portland cement is chiefly used as the binder. However, a large volume of CO_2 is emitted in the process of manufacturing Portland cement, and in recent years, this is viewed as a problem, as it is a contributing factor to global warming. Also, about 60% of the emitted CO_2 originates from thermal decomposition of limestone ($CaCO_3$), which is the prime ingredient of Portland cement therefore [1], such emissions are unavoidable. Thus, it is important to develop a replacement for Portland cement for use as a binder [2]. Finding a practical use for the coal ash generated by burning over ten million tons per year of coal in Japanese electrical power plants is also an important area of research. Many studies have addressed using fly ash (FA) as an admixture for concrete. However, the coal ash actually used for this only amount to a tiny 3.5% of the generated volume [3].

The above is a background for the ongoing research into geopolymers (GPs) composed of the amorphous polycondensates obtained with aluminum silicate powders (activated filler) such as blast furnace slag (BFS) or FA [4]. This filler could be a replacement for cement, thus reducing CO_2 emissions. Inukai et al. carried out basic research on GP using liquid glass, BFS, and FA in order to obtain a harder material with higher strength [5]-[7]. However, no detailed research has been conducted on GP finely ground FA as an activated filler, examining types and concentrations of aqueous alkaline solutions.

This research employed JIS Type II FA and "FA7" finely ground to a surface area of about 7000 cm^2/g, which was described in previous reports [8], [9]. In Exp. 1, the Influence of the mass mixing ratio between a 10 mol/L aqueous solution of sodium hydroxide (NH) and an aqueous sodium metasilicate (NS) solution on the compressive strength characteristics of GP mortar was examined. In Exp. 2, the fineness of the FA powder on the compressive strength characteristics of GP mortar was examined. In Exp. 3, anhydrous citric acid (ST) was added as a setting retarder and its effect on the aging of the flow value and on the compressive strength characteristics of GP mortar was examined.

2. Influence of 10 mol/L NH Aqueous: NS Aqueous Mass Mixing Ratio on Compressive Strength Characteristics of GP Mortar (Exp. 1)

2.1 Overview of Experiment

2.1.1 Experimental Factors

Table 1 shows the experimental factors. The mass mixing ratio for the 10 mol/L NH aqueous solution and the NS aqueous solution was varied.

Table 1. *Experimental Factors (Exp.1)*

Factors	Levels
Nsaq Concentration (mol/L)	0.5, 1.0, 1.5, 2.0
NH 10 mol/LSaq (mass)	0 (NS Only), 1, 2,3,4

2.1.2 Used Materials and Mix Proportion

Table 2 shows the materials used and Table 3 shows the mix proportion. FA7 was used as the activated filler. Alkaline solution (AW) was dissolved in water to produce aqueous solutions with the desired concentrations and NH/NS mixing ratios. The mix proportion was AW/FA7 50%, 0% air content, and for convenience, the density of the aqueous alkaline solution was always assumed to be 1.0 g/cm^3 in the calculations.

Table 2. *Used Materials (Exp. 1-3)*

Material Names	Marks		Types		Physical Properties
Active fillers	F	FA7	Fly ash JIS type II	Fine grinding Presence	Blaine (cm^2/g): 7480 Density (g/cm^3): 2.52
		FA		Fine grinding Absence	Blaine (cm^2/g): 3480 Density (g/cm^3): 2.28
Fine aggregate	S		Standard sand		Absolute dry density (g/cm^3): 2.64
Alkaline additives	NS		Sodium metasilicate (Na_2SiO_3)		Density (g/cm^3): 2.61
	NH		Sodium hydroxide (NaOH)		Density (g/cm^3): 2.13
Alkaline aqueous solution	AW		NSaq, NG10 mol/L.aq		-
Setting retarder	ST		Anhydrous citric acid ($C_6H_8O_7$)		Density (g/cm^3): 1.66

Table 3. *Mix Proportion (Exp.1)*

NSaq concentration (mol/L)	NHaq/NSaq (mass)	Flow	Air(%)	AW/FA7(%)	S/FA7	Unit weight (km/m3)		
						FA7	AW	S
0.5	0	190 ± 20	0	50	3.63	440	220	1597
	1				2.71	520	260	1410
	2				2.03	600	300	1220
	3				1.51	680	340	1030
	4				1.11	760	380	840
1.0	0				3.20	474	237	1518
	1				2.47	546	273	1346
	2				1.89	620	310	1172
	3				1.40	700	350	982
	4				0.93	780	390	792
1.5	0				3.14	480	240	1505
	1				2.18	580	290	1267
	2				1.51	680	340	1030
	3				1.02	780	390	792
	4				0.63	880	440	557
2.0	0				2.91	500	250	1457
	1				2.03	600	300	1220
	2				1.40	700	350	982
	3				0.93	800	400	747
	4				0.57	900	450	510

2.1.3 Mixing and Flow Tests

The mixing and flow tests were carried out in accordance with JIS R 5201 "Physical Testing Methods for Cement". The batch mixing volume was 1 L.

2.1.4 Compressive Strength Tests

The compressive strength tests were carried out in accordance with JIS A 1142 "Method of test for fine aggregate containing organic impurities by compressive strength of mortar". The test material was created in 2 layers. Each layer was compacted by vibration using a table vibrator for 30 s. The specimens in molds with their upper surfaces covered with clear film, and were sealed curing for 28 days at 20°C.

2.2 Results and Discussion

Table 4 shows the measurement results for the flow value. As can be seen in Table 3, the unit amount of aqueous alkaline solution providing the target flow value tends to increase with increasing NS aqueous concentration and with increasing amount of NH aqueous.

Fig. 1 shows how the compressive strength varies with NS aqueous concentration. Notably, most of the specimens made from only NS (NH/NS = 0) aqueous GP mortar with concentrations of 1.5 mol/L or less broke while being removed from their molds; therefore, only the results for NS aqueous concentrations of 2.0 mol/L are shown.

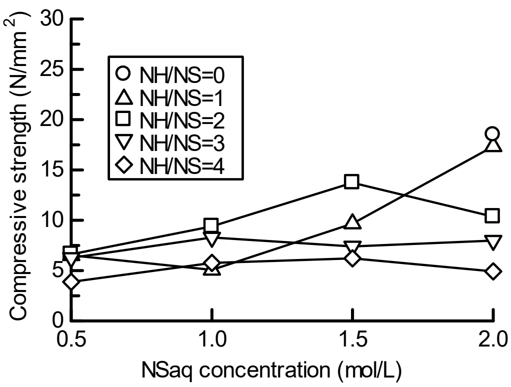


Figure 1. *Relationship between compressive strength and NSaq concentration (Exp. 1)*

The compressive strength varied with the mass mixing ratio for NH aqueous. The lower the ratio, the greater the concentration of NS aqueous at which the maximum compressive strength was reached. As suggested by the test results for the only NS aqueous specimens, when that concentration is high, it reduces the need to incorporate NH aqueous. We can therefore anticipate that higher compressive strength will be found for specimens using s only NS aqueous. Thus, we decided to employ only NS aqueous with concentrations of 2.0 mol/L or greater in the experiments described in the following sections.

Table 4. *Measurement results for flow values (Exp. 1)*

NSaq concentration (mol/L)	Flow value				
	NHaq/NSaq (mass)				
	0	1	2	3	4
0.5	185	181	192	197	192
1.0	182	186	194	202	196
1.5	205	188	188	196	201
2.0	207	202	186	194	198

3. Influence of NS Aqueous Concentration and Fineness of FA on Compressive Strength Characteristics of GP Mortar (Exp. 2)

3.1 Overview of Experiment

3.1.1 Experimental Factors

Table 5 shows the experimental factors. The NS aqueous concentration and the fineness of the FA particles were varied.

Table 5. *Experimental Factors (Exp.2)*

Factors	Levels
NSaq concentration (mol/L)	2.0, 2.5, 3.0, 3.5, 4.0
Fine grinding	Presence (FA7), Absence (FA)

3.1.2 Used Materials and Mix Proportion

The materials used were the same as in Exp. 1 (refer Table 2), and the mix proportion are shown in Table 6. The mix proportion were all calculated to have an AW/F ratio of 50%, assuming an air entrapment of 2%, and the densities of the NS aqueous in the mix design were as shown in Table 7.

The setting time was observed to become shorter when the NS aqueous concentration was 2.5 mol/L, and when the concentration was 3.0 mol/L, the GP mortar hardened instantly. Therefore, in mix proportion where the NS aqueous concentration were 3.0 mol/L or greater, ST was added as appropriate to the amount of added FA or FA7. Table 8 shows the addition ratio of ST, selected within the range that does not affect the mixing of the specimens. Incidentally, the mix proportion in Table 6 all have greatly reduced volumes of amount of NS aqueous, because of the added FA finely ground powder. A hypothesis was developed concerning this and was the basis for the discussions in Exp. 3.

Table 6. *Mix Proportion (Exp.2)*

NSaq concentration (mol/L)	Fine grinding	Flow	Air(%)	AW/F	S/F	Unit weight (km/m^3)			Weight (kg)
						F	AW	S	ST
2.0	Presence(FA7)	190 ± 20	2	50	3.55	450	225	1597	-
	Absence(FA)				2.31	560	280	1296	-
2.5	Presence(FA7)				3.23	480	240	1552	-
	Absence(FA)				2.12	590	295	1249	-
3.0	Presence(FA7)				3.05	500	250	1526	5.0
	Absence(FA)				2.08	600	300	1246	6.0
3.5	Presence(FA7)				2.37	580	290	1375	23.2
	Absence(FA)				1.72	660	330	1135	26.4
4.0	Presence(FA7)				2.11	620	310	1309	37.2
	Absence(FA)				1.42	720	360	1022	43.2

Table 7. *Nsaq density (Exp. 2,3)*

NSaq concentration (mol/L)	NSaq Density (g/cm^3)
2.0	1.15
2.5	1.19
3.0	1.23
3.5	1.26
4.0	1.30

Table 8. *Addition ration of ST (EXp.2)*

NSaq concentration (mol/L)	Extra addition ratio of ST to F (%)
3.0	1.0
3.5	4.0
4.0	6.0

3.1.3 Mixing and Flow Tests

The mixing and flow experiments were carried out in the same way as in Experiment 1. However, the mixing time for the mortars containing FA was extended by 1 min when the NS aqueous concentration was 3.0 mol/L, and by 2 min when it was 3.5 or 4.0 mol/L.

3.1.4 Air Content Tests

The air content was measured in accordance with JIS A 5002 "Lightweight aggregates for structural concrete" (5.12.d Measurement of weight per unit volume of mortar) and calculated in accordance with JIS A 1116 "Method of test for unit mass and air content of fresh concrete by mass" (6.2 Air content).

The test material was created in 2 layers. Each layer was compacted for 30 s by vibration using a table vibrator.

3.1.5 Compressive Strength Tests

The compressive strength was measured as in Exp. 1.

3.2 Results and Discussion

Table 9 shows the measurement results for flow values and air content. As stated in 3.1 (2), the amount of NS aqueous solution to obtain the target flow value showed a reduction of about 50 kg/m^3 (refer to Table 6) for any mix proportion when FA was ground fine. When FA7 was used as the activated filler, the entrapped air was in the range 0.9 - 2.3%, and when FA was used, the entrapped air fell to 0.2 - 0.8%. The entrapped air showed a tendency to increase when the FA was ground fine.

Table 9. *Measurement results for flow values and air contents (Exp. 2)*

NSaq concentration (mol/L)	Fine grinding	Flow value	Air content (%)
2.0	Presence(FA7)	180	2.3
	Absence(FA)	203	0.2
2.5	Presence(FA7)	197	1.9
	Absence(FA)	202	0.5
3.0	Presence(FA7)	181	1.5
	Absence(FA)	192	0.8
3.5	Presence(FA7)	209	1.5
	Absence(FA)	202	0.7
4.0	Presence(FA7)	196	0.9
	Absence(FA)	202	0.2

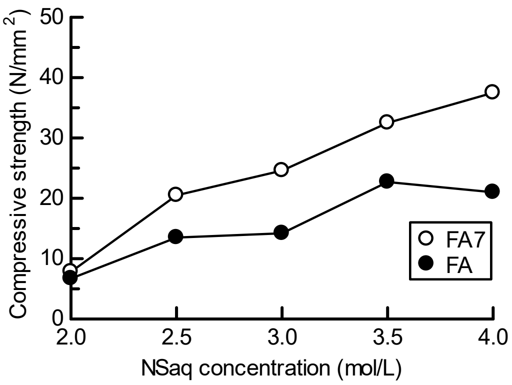


Figure 2. *Relationship between compressive strength and NSaq concentration (Exp. 2)*

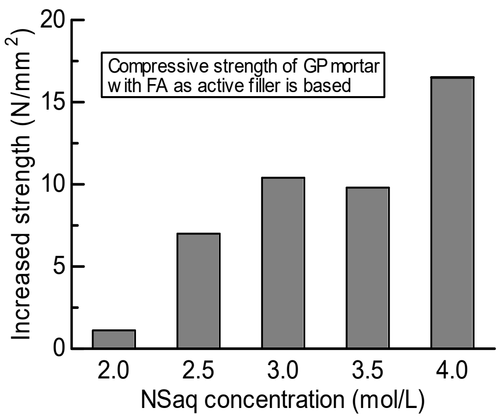


Figure 3. *Increased on compressive strength with NSaq concentration and fine grinding of FA (Exp. 2)*

Fig. 2 shows that the compressive strength increases with increasing NS aqueous concentration, regardless of which FA powder was used. However, the pattern of increase varies with FA fineness. When FA7 was added, the compressive strength increased linearly, while with unground FA, the increase was somewhat more gradual and the compressive strength levelled off at a concentration of 3.5 mol/L. This may be explained as follows. The higher the NS aqueous concentration, the larger the increase in compressive strength imparted by the use of the finer FA7 powder (refer Fig. 3). Thus, the compressive strength of GP mortar incorporating FA as the activate filler was strongly influenced by both the concentration of NS aqueous and the fineness of the FA powder. In comparison to the findings of Exp. 1 and a previous report [9], the compressive strength here is low. The reasons for this are not clear, but the mix design conditions and differences in the addition ratio of ST at may be affecting.

4. Influence of ST on Aging Flow Value and Compressive Strength Characteristics (Exp. 3)

4.1 Overview of Experiment

4.1.1 Experimental Factors

Table 10 shows the experimental factors. The NS aqueous concentration, the fineness of the fly ash, the addition ratio of ST, and the timing of initiation of the flow experiment were varied. The NS aqueous concentration was 3.0 - 4.0 mol/L, requiring addition of ST to retard setting. The addition ratio of ST was the same for specimens with FA and FA7 powder, and was determined as appropriate to the NS aqueous concentration, as described for Exp. 2.

Table 10. *Experimental Factors (Exp. 3)*

NSaq concentration (mol/L)	Fine grinding	Extra addition ratio of ST to F (%)	Start time of flow tests (min)
3.0	Presence(FA7)	1.0, 1.5, 2.0	After completion of mixing 0, 30, 60, 90, 120
	Absence(FA)		
3.5	Presence(FA7)	3.5, 4.0, 4.5	
	Absence(FA)		
4.0	Presence(FA7)	6.0, 6.5, 7.0	
	Absence(FA)		

4.1.2 Used Materials and Mix Proportion

The materials used were the same as in Exp. 1 (refer Table 2) and Table 11 shows the mix proportion. The mix design conditions were identical to those in Exp. 2.

Table 11. *Mix Proportion (Exp. 3)*

NSaq concentration (mol/L)	Fine grinding	ST addition ration (%)	Flow	Air (%)	AW/F (%)	S/F	Unit weight (km/m^3)			Weight (kg)
							F	AW	S	ST
3.0	Presence(FA7)	1.0	190±20	2	50	3.05	500	250	1526	5.0
		1.5								7.5
		2.0								10.0
	Absence(FA)	1.0				2.08	600	300	1246	6.0
		1.5								9.0
		2.0								12.0
3.5	Presence(FA7)	3.5				2.61	550	275	1436	19.2
		4.0								22.0
		4.5								24.8
	Absence(FA)	3.5				1.78	650	325	1156	22.8
		4.0								26.0
		4.5								29.2
4.0	Presence(FA7)	6.0				2.25	600	300	1349	36.0
		6.5								39.0
		7.0								42.0
	Absence(FA)	6.0				1.47	710	355	1045	42.6
		6.5								46.2
		7.0								49.7

4.1.3 Mixing and Flow Tests

The mixing and flow experiments were carried out in the same way as in Exp. 2, except that just before measuring the flow value after the scheduled time had elapsed, the specimens were re-mixed with a spoon for 30 s. The flow value was then measured and the decrease was calculated using Eq. (1).

$$FLd = (FLs - FLt) / FLs \times 100 \quad (1)$$

FLd: decrease in flow value (%)

FLs : flow value just after mixing

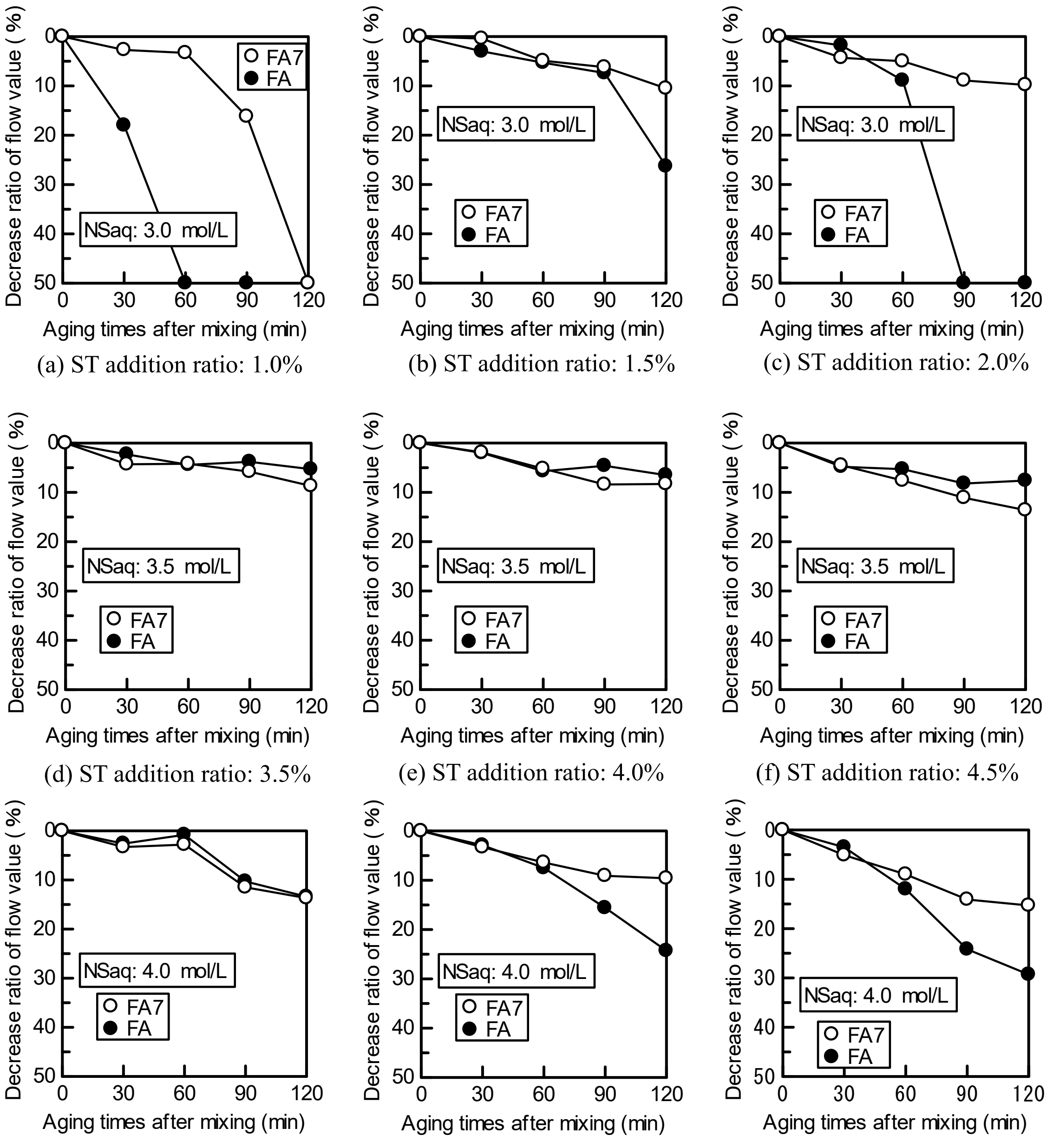
FLt : flow value at time "t" after completion of mixing

4.1.4 Compressive Strength Tests

The compressive strength was measured as in Exp. 1.

4.2 Results and Discussion

Table 12 shows the measurement results for the flow value, and Fig. 4 shows the relationship between the decrease in the flow value and the time since the completion of mixing. When the fluidity fell so low that the flow value could no longer be measured, the decrease was graphed at 50% for all affected mortars.

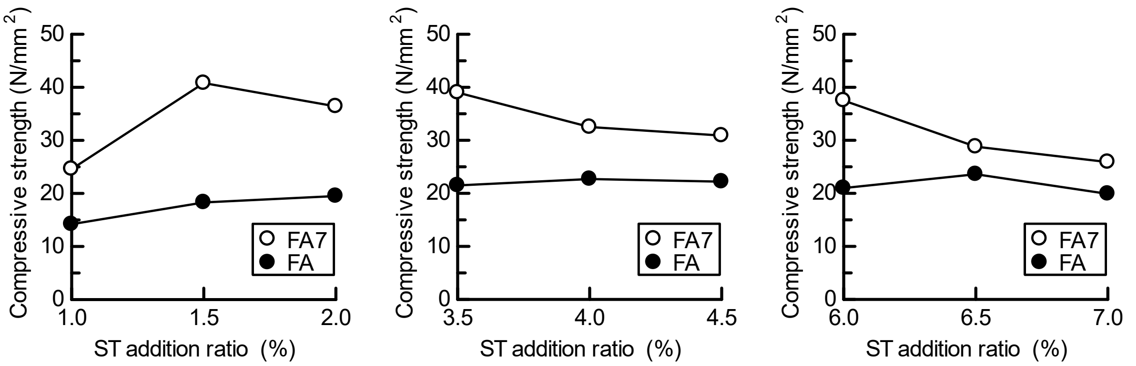


(a) ST addition ratio: 1.0% (b) ST addition ratio: 1.5% (c) ST addition ratio: 2.0%

(d) ST addition ratio: 3.5% (e) ST addition ratio: 4.0% (f) ST addition ratio: 4.5%

(g) ST addition ratio: 6.0% (h) ST addition ratio: 6.5% (i) ST addition ratio: 7.0%

Figure 4. *Relationship between flow values decrease and time after completion of mixing (Exp. 3)*



(a) NSaq: 3.0mol/L (b) NSaq: 3.5mol/L (c) NSaq: 4.0mol/L

Figure 5. *Relationship between compressive strength and addition ratio of ST (Exp. 3)*

Table 12. *Measurement results for flow values (Exp. 3)*

NSaq concentration (mol/L)	Fine grinding	ST addition ration (%)	Flow value				
			Aging times after mixing (min)				
			0	30	60	90	120
3.0	Presence(FA7)	1.0	186	181	180	156	
		1.5	189	188	180	177	169
		2.0	192	183	182	175	173
	Absence(FA)	1.0	178	146	-	-	-
		1.5	196	190	185	181	-
		2.0	192	188	175	-	-
3.5	Presence(FA7)	3.5	194	186	186	183	178
		4.0	195	191	185	179	179
		4.5	200	190	184	177	172
	Absence(FA)	3.5	193	188	184	185	182
		4.0	189	185	178	180	177
		4.5	200	190	189	184	185
4.0	Presence(FA7)	6.0	193	186	187	171	166
		6.5	193	186	180	175	174
		7.0	194	184	176	166	164
	Absence(FA)	6.0	188	183	177	168	163
		6.5	188	182	174	158	142
		7.0	194	187	171	147	137

*) Table in – indicates that it was impossible to measure the flow value

As the figure shows, the decrease in the flow value depended on the NS aqueous concentration, the fineness of the fly ash, and the addition ratio of ST. However, there was no consistent trend under the conditions used in this experiment. For an NS aqueous concentration of 3.0 mol/L, no consistent effect of the addition ratio of ST on aging of the flow value was observed, and the aging of the flow value for some of the GP mortars could not be evaluated for some addition ratio of ST. However, for addition ratio of ST fractions of 1.5% - 2.0% with FA7, and 1.5% with FA, a reasonably clear decrease in flow value with addition ratio of ST was found.

Also, there was a smaller decrease in flow value when the finer FA7 was used, regardless of the addition ratio of ST. Aging were also seen when the NS aqueous concentration was 3.5 mol/L. In contrast to the findings when the NS aqueous concentration was 3.0 or 4.0 mol/L, there was almost no influence of the fineness of the fly ash for any addition ratio of ST. Also, there was a small but visible trend for the drop in flow value to increase with increasing addition ratio of ST in mortars using both FA or FA7. This implies that the best time-dependences were obtained for an ST fraction of 3.5% or lower. When the NS aqueous concentration was 4.0 mol/L, the aging in the flow value again depended on the fineness of the fly ash and on the addition ratio of ST. With FA7, the best aging was found for an addition ratio of ST of 6.5%, and for ST addition ratios close to this, the rate of decrease was only slightly larger. The rate of decrease was lower when FA7 was replaced with FA. However, for higher ST addition ratios, the use of FA led to a higher rate of decrease. The addition ratio of ST where the best aging was obtained was 6.0% or lower.

Thus, the optimum addition ratio of ST depends on the NS aqueous concentration and the fineness of the fly ash, as well as other conditions. More detailed experiments are necessary in order to determine the optimal conditions.

Fig. 5 shows that the compressive strength depended on both the fineness of the fly ash and the addition ratio of ST. It exhibited a maximum for an NS aqueous concentration of 3.0 mol/L and when FA7 was used, which is similar to a previous result for an addition ratio of ST of 1.5%. The strength was lower for addition ratio of ST above and below that level. However, with FA7 and with NS aqueous concentrations of 3.0 mol/L and 4.0 mol/L, the compressive strength was a maximum when the addition ratio of ST was 3.5% and 6.0%, respectively; it tended to decrease with increasing addition ratio of ST. Accordingly, the addition ratio of ST that gave the maximum compressive strength were 1.5% when the NS aqueous concentration was 3.0 mol/L, 3.5% or lower when the NS aqueous concentration was 3.5 mol/L, and 6.0% or lower when the NS aqueous concentration was 4.0 mol/L. Conversely, the compressive strength using FA was independent of the addition ratio of ST. As was found for Exp. 2, the maximum strength occurred for an NS aqueous concentration of around 3.5 mol/L.

Thus, like the aging of the flow value, the compressive strength varied with the addition ratio of ST, the NS aqueous concentration, and the fineness of the fly ash. Again, more detailed investigations are needed in order to achieve higher compressive strength.

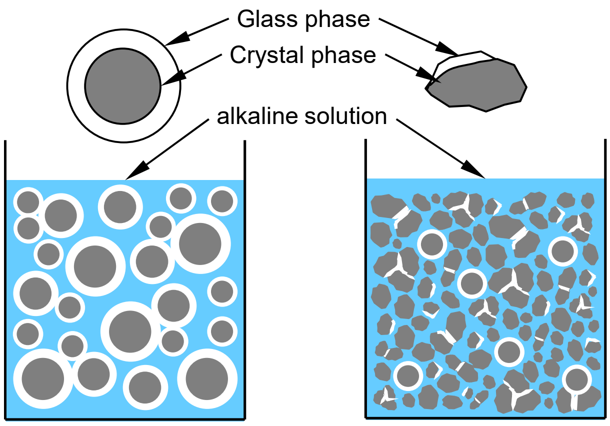


Figure 6. *Initial reaction conditions for FA and FA7 particles*

Fig. 6 presents models for the initial reaction conditions using FA and FA7 particles. These will have to be verified, but based on the above results, the following hypothesis is put forward for the large increase in the amount of NS aqueous when FA is employed. FA particles have a glass phase on their surface, and this initially reacts with the alkaline aqueous solution. In contrast, FA7 particles, being finely ground, have both glass and crystal phases on their surface, and these would be expected to react simultaneously with the alkaline solution. Also, in a concentrated alkaline solution, the glass phase would be expected to react more rapidly. It has been observed that FA particles with a glass surface fuse together more readily than FA7 particles. In addition, since FA particles are larger, they form larger flocks. Assuming that this hypothesis is correct, right from the time mixing begins, geopolymer mortar with FA would contain a greater number of large flocks of particles. This would explain why the amount of NS aqueous solution was observed to increase. Also, the NS aqueous concentration and the addition ratio of ST affect the rate of decrease of the flow value and the increase in the compressive strength when FA7 particles are used.

5. Conclusions

1. The use of finely ground fly ash and a high concentration of aqueous Na_2SiO_3 in a geopolymer mortar reduces the required volume of 10 mol/L NaOH aqueous solution, and only by aqueous Na_2SiO_3 solution can increase the compressive strength.

2. At Na_2SiO_3 concentrations of 3.0 mol/L or greater, anhydrous citric acid must be added to the geopolymer mortar, regardless of whether finely ground fly ash is used.
3. At increased aqueous Na_2SiO_3 concentrations, the compressive strength increases. Adding finely ground fly ash further increases the compressive strength.
4. The aging of the flow value and the compressive strength depend in an inconsistent way on the concentration of aqueous Na_2SiO_3, the fineness of the fly ash, and the anhydrous citric acid ratio added to the geopolymer mortar. More detailed investigations are necessary in order to identify the optimal conditions.
5. A compressive strength exceeding 40 N/mm^2 can be obtained by curing at normal temperatures with finely ground fly ash, and a suitable amount of aqueous Na_2SiO_3 and anhydrous citric acid.
6. Although further study is required, flocking of FA7 and FA particles appears to affect the required amount of aqueous Na_2SiO_3 solution, the time-dependence of the flow value, and the compressive strength.

In the future, more detailed investigations are needed concerning the influences of the Na_2SiO_3 concentration and the addition ratio of anhydrous citric acid on the compressive strength, the time-dependence of the flow value, and setting properties of geopolymer mortar. It is also necessary to clarify the reaction mechanisms of FA and FA7 with aqueous Na_2SiO_3 and with anhydrous citric acid.

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