

Fundamental Study on Techniques for Improving Activity of Fly Ash

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Abstract: The large quantities of coal ash produced by the growing number of coal-fired power plants in Japan incorporate an airborne component known as fly ash (FA). The most promising proposal for effectively turning FA into a useful resource is to employ it as an additive to be mixed with concrete. In this study, with the goal of achieving reliable improvements in FA activity using methods proposed in earlier study, we conduct a series of experiments to assess methods for improving FA activity, focusing in particular on the quantities of $\text{Ca}(\text{OH})_2$ and $\text{CaSO}_4(2\text{H}_2\text{O})$ additives and on their relationship with NaOH. Our results include the following findings. (1) Adding a combination of NaOH and $\text{Ca}(\text{OH})_2$ allows the findings of previous studies to be reproduced and yields a significant improvement in FA activity. (2) When the NaOH concentration lies in the range 0.025 - 0.100 mol, any quantity of the additives yields reliable and favorable improvements in FA activity. (3) Differences between cement production lots exhibit minimal impact on the improvement of FA activity. (4) The combination of NaOH and $\text{Ca}(\text{OH})_2$ yields reliable and reproducible improvements in FA activity.

Keywords: Fly ash, mixing agents, activity, fine powders, additives, improvement technique

1. INTRODUCTION

The increasing number of coal-fired power plants in Japan has led to consistent growth in the production of coal ash, with volumes in recent years exceeding 1 million tons per year [1]. The most promising proposal for effectively turning this massive volume of coal ash into a useful resource is to employ it as an additive to be mixed with concrete. Among the various constituents of coal ash, the airborne component known as fly ash (FA) consists of microscopic spherical particles and exhibits pozzolanic reaction, and for this reason many studies have been conducted to date on the effective use of FA as a mixing agent for concrete. However, FA has scant real-world track record of use as a concrete mixing agent, and comprises only 3.5% of all coal ash emitted in Japan (based on data from fiscal year 2006 [2]). The reasons for this include (a) degradations in initial strength due to the sluggishness and pozzolanic reactivity of FA, and (b) wide variations in quality [3] due to differences in sintering methods and types of coal.

In an effort to expand the effective use of FA, the authors have studied techniques for increasing the activity of FA to allow its use as a substitute for cement. One result of this study, illustrated in Figure 1, was the demonstration that a 25% substitution of cement for FA powder, finely crushed with an NaOH additive, yields a material with compressive strength equivalent to that of ordinary Portland cement [4]-[6]. However, in these studies it was found that the improvement in FA activity was unreliable and sometimes not reproducible depending on the curing conditions – specifically, on the concentration of OH in the curing solution – and that the addition of $\text{Ca}(\text{OH})_2$ from the earliest stages of the fabrication process was crucial to obtaining reliable improvements in FA activity (see Figure 2). Additionally, further study was needed to quantify the impact of SO_4 [7].

In this study, with the goal of achieving reliable improvement in the activity of FA, we experimentally assess techniques for improving FA activity, focusing in particular on the quantities of $\text{Ca}(\text{OH})_2$ and $\text{CaSO}_4(2\text{H}_2\text{O})$ additives and on their relationship with NaOH.

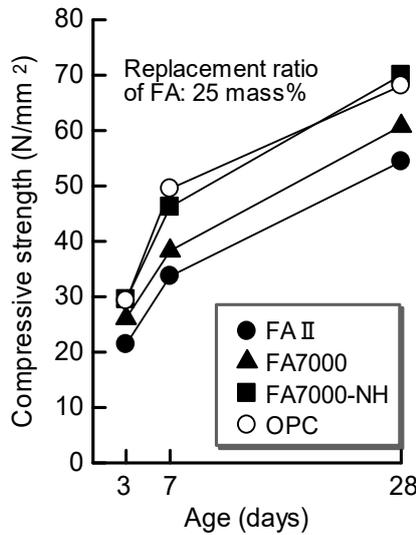
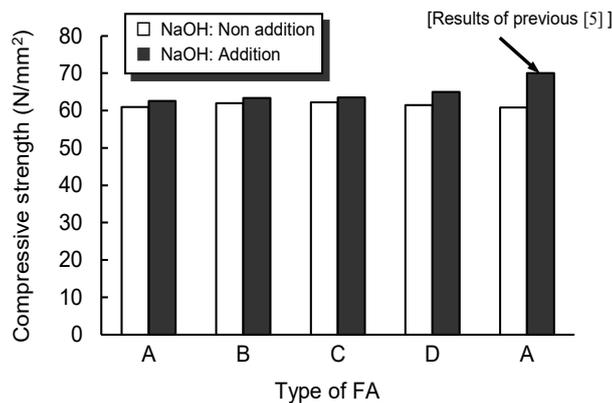
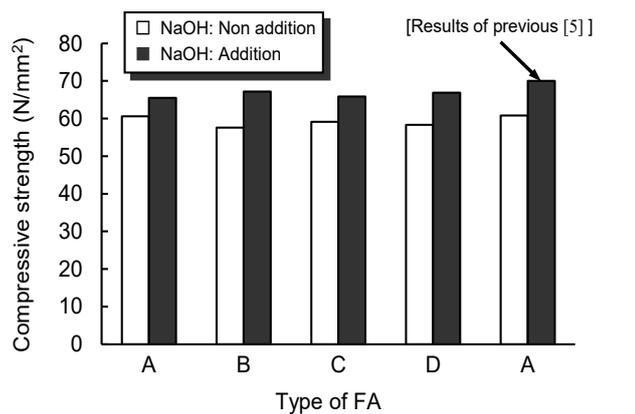


Figure 1. Techniques for improving activity of FA according to fine powder and NaOH [5]



(a) Curing water of tap water



(b) Curing water of 0.1mol aqueous solution of $\text{Ca}(\text{OH})_2$

Figure 2. Impact of curing conditions and type of FA on FA activity [7]

2. IMPACT OF COMBINATION OF NaOH AND $\text{Ca}(\text{OH})_2$ ON FA ACTIVITY (EXP. 1)

2.1 Overview of Experiment

Table 1 details the experimental conditions. $\text{Ca}(\text{OH})_2$ was diluted by mixing with a water-paste mixture. NaOH was dissolved in a water-paste mixture at a concentration of 0.1 mol and added.

Table 1. Experimental conditions (Exp. 1)

Type of FA*)	Addition Agent		Mark of test piece
	NaOH	Ca(OH) ₂ (W×mass%)	
-	-	-	OPC
FA (grade II)			FA
			F7
FA (grade II) : Fine powder to 7000 blaine	0.1 Molaqueous solution		F7-NH
		0.05	F7-NH-005C
		0.10	F7-NH-010C
		0.15	F7-NH-015C
	-	0.05	F7-005C
		0.10	F7-010C
		0.15	F7-015C
		0.20	F7-020C

*) Replacement ratio : 25 mass%

2.2 Mortar Materials and Experimental Procedures

Table 2 details the mortar materials used. Experimental procedures followed Appendix 2 of JIS A 6201, "Experimental methods for characterizing flow value ratios and activity indices for fly-ash mortars." The improvement in FA activity was characterized by the activity index for compressive strength at an age of 28 days.

Table 2. Mortar materials (Exp. 1-5)

Materials	Type
Cement	OPC (Type A, Type B)
FA	JIS (grade II) JIS (grade II): Fine powder to 7000 blaine
Sand	Standard sand
Water	Tap water
Addition agent	NaOH, Ca(OH) ₂ , CaSO ₄ (2H ₂ O)

2.3 Experimental Results and Discussion

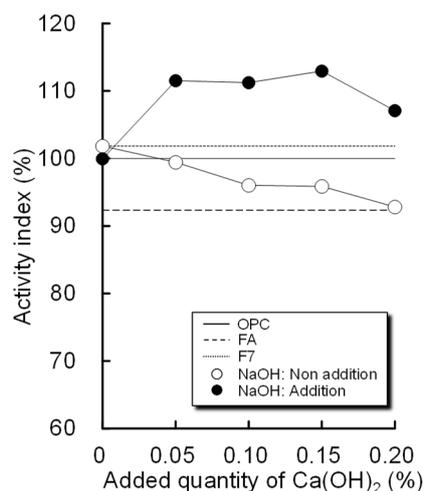


Figure 3. Impact of combination of NaOH and Ca(OH)₂ on FA activity (Exp. 1)

Figure 3 illustrates the impact of the combination of NaOH and Ca(OH)₂ on FA activity. As the figure indicates, the samples with an addition of Ca(OH)₂ but without an addition of NaOH exhibited no improvement in activity whatsoever; in fact, increasing the quantity of Ca(OH)₂ caused the activity index to decrease. In contrast, adding NaOH in addition to Ca(OH)₂ resulted in significant improvements in activity, with samples F7-NH-005C, F7-NH-010C, and F7-NH-015C all showing improvements in activity index of around 10% or more. Sample F7-NH-015C exhibited a particularly large improvement of more than 15%. In view of the experimental results reported previously [7], this

observation may be attributed to the impact of adding $\text{Ca}(\text{OH})_2$ from the early stages of the fabrication process, and suggests that the presence of $\text{Ca}(\text{OH})_2$ stimulates the improvement in activity arising from the addition of NaOH . Consequently, we may expect that by choosing the quantity of $\text{Ca}(\text{OH})_2$ added to be within 0.05 – 0.15% and by adding NaOH together with $\text{Ca}(\text{OH})_2$, it is possible to achieve significant and reliable improvements in FA activity – results which not only successfully reproduce the results of earlier study [5] but actually surpass those results. Note that, when the quantity of $\text{Ca}(\text{OH})_2$ added exceeds 0.15%, the activity index begins to decline. The reasons for this are not clear, but one possible factor is the degree to which $\text{Ca}(\text{OH})_2$ dissolves in the mixture.

3. IMPACT OF COMBINATION OF NaOH AND $\text{CaSO}_4(2\text{H}_2\text{O})$ ON FA ACTIVITY (EXP. 2)

3.1 Overview of Experiment

Table 3 details the experimental conditions. $\text{CaSO}_4(2\text{H}_2\text{O})$ was dissolved in FA. NaOH was dissolved in a water-paste mixture at a concentration of 0.1 mol and added.

Table 3. Experimental conditions (Exp.2)

Type of FA ^{*)}	Addition Agent		Mark of test piece
	NaOH	$\text{CaSO}_4(2\text{H}_2\text{O})$ (FA×mass%)	
-	-	-	OPC
FA (grade II)	-	-	FA
FA (grade II) : Fine powder to 7000 blaine	0.1 Molaqueous solution	-	F7
		-	F7-NH
		16	F7-NH-16S
		20	F7-NH-20S
		24	F7-NH-24S
	-	28	F7-NH-28S
		16	F7-16S
		20	F7-20S
		24	F7-24S
		28	F7-28S

*) Replacement ratio : 25 mass%

3.2 Mortar Materials and Experimental Procedures

The mortar materials used and the experimental procedures were similar to those of Experiment 1.

3.3 Experimental Results and Discussion

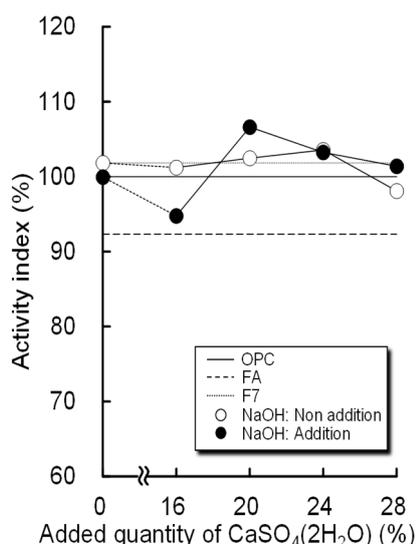


Figure 4. Impact of combination of NaOH and $\text{CaSO}_4(2\text{H}_2\text{O})$ on FA activity (Exp. 2)

Figure 4 illustrates the impact of the combination of NaOH and $\text{CaSO}_4(2\text{H}_2\text{O})$ on FA activity. As the figures indicate, the samples to which $\text{CaSO}_4(2\text{H}_2\text{O})$ was added but to which NaOH was not added exhibited essentially no improvement in activity; this finding is directly analogous to the results

obtained in Experiment 1 for samples to which only $\text{Ca}(\text{OH})_2$ was added. In contrast, adding the combination of NaOH and $\text{CaSO}_4(2\text{H}_2\text{O})$ in addition to $\text{Ca}(\text{OH})_2$ yielded no uniform change in activity, except in the case of sample F7-NH-20S, which exhibited an improvement in activity index of around 6%. Consequently, we conclude that the addition of $\text{CaSO}_4(2\text{H}_2\text{O})$ from the earliest stages of the fabrication process may play some small role – depending on the quantity added – in stimulating the improvement in activity arising from the addition of NaOH . However, the effect is small compared to the improvement in activity index that may be achieved from the combination of NaOH and $\text{Ca}(\text{OH})_2$ (as shown in Figure 3), and this fact – together with the large variation in activity observed for different quantities of $\text{CaSO}_4(2\text{H}_2\text{O})$ added – suggests that this is unlikely to be an effective technique for improving activity.

4. IMPACT OF COMBINATION OF NaOH AND $\text{Ca}(\text{OH})_2$ WITH $\text{CaSO}_4(2\text{H}_2\text{O})$ ON FA ACTIVITY (EXP. 3)

4.1 Overview of Experiment

Table 4 details the experimental conditions. $\text{Ca}(\text{OH})_2$ and $\text{CaSO}_4(2\text{H}_2\text{O})$ were each diluted with paste and FA. NaOH was dissolved in a water-paste mixture at a concentration of 0.1 mol and added.

4.2 Mortar Materials and Experimental Procedures

The mortar materials used and the experimental procedures were similar to those of Experiment 1.

4.3 Experimental Results and Discussion

Figure 5 illustrates the impact of the combination of NaOH and $\text{Ca}(\text{OH})_2$ with $\text{CaSO}_4(2\text{H}_2\text{O})$ on FA activity. As the figure indicates, the combination of NaOH and $\text{Ca}(\text{OH})_2$ with $\text{CaSO}_4(2\text{H}_2\text{O})$ exhibited no uniform trends, with each case giving rise to different behavior. Similar observations were noted in previous study [8]. However, samples F7-NH-005C-28S, F7-NH-010C-20S, F7-NH-015C-16S, and F7-NH-015C-20S exhibited significant improvement in activity, with activity indices improved by 10% or more. This demonstrates that, while the combination of NaOH and $\text{CaSO}_4(2\text{H}_2\text{O})$ alone yields minimal improvement in activity (cf. Figure 4), the further addition of $\text{Ca}(\text{OH})_2$ leads to favorable improvements in activity. This suggests that significant improvements in activity may be expected depending on the way in which $\text{CaSO}_4(2\text{H}_2\text{O})$ is combined with NaOH and $\text{Ca}(\text{OH})_2$. However, in some cases, depending on the quantities of $\text{Ca}(\text{OH})_2$ and $\text{CaSO}_4(2\text{H}_2\text{O})$ added – that is, on the way in which the substances are combined – the activity index may also decrease significantly. These findings, which are similar to the results observed in Experiment 2, suggest that this particular combination is unlikely to prove an effective technique for improving FA activity.

Thus, in view of the results of Experiments 1 and 2 and taking into account practical considerations, we conclude that the combination of NaOH and $\text{Ca}(\text{OH})_2$ – which reliably yields favorable improvements in activity – is the most effective technique for improving the activity of FA.

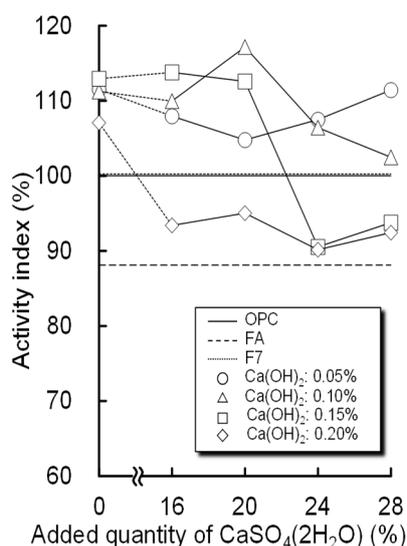


Figure 5. Impact of combination of NaOH and $\text{Ca}(\text{OH})_2$ with $\text{CaSO}_4(2\text{H}_2\text{O})$ on FA activity (Exp.3)

Table 4. Experimental conditions (Exp.3)

Type of FA*)	Addition Agent		Mark of test piece		
	NaOH	Ca(OH) ₂ (W×mass%) CaSO ₄ (2H ₂ O) (FA×mass%)			
-	-	-	OPC		
FA(grade II)			FA		
FA (grade II) : Fine powder to 7000 blaine			0.1 Molaqueous solution	0.05	F7
					F7-NH
	F7-NH-005C-16S				
	F7-NH-005C-20S				
	F7-NH-005C-24S				
	F7-NH-005C-28S				
	0.10	F7-NH-010C-16S			
		F7-NH-010C-20S			
		F7-NH-010C-24S			
		F7-NH-010C-28S			
	0.15	F7-NH-015C-16S			
		F7-NH-015C-20S			
		F7-NH-015C-24S			
		F7-NH-015C-28S			
	0.20	F7-NH-020C-16S			
		F7-NH-020C-20S			
F7-NH-020C-24S					
F7-NH-020C-28S					

*) Replacement ratio : 25 mass%

5. IMPACT OF NaOH CONCENTRATION ON FA ACTIVITY (EXP. 4)

5.1 Overview of Experiment

Table 5 details the experimental conditions, while Table 6 details additional experimental conditions. Ca(OH)₂ was diluted with a water-paste mixture, while NaOH was dissolved in a water-paste mixture and added.

Table 5. Experimental conditions (Exp.4)

Type of FA*)	Addition Agent		Mark of test piece		
	Aqueous solution of NaOH	CaSO ₄ (2H ₂ O) (FA×mass%)			
-	-	-	OPC		
FA (grade II)			FA		
FA (grade II) : Fine powder to 7000 blaine			0.1 mol	0.15	F7
			0.4 mol		F7-01NH-015C
	0.7 mol	F7-04NH-015C			
	1.0 mol	F7-07NH-015C			
		F7-10NH-015C			

*) Replacement ratio : 25 mass%

Table 6. Additional experimental conditions (Exp. 4)

Type of FA*)	Addition Agent		Mark of test piece		
	Aqueous solution of NaOH	CaSO ₄ (2H ₂ O) (FA×mass%)			
-	-	-	OPC		
FA (grade II)			FA		
FA (grade II) : Fine powder to 7000 blaine			0.025 mol	0.15	F7
			0.050 mol		F7-0025NH-015C
	0.075 mol	F7-0050NH-015C			
	0.100 mol	F7-0075NH-015C			
		F7-0100NH-015C			

*) Replacement ratio : 25 mass%

5.2 Mortar Materials and Experimental Procedures

The mortar materials and the experimental procedures were similar to those of Experiment 1. The improvement in FA activity was characterized by the compressive strength at a material age of 28 days.

5.3 Experimental Results and Discussion

Figure 6 illustrates the impact of NaOH concentration on FA activity. As indicated in the figure, when the NaOH concentration is in the range 0.1 – 1.0 mol, the compressive strength decreases significantly as the concentration increases, falling at 1.0 mol to just half of its value at 0.1 mol. On the other hand, as illustrated in Figure 7, under alternative experimental conditions with the NaOH concentration between 0.025 – 0.1 mol, the compressive strength remains essentially unchanged, indicating a favorable result for the improvement in FA activity.

Thus, we conclude that the optimal range for the NaOH concentration is 0.025 – 0.1 mol. Note that, although these results might seem to indicate that the NaOH concentration has minimal impact on FA activity improvement, this impact has also been investigated in previous study [7], as indicated in Figure 2.

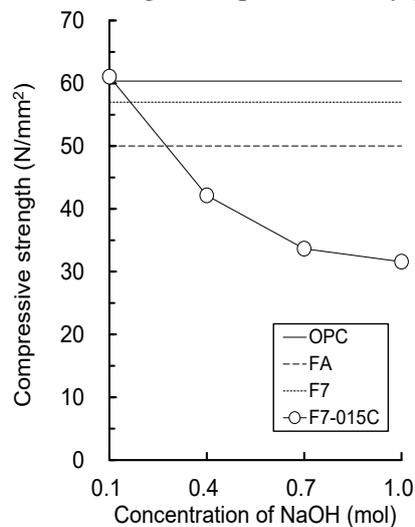


Figure 6. Impact of NaOH concentration on FA activity – No.1 (Exp. 4)

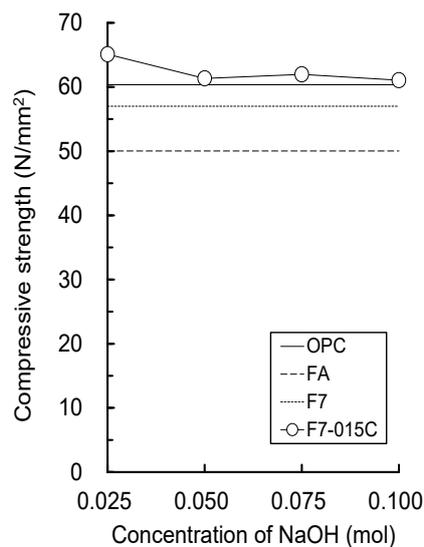


Figure 7. Impact of NaOH concentration on FA activity – No.2 (Exp. 4)

6. IMPACT OF DIFFERENT CEMENT PRODUCTION LOTS ON FA ACTIVITY (EXP. 5)

6.1 Overview of Experiment

Table 7 details the experimental conditions. $\text{Ca}(\text{OH})_2$ was diluted with a water-paste mixture. NaOH was dissolved in a water-paste mixture at a concentration of 0.1 mol and added.

Table 7. Experimental conditions (Exp. 5)

Type of FA ^{*)}	Production lot of OPC	Addition Agent		Mark of test piece
		NaOH	Ca(OH) ₂ (W×mass%)	
-	Type A Type B Type C	-	-	OPC
FA(grade II)				FA
FA (grade II) : Fine powder to 7000 blaine		0.1 Molaqueous solution	0.15	F7-NH-015C

*) Replacement ratio : 25 mass%

6.2 Mortar Materials and Experimental Procedures

The mortar materials were similar to those of Experiment 1. However, in addition to the research cement A used in experiments 1–3, research cements B and C were also used. Experimental procedures were similar to those of Experiment 1. The improvement in FA activity was characterized by the compressive strength at a material age of 28 days.

6.3 Experimental Results and Discussion

Figure 8 illustrates the impact of different cement production lots on compressive strength. The figure demonstrates that, although different cement production lots exhibit differences in OPC compressive strength, a consistent improvement in activity is observed. This finding again suggests that the combination of NaOH and Ca(OH)₂ depicted in Figure 3, which exhibits reproducible results minimally affected by the differences in cement production lots, is the most effective technique for reliably improving FA activation.

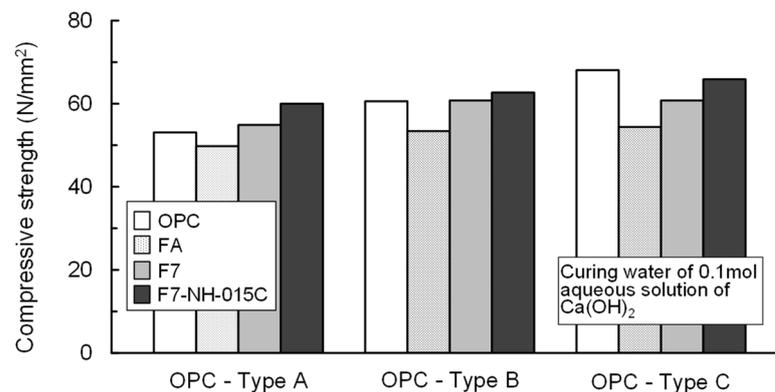


Figure 8. Impact of different cement production lots on compressive strength (Exp. 5)

7. CONCLUSIONS

In this study, with the goal of achieving reliable improvement in the activity of FA, we have experimentally assessed techniques for improving FA activity, focusing in particular on the quantities of Ca(OH)₂ and CaSO₄(2H₂O) additives and on their relationship with NaOH. The conclusions drawn from the series of experiments we performed are as follows.

1. By adding a combination of NaOH and Ca(OH)₂, we can reproduce the results of previous studies [5] and obtain significant improvements in activity.
2. The combination of NaOH and CaSO₄(2H₂O) can – depending on the quantity of CaSO₄(2H₂O) – yield relatively favorable results for the improvement of FA activity.
3. The combination of NaOH and Ca(OH)₂ together with CaSO₄(2H₂O) can – depending on the quantities of Ca(OH)₂ and CaSO₄(2H₂O) added – yield favorable results for the improvement of FA activity; however, the degree of activity improvement varies widely depending on the quantities added.
4. If the NaOH concentration is within the range 0.025 – 0.100 mol, an improvement in activity may be reliably achieved regardless of the quantity added.

5. Variations in cement production lots exhibit only minor impacts on the degree of activity improvement, but the combination of NaOH and Ca(OH)₂ yields reliable and reproducible improvements in activity.

In view of the above findings, we conclude that the combination of NaOH and Ca(OH)₂, which reliably yields favorable improvements in activity, is the most effective technique for achieving activity improvements in practical applications.

Topics for future study include elucidation of the mechanism of activity improvement, experiments in which the FA replacement ratio is varied as an experimental parameter, and the possibility of using FA materials as a replacement for cement.

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