KCI Concrete Journal
Vol. 13 No. 1 pp. 3-9, January 2001

Activating Temperature of Kaolin As a Cement Admixture

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(Received March 18, 2000, Revised February 2, 2001)

Abstract

This research concerns the effect of kaolin as material of cement admixture, which has the advantage of low price and high adaptability. Kaolin, a kind of soil, is well known as a raw material of pottery, which is widely scattered on the earth (especially in Korea). This research shows the method and process for activating kaolin to have the properties of a cement admixture through experiment. In the experiments, kaolin is baked in high temperature and then cooled suddenly to be activated. The temperature zone and time span, on which kaolin is activated, are examined. The research looks over the effect of the activated kaolin based on several criteria regarding to chemical and physical characteristics of general admixtures. The results of this research are as follows; kaolin start activation at the temperature above 500℃ and make ends of activation at the temperature below 950℃ and there was little effect by the change of duration. It is concluded that compressive strength can be increased by putting activated kaolin in the concrete and the activated kaolin is good for water resistance and anti-chemical, and that it is effective for protecting the leakage of hazardous article like Cl- and for protecting damage by an organic salt like acid. The activated kaolin of proper temperature and time is effective in compressive strength, salt resistance and acid resistance. The adaptability of activated kaolin as a cement admixture was shown through this research.

keywords : kaolin material, kaolin activation, activating temperature, pozzolan reaction

1. Introduction

Pozzolan materials such as silica fume and fly ash have been used to improve the quality of concrete and cement mortar. These materials as cement admixtures play important roles according to their chemical or physical characteristics. [1~3, 11~13]

However, silica fume is limited to be used in the cement mixture because of its high price.

Fly ash also has the limited adaptability since it is hardened slowly during curing time. The purpose of this research is to develop a new pozzolan material with kaolin, which satisfies the requirements of typical cement admixtures in the lower price and the higher adaptability.

Kaolin, a kind of soil, was well known as a raw material of pottery, which is widely scattered on the earth (especially in Korea).

This research shows the method and process for activating kaolin to have the properties of a cement admixture through an experiment. In the experiment, kaolin is baked in high temperature and then suddenly cooled to activated.

The temperature zone and time span, on which kaolin is activated, are examined. The research looks over the effects of the activated kaolin based on several criteria regarding to chemical and physical characteristics of general admixtures.

2. Experimental Procedures

2.1 Experimental Plan

A comprehensive experimental program was designed to investigate the process of activating kaolin. The experiment

Table 1 The factors and levels of kaolin activation experiment

Experiment steps		Experiment factor	Experiment level	Inspection method
Step 1. Experiment to investigate the activating temperature and time	Fundamental experiment to investigate the activating temperature	Baking temperature	Original kaolin 200, 400, 600, 800, 1000, 1200°C	• Compressive strength of mortar • Phenolphthalein reaction • Coagulation and setting
	Additional experiment for investigate the activating temperature	Baking temperature	1) 400, 450, 500, 550, 600°C 2) 800, 850, 900, 950, 1000°C	• Compressive strength of mortar
	Experiment to investigate the activating time	Baking temperature	1) Initial time period 20,40,60,80,100 min 2) Terminating time period - 40, 60, 80, 100min	• Compressive strength of mortar
Step 2. Experiment for examine the activating effect		Activation, non-activation	1) Non-activation 2) Activation: baking till 950°C	• Compressive strength of concrete • Anti-salt & anti-chemical

Table 2 The properties of materials

Materials		Properties
Cement	Portland cement	Blain value 3200cm^2/g, specific gravity 3.28, initial setting 234minute, final setting 5hour 47minute, autoclave explosion 0.09%, Compressive strength (3days, 7days, 28days) 202, 263, 361kg/cm^2
Kaolin	Activated kaolin Non-activated kaolin	Diameter< 5μm, specific gravity 2.55
Gravel	Crushed stone	FM(fineness modulus) 4.76, specific gravity 2.83
Sand	Sea sand	FM 2.71, specific gravity 2.61, Absorption 0.77%, Unit weight 1.654kg/m^3

Table 3 Mortar mix proportion

Experiment symbol	Cement (g)	Kaolin (g)	Sand (g)	Water (g)
Plain Mortar (P-M)	500	0	1500	325
Activated Kaolin Mortar (AKM)	450	50	1500	325
Non-activated Kaolin Mortar (NKM)	450	50	1500	325

Table 4 Concrete mix proportion

Experiment symbol	Cement (kg/m^3)	Kaolin (kg/m^3)	Sand (kg/m^3)	Gravel (kg/m^3)	Water (kg/m^3)
Plain Concrete (P-C)	340	0	798	906	187
Activated Kaolin Concrete (AKC)	306	34	795	903	187
Non-activated Kaolin Concrete (NKC)	306	34	795	903	187

was performed through of two steps. In the first step, the temperature zone and period in which kaolin can be activated, were extracted. In the second step, the effects of activation were examined by comparing the activated kaolin with the non-activated kaolin in condition of temperature and period beyond the adequate ranges.

The experiment factors and levels are shown in Table 1, and the experimental materials in Table 2, The mix proportion of the mortar in kaolin-activating effect experiment in Table 3, and the mixture of the concrete in Table 4.

2.2 Experiment Methods

2.2.1 Compressive Strength Experiment

This experiment was performed to examine the changes in the strength of concrete and mortar which kaolin is added to. Samples of concrete were taken out in 150×300mm cylinder mold and samples of mortar are taken out in 4×4×16 ㎝ mold. Then the sample molds cured wet conditions for 24hours, and was water curing continuously from the age of 1 day to 28 day in accordance with the regulations of KS F 2405 and KS L 5105.

2.2.2 Phenolphthalein reaction Experiment

This experiment was performed to find the pozzolanic reaction of kaolin in the saturated solution of $Ca(OH)_2$. If kaolin consumes $Ca(OH)_2$ wholly in saturated solution by a pozzolanic reaction, it won't show any change in color of the phenolphthalein solution. If kaolin does not react with $Ca(OH)_2$ in the saturated solution, its color changes to red. This experiment was useful to perceive the condition of activating kaolin.

2.2.3 Coagulation and Setting Experiment

This experiment was performed to examine whether the kaolin mass is soluble or not. Kaolin is kneaded with $Ca(OH)_2$ at the rate of 3 to 1, and then it is hardened to be a mass. If the kaolin mass causes a pozzolanic reaction, it will make a compound called Afwillite, which is in insoluble. If it was coagulated physically, it is soluble

2.2.4 Salt Resistance Experiment (Cl^-)

This experiment was performed to find the salt resistance effect of kaolin by measuring the infiltration degree of a chloride ion (Cl^-). A specimen of the mortar mixed with kaolin in 40×40×160mm mold is coated with up and down. After the specimen is soaked in chloride sodium (NaCl) solution for 7 days, it cut two parts. Then, 0.1% of fluorescein sodium solution is sprayed on six sections of the specimen. It is assumed that fluorescein territory is anti-salt area. The infiltration depth of a chloride ion is measured in each section.

2.2.5 Acid resistance Experiment (HCl)

This experiment was to find the acid resistance effect of kaolin by measuring the rate of gravity change. A specimen of mortar mixed with kaolin in 40×40×160mm mold is settled in 3% of chloride hydrogen (HCl) solution at 20℃ for 28 days. The erosion part of the specimen is eliminated with a brush. Then, the rate of gravity change is measured. It was assumed if the chemical erosion occurs, the gravity of the mortar will be reduced.

3. Results and Analysis

3.1 Tests of Temperature and Time for Activating Kaolin

3.1.1 Activating on Temperature Zone

To figure out the adequate temperature of activating kaolin, the experiments are performed in every 200℃ from the current temperature to 1,200℃, and the results are shown in Table 5.

There was no change in 400℃ and below. A response happens in 600℃, no change in above 1,000℃ again, the result of the compressive strength experiment is shown in Table 6 and Fig. 1. It was the same to the above result. The change of the strength didn't happen from a current temperature to 400℃, the strength increase happened beyond that temperature, and the same strength as first strength appeared in the temperature above 1,000℃.

Table 5 Phenolphthalein reaction at activating temperatures

Experiment symbol	Phenolphthalein reaction	Coagulation & Setting of experiment	Baking temperature (°C)
K00	(red)	Physical coagulation (soluble)	Original kaolin
K02	(red)	Physical coagulation (soluble)	200
K04	(red)	Physical coagulation (soluble)	400
K06	O (no color)	Chemical setting (insoluble)	600
K08	O (no color)	Chemical setting (insoluble)	800
K10	(red)	Physical coagulation (soluble)	1000
K12	(red)	Physical coagulation (soluble)	1200

Table 6 Compressive strength of mortar at activating temperatures (28 days)

Experiment symbol	Compressive strength (kgf/cm^2)	Baking temperature (°C)
K00	215	Original kaolin
K02	226	200
K04	234	400
K06	328	600
K08	352	800
K10	245	1000
K12	231	1200

Table 7 Compressive strength of mortar at initial activating temperatures (28 days)

Experiment symbol	Compressive strength (kgf/cm^2)	Baking temperature (°C)
K40	231	400
K45	236	450
K50	312	500
K55	321	550
K60	330	600

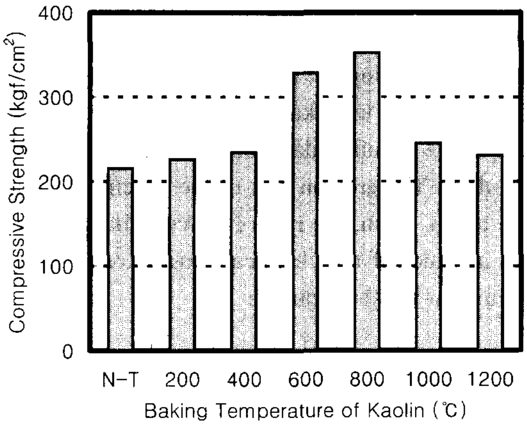

Fig.1 Compressive strength according to the mortar at activating temperatures

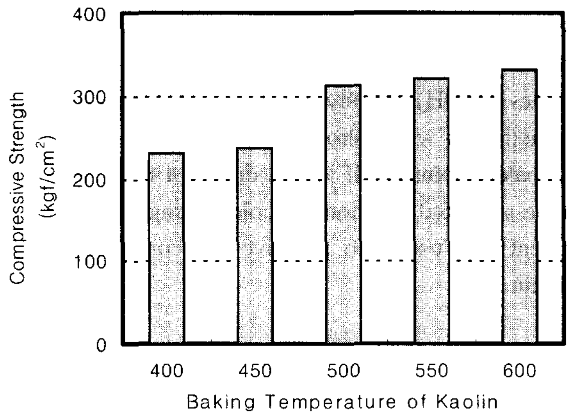

Fig. 2 Compressive strength according to the mortar at initial activating temperatures

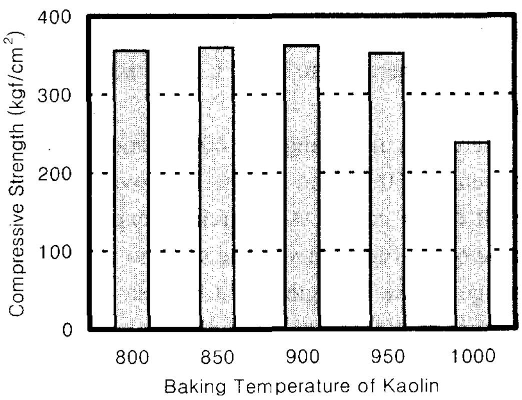

Fig. 3 Compressive strength according to the mortar at final activating temperatures

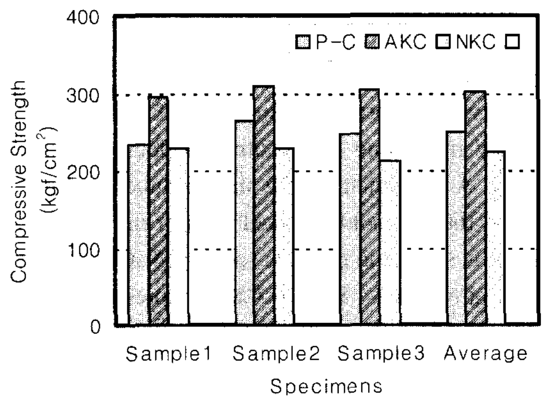

Fig. 4 Compressive strength according to the types of concrete (PC, AKC,NKC)

3.1.2 Activating Temperature

The experiment was to figure out the initial activating temperature of the mortar mixed with kaolin. The baking temperature is increased from 400℃ to 600℃ in every 50℃.

The compressive strength was not increased until 450℃. The activating response happened at 500℃. The results are shown in Table 7 and Fig. 2. Therefore, it was considered that kaolin begins to be activated at over 500℃.

To know the activation temperature the experiments in which baking temperature, has increased by 50℃at each step from 800℃ to 1,000℃, lead to the results in Table 8 and Fig. 3.

The activation has happened at 950℃ and the fit temperature increased, but there was no activation at 1,000℃ and above 900℃. It was found that the baking temperature to activate kaolin should be over 500℃ and below 950℃.

3.1.3 Activating Time

To find the adequate baking time required to activate kaolin, a series of experiments were performed by changing the baking time at 500℃ as the highest temperature. The results of the experiments are shown in Table 9. There is no significant variation in the baking time. However, the compressive strength began to decrease after 60 minutes. Therefore, it was expected that the baking time should not be beyond 60 minutes from the viewpoint of economy.

To find the time at which the activation is terminated, the experiments were performed by changing the activating time from 40 to 100 minutes with 20 minutes period at 950℃.

Table 8 Compressive strength of mortar at final activating temperatures period

Experiment symbol	Comp. strength (kgf/cm²)	Baking temp. (°C)
K80	356	800
K85	360	850
K90	362	900
K95	352	950
KT	238	1000

Table 9 Activating time at initial activating period

Experiment symbol	Comp. strength (kgf/cm^2)	Activating time (min.)	Baking temp. (°C)
K20ms	298	20	500
K40ms	308	40	500
K60ms	311	60	500
K80ms	305	80	500
K100ms	296	100	500

Table 10 Activation time at final activating period

Experiment symbol	Comp. strength (kgf/cm^2)	Activating time (min)	Baking temp. (°C)
K40mf	342	40	950
K60mf	349	60	950
K80mf	361	80	950

Table 11 Concrete strength with activated kaolin and nonactivated kaolin (28days)

Experiment symbol	Sample 1 (kgf/cm^2)	Sample 2 (kgf/cm^2)	Sample 3 (kgf/cm^2)	Average (kgf/cm^2)	Remarks
P-C	234	264	248	249	Plain concrete
AKC	294	309	303	302	Concrete mixed with activated kaolin
NKC	229	228	212	223	Concrete mixed with non-activated kaolin

Table 12 Infiltration depth of chloride for mortar mixed with kaolin

Experiment symbol	Sample 1 (mm)		Sample 2 (mm)		Sample 3 (mm)		Total average (mm)
Plain Mortar (P-M)	13.3	14.5	15.3	15.0	13.9	14.9	14.8 (100%)
	14.7		16.0		15.8		
	15.3		14.7		14.9		
	15.4		14.2		16.0		
	13.5		16.0		13.7		
	14.8		13.8		15.1		
Mortar mixed with Activated Kaolin (AKM)	10.2	9.3	8.9	9.8	10.2	9.4	9.5 (64.2%)
	9.4		10.7		8.6		
	8.5		8.5		8.5		
	8.1		10.1		9.3		
	10.0		9.9		10.7		
	9.6		10.7		9.1		
Mortar mixed with Non-activated Kaolin (NKM)	13.3	14.6	15.5	16.3	15.5	15.0	15.3 (103.4%)
	14.8		16.1		13.6		
	15.9		17.4		15.0		
	15.4		15.2		16.4		
	13.5		16.5		16.0		
	14.7		17.1		13.5		

Table 13 Antichemical effects of kaolin to weight reduction

Experiment symbol	Sample 1			Sample 2			Sample 3			Average
	Pre-weight (g)	Post-weight (g)	Reducing rate (%)	Pre-weight (g)	Post-weight (g)	Reducing rate (%)	Pre-weight (g)	Post-weight (g)	Reducing rate (%)	Reducing rate (%)
P-M	535.7	409.1	23.6	542.6	411.3	24.2	539.3	410.9	23.8	23.9 (100)
AKM	547.5	450.1	17.8	537.8	440.5	18.1	546.8	452.8	17.2	17.7 (74.1)
NKM	539.3	404.3	24.3	548.1	417.1	23.9	531.2	400.5	24.6	24.3 (101.7)

The results of the experiments are shown in Table 10.

It was figured out that there are no significant changes in each time period. Therefore, the advantages due to time are no longer valid to increase the compressive strength of the mortar mixed with the activated kaolin.

3.2 Activating Effects

3.2.1 Compressive Strength

The compressive strength of concrete mixed with the activated kaolin (AKC) was increased by 22.2% in comparison with that of plain concrete as shown in Table 11 and Fig. 4.

The compressive strength of the concrete mixed with non-activated kaolin (NKC) was decreased by 7.7% in comparison with that of plain concrete.

Based upon these results, it was verified that the activated kaolin increases the concrete strength and there was an obvious difference between the activated kaolin and non-activated kaolin. Therefore, the activated kaolin could significantly improve properties of concrete.

3.2.2 Salt Resistance Function

The chloride infiltration of the mortar mixed with the activated kaolin was decreased by 35.8% in comparison with that of plain concrete. However, the chloride infiltration of the mortar mixed with the non-activated kaolin was increased by 3.4% in comparison with that of plain concrete as shown in Table 12 and Fig. 5.

Based upon these results, it was confirmed that the activated kaolin is effective to protect the mortar from the infiltration of chloride materials by improving the water resistance.

3.2.3 Acid Resistance Function

The weight reduction rate of the mortar mixed with the activated kaolin was lower by 25.9% and Fig. 6.

However, the weight reduction rate of the mortar mixed with the non-activated kaolin (NKM) was higher by 1.7% than that of plain mortar. Based upon these results, the activated kaolin had a good acid resistance function and was effective for protecting concrete from the damage of organic salts like acid.

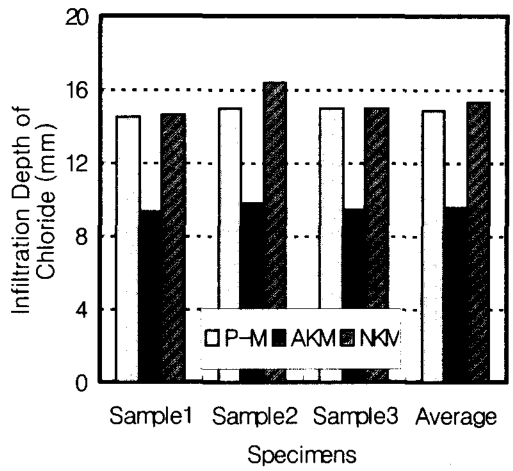

Fig. 5 Infiltration depth of chloride according to the types of mortar (PM, AKM, NKM)

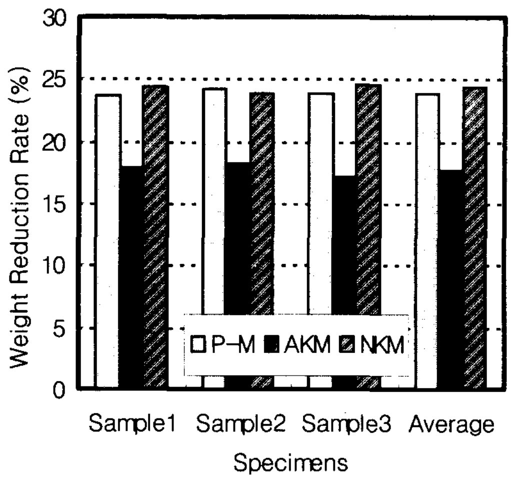

Fig. 6 Anti-chemical effects of the types of mortar to weight reduction

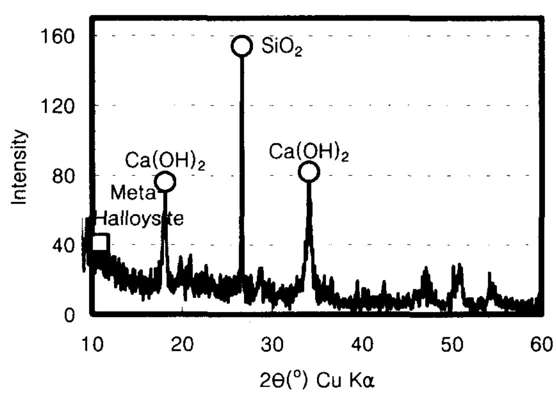

Fig. 7 XRD analysis of power (Activated Kaolin+ Quicklime)

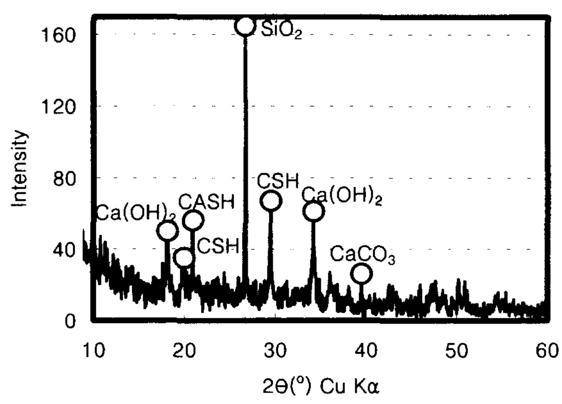

Fig. 8 XRD analysis of mortar (Activated Kaolin+ Water+Quicklime)

3.2.4 X-ray Diffraction Analysis of Kaolin Mortar

The followings were the results through XRD analysis on Kaolin Mortar.

Fig. 7 showed XRD analysis on the powder condition, while Fig. 8 showed XRD analysis on the sample powder extracted from mortar condition.

While Fig. 7 shows the activated components of kaolin, such as Metahalloysite, $Ca(OH)_2$, and SiO_2, Fig. 8 indicates the results of CSH gel and CASH gel after reaction of water and $Ca(OH)_2$

mortar was pozzolan reaction and stratlingite reaction, the mechanism of reactions is as follows.

1) $2SiO_2 + 3Ca(OH)_2 \rightarrow CaO \cdot 2SiO_2 \cdot 3H_2O$
(Afwillite–CSH gel)

2) $2Ca(OH)_2 + Al_2O_3 + SiO_2 + 6H_2O \rightarrow$
$2CaO \cdot Al_2O_3 \cdot SiO_2 \cdot 8H_2O$
(stratlingite – CASH gel)

4. Conclusions

The purpose of this research was to develop a new pozzolan material with kaolin that satisfies the requirements of typical cement admixtures in the lower price and the higher adaptability and the results of this research were as follows.

1) We got to know that kaolin start activation at the temperature above 500℃ and make ends of activation at the temperature below 950℃.

There was little change by the time, but we could conclude that we have some benefit when we set the time as long as we can.

2) The concrete that contains activated kaolin showed the increase of strength about 21.3 % more than plain concrete usually used.

The mortar that contains nonactive kaolin showed the decrease of strength about 10.4 %. By this fact, there is difference of strength in the activated kaolin and non-active kaolin, and you could raise compressive strength by put activated kaolin in the concrete.

3) The mortar that contains activated kaolin showed the decrease in invasion depth 35.8 % and the mortar that contains nonactive kaolin showed the increase in invasion depth 3.4 %. Activated kaolin was good for water resistance and it was effective for protecting the invasion of hazardous article like Cl^-

4) The mortar that contains activated kaolin, showed 25.9 % decrease, and the mortar that contains nonactive kaolin showed 1.7 % increase in the specimen decrease rate by HCl. Activated kaolin was good for antichemical and it was effective for protecting damage by an organic salt like acid. We were convinced of the adaptability of activated kaolin as a cement admixture through this research.

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