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THRESHOLD OH^- ION CONCENTRATION IN PORE SOLUTION OF MORTAR USING ALKALI REACTIVE AGGREGATES

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ABSTRACT

Predicting the future progress of ASR damage in concrete structures with a combination of the alkalinity of extracted pore solution and residual expansion of concrete cores taken from the structures. In this study, focusing on the alkali hydroxide in the pore solution, experiments were conducted to clarify the threshold OH^- ion concentration to initiate alkali-silica reaction (ASR). Mortar specimens were produced using Pyrex glass and 2 types of natural reactive aggregate and cured in sealed containers at 40°C and 100% relative humidity. Some ion concentrations in the pore solution and the expansion ratio of the specimens for each curing period were then measured. It was clarified that as conventionally proposed, approx. 250mmol/l was appropriate for values as threshold OH^- ion concentration and that caution was required when substituting glass cullet for concrete aggregate for recycling purposes since the threshold OH^- ion concentration of Pyrex glass was as low at 150mmol/l.

Key words: alkali concentration, ASR, expansion ratio, glass recycle, pore solution

1 INTRODUCTION

ASR is a chemical reaction[1] between reactive silica mineral and alkali hydroxide. Precautionary measures[2] are, therefore, advised, such as avoiding the use of reactive aggregate, and controlling the total alkalis in the concrete to under 3.0kg/m^3.

ASR develops with the pore solution containing alkali hydroxide. It is, therefore, important to understand the reaction by focusing on the composition of this pore solution.

Research on the liquid phase of concrete has started to determine the composite change of the liquid phase due to hydration of the cement in the initial hydration stage. Various other researches regarding ASR have also been conducted [3],[4],[5].

In the research[6] conducted to evaluate the influence of environmental conditions on ASR deterioration, we analyzed the pore solution inconcrete cores taken from existing structures. We also evaluated the deterioration level caused by ASR, and indicated the possibility of predicting the further deterioration of structures in the future that had prematurely deteriorated due to ASR.

In this research, we conducted experimental studies regarding the threshold OH^- ion concentration ([OH^-]); namely the threshold [OH^-] required to initiate ASR.

2 POSSIBILITY OF PREDICTING PROGRESS OF ASR DAMAGE IN CONCRETE STRUCTURES

Generally speaking, the progress of ASR deterioration in a concrete structure is determined by

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Table 1 Criteria for determining possibility of ASR progress in future

		OH^- ion concentration	
		≧ 250 (mmol/l)	≦ 250 (mmol/l)
Expansion in NBRI test (14days)	≧ 0.1%	A	B
	≦ 0.1%	C	D

Note:

A : high possibility because of high OH^- ion concentration and expansion

B : little possibility because of low OH^- ion concentration (※)

C : little possibility because of low expansion

D : little possibility because of low OH^- ion concentration and expansion

※ In the evaluation of OH^- ion concentration, variations from place to place must be taken into consideration.

the existence or otherwise of reactive components in the aggregates, and whether the OH^- ion concentration in the pore solution exceeds the threshold for ASR. In order to evaluate these criteria, we can use, for example the NBRI test[7], which is an accelerated test for determination of the existence of reactive components. There have been several proposals [8] for the threshold of the test period and the expansion rate of cores, and here we adopt 14 days and 0.01% for the expansion rate. As the threshold of OH^- ion concentration, we use the value of 250mmol/l as proposed [9].

Using the accelerated test and the thresholds described above, we have already proposed the possibility of predicting progress of ASR damage in concrete structures and can predict the progress of deterioration (Table 1).

In this study, focusing on the alkali hydroxide in the pore solution, experiments were conducted to clarify the threshold OH^- ion concentration to initiate alkali-silica reaction (ASR).

3 CHANGES IN ION CONCENTRATIONS AND EXPANSION OF MORTAR SPECIMENS OF DIFFERENT ALKALI LEVELS

Acceleration tests were conducted in sealed containers at 40°C and 100% relative humidity to measure the changes of pore solution concentration and of the expansion ratio in the specimens for each curing period.

3.1 Materials

Three types of aggregate (Pyrex glass, reactive crashed sand (pyroxene andesite) and reactive riversand) were used as reactive aggregate. Standard sand was used as non-reactive aggregate.

Sodium hydroxide was also used to adjust the alkali content of the mortar. Table2 shows the cement composition.

3.2 Mix-proportion and specimens

The mortar mix-proportions were determined according to the ASR test method for aggregate (mortar bar method) described in JIS A 5308, Appendiz 8. The water/cement ratio was 60%. Regarding the adjustment of alkali concentration in

Table 2 Composition of Cement (%)

	Ordinary Portland Cement	White Cement
SiO_2	20.90	23.13
Al_2O_3	4.96	4.42
Fe_2O_3	2.91	0.19
CaO	64.48	65.79
MgO	1.26	0.51
SO_3	2.00	2.78
Na_2O	0.48	0.36
K_2O	0.36	0.10
Na_2O eq.	0.61	0.35
Ig.loss	1.73	2.63

Table 3 Experimental Condition

【Materials】
- Reactive Aggregate : Pyrex Glass, Pyroxene Andesite, River Sand
- Non-reactive Aggregate : ISO standard sand

【Proportions】
- Proportion Ratio (Mass) : Cement : Sand : Water = 1: 2.25: 0.6
- Adjustment of Alkali Concentration

①High Concentration Sample: $[OH^-]$ = 800 (mmol/l) Equivalent
Ordinary Portland Cement + Sodium Hydroxide
(Approx. 750mmol/l of Mixing Water Concentration)

②Medium Concentration Sample: $[OH^-]$ = 500 (mmol/l) Equivalent
Composition the same as Above
(Approx. 250mmol/l of Mixing Water Concentration)

③Low Concentration Sample: $[OH^-]$ = 200 (mmol/l) Equivalent
White Cement (Approx. 200mmol/l of Mixing Water Concentration)

【Cure Condition】
- Temperature : 40℃, Relative Humidity : over 95%
  High, Medium and Low Concentration Samples
- Temperature : 20℃, Relative Humidity : 95% and Above
  Only for high Concentration Samples

【Experimental Ages】
- Pore Solution Extraction test : ϕ 48×100mm
  1 day, 7days, 14 days, 28 days, 56 days, 90 days, 180 days, 365 days
- Measurement of Mortar Bar Expansion Ratio : 40×40×160mm
  1 day, , 14 days, 28 days, 56 days, 90 days, 180 days, 365 days

pore solution, the amount of sodium hydroxide to be added was assumed from the $[OH^-]$ in pore solution measured previously with standard sand for the7 day-old sodium hydrate additive mortar. The addition was determined to get the concentration to be approx. 800mmol/l, 500mmol/l, and 200mmol/l.

Regarding those mortars, the goals of which $[OH^-]$ were 800mmol/l and 500mmol/l, sodium hydrate was added to ordinary Portland cement. Only white cement was applied to the 200mmol/l mortar.

Here, each mortar is referred to as high, medium, or low alkali concentration specimens. Preparation of all materials and specimens was conducted according to the previously mentioned [mortar bar method].

Specimens were demolded after 1day. Testing was carried out immediately.

The specimens were ϕ48mm × 100mm cylinders for pore solution extraction, and 40mm× 40mm × 160mm prisms for expansion ratio measurement. (Table 3)

3.3 Experiment method

Demolding the specimens 1day after placing, analysis of extracted pore solution were conducted for the cylindrical specimens. Using the prism

specimens, length was also measured as the initial value.

The cylindrical and prism specimens were then cured in sealed containers at 40°C and 100% relative humidity. The high alkali concentration specimens were also cured in sealed containers at 20°C and 100% relative humidity.

The experiment duration was 6 months or partially 12 months. During this period, the pore solution of two cylindrical specimens was extractedand analyzed at 7, 14, 28, 56, 90, 180 days (and 365 days). Simultaneously, sample expansion was measured to calculate the expansion ratio at each age. Table 3 summarizes the materials, conditions, and ages in the experiment.

Table 4 Result of Analysis of Pore Solution (Pyrex Glass)

Days	ph	Ion Concentration (mmol/l)			Σ +	Σ −	Expansion (%)
		Na^++K^+	Ca^{2+}	OH^-			
High Concentration Specimen (40℃,100%)							
1	13.8	681.6	1.8	787.5	685.2	787.5	0.00
14	13.8	515.5	1.7	523.0	519.0	523.0	0.26
28	13.7	360.2	1.9	371.5	364.0	371.5	0.48
90	13.0	137.7	0.9	153.0	139.6	153.0	0.53
180	13.0	151.2	1.6	150.0	154.5	150.0	0.55
365	13.0	136.3	0.3	104.0	136.8	104.0	0.57
Medium Concentration Specimen (40℃,100%)							
1	13.5	516.0	3.0	463.0	522.1	463.0	0.00
14	13.5	306.1	4.1	356.0	314.4	356.0	0.16
28	13.4	272.5	1.4	290.5	275.3	290.5	0.33
90	13.1	142.9	4.9	156.5	152.8	156.5	0.43
180	13.0	125.5	1.4	116.0	128.2	116.0	0.44
365	13.2	121.3	3.6	98.0	128.5	98.0	0.43
Low Concentration Specimen (40℃,100%)							
1	13.3	139.4	5.9	151.5	151.3	151.5	0.00
14	13.4	207.9	2.8	211.5	213.5	211.5	0.01
28	13.4	185.0	2.3	187.5	189.6	187.5	0.07
90	13.0	111.1	8.6	139.0	128.4	139.0	0.22
180	13.0	111.2	1.5	108.0	114.2	108.0	0.23
365	13.2	109.1	0.0	93.3	109.2	93.3	0.23
High Concentration Specimen (20℃.100%)							
1	13.8	656.5	2.5	771.0	661.5	787.5	0.00
14	13.9	708.0	2.9	785.0	713.8	785.0	0.04
28	13.9	705.9	1.4	750.0	708.7	750.0	0.10
90	13.9	627.5	1.2	672.5	629.9	672.5	0.36
180	13.5	537.0	1.9	439.5	540.8	439.5	0.62
365	13.5	409.3	0.1	305.0	409.5	305.0	0.98

4 EXPERIMENT RESULTS AND DISCUSSION

Tables 3 to 6 show the concentrations of negative ion (OH^-) and positive ions (Na^+, K^+, Ca^{2+}) in the pore solution of the specimens made from Pyrex glass, standard sand, pyroxene andesite, and reactive river sand,respectively. The difference between the concentration of total positive ions and the negative ion is approx. 10%. The the analysis precision is, therefore, deemed sufficient.

4.1 Standard sand and Pyrex glass mortars

Fig.1 shows the changes of the [OH^-] of Pyrex glass mortar and of the expansion ratio of the specimens. [OH^-] of high, medium and low alkali concentration mortar specimens just after demolding were 788mmol/l, 463mmol/l, and 152mmol/l, respectively.

When cured in sealed containers at 40°C and 100% relative humidity, the [OH^-] in pore solution of high and medium alkali concentration mortars dropped sharply to reach the steady state at the age of 90 days. It then stabilized at approx. 100mmol/l until 365 days. The specimens also expanded largely just after experiment commencement. At 90 days, their expansion almost reached the steady state and changed little until 365 days.

Regarding the low alkali concentration specimens, [OH^-] continued to increase until 14 days. Then, alkali consumption in response to ASR development occurred and caused specimens expansion accordingly. Similar to the high and medium alkali concentration specimens, however, it settled at 90 days. The initial low [OH^-] of 150mmol/l is believed to have caused the different behaviors in the low alkali concentration specimens compared to the high and medium alkali concentration. The [OH^-] in pore solution increased due to the reduction of free water in response to the cement hardening reaction according to the age and the dissolution of alkalis in Pyrex glass, etc., reaching 212mmol/l at 14 days. The concentration then dropped since the OH^- ion consumption exceeded due to ASR development. The expansion ratio of 0.23% at 365 days suggests that despite the use of low alkali white cement. There is a possibility

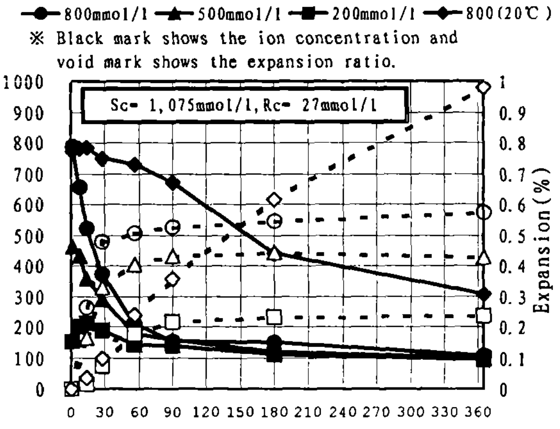

Fig.1 OH ion concentration in the pore solution (Pyrex glass)

Table5 Result of Analysis of Pore Solution

(Standard Sand)

Days	ph	Ion Concentration (mmol/l)			Σ +	Σ -	Expansion (%)
		Na^++K^+	Ca^{2+}	OH^-			
High Concentration Specimen (40℃,100%)							
1	14.0	664.6	3.6	748.0	671.8	748.0	0.00
14	14.0	809.6	2.6	828.0	814.8	828.0	0.01
28	14.1	829.1	2.7	806.5	834.5	806.5	0.01
90	14.0	693.5	3.5	779.0	700.5	779.0	0.02
180	13.8	709.3	2.4	708.0	714.0	708.0	0.01
High Concentration Specimen (20℃,100%)							
1	14.0	664.6	3.6	748.0	671.8	748.0	0.00
14	13.9	766.2	1.7	816.5	769.6	816.5	0.00
28	14.0	759.9	2.2	788.0	764.3	788.0	0.00
90	13.9	665.4	2.0	800.0	669.4	800.0	0.01
180	13.8	775.2	2.0	784.5	779.1	784.5	0.02

of harmful expansion due to ASR when glass materials including Pyrex are used.

All samples are also assumed to finalized ASR at

approx. 90 days. [OH$^-$] in pore solution at this stage was approx. 100mmol/l. The convergence values of expansion ratio of high, medium, and low alkali concentration were 0.57%, 0.43%, and 0.23%, respectively. Thus, the expansion ratios related to differences in their initial [OH$^-$].

Regarding the specimens cured in sealed containers at 20°C and 100% relative humidity (high alkali concentration indicated by a fine line in the figure), expansion due to ASR occurred at an early age. [OH$^-$] in its pore solution, however, hardly changed until 14 days due to the alkali dissolution from the Pyrex glass and free water reduction due to development of the cement hardening reaction. It dropped later due to consumption by ASR. At 365 days, the [OH$^-$] in the pore solution was still high at 305mmol/l. However, the expansion ratio exceeded that of the samples cured in sealed containers at 40°C and 100% relative humidity, and reached 0.98% and continued to increase.

The reason for a large, long lasting expansion ratio despite small [OH$^-$] consumption by ASR (small gel generation) is that the viscosity of ASR gel generated during the low temperature curing is relatively higher than that cured at a high temperature, thus generating a large, long lasting expansion pressure.

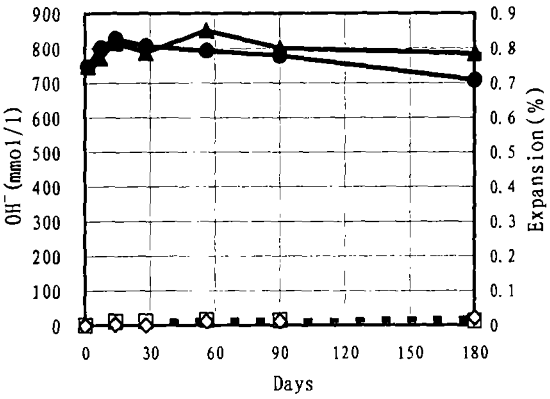

Fig.2 OH$^-$ ion concentration in the pore solution (Standard Sand)

Fig.2 shows the changes in the time sequence of the [OH$^-$] and expansion ratios of the specimens using standard sand. In both cases the concentration increased until 14 days. This is caused by water reduction due to the cement hardening reaction.

Although the specimens cured at 40°C indicated a slight concentration drop at 180 days, its [OH$^-$] was retained at the steady level. Also, no expansion was indicated during the test period.

4.2 Reactive Pyroxene Andesite and Reactive River sand mortars

Fig.3 and 4 show the time sequence changes of [OH$^-$] and the expansion ratios of the reactive pyroxene andesite and reactive river sand specimens.

Regarding the pyroxene andesite mortar specimens, just after demolding the [OH$^-$] of high, medium, and low alkali samples were 538mmol/l, 210mmol/l, and 85mmol/l, respectively. These figures were significantly lower than initially assumed. The reason for this is that OH$^-$ ions were consumed largely in approx. 1 day before demolding by the quick reaction occurred between the reactive components in the pyroxene andesite and the alkali hydroxide.

Regarding the high alkali specimens, [OH$^-$] in pore solution dropped largely in response to ASR development at 40°C, thereby increasing the

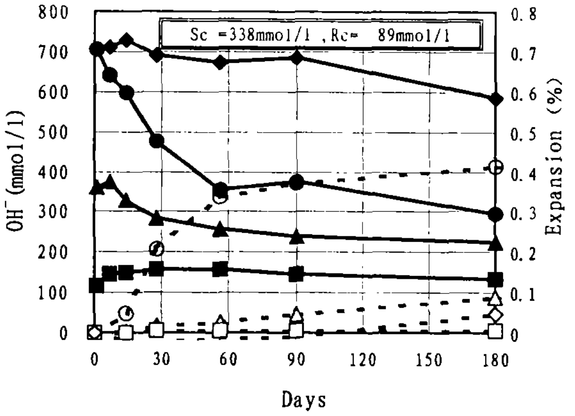

Fig.3 OH^- ion concentration in the pore solution (Reactive Pyroxene Andesite)

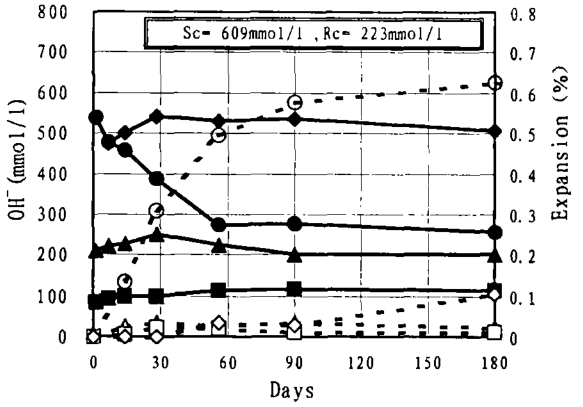

Fig.4 OH^- ion concentration in the pore solution (Reactive River Sand)

Table6 Result of Analysis of Pore Solution (Reactive Pyroxene Andesite)

Days	ph	Ion Concentration (mmol/l)			Σ +	Σ −	Expansion (%)
		$Na^+ + K^+$	Ca^{2+}	OH^-			
High Concentration Specimen (40℃,100%)							
1	14.0	435.0	2.1	538.0	439.3	538.0	0.00
14	13.9	366.2	1.5	456.0	369.2	456.0	0.13
28	14.0	344.8	1.1	387.5	347.0	387.5	0.31
90	13.5	255.3	0.3	277.0	255.9	277.0	0.58
180	13.2	241.6	1.2	256.0	244.0	256.0	0.63
Medium Concentration Specimen (40℃,100%)							
1	13.5	151.7	4.7	209.5	161.0	209.5	0.00
14	13.5	209.1	4.3	228.0	217.7	228.0	0.03
28	13.6	251.2	3.8	251.0	258.9	251.0	0.03
90	13.3	208.0	2.1	201.0	212.2	201.0	0.03
180	13.1	196.1	2.7	200.0	201.6	200.0	0.02
Low Concentration Specimen (40℃,100%)							
1	13.0	45.7	12.2	84.5	70.0	84.5	0.00
14	13.2	76.6	6.8	99.0	90.2	99.0	0.00
28	13.2	82.2	6.4	99.5	95.1	99.5	0.02
90	13.2	91.9	3.6	115.0	99.1	115.0	0.01
180	13.0	106.8	2.7	114.0	112.2	114.0	0.01
High Concentration Specimen (20℃,100%)							
1	14.0	435.0	2.1	538.0	439.3	538.0	0.00
14	13.8	396.4	2.6	499.5	401.6	499.5	0.00
28	14.1	462.5	2.6	540.0	467.7	540.0	0.00
90	13.8	394.0	1.4	536.0	396.8	536.0	0.03
180	13.6	443.7	2.3	505.0	448.2	505.0	0.11

Table 7 Result of Analysis of Pore Solution (Reactive River Sand)

Days	ph	Ion Concentration (mmol/l)			Σ +	Σ −	Expansion (%)
		$Na^+ + K^+$	Ca^{2+}	OH^-			
High Concentration Specimen (40℃,100%)							
1	13.9	594.9	2.9	704.5	600.8	704.5	0.00
14	13.8	492.8	2.9	595.5	498.6	595.5	0.05
28	13.6	435.6	1.2	474.5	438.1	474.5	0.21
90	13.4	359.3	3.8	375.6	366.9	375.6	0.37
180	13.3	327.7	1.2	295.0	330.1	295.0	0.41
Medium Concentration Specimen (40℃,100%)							
1	13.8	284.6	3.2	361.0	291.0	361.0	0.00
14	13.5	283.9	2.2	329.0	288.3	329.0	0.00
28	13.7	296.6	1.2	283.0	299.1	283.0	0.02
90	13.5	224.9	1.5	240.0	227.9	240.0	0.05
180	13.2	226.3	1.1	223.0	228.5	223.0	0.09
Low Concentration Specimen (40℃,100%)							
1	13.2	82.1	9.7	116.0	101.5	116.0	0.00
14	13.1	137.5	4.3	147.0	146.1	147.0	0.00
28	13.3	140.6	2.2	155.0	145.0	155.0	0.01
90	13.2	116.6	3.5	146.0	123.6	146.0	0.01
180	13.1	133.5	2.6	134.0	138.8	134.0	0.01
High Concentration Specimen (20℃,100%)							
1	13.9	594.9	2.9	704.5	600.8	704.5	0.00
14	13.9	617.1	3.6	729.5	624.3	729.5	-0.02
28	13.8	630.2	1.2	691.0	632.6	691.0	-0.02
90	13.7	590.2	4.3	683.6	598.8	683.6	-0.01
180	13.7	521.3	1.3	585.0	523.9	585.0	0.05

expansion ratio of the specimens.

After 56 days the $[OH^-]$ in the pore solution also settled at approx. 250mmol/l. The final expansion ratio of the specimens was approx. 0.63%. Regarding the medium alkali concentration specimens, the initial $[OH^-]$ was low at 210mmol/l thereby indicating a tendency similar to the low alkali Pyrex glass specimens. It increased to 251mmol/l until 28 days and stabilized at 200mmol/l as of 180 days.

The sample, however, indicated no expansion.

Regarding the low alkali specimens, the initial

$[OH^-]$ was extremely low (85mmol/l). Therefore, the alkali concentration increased according to its age. It was, however, approx. 114mmol/l as of 180 days.

The sample indicated no expansion.

The mortar specimens formed from reactive river sand also indicated a tendency similar to the reactive pyroxene andesite (Fig.4). Regarding the medium alkali concentration specimens, especially, upon removal from the mold the $[OH^-]$ dropped gradually from 361mmol/l in a manner similar to the high alkali concentration specimens. ASR is assumed to have completed at 180 days with approx. 220mmol/l.

The specimens expanded gradually from an early stage. Considering the approximately 0.1% of expansion ratio at 180 days, an initial $[OH^-]$ of 360mmol/l may cause harmful expansion.

Regarding the high alkali concentration specimens, its $[OH^-]$ continued to drop as of 182 days. The specimens also continued to expand.

4.3 Threshold alkali ion concentration in pore solution

ASR depends on the silica quantity in the pore solution[10]. Since one type of "catalyst" is required for silica to dissolve, OH^- ion works as the "catalyst" in a high alkali solution[11].

Silica generally dissolves faster when the pH exceeds 10. Regarding the composition of pore solution in concrete containing reactive aggregate, therefore, a minimum quantity of alkali hydrate possibly exists to initiate ASR.

Assuming that ASR expansion is almost completed before 180 days, the threshold $[OH^-]$ required for ASR initiation is concluded for each aggregate.

Regarding the Pyrex glass, the expansion ratio at 180 days exceeded 0.2% despite a low initial $[OH^-]$ of 152mmol/l for the low alkali concentration specimens.

Thus, in the case of Pyrex glass, the threshold $[OH^-]$ to initiate ASR is lower than 150mmol/l. In this case, also, white cement was used to produce the specimens. When substituting glass cullet for concrete aggregate for recycling purposes, however, the use of white cement requires caution despite its low alkali content.

When using a natural aggregate, reactive pyroxene andesite did not initiate ASR at the initial $[OH^-]$ of 210mmol/l (medium alkali concentration sample), nor provide harmful expansion.

When using reactive river sand, the expansion ratio was approx. 0.1% at 180 days despite an initial $[OH^-]$ of 360mmol/l (medium alkali concentration sample) and continued to expand. Thus, minimum harmful expansion is assumed to be slightly lower than 360mmol/l.

According to reference[9], the threshold $[OH^-]$ necessary to initiate ASR is approx. 250mmol/l. From the results of experiments using natural aggregate in this research, its value is deemed appropriate.

Since the above mentioned two types of natural aggregate were used in this experiment, it is, however, necessary to collect and examine more data in the future.

As long as the data acquired from this research is concerned, the threshold $[OH^-]$ required for ASR initiation accompanying harmful expansion varies depending on the reactive aggregate,including artificial glass.

5 SUMMARY

Mortar specimens made from 2 to 3 types of reactive aggregate were cured in sealed containers at 40°C and 100% relative humidity. The threshold alkali hydrate concentration required for ASR initiation was examined accordingly.

The data acquired from this research is summarized hereafter.

(1)Regarding the high alkali concentration specimens, ASR developed from the initial stage of aging and alkali ion concentration in the pore solution decreased. It indicated a steady value after 90 days and ASR completed.

(2)The alkali ion concentration of the low alkali concentration specimens indicated an increasing tendency at early stage, and dropped after a certain periods due to ASR development. It indicated a steady value after 90 days and with ASR completed.

(3) The threshold alkali hydrate concentration required for ASR initiation varies depending on the aggregate.

(4) Regarding Pyrex glass, the expansion ratio of the specimens at 180 days exceeded 0.2% despite the use of low alkali white cement. Caution is, therefore, required when substituting glass cullet for concrete aggregate for recycling purposes.

(5) Regarding natural aggregate, [OH^-] of 250mmol/l seems appropriate as proposed for the threshold [OH^-] to initiate ASR.

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