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Determining the pH of concrete pore water for alkali-wrapping considering alkali dissolution from aggregate

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Abstract

The alkali-silica reaction (ASR) is governed by the pH of pore water in concrete. Furthermore, for reliable testing for the quantitative estimation of long-term ASR expansion, experimental conditions such as alkali content and moisture supply must be controlled, with alkali-wrapping of a concrete prism having been proposed as a potential solution. To prevent inconsistent alkali-leaching during some accelerated tests in various conditions, concrete prisms are wrapped with a water-holding material containing an alkaline solution with the same pH as the pore water in the concrete. However, the method of determining the pH is rather complicated. To design the pH for the wrapping material, a detailed discussion for pH of pore water in concrete was presented.

The pH of the pore water results from alkalis from the cementitious materials. The pH is affected by various factors such as alkali binding by C-A-S-H with low Ca/(Si+Al), alkali absorption by the aggregate, alkali release from the aggregate, alkali-leaching during exposure (especially from small specimens or members), and alkali absorption in the neutralized state.

Based on the detailed mechanism of alkali balance between solids and solution, and its transfer, the fundamental concepts for determining the pH of the wrapping material is discussed, with a simple equation considering alkalis from Portland cement and the SCMs, and water-to-cement ratio is proposed.

Keywords: *alkali dissolution; pH; pore water composition; alkali-silica gel; alkali adsorption*

1. INTRODUCTION

A key factor in the alkali-silica reaction (ASR) in concrete is the pH of the pore water in concrete. Many factors affect the pH, having been studied intensively from several angles, as presented in the review of [1]. However, some uncertainty remains as to how to quantitatively determine or estimate the pore solution pH.

A new test protocol, the alkali-wrapped concrete prism test (AW-CPT), has been proposed for assessing the potential expansion of concrete through the ASR [2]. To prevent the loss of alkalis (alkali-leaching) from concrete test specimens during accelerated curing, the concrete test prism is wrapped with a water-holding material containing an alkali solution that mimics the pH of the concrete pore water. Thus, it is necessary to have a suitable determination method for the pH for the design of the AW-CPT.

In the present study, major factors affecting the pH of the pore solution are summarized and the background for the calculation method is discussed for proposing a method to calculate the pH for the AW-CPT. In this process, the effect of aggregate is considered, even though as a conclusion, such effects are not taken into account for the calculation of the pH for AW.

2. PRIMARY FACTORS AFFECTING PORE WATER PH

Before discussing the factors that affect pH, it is worth describing the pore water itself, the measurement method, and the difference between pH and alkali concentration. The pH of pore water or more simply, the exact nature of pore water containing alkalis is unclear. Pore water exists in the pores of concrete. However, the nature of this water varies depending on the size of the pore. In gel pores, water is significantly confined, differing greatly from that in larger pores [3]. At the microscopic scale, pore water is inhomogeneous. Thus, the "pH of pore water" contains some uncertainty.

As for the measurement method, assuming that one can measure or determine the "pH of pore water", each measurement method has limitations. Pore water squeezing has been assumed as the most reliable method; however, it has pressure level dependency [4]. Another popular method is washing alkalis from ground concrete via hot and cold water. As the ion concentration of pore water is in equilibrium with various ions, the dilution affects the equilibrium, and thus the accuracy of this method is doubtful.

In regard to the difference between pH and alkali concentration, there is an assumption in pH estimation by alkali washing that the only counter anion is OH^-. However, significant amounts of sulfate and silicate anions can be detected, especially when ASR is observed. Therefore, when alkalis are evaluated by pore water squeezing or alkali washing, the sulfate and silicate ion concentrations must be measured simultaneously and the counterbalance between cations and anions must be checked.

Given the uncertainty in the pH/alkalinity of pore water in concrete, there are three major factors affecting the pH of pore water as summarized in Figure 2.1 [1]: the alkalis from cement paste, effects from concrete aggregate, and interaction with the environment. The details of these three factors are explained in the following chapters.

The primary supply of alkalis in concrete comes from Portland cement, as will be described in Section 3. Some supplemental cementitious materials (SCMs) or admixtures such as water-reducers contribute or absorb alkalis. SCMs contain some alkalis; however, its pozzolanic reaction reduces alkalinity by absorbing alkalis.

The role of aggregate is also multi-fold. Aggregate can absorb alkalis through clays contained in the aggregate, while aggregate may also demonstrate a phenomenon known as "alkali-release" [5]. Through the alkali absorption by aggregate, the pH decreases. However, alkali-release from aggregate does not necessarily indicate an increase in pH. The details are discussed in Section 4. The ASR is an alkali consuming reaction and through the ASR, the pH decreases. Some researchers have suggested alkali-recycling through the exchange of alkalis in alkali-silica gel (ASR gel) with Ca from portlandite. However, is it evident that the generated C-S-H has lower C/S than the products from C_3S, absorbing alkalis. Further, alkalis in ASR gels in cracked aggregate are not easily replaced by Ca, with the time required for replacement exceeding a reasonable duration for this mechanism to be effective. Moreover, ASR gel changes its form to a rosette-like crystalline phase, possibly stabilizing alkalis in a water-insoluble form.

The interaction of concrete with the environment can modify the pH in several different manners. The supply of water such as rainfall may cause alkali-leaching, while alkali-ingress may result from deicing salts. When NaCl ingresses into concrete, Cl is trapped as Friedel's salt and OH^- is released instead, with the pH then can be increasing. However, the primary cause is the increase in alkali concentration but not the exchanging of anions. The alkali ion concentrations of pore water and seawater is in a similar range and direct contact with seawater may not increase pH. In splash zones, pH increase can be expected due to concentration of alkalis increases from drying. Additional pH changes result from carbonation. When cement paste is carbonated by atmospheric CO_2, alumino-silica gel is formed, which absorbs many alkalis [6] and results in a decreased pH. Alkali-leaching and the effect of carbonation are detailed in Section 5.
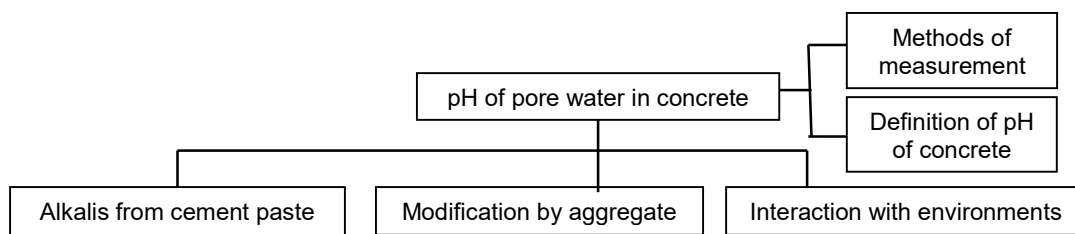
Figure 2.1: Factors affecting the pH of pore water in concrete [1]

3. ESTIMATION OF THE PH OF CEMENT PASTE

3.1 Portland cement

There are many ways to estimate the alkali concentration of the pore water in concrete, typically expressed in mol/L. For pure Portland cement paste, the alkali concentration of pore water is equivalent with pH. Based on experimental results, Helmuth [7] formulated Equation (1) to demonstrate the relationship between the alkali content of cement and the OH$^-$ concentration in pore water.

$$[OH^-] = 0.339 \times Na_2O_{eq,\,cem} /(W/C) + 0.02 \pm 0.06, \tag{1}$$

where [OH$^-$] is the hydroxide ion concentration in the pore water, $Na_2O_{eq,\,cem}$ is the equivalent alkali content of the cement, and W/C is the water-to-cement mass ratio.

Thomas et al. [8] presented an alternative formulation based on a review of pore water data. Kawabata and Yamada [9] calculated the hydroxide ion concentrations of cement with various SCMs, considering the interaction of alkali metal ions (Na, K) with C-S-H gel. As a result, a simple relationship between the alkali content of Portland cement and the hydroxide ion concentration for a water-to-cement ratio of 0.50 was determined.

A comparison of these relationships along with some experimental results is summarized in Figure 3.1 [2]. As is shown in the figure, there are large variations in the hydroxide ion concentration [10].
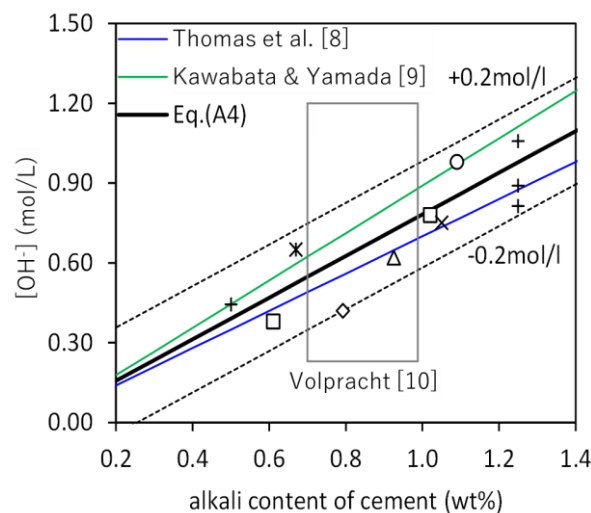

Figure 3.1: Alkali content of ordinary Portland cement and the hydroxide ion concentration in its pore water (modified from [2])

To calculate [OH$^-$] in the pore water from the equivalent alkali content of the cement, Kawabata and Yamada reviewed the results of prior studies [2], proposing a representative and simple relationship as

$$[OH^-] = 0.773 \times Na_2O_{eq,\,cem}. \tag{2}$$

This equation was developed for Portland cement paste with a water-to-cement ratio of 0.50.

Finally, Eq. (3) was proposed to apply this calculation to various mixture proportions, especially for calibrating the effect of water-to-cement ratios with reference to Eq. (1):

$$[OH^-] = 0.773 \times Na_2O_{eq,\,cem} \times 0.5/(W/C) = 0.386 \times Na_2O_{eq,\,cem} /(W/C). \tag{3}$$

Eq. (3) was established based on experiments on cement paste or mortar with water-to-cement ratios higher than 0.45. Therefore, care should be taken when applying this formulation to concrete with very low water-to-cement ratios (e.g., 0.35).

3.2 Concrete with SCMs

To extend Eq. (3) from cement paste to the estimation of the alkali concentration of concrete with SCMs, the equivalent alkali content of the total binder, which is calculated from the entire binder as well as the alkaline solution included for boosting, is used as shown in Eq. (4).

$$Na_2O_{eq,binder} = Na_2O_{eq,cem} \times C/B + Na_2O_{eq,SCM1} \times S1/B +$$
$$+ Na_2O_{eq,SCM2} \times S2/B + 61.98/(39.997 \times 2) \times NaOH/B \times 100, \tag{4}$$

where $Na_2O_{eq,binder}$ is the equivalent alkali content of the total binder (wt%), $Na_2O_{eq,cem}$ is the equivalent alkali content of the Portland cement (wt%), $Na_2O_{eq,scm1}$ is the equivalent alkali content of the first supplementary cementitious material (SCM1, wt%), $Na_2O_{eq,scm2}$ is the equivalent alkali content of the second supplementary cementitious material (SCM2, wt%), etc. B is the total unit content of the binder (kg/m^3), C is the unit content of the Portland cement, NaOH is the unit content of boosted NaOH (kg/m^3), $S1$ is the unit content of the SCM1 (kg/m^3), $S2$ is the unit content of the SCM2 (kg/m^3), etc..

Finally, replacing $Na_2O_{eq,cem}$ and C by $Na_2O_{eq,binder}$ and B, respectively, Eq. (5) is obtained:

$$[OH^-] = 0.386 \times Na_2O_{eq,binder} \times B / W. \tag{5}$$

Examples of results given by Eq. (5) are shown in Figure 3.2. In this estimation, alkali uptake by C(-A)-S-H with lower a Ca/(Al+Si) ratio is not considered due to a lack of experimental data. A specific example considering the effect of alkali uptake is shown in [2].

(a) Influence of water-binder ratio

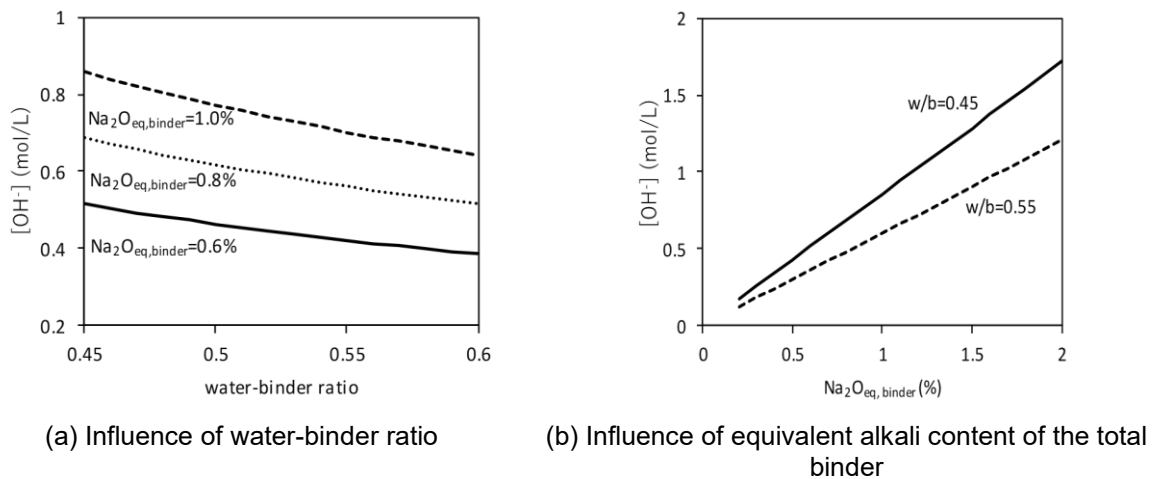(b) Influence of equivalent alkali content of the total binder

Figure 3.2: Examples of calculation with Eq. (5)

4. THE EFFECT ON PH OF ALKALI ABSORPTION BY AGGREGATE

4.1 Experimental procedure

As there is some ambiguity in the direct measurement of the pH of pore water, a series of experiments were carried out to evaluate the pH indirectly. If the pH of the pore water and wrapping material is equal and both the pore and wrapping solutions are in equilibrium, the pH of the wrapping material will be constant.

In the early hydration stage, some alkalis are not released from the cement and free water not bound by cement hydration remains in limited amounts in the cement. Therefore, cement paste having a 0.50 water-to-cement ratio (W/C) was cured in sealed condition at 20 ºC and 60 ºC for three months with the pore water composition measured after pore water squeezing. The alkali content in the Portland cement was 0.55 wt%. Then, concrete specimens containing 5.50 kg/m^3 of total alkalis by boosting and with a 0.50 water-to-cement ratio were fabricated and wrapped with a non-woven polypropylene cloth containing solutions with varying NaOH concentrations. These specimens were cured at 60 ºC for six months. Crushed pure limestone fine and coarse aggregates were used. Finally, the alkali concentration of the solution in the cloth and the [OH$^-$] in the pore water in the concrete were measured.

4.2 Cement paste

The measured [OH$^-$] in the pore water in the cement paste are shown in Figure 4.1 [11]. The alkali was boosted from 0.55 wt% to 1.72 wt%, which corresponds to concrete with 320 kg/m^3 of cement and 5.50 kg/m^3 of total alkalis. The [OH$^-$] was proportional to the alkali content in the cement; however, the [OH$^-$] was slightly lower at the higher temperature. Although the regression lines are not equal to Equation (5), the difference is limited and thus Equation (5) can be used to describe the pH of the pore water in the cement paste.
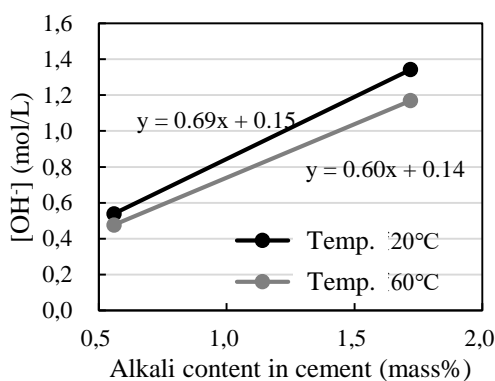

Figure 4.1: [OH$^-$] in the pore water in the cement paste for varying alkali content and curing temperature [11]

4.3 OH$^-$ concentration of pore water in concrete [11]

The original [OH$^-$] of the solution in the wrapping cloth was modified to be 0.00, 0.60, and 1.33 mol/L, and the [OH$^-$] after curing were measured. 1.33 mol/L corresponds to the [OH$^-$] of the cement paste for the cement having 1.72 wt% of alkali content. The results are shown in Figure 4.2 (left). In all cases, the Na$_{eq}$ concentrations were lower than the original [OH$^-$] in the wrapping cloth, even in the case of 1.33 mol/L. Significant alkali reductions can be observed.

One possible reason for this reduction in alkali concentration is alkali absorption by the aggregate. The limestone used is relatively pure with a calcite content of greater than 97%, yet it still contains some amount of clay minerals. From a separate experiment, the alkali absorption ability of aggregate was evaluated by adsorption equilibrium experiments. Crushed limestone with a size of 150 – 300 μm was dipped in NaOH solutions with a pH of 0.2, 0.5, or 1.0 for twenty-four hours and the [OH$^-$] reduction was measured. Then, the distribution ratio of alkalis between the solution and aggregate was calculated. Based on these results, in the concrete examined, the aggregates are estimated to have absorbed approximately 1.1 to 1.2 kg/m^3 of alkalis, corresponding to a reduction of 0.2 mol/L of the [OH$^-$] in this concrete. In Figure 4.2 (right), the measured [OH$^-$] and the [OH$^-$] calculated from the cement paste and aggregate alkali absorption results are shown against the original [OH$^-$]. The difference between the original [OH$^-$] in the wrapping cloth and that of the concrete pore water reduced and the difference between the measured and estimated [OH$^-$] is approximately 0.1 mol/L. This suggests a significant contribution from alkali absorption by the aggregate to the pH of the concrete pore water.

As explained in Section 5, it is known that the alkali absorbing ability of aggregate depends on the nature of aggregate used, and even if the alkali absorbing ability can be evaluated through chemical methods such as ASTM C289-07 (withdrawn in 2016 yet still popular in Japan), it is quite difficult to estimate the

long-term absorption ability due to the possible decomposition of clay minerals and alkali bearing minerals such as feldspar and feldspathoid.
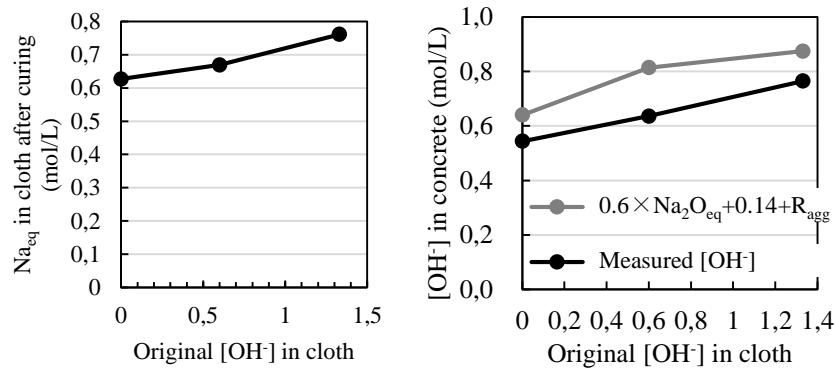

Figure 4.2: The effects of the original [OH$^-$] in the wrapping cloth solution on (left) the Na_{eq} concentration in the wrapping cloth after curing and (right) [OH$^-$] in the concrete pore water in concrete

5. ALKALI DISSOLUTION FROM AGGREGATE AND THE PH OF PORE WATER

As discussed in Section 2, if the pH is assumed as equal to the soluble alkali content, it is necessary to proceed carefully to avoid misleading phenomena. "Alkali release", or the dissolution of alkalis from aggregates, is a well-known phenomenon [5, 12]. Alkali feldspar, felspersoid, volcanic glass, clay minerals, and other alkali bearing minerals may contribute to this dissolution. The reaction of these minerals generates ASR gel in the aggregate. When the ASR gel penetrates the cement paste, a low C/(S+A) ratio C-A-S-H forms from the exchange of Ca in the paste and alkali in the ASR gel. By this reaction, alkali is consumed to some extent. Once the reaction sequence is clarified, it can be understood that the alkali release is not equal to the pH increase. Figure 5.1 presents the change in solution chemistry when a ground aggregate (andesite) is immersed in a solution of 1M-NaOH saturated with Ca(OH)$_2$ [13]. Aggregates A8 and A9 contained insignificant amounts of glass and clays, Aggregate B3 contained significant amounts of glass, and Aggregate C2 contained clays. Except for A9, significant amounts of K$_2$O were released. Simultaneously, a significant amount of silica was dissolved. Subsequently, the [OH$^-$] did not increase from 1 M; instead, there was a decrease. In summary, as alkali is released from the aggregate, the pH of the pore water decreases. When alkali alumino-silicate dissolves, alkalis can be released but simultaneously, anionic amorphous alumino-silicate is generated and this has strong alkali absorbing ability even in the condition of Portland saturated condition. More detailed thermodynamic quantification is required.
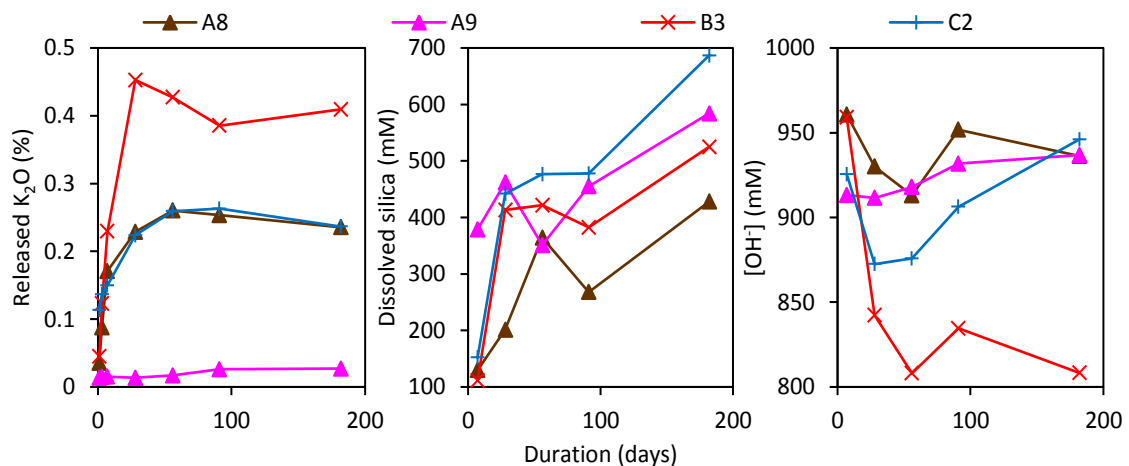

Figure 5.1: Solution chemistry when a ground aggregate (andesite) was immersed in a 1M-NaOH solution containing lime [13]

6.   EXAMPLE OF ALKALI-LEACHING IN FIELD CONDITIONS [14]

6.1   Outline of experiments

To investigate the environmental effects on ASR expansion, concrete blocks (40×40×60 cm) and concrete cylinders (φ10×L35 cm) were exposed to the environment at three sites in Japan [15]. The coldest site was Monbetsu in Hokkaido and the hottest was Okinawa with annual average temperatures of 7 and 28 ºC, respectively. The expansion behaviors and important factors affecting expansion will be discussed in another paper [15], however in the present study, alkali-leaching is introduced. Three concrete mixtures were examined: highly reactive andesite "N10" with Portland cement and 3.0 kg/m³ of total alkali content; the same andesite and alkali content with 15% replacement of cement with siliceous fly ash "FA"; and late reactive hornfels "EH100". The mixed ratio of N aggregate was 10% with 90% of non-reactive pure limestone aggregate while the EH aggregate used was 100% pure coarse aggregate. For each mixture, the fine aggregate was crushed non-reactive pure limestone. The water-to-cement ratio was 0.50 and the air content was maintained at 4.5±1.5 vol%.

Among these four samples, only N10 concrete expanded significantly and showed cracks of one or two mm width. The other mixtures showed no expansion or cracks.

6.2   Experimental alkali-leaching results

Cylinders were taken as samples from the outer and inner parts of the test specimens as shown in Figure 6.1 (left) [14]. The samples were crushed and their water-soluble alkalis measured. The measurement results are shown in Figure 6.1 (right). There are significant differences between the central and edge samples. Evidently, water-soluble alkalis are lost near the surface of the test specimens.

To better understand the alkali distribution, an electron microprobe analysis, EPMA [16] was carried out for a cross section of test samples for the N aggregate in Okinawa, N aggregate and FA in Okinawa, and EH aggregate in Okinawa and Monbetsu. The results by EPMA indicate the total alkali concentration at the point of analysis, which is 50 µm of a diameter. The neutralization depth was additionally validated by spraying phenolphthalein ethanol solution. In Figure 6.2, images of the results are shown.
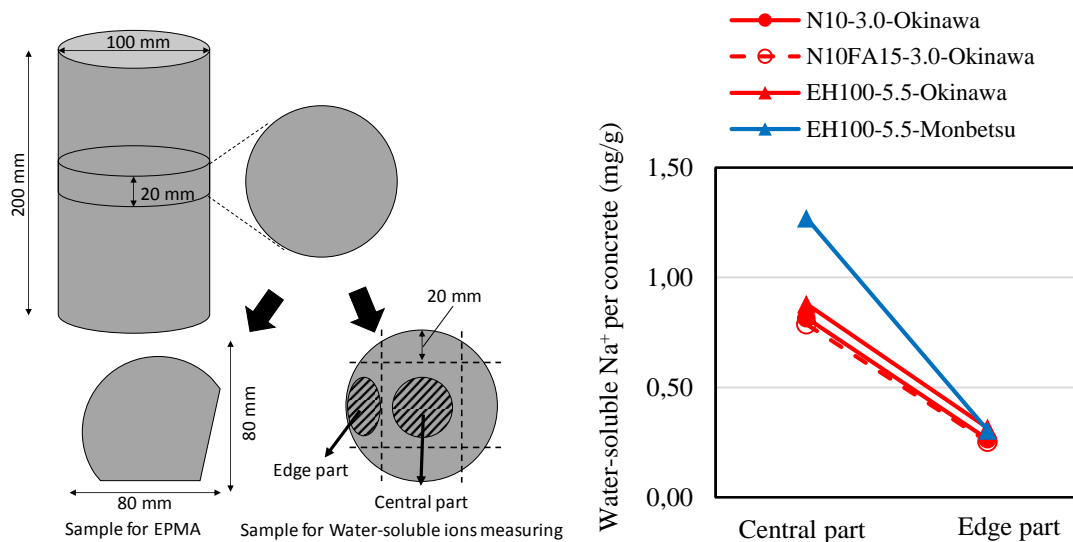

Figure 6.1: Water-soluble Na from a cylinder sample exposed in field [14],
(Left) The position of sampling, and (Right): The content of water-soluble Na

From Figure 6.2, the neutralization areas are limited, with their location independent of the concrete mixture and exposure locations. For the Na distribution, significant differences can be observed. Na was lost in the N10 aggregate in Okinawa, even from the center. However, by adding FA, Na leaching was limited, suggesting that FA causes the cement paste texture to become impermeable. Na leaching was more limited for the EH aggregate in Okinawa than for the N aggregate, suggesting that ASR expansion accelerates alkali-leaching. Comparing EH aggregate concrete in Okinawa and Monbetsu, it can be observed that in a colder climate, the movement of Na was more limited.
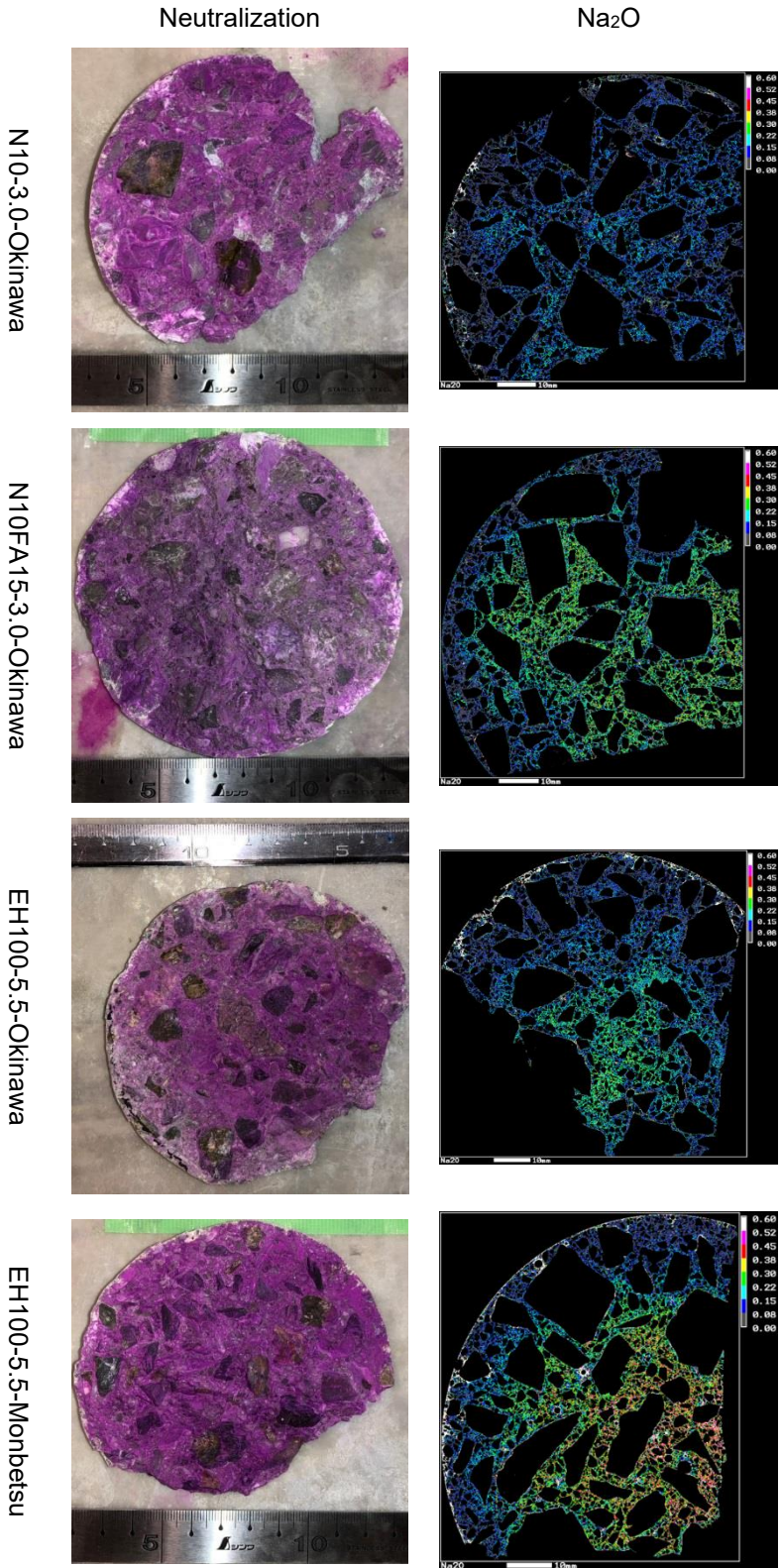
Figure 6.2: Neutralization area and Na distribution in the cross sections of exposed samples [14]

These results suggest that for the expansion test in the field, after only in three years, alkalis are lost at a depth of 3-4 cm. Therefore, sample sizes greater than several tens of centimeters are required for field exposure to test ASR expansion.

7. DISCUSSION ON THE DESIGN OF PH FOR AW

As the alkali concentration of the pore water in concrete changes with time and is dependent on the material used, it is quite difficult to quantify the concentration accurately. The following is a non-exhaustive list of factors that affect the alkali concentration:

1) Alkali concentration of cement and SCMs.
2) Degree of hydration of alkali bearing phases of cement and SCMs.
3) Alkali absorption by or release from aggregate depending on the nature of aggregate.
4) Alkali adsorption by C-A-S-H with a low Ca/(Si+Al) ratio.
5) Alkali consumption by alkali-silica gel.
6) Water suction from wrapped solution-holding material by water consumption due to cement hydration and ASR.
7) Age and curing condition depending on the above factors.
8) Alkali absorption in the carbonated state.

Therefore, it is necessary to make an assumption for determining the alkali concentration of the pore water in concrete for the design of AW. The highest alkali concentration of pore water is obtained for the case considering only 1) with all other effects considered as negligible. For the AW-CPT, assuming the worst case, the calculation using only the contribution from 1) is reasonable. Furthermore, if all the alkalis originally present in the wrapping material are absorbed by a concrete prism, the total increase of total alkalis is limited to as much as 20% of the originally present alkali level in the pore water of the concrete prism in the case for 7.5×7.5×25 cm size [2]. The goal of the wrapping in the AW-CPT is to control the total alkali content in the concrete prism without leaching alkalis while maintaining sufficient supply of water.

According to results of the most recent research [11], alkalis in the wrapping material may move from the wrapping material to the concrete prism even when there is an equilibrium in alkali concentration between the wrapping material and the pore water of the concrete prism. Even for non-reactive aggregates, carbonation of the cement paste and moisture imbibition affect the movement of alkalis. Through carbonation of the cement paste, an alumino-silica gel is formed and significant amounts of alkalis are absorbed. Alkali-silica gel formed inside the concrete consumes alkalis and reduces the alkali concentration in the pore water, resulting in ingress of alkalis from the wrapping material by diffusion. A further complication arises from, for certain aggregates, significant amounts of alkali-silica gel exuding or leaking from prisms, being changed by the reactivity of the aggregate and the acceleration conditions. Higher reactivity and more severe conditions can thus result in more exudations and less final expansion.

Based on these complicated mechanisms, for perfect quantitative expansion estimation, all these mechanisms should be included. However, for a laboratory test to examine the basic behavior of expansion, as the effect of alkalis from wrapping is limited and constant, the alkali concentration should be designed based on the pH of the cement paste.

8. CONCLUSION

A method to select or design the pH for the alkali solution used for wrapping of the concrete prism for ASR expansion test was proposed through a discussion of the determining factors of the pH of pore water in concrete.

The primary fundamental mechanism and calculation method of the pH in concrete is the pH of the pore solution in the cement paste. Based on the literature survey and experiments, a basic equation to design pH considering alkalis from Portland cement and SCMs was proposed.

The effects of alkali absorption and release by aggregate were summarized to provide a basis for this method. An interesting phenomenon, the alkali concentration in carbonated areas in the surface of concrete samples, was also introduced.

9. AKNOWLEDGEMENT

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