Current State of the JSCE Standard on Test Method for Leaching of Trace Elements from Hardened Concrete

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ABSTRACT: Current increased demand to utilize industrial by-products and wastes for concrete materials has urged to provide appropriate test methods to investigate the leaching of trace elements from the hardened concrete and/or concrete structures. This is due to the fact that test methods currently applied to hardened concrete need to crash into fine particles so as to lead to misinterpret on leaching behaviors of trace elements from the concrete. In response to these circumstances the Concrete Committee in the JSCE (Japan Society of Civil Engineers) had appointed to propose a Standard Test Method for Leaching of Trace Elements from Hardened Concrete (JSCE G575-2005). In this present paper the standard test method is introduced. In addition some test results are shown. Target trace elements are limited to Boron (B), Fluorine (F), Hexavalent Chromium (Cr6+), Arsenic (As), Selenium (Se), Cadmium (Cd), Mercury (Hg) and Lead (Pb). A Tank Leaching Method is employed to carry out leaching test from hardened concrete where the leachant are either ion-exchanged water or distilled water. Test results on concrete with normal portland cement with known contents of the trace elements are shown. The concentrations of trace elements mostly exhibit below the lower limit specified by each apparatus used for the chemical analysis. In addition the effect of the repetition times for renewal of the leachant on the leaching behavior is discussed.

1 INTRODUCTION

Environmental aspects have become of a significant research subject in concrete engineering [Kawai et al 2005, Kawai & Sugiyama 2005]. Leaching behavior of trace elements from hardened concrete is one of subjects necessary to be clarified. Evaluation of the leaching of trace elements from hardened concrete has widely been made according to the Notification of No.13 and No.46 of Environmental Agency of Japan issued in 1973 and in 1991 respectively [JSCE 2003]. Test method by the Notification of No.13 has been applied to examine the amount of toxic metals in industrial wastes such as sludge, cinders and particle matters. Then industrial wastes are judged whether or not to be treated under controlled conditions. For applying this method to hardened concrete it must be crashed and ground less than 5mm or lower in the particle size. Similar treatment is required in a test method by the Notification of No.46 that is normally used to investigate pollution of soils. A massive soil needs to be crashed and ground less than 2mm or lower in the particle size. With results of either these tests judgment will be made according to the Environmental Quality Standard of Japan for Water pollution, soil pollution and groundwater pollution, whichever is specified depending on a given purpose. In this way powder of concrete will be used to investigate the leaching of trace elements from hardened concrete, which is considered to be far from real condition.

Upon circumstances explained above more realistic test method for hardened concrete had been requested. Then the Research Committee on Concrete in the Japan Society of Civil Engineers has proposed a tank leaching test method in 2006 on the basis of the previous version issued as a tentative plan in 2003 [JSCE 2006]. This present paper introduces and summarizes the proposed method for the leaching of trace elements from hardened concrete.

2 SCOPE AND DEFINITIONS OF THE STANDARD TEST

2.1 *Scope*

This standard specifies requirements for testing trace elements leached from hardened mortar or concrete. To note that the term 'trace elements' as used in this standard refers to elements including boron, fluorine, hexavalent chromium, arsenic, selenium, cadmium, mercury and lead.

2.2 *Definitions*

The following terminology is used in this standard;

a) <u>A tank leaching test</u> refers to a test carried out for the purpose of determining the amount of trace elements leached from an untreated sample (a mortar specimen, concrete specimen or core sample) that has been immersed in a leachant. The sample is maintained at rest in an unstirred leachant during the test.

b) Leachant refers to a solution used for leaching.

c) Leachate refers to a solution obtained by leaching with particles removed by solid-liquid separation, such as filtration.

3 PREPARATION OF SPECIMENS

Specimen should be of mortar or concrete. In case of mortar specimen the unit weight of cement is larger than the case of concrete specimen. Then the amount of trace elements which may be leached out can be increased so that it is relatively easier to obtain results. In addition mortar specimen can be easy to handle as compared with concrete specimen. However to investigate leaching behavior from hardened concrete results obtained with mortar specimen must be converted for the corresponding concrete mixture proportion depending on the different unit weight of cement. The conversion will be explained later in this present paper.

The temperature at which the specimen is prepared and cured and also at which the tank leaching test is conducted in the laboratory must be 20±2°C. The specimen may be cured on site.

3.1 *Preparation of mortar specimen*

Cement, fine aggregate and water for the preparation of a mortar specimen are selected in accordance with the objectives of the test. For water ion exchanged water or distilled water is specified except when testing the effects of water quality on the leaching of trace elements. Materials used in preparation of the mortar specimen are moved into a laboratory in advance of the test so as to ensure they are at room temperature. If an admixture is used, the term cement here means ‘cement plus admixture’ according to the proportion of admixture used.

As a general rule, the water-to-cement ratio and ratio of fine aggregate to cement of the mortar shall be 0.50 and 3.0, respectively. These ratios may be varied according to the objectives of the test. Mortar is mixed in accordance with JIS R 5201. JIS R 5201 prescribes that the clearance between the outermost part of a paddle and the inner wall of a mixing bowl be 3±1 mm. When using fine aggregates 2 mm or more in maximum grain size, the clearance needs to be increased if it is adjustable. If the clearance is not adjustable, the mortar must be mixed with care to ensure that no aggregate becomes caught in the clearance.

Mortar is placed in two layers in a ø50 mm x 100 mm plastic form and compact each layer with a tamper 10-15 mm in diameter 25 times to ensure that no air bubbles remain on the surface of the specimen. To note that do not apply a mold releasing agent to the inner surfaces of the plastic form.

The top of mortar leveled by a screed is covered with plastic film for sealed curing for seven days.

3.2 *Preparation of concrete specimen*

Materials for the preparation of concrete specimen are selected in accordance with the objectives of the test. For water ion exchanged water or distilled water is specified except when testing the effects of water quality on the leaching of trace elements. Materials used in preparation of concrete specimen are moved into a laboratory in advance of the test so as to ensure they are at room temperature.

Mixture proportion of concrete is selected in accordance with the objectives of the test. Concrete is placed in a ø100 x 200 mm plastic form. To note that do not apply a mold releasing agent to the inner surfaces of the plastic form.

The top of concrete leveled by a screed is covered with plastic film for sealed curing for seven days.

3.3 *Core sample*

When used core sample it is taken in accordance with JIS A 1107. To note that as little water as possible should be used in taking the core sample.

A core sample must be 50 mm or more in diameter. If the end faces of the core sample are uneven, smooth them by cutting or other means.

Fine powder needs to be removed away from the surfaces of core sample. After lightly washing off any fine powder with ion exchanged water or distilled water, water is blotted away and any remaining fine powder are wiped off using filter paper as used for chemical analysis.

4 TANK LEACHING TEST

4.1 *Leachant*

To prepare a leachant, 5 ml of the water is used per 100 mm^2 of the surface area of a specimen into a test chamber. This standard specifies ion exchanged water or distilled water as the leachant because it is assumed that the environment is not one in which concrete structures are especially subject to chemical erosion and water with lower hardness tends to leach various ions out of concrete. In addition, this standard specifies the quantity of leachant as 5 ml per 100 mm^2 of the surface area of the specimen in consideration of the draft proposal of CEN/TC51.

A substantial quantity of leachate is required to analyze the eight trace elements described in Section 2.1. The number of ø50 x 100 mm and ø100 x 200 specimens required to obtain enough leachate is 2 and 1, respectively. Correspondingly 1.962 *l* of water and 3.925 *l* of water are required for 2 mortar specimens and 1 concrete specimen, respectively.

4.2 *Procedures*

A specimen is immersed in the leachant and is left at rest. It is ensured that the leachant has reached a steady temperature of 20±2°C in advance. Initial immersion is immediately carried out after removal of the specimen from the form. The specimen is placed in a test chamber in a way that minimizes the area of contact between the surface of the specimen and the bottom of the test chamber.

The leachant shall be completely renewed every 24 hours. The leachant removed from the test chamber is filtered by suction through a glass fiber filter or membrane filter with a pore diameter of 1 micrometer. This operation shall be repeated four times to prepare a leachate for the analysis of trace elements. The concentration of trace elements in the leachate falls with the number of leachant replacements. For this reason, this standard specifies the number of repetitions as four when the concentration of trace elements in the leachate is relatively high.

4.3 *Analysis of trace elements in leachate*

Concentrations of boron, fluorine, hexavalent chromium, arsenic, selenium, cadmium, mercury and lead are measured in the leachates taken from the first to fourth cycles of immersion in Section 4.2 in accordance with JIS K0102 or by the method prescribed in Notification Concerning Quality Tests of Materials and Equipment (Notification No. 45 of the Ministry of Health and Welfare of February 23, 2000).

JIS K0102 prescribes two or more analysis methods for each element to be analyzed. Because the minimum determinable level of a trace element in the leachate depends on the analyzer used and the analysis method, the choice of analysis method must be made in consideration of expected trace element concentrations. To measure concentrations of trace elements that are below the minimum determinable level of the analyzer, pre-treat the leachate with an appropriate separation and concentration method chosen from those prescribed in JIS K0102.

In Section 3 (Analysis Method) of the Notification Concerning Quality Tests of Materials and Equipment, electrothermal atomic absorption spectrometry is specified for the analysis of arsenic and selenium in addition to the test methods prescribed in JIS K0102.

Concentrations are expressed in a unit of mg/l, and round figures down to two decimal places for fluorine, three decimal places for boron, hexavalent chromium, arsenic, selenium, cadmium and lead, and four decimal places for mercury.

If it is impossible to carry out trace element analysis on the leachate immediately, the following steps with regard to storage are taken: for a solution to be analyzed for boron, selenium, cadmium, mercury or lead, add nitric acid (for measurement of harmful metals) to adjust the pH of the solution to about 1 and place into storage; for a solution to be analyzed for arsenic, add hydrochloric acid (for analysis of arsenic) or nitric acid (for the measurement of harmful metals) to adjust the pH of the solution to about 1 and place into storage; and for a solution to be analyzed for hexavalent chromium and fluorine, store at a temperature of 0-10°C in a dark place without adding nitric acid or hydrochloric acid and carry out analysis as soon as possible.

5 CONVERSION INTO TRACE ELEMENT CONCENTRATION IN LEACHATE FROM CONCRETE USING MORTAR SPECIMEN TEST RESULTS

A correction factor is calculated from Equation (1) based on the cement content per unit volume of the concrete being evaluated and the cement content per unit volume of the mortar specimen. Mortar specimen test results are multiplied by the correction factor to obtain results corrected for the concrete. Round off figures as in Section 4.3.

$$F = \frac{C}{M} \qquad (1)$$

where F = correction factor, C = cement content per unit volume of concrete being evaluated (kg/m^3), M = cement content per unit volume of mortar specimen (kg/m^3)

If an admixture is used, the sum of the cement and admixture contents is treated as the binder content. For the use of admixtures the determination of the concentrations of trace elements in the leachate are the same as the procedures explained in Section 4. Then the term "cement content" in the definitions of C and M in Equation (1) is replaced with "binder content."

6 REPORT

This standard says that the report must give the following information:

a) Type and dimensions of test specimen.

b) Materials used for preparation of test specimen and mix proportion of the materials. This may be omitted if a core sample is used.

c) Surface area of the specimen (mm^2)

d) Results for the first to fourth repetitions of immersion.

e) Analysis methods used and minimum determinable level of the methods. The minimum determinable level by an appropriate method must be determined because no specific method is given in this standard. If the minimum determinable level is 0.008 mg/l, for example, report with an inequality sign followed by this value (i.e. <0.008) to indicate a measurement that is less than the minimum determinable level.

f) The above information to be reported as shown in tables in Section 7.

g) Correction factor if concentrations of trace elements in leachate from concrete are obtained by conversion from test results using a mortar specimen.

7 CASE STUDY

7.1 *Concrete*

This standard test method has been applied to concrete. normal portland cement was used and Table 1 gives the content of trace elements in the cement used in this present test. Mixture proportion and other details on specimen are shown in Table 2.

Table 1. Content of trace elements in normal portland cement used in this present test for concrete (mg/kg)

Boron	Fluorine	Hexavalent chromium*	Arsenic	Selenium	Cadmium	Mercury	Lead
42	844	10.3	24	<1.0	3.8	0.0190	98

* Water-soluble

Table 2. Report on specimen

Item		Values
Type and dimensions of test specimen		Concrete, ø100 x 200 mm
Materials used for preparation of the test specimen and mix proportion of the materials	Water	170 kg/m^3
	Cement	340 kg/m^3 (normal portland cement)
	Fine aggregate	758 kg/m^3
	Coarse aggregate	1,049 kg/m^3
	Other materials	850 g/m^3 (air entraining and water reducing agent)
Surface area of specimen		78,500 mm^2
Correction factor if concentrations of trace elements in leachate from concrete are obtained by conversion from test results using a mortar specimen		-

Table 3 Report on results of chemical analysis (Unit: mg/*l*)

Element	Test results				Minimum determinable level	Analysis method
	1st repetition	2nd repetition	3rd repetition	4th repetition		
Boron	<0.010	<0.010	<0.010	<0.010	0.010	Inductively coupled plasma atomic emission spectrometry
Fluorine	<0.2	<0.2	<0.2	<0.2	0.2	Lanthanum-alizarin complexon absorptiometry
Hexavalent chromium	<0.004	<0.004	<0.004	<0.004	0.004	diphenylcarbazide absorptiometry
Arsenic	<0.005	<0.005	<0.005	<0.005	0.005	Electrothermal atomic absorption spectrometry
Selenium	<0.005	<0.005	<0.005	<0.005	0.005	Inductively coupled plasma atomic emission spectrometry (hydrogen compound emission)
Cadmium	<0.001	<0.001	<0.001	<0.001	0.001	Electrothermal atomic absorption spectrometry
Mercury	<0.0001	<0.0001	<0.0001	<0.0001	0.0001	Reduction vapor atomic absorption spectrometry
Lead	0.004	0.003	<0.002	<0.002	0.002	Electrothermal atomic absorption spectrometry

Table 4. Contents of trace elements in normal portland cement used for Mortar A and Mortar B (mg/kg)

	Boron	Fluorine	Hexavalent chromium*	Arsenic	Selenium	Cadmium	Mercury	Lead
Mortar A	20	340	7.0	8	<0.8	3.0	0.0280	52
Mortar B	33	450	10.3	6	<0.8	2.4	0.0040	38

* Water-soluble

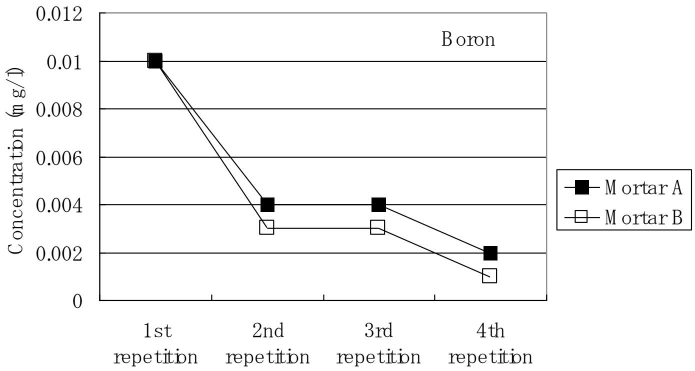

Figure 1 Change in the concentration of Boron with repetitions

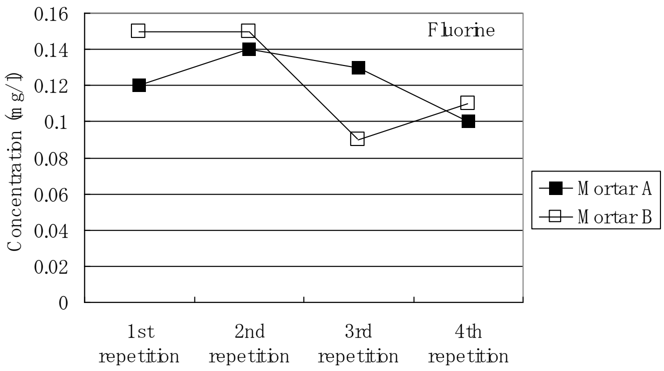

Figure 2 Change in the concentration of Fluorine with repetitions

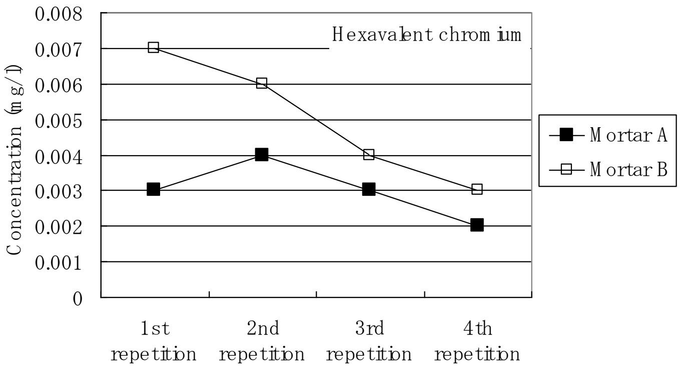

Figure 3 Change in the concentration of Cr((IV) with repetitions

It is noted that the contents of Arsenic, Cadmium and Lead are determined in accordance with the Japan Cement Association Standard method (JCAS I-52-2000). The contents of Water-soluble Hexavalent chromium and Fluorine are determined in accordance with the Japan Cement Association Standard method (CAJS I-51-1981). For the determination of the content of Water-soluble Hexavalent chromium the diphenylcarbazide method was employed in which a diphenylcarbazide solution was added 1 minute after the addition of sulfuric acid.

Results on chemical analysis for trace elements are reported in Table 3. Every trace elements except Lead exhibited concentrations that are below the minimum determinable levels. The concentration of Lead gradually decreased with increased repetitions in the tank leaching test.

7.2 *Effect of the number of repetitions on leaching*

To investigate the effect of the number of repetitions on the leaching behavior of trace elements mortar specimens were tested. Two different normal portland cements were used for the preparation of mortar specimens (Mortar A and Mortar B) although mixture proportion was the same. The contents of trace elements in those cements are given in Table 4.

Water-cement ratio was 0.5 and the unit cement content was 450 kg/m^3. Standard fine aggregate in accordance with JIS R5201 was used for the two mortar specimens.

The concentrations of Boron, Fluorine and Hexavalent chromium are shown in Figure 1, 2 and 3, respectively. The minimum determinable levels of the concentration were 0.001, 0.01 and 0.001 mg/l for Boron, Fluorine and Hexavalent chromium, respectively. Concentrations of these trace elements tend to be reduced with the repetitions. In this respect the number of the repetition of four times is considered to be appropriated. It is noted that the concentration of other trace elements exhibited the minimum determinable levels even with the first repetition so that the leaching behaviors were hardly examined.

8 CONCLUSIONS

Current State of the JSCE Standard on Test Method for Leaching of Trace Elements from Hardened Concrete (JSCE G575-2005) was introduced. The method of tank leaching employed in this standard test was thoroughly explained.

In addition leaching test results were given for concrete and mortar specimens. The concentration of Boron, Fluorine and Hexavalent chromium were determined for mortar because they were above the minimum determinable levels. These concentrations were in general reduced with the increased repetition times.

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