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Long-term leaching of toxic trace metals from Portland cement concrete

S.R. Hillier [a], C.M. Sangha [a,*], B.A. Plunkett [b], P.J. Walden [a]

[a]*Department of Civil Engineering, University of Portsmouth, Burnaby Building, Burnaby Road, Portsmouth P01 3QL, UK*
[b]*Department of Chemistry, University of Portsmouth, Burnaby Building, Burnaby Road, Portsmouth P01 3QL, UK*

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Abstract

Over the past 30 years, environmental regulations in the United Kingdom have become increasingly stringent. Concerns recently were aired over cementitious materials coming into contact with water for human consumption. Some of these materials, by virtue of their origin, can contain toxic trace metals in their composition. Results of a study investigating the long-term leaching of toxic trace metals from various Portland cement mortars in an aqueous environment are presented. Test samples were subjected to a leaching procedure based on criteria detailed by the Netherlands "diffusion" method. The leachates generated were analysed for various toxic metals outlined in directive 80/778/EEC using atomic absorption spectroscopy. The analytical results revealed only vanadium leached in detectable quantities from poorly cured concrete, and its removal was restricted from the surface only.

Keywords: Toxic metal; Diffusion; Curing

Environmental pollution has been recognised as both a national and world problem since the late 1960s [1]. The immense pollution pressures exerted on the environment stemmed from a drastic increase in environmental pollution by the industrialised nations over the previous 100–150 years, due to rapid development and population growth [2]. In the United Kingdom (UK), these pressures have slowly been addressed over the past 3 decades, through the introduction of numerous directives and legislations from the EEC. These have tackled the three main areas of pollution: water pollution, air pollution, and land pollution [3].

From the directives and legislations so far introduced, the most stringent are concerned with water pollution. At present, from the numerous EEC directives governing the UK water sources, directive 80/778/EEC [4] is responsible for the quality of water fit for human consumption. This directive contains limits for 62 parameters, a large number of which are inorganic in nature. With the help of significant advances recently made in analytical instrumentation and techniques [5], very stringent limits have been established for these inorganic constituents, ranging from parts per million (ppm) to parts per billion (ppb) levels, depending on the toxicity of the individual inorganic constituent.

Since the implementation of directive 80/778/EEC [4], concerns have been aired over the type of materials coming into contact with water for human consumption. These concerns surround the potential of materials to leach inorganic constituents, with possible resulting contamination of drinking water. One of these materials presently causing great concern is "concrete," which accounts for the largest bulk of man-made material coming into contact with water. The concern is centred on the cementitious materials used in the production of concrete. This stems from the known presence of most of the naturally occurring trace toxic metals in the raw materials used in the manufacture of cement. This is especially true for coal, which contains toxic metals in widely varying concentrations, depending on the rank and geological origin of the coal [6]. This concern over cementitious materials is further fuelled by results from a past leaching investigation involving its unhydrated form by the PCA Kiln Dust Task Force [7]. This investigation conducted a Toxicity Characteristic Leaching Procedure (TCLP) with acetic acid on cement samples from 97 cement plants in North America. The results showed As, Be, Cd, Cr, Hg, Ni, Pb, Sb, Se, and Th leached in detectable concentrations.

This recent concern over cementitious materials in concrete has seen three research teams, Kanare and West [8], Rankers and Hohberg [9], and Germaneau et al. [10], conducting leaching investigations on hydrated cementitious materials during the 1990s. Kanare and West [8] investigated the leaching of harmful trace metals from eight Portland cement concretes, made from four cements and two aggregates, using the TCLP with two different leachants. The first test used the traditional TCLP leachant of acetic acid, whereas the second test replaced this leachant with de-ion-

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[*] Corresponding author. Tel.: 44-1705 876543; Fax: 44-1705-842521.

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ised water. Results for the acetic acid test showed that Cr, Hg, Ni, and Pb were leached, whereas results for the de-ionised water test showed partial leaching of Cr, Hg, and Ni. Rankers and Hohberg [9] undertook various leaching tests on cement mortars. The tests used included two agitated extraction tests (similar to the TCLP), a flow around dynamic test (called a column test), and a serial batch test (called a tank test). The first agitated extraction test used was from a German standard (DIN 38414-S4). This test involved placing a crushed sample into agitated de-ionised water for 24 h, similar to the TCLP. Their second agitated test determined the maximum leachability of the crushed samples, by subjecting a very small sample to agitated nitric acid, maintained at pH 4, for 5 h. The third test involved placing the crushed samples into a polyethylene column and then percolating mineralised water adjusted to pH 4 using nitric acid. The fourth test involved uncrushed cubes in a tank-leaching test and was carried out over a substantial period of time in dilute nitric acid, which was renewed at increasing periods. Results showed that Cd, Cr, and Pb leaching occurred with the second agitated test, but leaching with the first test gave metal solutions too dilute for their concentrations to be established. No results were given for the last two tests, but the paper inferred leaching did take place. Germaneau et al. [10] carried out a 5-day serial batch test with de-ionised and natural mineral water to investigate the preconditioning of water supply mains. Their work showed traces of Cr, Ni, and Pb were leached from iso-rilem mortar prisms containing a range of commercial binders.

The results from these three research teams [8–10] detected the presence of some trace toxic metals in the leachates taken. These results would seem to justify the concern being aired over the potential for concrete to leach toxic metals. However, the relevance of these tests to concrete placed in the environment must be questioned. The three agitated extraction tests (TCLP [8], DIN 38414-S4 [9] and the maximum leachability test [9]) and the column test [9] are tests that provide quick assessment (less than 24 h) of the materials' harmful element leaching potential. Thus, they failed to simulate environmental conditions not only in terms of their duration, but also by incorporating the following artificially aggressive conditions:

1. Crushing the samples,
2. Agitating the samples throughout the test (except the column test), and
3. Placing samples in artificially aggressive leachants.

All three of these artificially aggressive conditions deflect from true conditions experienced in the environment. The crushing up of the samples exaggerates the leaching potential in three ways. First, it leads to a drastically increased surface area, being exposed to a leachant than what would occur in reality. Second, the crushing of samples, sometimes to finenesses passing a 150-μm sieve, i.e., with TCLP [9], would drastically alter the structural makeup of the hydrated product. This would manifest itself in the breaking of both physical and chemical bonds. Third, crushing the samples has led to concerns as to whether the crushing process contaminates the samples with metals from the processing equipment, such as jaw crushers, disc pulverisers, or swing mills. This was found to hold true for nickel and chromium with the jaw crusher process and the chrome alloy disk pulverised plates [8]. Although agitation and artificially aggressive leachants are not as influential as crushing in exaggerating the materials leaching potential, they do both influence the final result. The agitation of samples throughout the three agitated extraction tests creates an environment in which friction between particles can remove trace metals from their surface. The use of nitric acid and acetic acid as leachants gives an overly aggressive leachant, not commonly encountered by concrete in the environment.

The two tank-leaching tests (unnamed) used by Rankers and Hohberg [9] and Germaneau et al. [10] provided the greatest relevance to actual field conditions of all the tests. These tests not only provided the basis for a long-term study of concrete, but also used samples more representative of concrete placed in the environment, i.e., uncrushed. However, the information provided concerning the exact procedure and the results obtained for the investigation by Rankers and Hohberg [9] was sketchy and inconclusive, and the test by Germaneau et al. [10] was aimed at simulating the preconditioning of water supply mains and thus only lasted 5 days. Hence, from the investigations conducted to date, there is a severe shortage of credible research into the long-term leaching potential of harmful constituents from concrete placed in the environment.

This investigation will seek to clarify the long-term potential of concrete to leach trace toxic metals into the environment. The concrete specimens to be used are selected on the basis of being representative of those used in civil engineering. Portland cement was used, as it accounts for 90% of the cement used in the UK [11]. Test samples were subjected to a laboratory leaching test that simulates environmental conditions.

1. Materials

Castle Cement Ltd. supplied the Portland cement used in making the concrete specimens. Its chemical composition is shown in Table 1.

The aggregate was 5-mm maximum size, land-based flint, oven dried at 105°C for 24 h. The free water-to-cement ratios used were 0.45 and 0.65 and represented the range generally used for structural concrete. Because the aggregate was oven dry, water equivalent to 1.5% absorption by weight of aggregate was added. The water used throughout was de-ionised. The ratio of aggregate to cement was 4:1.

2. Specimen preparation

The specimens used throughout this investigation were 100-mm cubes. Each mould used was covered inside with

Table 1
Main composition of Portland cement

Compound	SiO_2	$A1_2O_3$	Fe_2O_3	CaO	MgO	Na_2O	K_2O	TiO_2	Mn_2O_3
Percent of cement by mass	20.7	5.0	2.9	63.3	2.9	0.31	0.73	—	—

cling film instead of the usual de-moulding oil to avoid contamination of the sample surface. Mixing and compaction was achieved using procedures set out in EN 196, Part 1 [12]. After compaction each mould was wrapped in four layers of cling film before being de-moulded at 24 h. Two curing conditions were used. Concretes whose initial curing period was 1 day were left in air, whereas those whose moist curing period was 28 days were rewrapped in four layers of cling film for another 27 days. This was necessary so that the samples were not contaminated or leached in any way before the leaching investigation, which would occur if they were moist cured using the conventional method involving a curing tank.

3. Leaching test

Due to the severe lack of work involving the long-term leaching of toxic metals from uncrushed concrete samples, no standard recognised leaching test has been cited in past literature for the task presented by this investigation. With the existence of hundreds of different leaching tests, the decision was taken to select one sponsored by a recognised organisation or associated with regulatory standards. In the UK, the only standard leaching test available that remotely approximated the requirements is the BS 6920 "Suitability of non-metallic products for use in contact with water intended for human consumption with regard to their effect on the quality of the water" [13]. Unfortunately, this test is inadequate for providing a long-term investigation. The total duration of the test is only 9 days, and it cannot be extended due to the unusual time periods for extraction.

The test chosen was selected from more than 100 outlined in Environment Canada [14]. This publication provides a comprehensive classification of the numerous different leaching tests developed before 1990. The list of tests was first reduced by choosing the classification of test that best met the following criteria:

- The closest representative of conditions encountered by concrete in the environment,
- Simple and inexpensive to set up and run, and finally
- Suitable for the testing of a large number of samples.

A "dynamic test" in the form of a serial batch test was chosen, as this type of test provided the best solution to the task at hand. From the 12 tests outlined in Environment Canada [14], only one specified uncrushed samples, was longer than 24 h in duration, and did not involve the use of expensive rotary equipment. Thus, the test chosen was the NVN 5432 maximum leachability test [15].

Despite this test being the most suitable of all the standard reference tests available from Environment Canada [14], it was not totally adequate for the task at hand. This test needed to be adapted by making adjustments in terms of the leaching duration, size of specimen, and the volume of leachant. The duration of 64 days was considered insufficient for an investigation into the long-term leaching potential of concrete. Hence, the leachant renewal times given in Table 2 were adopted. The schedule for leachant renewal was based on the assumption that bulk diffusion would be the dominant leaching mechanism. If this held true, then the leaching rate would decrease with time.

For the size of specimen and the volume of leachant used, the volume-to-surface ratio of 5 cm outlined in NVN 5432 maximum leachability test [15] was retained.

4. Leachate generation

The leaching test was initiated 28 days after the casting of the concrete specimens. Each specimen was placed inside a separate uncontaminated 4-L polypropylene container containing 3 L of de-ionised water. The leachant had stood for 24 h before the test to achieve carbonate and temperature equilibrium. Each specimen was positioned on two thin spacers to expose all six surfaces to the leachant. A polypropylene lid was placed on top of the container to eliminate contamination from dust. The containers were stored in a static state (i.e., no stirring, shaking, agitation, or circulation) until leachant renewal. At each designated leachant renewal the concrete specimen was removed from the leachant, with any excess liquid carefully shaken off into the used leachant. The specimens were transferred to a new container housing fresh leachant.

From every used leachant, one 52.5-mL leachate sample was generated using the following procedure. After removing the concrete specimens from the used leachant solution, it was vigorously stirred for 30 s with a clean TFE-fluoro-

Table 2
Times for leachant renewal

N	Time of leachant renewal from initiation of leaching test
1	1.5 h
2	6 h
3	24 h
4	4 days
5	16 days
6	64 days
7	256 days

carbon-coated stirring rod. While the leachate was still circulating, a 10-mL sample was removed quickly using an uncontaminated 10-mL pipette and placed inside a 60-mL polypropylene laboratory bottle. The leachate was stirred for another 10 s before another 10-mL sample was taken. This was repeated three times, giving a final sample of 50 mL inside the 60-mL polypropylene laboratory bottle. This technique was chosen to reduce the number of 60-mL sampling bottles to be analysed, while still giving a representative sample of the leachate produced. Immediately after pipetting each 50-mL leachate sample, 2.5 mL of concentrated AnalaR grade hydrochloric acid was added to the 60-mL polypropylene laboratory bottle. This was to eliminate any loss of metal ions through chemisorption and hydrolysis. The remaining leachant was discarded, and the 52.5-mL leachate samples were stored at room temperature until analysis.

5. Inorganic constituents investigated

From the five tables outlined in the directive 80/778/EEC [4], one contained the toxic metals to be investigated. This table has been abstracted with its corresponding toxic metals and is shown in Table 3.

6. Digestion of Portland cement

To express the leaching results in terms of an "equivalent leaching depth (D_X)," the chemical composition of the Portland cement needed to be known in terms of the toxic metals being investigated. Furthermore, their presence in the cement needed to be confirmed, as many of these trace elements may not even be present. However, before chemical analysis of the Portland cement could commence it needed to be digested. This was accomplished using the "lithium tetraborate ($Li_2B_4O_7$) fusion method" outlined in ASTM E886-94 [16]. This method was selected as it used hydrochloric acid, thus significantly reducing the number of possible matrix effects present in the digestion, which can develop when digesting complex matrices such as cement in various acids. Furthermore, the use of hydrochloric acid with this method provided compatibility with the digestions and leachates, which both used hydrochloride acid for acidification, thus limiting the number of variables in this investigation.

Table 3
Parameters concerning toxic metals taken from Table D in Annex 1 of Directive 80/778/EEC [4]

Parameter	Expression of the results	Guide level (GL)	Maximum admissible concentration (MAC)
Arsenic	As μg/L	—	50
Beryllium	Be μg/L	—	—
Cadmium	Cd μg/L	—	5
Chromium	Cr μg/L	—	50
Mercury	Hg μg/L	—	1
Nickel	Ni μg/L	—	50
Lead	Pb μg/L	—	50
Antimony	Sb μg/L	—	10
Selenium	Se μg/L	—	10
Vanadium	V μg/L	—	—

To optimise the $Li_2B_4O_7$ fusion method for this investigation, a few amendments were made with regard to the weight of the sample/fusion material used and the furnace time. As the toxic metals were present in trace amounts, it was decided to double the mass of cement digested from 0.1 to 0.2 g. Consequently, the mass of the $Li_2B_4O_7$ was increased to 0.75 g and the furnace time to 20 min. The rest of the ASTM method was strictly adhered to.

7. Quantitative analysis

To relate the quantities of toxic metals originally present in concrete to the quantities leached, the same quantitative analytical method was performed on both Portland cement digestions and the leachates produced. As no standard analytical method exists for leachates, the method chosen was associated with the analysis of cementitious materials. Due to the number of leachates being generated and the type of inorganic constituents being investigated, a rapid analysis method was used. However, with the chemical analysis of cementitious materials in the UK governed by the wet methods set out in EN 196: Part 2 [17] and EN 196: Part 21 [18], the analysis was carried out in accordance with ASTM E885-88: Standard Test Methods for Analyses of Metals in Refuse-Derived Fuel (RDF) by Atomic Absorption Spectroscopy [19]. This method was selected as it not only incorporated the chemical analysis of trace elements, but it also provided criteria for interference checks. The latter is extremely important, as unlike the major/minor elements, the instrument specifications used throughout cannot be checked with reference materials and precision/accuracy criteria, as the trace amounts detected are too small.

The quantitative analysis of both the leachates and digestions was carried out using the Hitachi model Z-8100 polarised Zeeman atomic absorption spectrometer (Hitachi Z-8100). This instrument has flame atomic absorption (FAAS), flame atomic emission (FAES), and electrothermal atomisation atomic absorption (ETA-AAS) capabilities. It is a single-beamed instrument that contains a grating monochromator, photomultiplier detector, adjustable slit widths, a wavelength range of 190 to 900 nm in increments of 0.1 nm, and computerised data measuring and recording facilities. A variety of attachments are used in conjunction with the Hitachi Z-8100. These included:

- Two types of burners (atomisers)—standard burner and high temperature,
- A single-element "hollow cathode" lamp for each of the elements, and
- The model SSC-200 auto sampler for the graphite furnace.

Table 4
Toxic metal composition of the Portland cement used for this leaching investigation

Element	Sb	As	Be	Cd	Cr	Pb	Hg	Ni	Se	V
Concentration (μg/g)	Not detected	19.9 ± 2.4	1.4 ± 0.2	Not detected	72.7 ± 6.9	75.3 ± 3.2	Not detected	72.0 ± 4.8	Not detected	44.1 ± 3.3

With the elements measured being in the ppb range, the majority of the analytical methods chosen from ASTM E885-88 [19] involved electrothermal atomisation techniques. The only exceptions were beryllium, cadmium, chromium, and mercury. For these elements, flame absorption techniques were chosen for sensitivity and equipment reasons. Furthermore, due to the great variety of AAS instruments available, not all the conditions suggested in ASTM E885-88 [19] were applicable for the Hitachi Z-8100 to pass the interference checks. Although primarily set up using ASTM E885-88, the conditions used also incorporated instrumentation adjustments suggested by the instruments operation manual [20]. The analytical conditions that passed the interference checks were applied throughout the leaching investigation, giving consistency to all the digestion and leaching results obtained.

8. Results and discussion

8.1. Composition of Portland cement

The mean and 95% confidence limits values obtained from analysing Portland cement for the toxic metals outlined in Table 3 are given in Table 4.

The results for the Portland cement digestions showed that all the toxic metals investigated were detected, except for antimony, cadmium, mercury, and selenium. Their concentrations were below 5 μg/g for antimony and cadmium, 12 μg/g for mercury, and 2 μg/g for selenium. The very low/undetectable concentration for antimony was not unusual. This finding agreed with concentrations of antimony found in cements analysed by the PCA Kiln Dust Task Force [7] and Krcmar et al. [21]. The three cements tested by the two research teams each gave antimony values below 4 ppb. Such low concentrations were shown by Krcmar et al. [21] to be due to its naturally low levels in the raw materials and fuel (coal ash) used to produce Portland cement. Any high concentrations in the raw material would have been detected in the cement, as Weisweiler and Krcmar [22] proved antimony to exhibit poor volatile behaviour in the kiln, with only 0.1% reaching the dust collection system at the end of the kiln.

The undetectable concentrations of cadmium, selenium, and mercury were due to insufficient concentrations in the raw materials and fuel (coal ash) to overcome the high volatility of their compounds. Although the PCA Kiln Dust Task Force [7] and Krcmar et al. [21] have proved cadmium, selenium, and mercury to represent the most volatile trace elements in the kiln, both research teams have detected their presence in cement. However, the limits detected were extremely small when compared to the concentrations found in the raw materials and fuel (coal ash) used.

The concentrations of arsenic, chromium, beryllium, lead, and nickel were all within the range of value submitted by the PCA Kiln Dust Task Force [7]. The concentrations of vanadium could only be compared to values obtained by Krcmar et al. [21], who only analysed cement from one plant. Hence, values differed considerably, with the vanadium value being one third the value obtained by Krcmar et al. [21].

Allowing for variations in the raw materials and fuel (coal ash), the results for the trace toxic metal analysis of the Portland cement used in this investigation generally were consistent with data from other research teams [7,21–25]. Hence, the Portland cement used in this leaching investigation can be termed as normal, with no abnormally high concentrations of any trace toxic metals.

8.2. Trace toxic metals leached

The leaching results for the trace toxic metals proved that the elements shown in column 1 of Table 5 were not detected in any of the leachates produced. This applied to all the concretes tested. This meant that for each concrete tested, each of the seven leachate samples produced over the 256-day investigation failed to contain these toxic metals in concentrations greater than the detection limits outlined in column 2. These detection limits are shown as an equivalent leached mass in column 3 of Table 5.

The lack of detectable leaching proves that these toxic metals are:

- Originally present in the cement matrix in insignificant concentrations, or

Table 5
Trace toxic metals not detected in the leaching investigation

Toxic metal	Detection limit (ppb)	Equivalent mass required to leach in 3 L of leachant (g)
Antimony	5	15×10^{-6}
Arsenic	1	3×10^{-6}
Beryllium	1	3×10^{-6}
Cadmium	5	15×10^{-6}
Chromium	28	84×10^{-6}
Lead	5	15×10^{-6}
Mercury	12	6×10^{-6}
Nickel	3	9×10^{-6}
Selenium	2	6×10^{-6}

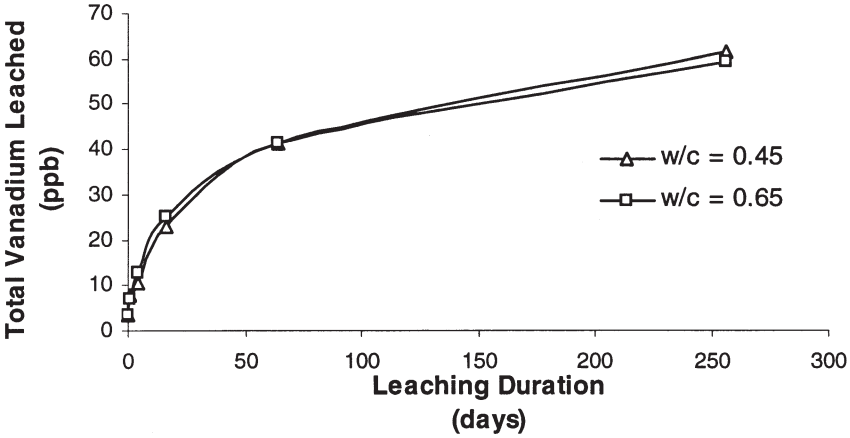

Fig. 1. Vanadium leached from 1-day, moist-cured Portland cement concrete.

- Bound up in the concrete matrix, or
- Present in the form of an insoluble compound, as both the initial surface wash and long-term diffusion were shown, by the first three leachant renewal times of 1.5, 6, and 24 h and the last two leachant renewal times of 64 and 256 days, respectively, to give undetectable concentrations.

The one trace toxic metal found to leach was vanadium. The leaching results obtained are shown in Fig. 1. These results reveal that the dominating factor in vanadium mobilisation is the degree of hydration achieved by the concrete, as detectable vanadium leaching only occurred with 1-day, moist-cured cubes. The leaching of vanadium was undetected (below 2 ppb) for all the leachates generated from the 28-day, moist-cured cubes. This difference in vanadium leaching between the two moist-curing periods is attributed to the degree of hydration reached. Good hydration of the concrete is important in creating a concrete matrix sufficient to encase vanadium. The results shown in Fig. 1 also revealed the water-to-cement ratio to have an insignificant effect on vanadium leaching.

The vanadium results for the 1-day, moist-cured concretes revealed detectable vanadium leaching occurred with each leachate sample taken. Furthermore, the quantity of vanadium leached increased as the leaching period increased. This finding suggests that the vanadium present was in the form of VO_4^{3-} (oxyanions), as work by de Groot et al. [26] found the maximum leachability of vanadium in this form occurred in the pH range from 7 to 10. Hence, each time the leachant was renewed, the time taken for the pH of the water to reach 10 would increase due to a reduction in the available hydroxide ions of calcium, potassium, and sodium.

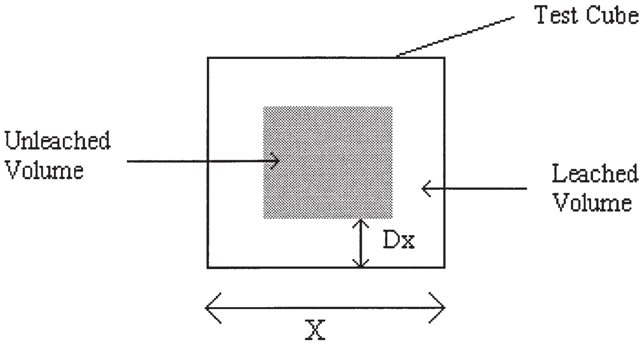

Fig. 2. Leached and unleached areas of a test cube.

8.3. Depth of vanadium leaching

The total volume of vanadium leached failed to give a true picture of the possible depths to which leaching occurred and the volume leached with respect to the volume of material originally present. This was determined using the equivalent leaching depth (D_X) method, which is based on the following assumptions:

- All the element is leached to that depth,
- Uniform leaching from all six sides, and
- No concentration profile exists within the concrete specimen being leached.

To transform the total volume leached to its equivalent depth of leaching, Eqs. (1) and (2) were used. Eq. (1) was used first to find the volume of the cube from which the element had leached after time t:

$$V_L(t) = [M_L(t)/M_C(o)] * X^3 \tag{1}$$

where$V_L(t)$ is the volume of the cube from which the element has been leached after time t (mm^3), $M_L(t)$ is the mass of element leached after time t (g), $M_C(o)$ is the mass of element originally present in the cube (g), and X is the size of the cube (mm).

For a typical cube (Fig. 2), the equivalent volume value obtained from Eq. (1) can be represented by Eq. (2):

$$V_L(t) = X^3 - (X - 2D_X)^3 \tag{2}$$

where$V_L(t)$ is the volume of the cube from which the element has been leached after time t (mm^3), X is the size of

Table 6
Depth of vanadium leaching for 1-day, moist-cured concretes

Water-to-cement ratio	Total vanadium leached (ppb)	Depth of vanadium leaching (mm)
0.45	61.7	0.151
0.65	59.6	0.149

the cube (mm), and D_X is the equivalent leaching depth (mm).

With D_X being an unknown, Eq. (2) became a quadratic, which was solved using the Newton-Raphson iterative method [27].

The equivalent leaching depth (D_X) method was applied to total vanadium leached values obtained from the 1-day, moist-cured specimens. The depth results are given in Table 6. These two values show the depth of vanadium leaching to be confined to the surface.

9. Conclusions

1. Well-cured Portland cement concrete released no detectable concentrations of the toxic metals outlined in directie 80/778/EEC [4] (water fit for human consumption).
2. Poorly cured Portland cement concretes released detectable concentrations of vanadium; however, the leaching was restricted to the surface only.
3. The water-to-cement ratio had no significant effect on the leaching potential of vanadium from concrete.

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