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Study of the leaching behaviour of paving concretes: quantification of heavy metal content in leachates issued from tank test using demineralized water

Anne-Marie Marion*, Michel De Lanève, Alain De Grauw

Centre de Recherches Scientifiques et Techniques de l'Industrie Cimentière Belge (C.R.I.C.), Laboratoire-Service Recherches, Avenue Adolphe Buyl, 87-1050 Brussels, Belgium

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Abstract

The leaching behaviour of concretes made from porphyry aggregate, river sand and Portland cement or blast furnace slag cement has been studied by means of a tank test using demineralized water in accordance with NEN 7345. The results show that the amount of heavy metals leached is small, much lower than the parametric values specified by the European Directive defining the quality of drinking water, and becomes negligible after prolonged immersion.

At the end of the tank test, the fraction of heavy metals leached by the concrete represents less than 1% of the total heavy metal content of the cement. To refer to the bulk content as a criterion of environmental quality is therefore unjustified and unduly restrictive.

The results suggest that the controlled use of alternative fuels and raw materials to replace natural materials does not in any way alter the leaching behaviour. Similarly, the replacement of a defined fraction of the clinker with blast furnace slag does not compromise the environmental compatibility of the concrete in terms of the heavy metals it releases.

A second finding of these experiments is that the risk of contamination due to release of heavy metals from concrete on-site appears small. The total heavy metal content of concrete is of the same order of magnitude as (or even smaller than) that of a soil considered as being unpolluted in Belgium. Moreover, the fraction of heavy metals released by the concrete as a proportion of this total heavy metal content is nonsignificant.

Keywords: Concrete; Characterisation; Heavy metals; Diffusion; Leaching behaviour

1. Introduction

The use of industrial by-products in replacement of natural materials is widely encouraged in construction: this practice enables residual materials to be recycled and valorized, while at the same time saving natural resources and energy.

In cement production the residual materials can be used both as substitute fuels and raw materials as well as supplementary cementing materials replacing part of the clinker. The question of whether the incorporation of alternative materials in the production of the cement is liable to increase the quantity of heavy metals it contains is legitimate insofar as there could be a risk of contamination of the ground water by exposed concrete on-site.

The numerous investigations carried out internationally tend to show that this risk is nonexistent [1–3] and that, for cementitious materials, there is no relationship between the bulk heavy metal content and the leachable fraction of these elements [4–6]. To date, however, most international studies into the leaching of cementitious materials have provided only indicative values as the heavy metal content of the leachates is generally below the limit of detection of the analytical methods used.

This paper aims to document further the published data relating to the environmental compatibility of concrete. The quantities leached during the course of the NEN 7345 tank test with demineralized water (diffusion test) are compared with the parametric values of the European Directive relating to "the quality of water intended for human consumption" (98/83/EC). By using a plasma torch coupled with a mass spectrometer (ICP-MS) it was possible to refine the analytical observation and properly

* Corresponding author. Tel.: +32-2-645-52-73; fax: +32-2-648-51-20.
E-mail address: am.marion@cric.be (A.-M. Marion).

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quantify most heavy metals that are present as trace elements in the concrete leachates (concentrations of the order of micrograms per liter or nanograms per liter). The fraction of heavy metals leached could then be calculated, taking into account the total heavy metal content of the cement part in concrete.

The effect of the controlled and partial use of alternative fuels is also examined, as is the influence on the leaching behaviour of concrete of replacing the clinker with blast furnace slag within the limits imposed by EN 197-1[7] for type CEM III/A blast furnace slag cements (36% to 65% of slag).

Finally, comparison with natural soils allows a relationship to be established between the results of the leaching tests and the probable impact on the environment of exposed concrete on-site.

2. Experimental procedure

2.1. Materials

The investigations concern concrete samples made from porphyry aggregate, river sand, and Portland cement (CEM I 42.5 R) or blast furnace slag cement (CEM III/A 42.5 LA), with a cement content of about 400 kg/m^3 and a water/cement ratio close to 0.45, which are the aggregates, types of cement, and concrete mixes commonly used in Belgium for road applications. The cements used are of industrial source; consequently, the chemical characterisation of the incorporated slag has not been possible. The bulk heavy metal content of cements is presented in Table 1. The minimum and maximum values are issued from the chemical characterization of the 2 × 3 investigated cements (three CEM I and three CEM III/A).

The three cements of each type were taken from different production sites [A-B-C (CEM I)/A′ -B′ -C′ (CEM III/A)]. At the time these cements were produced, the Belgian cement producers replaced natural fuels and raw materials with controlled proportions of alternative fuels and industrial by-products.

Table 1
Bulk concentration of heavy metals in cement (mg/kg)

Elements	CEM I cements		CEM III/A cements	
	minimum	maximum	minimum	maximum
As	7.1	11	2.9	5.9
Cd	0.51	1.5	0.53	0.89
Cr	74	121	45	71
Cu	22	60	15	30
Hg	0.71	3.5	< 0.19	0.44
Mn	234	465	746	1117
Ni	56	84	36	45
Pb	12	145	15	56
Sb	4.4	9.4	1.5	13
Zn	91	514	75	199
Se	4.0	9.9	2.6	5.1
Ag	0.49	0.61	0.30	0.74
Ba	205	577	534	689

Table 2
Typical concrete composition (kg/m^3)

Porphyry aggregate 7/20		745	
Porphyry aggregate 2/7		466	
River sand 0/5		652	
CEM I 42.5 R		398	
Water		179	
Total		2440	
W/C		0.45	
Fresh concrete			
Consistency			
Slump test (mm)		40	
Vebe test (s)		4.0	
Density (kg/m^3)		2441	
Real concrete volume placed with the tested composition (l)		1004	
Air content (% vol)		0.80	
Hardened concrete			
Compressive strength on cubes, d = 158 mm	Age (days)	R_c (N/mm^2)	
	7d	50.9	
	28d	66.1	
	91d	72.3	
Water absorption in % of starting mass (W) and in % of the dry mass (A)		W (%)	A (%)
On slice (S = 100 cm^2, h = 4.5 cm)	56 days	1.4	5.3

Table 2 shows the mix design of the concretes and gives some of the physicomechanical properties of the concrete in its fresh and hardened states.

The leaching solution was demineralized water (pH = 6 ± 0.2, conductivity ≤ 0.06 μS/cm).

2.2. Methods

According to international experts [8,9], the leaching of monolithic concrete under normal conditions of exposure is essentially governed by diffusion. Consequently, the most suitable test for laboratory simulation of the behaviour on site is a tank test.

The tank test used was that defined by NEN 7345 [10]: concrete cubes (10 × 10 × 10 cm) were cured for 56 days at 20 ± 2°C and > 90% RH, then immersed in a given volume of demineralized water (liquid/solid = 6) and kept in static conditions at a temperature of 20 ± 2°C.

At the end of each normalized immersion step (6 h and 1, 3, 7, 14, and 36 days), the eluate was separated, filtered, acidified, and stored for analysis and also replaced with 6 l of fresh demineralized water. The final sample was taken after an immersion period of 64 days.

The concrete leachates corresponding to each immersion step were analyzed by ICP-MS to quantify each of the heavy metals in the list of chemical parameters specified by

European Directive 98/83/EC [11] (Ni, Cr, Sb, Se, Mn, Hg, As, Pb, Cd, and Cu). Occasionally, the analysis was extended to include other heavy metals (Ba, Zn, and Ag).

3. Results and discussion

3.1. Bulk heavy metal content of concretes

Within the context of leaching, the bulk heavy metal content is an essential parameter for the chemical characterization of a concrete. The bulk heavy metal contents of the concretes investigated are given in Table 3, which shows the minimum and maximum values obtained from the chemical characterization of the six investigated concretes: three concretes with Portland cement (CEM I 42.5 R) and three concretes with blast furnace slag low-alkali cement (CEM III/A 42.5 LA with a percent slag content of $35 < S < 65$ in conformity with EN 197-1 [7]). All other composition parameters remaining similar, the replacement of a type CEM I cement by a type CEM III/A cement does not significantly modify the bulk heavy metal content of concrete.

3.2. Variation of heavy metals leached during the tank test using demineralized water

The interpretation of the data is based only on the heavy metal contents of the concrete leachates; any possible (re)precipitation phenomena are not taken into account [8,12]. When it comes in contact with concrete, demineralized water becomes very alkaline, especially when the water volume is limited.

The heavy metal content of the leachates, normalized to release per 24 h, falls sharply with increased length of immersion (Table 4), regardless of the cement type. In most cases, the leaching of heavy metals became insignificant after prolonged immersion. This was despite renewal of the demineralized water, thereby requiring a new solubility equilibrium to be established, with a consequent possibility of increased leaching activity. The six concretes behaved in a similar manner. This behaviour supports a diffusion mechanism for concrete submitted to the tank test: according to Fick's law, indeed, the leach rate would be proportional to root time. Real conditions on the field could differ significantly from those in the tank test: in natural exposure, concrete is susceptible to be exposed to unlimited water volume inducing quite different solubility conditions according to the pH. However, it is likely that the natural system "concrete–water" would be in most cases in static conditions: poor renewing of the leaching medium will induce pH close to alkalinity, which means close to the tank test conditions with regard to that particular parameter. Another crucial issue refers to the leaching mechanism: if the leaching is essentially governed by diffusion in the tank test conditions, other leaching processes (oxidoreduction, dissolution, precipitation) are susceptible to play a predom-

Table 3
Bulk concentration of heavy metals in concrete (mg/kg)

Elements	CEM I concretes		CEM III/A concretes	
	minimum	maximum	minimum	maximum
As	1.8	12	1.4	1.8
Cd	0.14	0.28	0.08	0.17
Cr	55	64	24	31
Cu	18	30	9.3	12
Hg	4.6	7.3	8.7	9.9
Mn	382	465	341	383
Ni	17	26	9.3	12
Pb	11	27	8.6	12
Sb	1.6	2.1	0.64	1.6
Zn	56	94	30	38
Se	< 0.01	4.5	< 0.01	< 0.01
Ag	0.33	0.74	0.27	0.58
Ba	327	400	366	393

Table 4
CEM I-C concrete—leached concentrations normalized to release per 24 h (μg/l)

Elements	Immersion steps of the tank test					
	1 day	3 days	7 days	14 days	36 days	64 days
Ba	143	90	53	32	14	8.3
Ni	0.88	0.57	0.28	0.16	0.29	0.19
Cr	6.2	3.7	1.8	0.94	0.33	0.21
Sb	0.15	0.11	0.071	0.048	0.016	0.015
Zn	0.17	0.067	0.042	0.015	0.016	0.009
Pb	0.15	0.10	0.058	0.033	0.018	0.010
Cd	0.025	0.011	0.006	0.003	0.001	< 0.001
Hg	0.029	0.015	0.005	< 0.003	< 0.001	< 0.001
Se	< 0.60	< 0.60	< 0.60	< 0.60	< 0.60	< 0.60
Mn	< 0.060	< 0.060	< 0.060	< 0.060	< 0.060	< 0.060
As	< 0.015	< 0.015	< 0.015	< 0.015	< 0.015	< 0.015
Ag	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005
pH	11.9	11.8	11.8	11.6	11.4	12.0

< Values: concentrations lower than the detection limit.

Table 5
Heavy metal released versus the requirement of the Directive (μg/l)

Element	LΣ64d/24h00				Parametric values 98/83/EC
	CEM I, minimum	CEM III/A, minimum	CEM I, maximum	CEM III/A, maximum	
Ba	6.4	8.0	22	8.8	Not required
Ni	0.19	0.096	0.45	0.28	20
Cr	0.31	0.13	0.71	0.29	50
Sb	0.011	0.010	0.028	0.068	5
Se	< 0.060	< 0.060	< 0.060	< 0.060	10
Mn	< 0.006	< 0.006	< 0.006	0.008	50
Hg	< 0.002	< 0.002	< 0.002	< 0.002	1
As	< 0.002	< 0.002	0.006	0.007	10
Ag	< 0.001	< 0.001	< 0.001	0.003	Not required
Zn	0.014	0.005	0.020	0.022	Not required
Pb	< 0.001	< 0.001	0.027	< 0.001	10
Cd	< 0.001	< 0.001	0.002	< 0.001	5
Cu	< 0.004	< 0.004	0.015	< 0.004	2000

< Values: concentrations lower than the detection limit.

Table 6
Bulk heavy metals content in cement (Bc) versus released concentration from concrete

Elements	CEMI/A		CEMI/B		CEMI/C		Parametric values 98/83/EC (μg/l)
	Bc cement (mg/kg)	LΣ64d/24h00/ concrete (μg/l)	Bc cement (mg/kg)	LΣ64d/24h00/ concrete (μg/l)	Bc cement (mg/kg)	LΣ64d/24h00/ concrete (μg/l)	
Ni	69	0.45	56	0.19	84	0.26	20
Cr	74	0.37	85	0.31	121	0.71	50
Sb	4.4	0.011	5.5	0.018	9.4	0.028	5
Se	4.0	< 0.060	9.4	< 0.060	9.9	< 0.060	10
Mn	234	< 0.006	453	< 0.006	465	< 0.006	50
Hg	0.71	< 0.002	3.0	< 0.002	3.5	< 0.002	1
As	8.0	0.002	11	0.006	7.1	< 0.002	10
Pb	12	< 0.001	29	0.003	145	0.027	10
Cd	0.51	< 0.001	0.55	0.002	1.5	0.002	5
Cu	22	< 0.004	36	0.011	59	0.015	2000

inant role when concrete is exposed on the field. The quality of the water and the dissolved species are likely to be determining parameters with regard to those secondary chemical reactions.

3.3. Leachate concentrations versus the parametric values (Directive 98/83/EC)

There are currently no European criteria for the environmental compatibility of concrete. Comparison of the leached heavy metal concentrations (normalized per 24 h) against the parametric values of Directive 98/83/EC has been used for interpreting the results of the leaching tests. It is a matter, so to speak, of assessing the risk of contamination due to the leaching of heavy metals from the concrete.

Table 5 shows an "average" leached concentration per 24-h period (LΣ64d/24 h00): these are the minimum and maximum values for the six investigated CEM I (three) and CEM III/A (three) concretes.

LΣ64d/24h00 is calculated by taking into account the leached quantities cumulated over the seven immersion steps of the tank test (LΣ64d), and dividing the obtained cumulated value by the total time of immersion (64 days).

Those "average" quantities of heavy metal released per 24 h by a paving concrete in contact with demineralized water are very small and, for undesirable heavy metals, well below the parametric values given in the Directive.

The presence of slag used as a clinker substitute within the limits allowed by EN 197-1 for CEM III/A type cements does not modify the leaching behaviour of the concrete.

3.4. Bulk heavy metal content of the cement versus concentrations released by the concrete: quantification of the leached fraction

A higher total heavy metal content in the cement would not necessarily lead to greater amounts being leached from concrete, as shown in Tables 6 and 7 for CEM I and CEM III/A concretes, respectively. This observation holds for the same element present in the different cements originating from the different production sites (horizontal comparison: Mn, Ni, Se, Cd, etc.) as well as for different elements present in increasing concentrations in the same cement (vertical comparison: Cd, Sb, Cu, Mn, etc.).

This confirms the findings recorded in the literature [4–6]: for cementitious materials, there is no systematic relationship between the bulk heavy metal content of the cement

Table 7
Bulk heavy metals content in cement (Bc) versus released concentration from concrete

Elements	CEM III/A-A′		CEM III/A-B′		CEM III/A-C′		Parametric values 98/83/EC (μg/l)
	Bc cement (mg/kg)	LΣ64d/24h00/ concrete (μg/l)	Bc cement (mg/kg)	LΣ64d/24h00/ concrete (μg/l)	Bc cement (mg/kg)	LΣ64d/24h00/ concrete (μg/l)	
Ni	40	0.096	36	0.28	45	0.28	20
Cr	71	0.29	45	0.13	59	0.21	50
Sb	5.3	0.038	13	0.068	1.5	0.010	5
Se	2.6	< 0.060	4.4	< 0.060	5.1	< 0.060	10
Mn	1117	< 0.006	792	< 0.006	746	0.008	50
Hg	0.44	< 0.002	< 0.002	< 0.002	0.030	< 0.002	1
As	3.7	0.002	2.9	< 0.002	5.9	0.007	10
Pb	56	< 0.001	31	< 0.001	15	< 0.001	10
Cd	0.83	< 0.001	0.89	< 0.001	0.53	< 0.001	5
Cu	21	< 0.004	30	< 0.004	15	< 0.004	2000

Table 8
Fraction of heavy metal leached—CEM I concretes

Elements	Bulk content due to cement (Bc) in a concrete cube (mg)			Leached concentration from a concrete cube over 64 days (mg)			Leached fraction (% Bc)		
	CEM I-A	CEM I-B	CEM I-C	CEM I-A	CEM I-B	CEM I-C	CEM I-A	CEM I-B	CEM I-C
Ni	27.5	22.3	33.6	0.173	0.073	0.102	0.631	0.328	0.302
Cr	29.5	33.9	48.3	0.142	0.119	0.272	0.480	0.352	0.563
Sb	1.8	2.2	3.8	0.004	0.007	0.011	0.248	0.305	0.290
Se	1.6	3.8	3.9	0.000	0.004	0.003	0.007	0.106	0.088
Mn	93.6	181.2	185.9	0.000	0.001	0.000	0.000	0.000	0.000
Hg	0.3	1.2	1.4	0.000	0.000	0.001	0.134	0.014	0.038
As	3.2	4.3	2.8	0.001	0.002	0.001	0.021	0.052	0.018
Pb	4.8	11.9	58.1	0.000	0.001	0.010	0.006	0.011	0.018
Cd	0.2	0.2	0.5	0.000	0.001	0.001	0.203	0.292	0.137
Cu	8.8	14.3	23.8	0.001	0.004	0.006	0.009	0.029	0.024

and the leachable fraction of these elements. The heavy metals are in fact trapped, either in the constituent phases of the clinker or in the hydrates of the cement [13].

The bulk heavy metal content will not vary greatly between one CEM I cement and another (of the order of a factor of 2), except for Pb and Hg. The quantities of these elements leached are very small, however, and do not require to be considered further here.

At the time of production of the cements investigated in the present study, the use of secondary materials as substitute for natural fuels was known to be applied in various but controlled proportions, according to the cement plant. On the basis of the leaching data, the following statement could be made: the controlled use of varying amounts of secondary fuels does not therefore necessarily contribute to an increase in the heavy metal content of the cement and does not alter the leaching behaviour of the concrete [14–18].

The fraction leached from concrete at the end of the tank test (64 days) was calculated and expressed as a percentage of the bulk heavy metal content due to cement. This quantification is based on a simplifying assumption, whereby only the bulk heavy metal content considered to be "leachable" has been taken into account, i.e., only that contained in the cement, that in the aggregates being ignored.

The data presented in Tables 8 and 9 have been calculated for a concrete cube with a cement content considered to be equivalent to the mix design (400 kg/m^3). The density is the average value of all the mixes tested. Values marked 0.00 indicate bulk contents of less than 10 μg, and 0.000 indicates leached quantities of less than 1 μg or leached fractions of less than 0.001%.

These data show that the heavy metals leached out by the concrete in contact with demineralized water cumulated over a 64-day period constitute less than 1% of the total heavy metal content of the cement. This finding confirms that there is no relationship between the total content and the leachable fraction. This conclusion applies for paving concretes made from CEM I and CEM III/A cements.

3.5. Bulk heavy metal content of concrete versus bulk heavy metal content of soil

The comparison between artificial and natural materials is of particular interest where leaching is concerned [15].

The bulk heavy metal contents of the concretes studied are, for the most part, of the same order of magnitude as the bulk heavy metal contents of Belgian soils [19,20] (Table 10). Only the Hg content is significantly higher in concrete, but as the quantities released during leaching tests are very small, this is not cause for alarm.

Table 9
Fraction of heavy metal leached—CEM III/A concretes

Elements	Bulk content due to cement (Bc) in a concrete cube (mg)			Leached concentration from a concrete cube over 64 days (mg)			Leached fraction (% Bc)		
	CEM III/A-A′	CEM III/A-B′	CEM III/A-C′	CEM III/A-A′	CEM III/A-B′	CEM III/A-C′	CEM III/A-A′	CEM III/A-B′	CEM III/A-C′
Ni	16.1	14.4	17.9	0.037	0.106	0.107	0.228	0.734	0.597
Cr	28.2	18.1	23.6	0.111	0.052	0.082	0.392	0.286	0.346
Sb	2.1	5.2	0.6	0.014	0.026	0.004	0.680	0.498	0.613
Se	1.0	1.8	2.0	0.000	0.004	0.010	0.000	0.199	0.495
Mn	446	316	298	0.002	0.001	0.003	0.001	0.000	0.001
Hg	0.2	0.00	0.01	0.000	0.000	0.000	0.000	0.000	2.132
As	1.5	1.2	2.3	0.000	0.001	0.003	0.006	0.122	0.110
Pb	22.3	12.5	6.1	0.000	0.000	0.000	0.000	0.000	0.000
Cd	0.3	0.4	0.2	0.000	0.000	0.000	0.052	0.008	0.166
Cu	8.4	11.4	5.9	0.001	0.000	0.000	0.017	0.000	0.002

Table 10
Bulk heavy metal content in concrete and in Belgian soils (mg/kg)

Elements	CEM I concretes		CEM III/A concretes		Soils[a]	Soils[b]	
	Minimum	Maximum	Minimum	Maximum		(a)	(b)
As	1.8	12	1.4	1.8	10–30	22	100
Cd	0.14	0.28	0.08	0.17	1	1	8
Cr	55	64	24	31	60–300	65	230
Cu	18	30	9.3	12	15–30	50	210
Hg	4.6	7.3	8.7	9.9	0.20	1.6	15
Mn	382	465	341	383	500–2000	No value	No value
Ni	17	26	9.3	12	10–80	40	180
Pb	11	27	8.6	12	50	70	1150
Sb	1.6	2.1	0.64	1.6	1	No value	No value
Zn	56	94	30	38	No value	150	680
Se	< 0.01	4.5	< 0.01	< 0.01	No value	No value	No value

[a] Indicative values of the maximum "normal" bulk concentration in soils, whatever the texture.
[b] Maximum threshold values for uncontaminated (a) and decontaminated soils (b).

This finding, together with the fact that the released concentrations are negligible, allows one to suggest that the risk of contamination of the soil due to the leaching of heavy metals from concrete on-site appears nonsignificant, and, consequently, the probability of pollution of the groundwater should be very low.

4. Conclusions

Paving concretes made from porphyry aggregate, river sand, and CEM I 42.5 R or CEM III/A 42.5 LA industrial cements, of the type commonly produced in Belgium, leach heavy metals at very low concentrations, significantly lower than the parametric values given in European Directive 98/83/EC, which defines "the quality of water intended for human consumption." Consequently, on the basis of the behaviour of such concrete submitted to a tank test with demineralized water according to NEN 7345, this type of concrete could be considered not to be harmful for the environment in terms of the leaching of heavy metals.

The partial replacement of clinker with blast furnace slag, within the limits defined in EN 197-1 for CEM III/A type cements (36% to 65% of slag), has no effect on the leaching behaviour of the concrete.

There is no systematic correlation between the bulk heavy metal content of CEM I and CEM III/A cements and the leachable fraction of these elements from concrete. Therefore, the use of the bulk content as a parameter to indicate the leaching potential and/or as a criterion of environmental quality is unfounded and unduly restrictive.

The partial and controlled replacement of fossil fuels and raw materials of natural origin by alternative materials in the cement production does not seem likely to influence the leaching behaviour of concrete.

The bulk heavy metal contents of road-paving concretes are of the same order of magnitude as those of unpolluted and decontaminated Belgian soils; in view of the very small amounts leached, the risk of contamination of the soil and the water that seeps through it seems nonsignificant.

The possible influence of pH, however, has to be kept in mind when establishing a relationship between the data from the tank test and the field conditions: the leaching behaviour of heavy metals is indeed known to be pH dependent.

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