



# CARACTERISTICI DE MEDIU ALE BETONULUI CU CIMENT FABRICAT CU COMBUSTIBIL ALTERNATIV<sup>▲</sup>

## ENVIRONMENTAL CHARACTERISTICS OF CONCRETE WITH CEMENT MANUFACTURED WITH ALTERNATIVE FUEL

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*A major environmental risk factor, associated with the use of construction materials, is their potential emission of pollutants and their migration in the aqueous medium (groundwater, surface water and precipitation).*

*The paper aim is to evaluate the environmental properties of concrete with cement manufactured by using of the alternative fuel.*

*Concrete matrix ability of heavy metals immobilization was assessed experimentally by appropriate leaching tests. Dynamic surface leaching test on monolithic concrete samples simulates release of contaminants from concrete products during service life. The potential leaching characteristics of the concrete after demolition were estimated by the extraction test, with assessment of the leaching availability (or the maximum leaching fraction). Also, there carried out the extractions at different values of pH for establishment the pH influence on heavy metals leaching.*

*The emission values of heavy metals from monolithic concrete samples after 64 days were compared with the regulated, as according to Dutch Building Materials Decree (DMBD). The release values of the heavy metals from concrete were well below of the DMBD regulated criteria, referring to categoria I of construction materials.*

*The extraction test results showed that pH plays a crucial role on the heavy metals leachability. The cationic contaminants (Mn, Ni, Co, and Cu) were leached at pH values < 7. The cations solubility is much lower than that of the oxanionic in the alkaline aqueous medium of concrete pore with pH ≥ 12.5. The higher alkalinity of leachate provided from alkaline character of cement matrix was favorable to leach of Cr (VI), As (V), Sb (V) and V (V) as oxanions.*

*The experimental results obtained can be a contribution referring to assessing the environmentally friendly character of the concrete due cementitious matrix ability to heavy metals immobilization.*

*Un factor de risc major asupra mediului, asociat cu utilizarea materialelor în construcții, este potențialul acestora de emisie a substanțelor poluante și de migrare a acestora în mediul apos (pânza freatică, apa de suprafață și de precipitații).*

*Scopul lucrării constă în evaluarea proprietăților de mediu ale betonului cu ciment fabricat prin utilizarea de combustibil alternativ.*

*Abilitatea matricei betonului de fixare a metalelor grele s-a apreciat, experimental, prin teste de lixiviere adecvate. Testul de lixiviere pe probe monolit de beton a simulat comportarea betonului pe durata de viață a aplicației. Caracteristicile potențiale de lixiviere ale betonului după demolare au fost determinate prin testul de extracție (availability) care a permis evaluarea fracției maxime lixivabile. De asemenea, s-au efectuat extracții la diferite valori de pH pentru stabilirea influenței pH-ului asupra lixivierii metalelor grele.*

*Valorile concentrației metalelor grele în eluatele obținute pe probe monolit după 64 de zile au fost comparate cu cele reglementate, conform cu Decretul olandez pentru materiale de construcție (DMBD). Valorile concentrației metalelor grele în extractele apoase conform testului monolitic au fost mult sub limita reglementată de DMBD referitor la material de categoria I (cu utilizare fără risc).*

*Rezultatele testului de extracție au arătat că pH-ul influențează semnificativ lixivierea metalelor grele din matricea betonului. Contaminanții cationici investigați ((Mn, Ni, Co și Cu) au trecut în soluție, semnificativ, la valori de pH < 7. Solubilitatea cationilor a fost mult mai mică decât a oxoanionilor în mediul alcalin cu pH ≥ 12,5, specific soluției apoase din porii betonului. Alcalinitatea ridicată a fost favorabilă lixivierii Cr (VI), As (V), Sb (V) și V (V), ca oxoanioni.*

*Rezultatele experimentale obținute pot fi o contribuție la descrierea caracterului prietenos față de mediu al betonului, prin capacitatea de fixare a metalelor grele.*

**Keywords:** concrete, cement, alternative fuels, leaching test, heavy metals

### 1. Introduction

The industrial Portland cements are containing heavy metals in low and variable concentration. Heavy metals in cement come from natural and secondary raw materials and from alternative fuels ash in the process of the clinker/cement obtaining.

In cement kilns at the very high temperatures possible organic contaminants from

alternative fuels, are completely destroyed [1-5]. Example of wastes used as alternative fuel in Europe are: used tires, spent solvents, waste oils, paper sludge, municipal waste of paper and plastics, auto textile waste, scrap woods, bone meals, biomass ash and refuse derived fuel [3-5]. The variability in major and minor elements content of the cement is quite small. Trace elements show a limited range in spite of the use of alternative fuels [6-8].

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The concrete produced with cement or with cement and fly ash addition presents the heavy metals immobilization ability [8-13]. The level of heavy metals concentration in standard concrete is comparable with the heavy metals content in natural soil [2, 9, 10]. But release of heavy metals from cement matrix is the most important criterion of the environmental properties of the concrete. This depends on the aqueous solution pH of the concrete pores and of concrete environment [2, 5, 12 - 14].

The heavy metals content of concrete is not a decisive criterion of their mobility into aqueous medium, due to low solubility of precipitates (hydroxides, carbonates and sulfates of heavy metals) in  $\text{pH} \geq 12.5$  aqueous solution of concrete pore. For example, release of hazardous  $\text{Cr}^{6+}$  does not increase with its content in the concrete matrix, but is significantly enhanced by highly basic pH of the solution in concrete pores [8, 15]. The same conclusion as for Cr may be valuable that there is no relationship between leachability and heavy metal content of the concrete [15]. For an understanding of material leaching behavior more elaborate tests are needed that can take into account aspects that relate to practice.

The heavy metals immobilization can be appreciate by experimental static or dynamic leaching tests carried out under specific conditions of pH, liquid/solid (L/S) ratio and during contact with aqueous medium [2, 16, 17]. Specific parameters of leaching tests simulate real exposure conditions of concrete, either as monolithic building component or as granular material (waste) from demolition. Purpose of the work consists to testing the leaching properties of cement-based concrete produced by using an alternative fuel.

We studied the transfer of heavy metals from concrete binder matrix into the aqueous medium, depending on the specific parameters of the leaching tests. The leaching tests performed in this paper have sought to assess the chemical stability of concrete in two scenarios: during to the service cycle, as element of construction and after demolition, as recyclable aggregate. Thus the concrete has been tested by tank leaching test - dynamic surface leaching test (DSLTL), for determination of surface dependent release of substances from monolithic or plate-like or sheet-like construction products [18] and by availability extraction test [19] combined with pH control leaching test [20].

There have been two series of concretes with industrial cement CEM I, produced with alternative fuel. The concrete samples were carried out with cement and limestone filler addition and another series of samples with cement and fly ash addition, as fine sand substitutes.

## 2. Experimental conditions. Materials and methods

The industrial Portland cement CEM I, according to SR EN 197-1 [21], was used. As according to research project results [22], the values of traces elements in cement (mg/kg) were as follows: As = 28.1, Cd=0.06, Cr=41.1, Cu=94, Ni=35.7, Pb=10.4, Sb=9.69, V = 38,07 TI=0.40. Limestone filler has the following characteristics: density  $2780 \text{ kg/m}^3$ ,  $\text{CaCO}_3$  content 95.6% and particle with  $d < 90 \mu\text{m}$  up to 91.3%. The fly ash, according to SR EN 450-1 [23] had the features: density of  $1790 \text{ kg/m}^3$ ,  $R_{45\mu\text{m}}=59\%$ , pozzolanic activity index of 78.6%,  $\text{SiO}_2=51\%$ ,  $\text{Al}_2\text{O}_3=24.4\%$ ,  $\text{CaO}=8.3\%$ . The river aggregate as 0/4 mm size of sand and as 4/16mm size of coarse aggregate characteristics were according to SR EN 12620 [24].

The concretes studied in this work were prepared with a cement dosage of  $440 \text{ kg/m}^3$ . The concrete, considered the reference, noted R82, was obtained with industrial cement CEM I and limestone filler addition of 20% and the concrete noted C82 was obtained with the same industrial cement CEM I and fly ash addition of 20%. The admixture was used as fine sand substitute. These concretes were obtained with  $D_{\text{max}}$  of 16 mm river aggregate and water/cement (W/C) ratio of 0.38.

The tank leaching test allows characterization of the leaching mechanism of pollutants transfer from concrete as monolithic construction material in the aqueous medium and the long term prediction of the heavy metals emission process [15, 18]. The concrete samples were immersed in a volume of leachant (demineralised water) at own pH. The leachant volume / total surface area of concrete sample in contact ratio was of  $8 \text{ cm}^3/\text{cm}^2$ . The test duration was 64 days which included the 8 consecutive sequences as follows: 0.25; 1; 2.25 ; 4; 9; 16 ; 36 and 64 days. The leachant renewal was achieved after each of 8 sequences to not reach steady state. The eluates were obtained by vacuum filtration, on the  $0.45 \mu\text{m}$  filters.

The measured parameters after each sequence were: pH, electrical conductivity and heavy metals concentration. Analysis of eluates was performed by atomic absorption spectral analysis AAS, at As, Cd, Co, Cr, Cu, Mn, Ni, Sb, V, Hg concentration level of ( $\mu\text{g/L}$ ).

To assess the ability of concrete matrix to immobilize heavy metals, including those from secondary fuel ash embedded in clinker, we used appropriate leaching tests. The Dutch leaching test NEN 7341 uses demineralised water [19]. The concrete samples were crushed until they passed through a  $0.125\text{mm}$  sieve. The protocol consists of extracting the eluates in two steps with a liquid



The sequential release of the Cu, Cr, Ni, Pb, Sb and V seems more significant. The measured leaching of the heavy metals per sequence and calculated cumulative leaching of the heavy metal component, for all test sequences, were the basis for emission calculation.

The variation on entire test duration of the pH and electrical conductivity of the each eluate fraction per sequence, are presented in Figures 1 and 2.

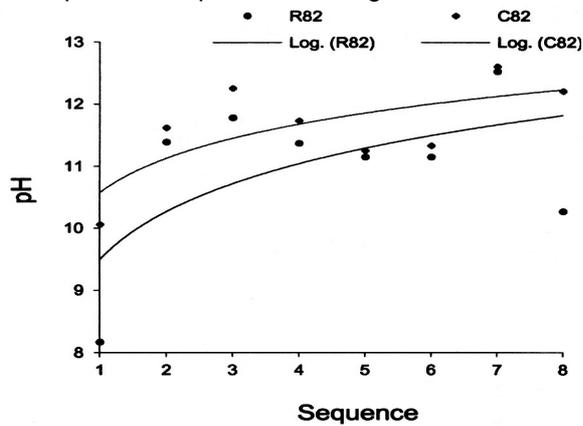


Fig. 1 - The pH variation of the eluate fractions during tank test-DSLTL/Variația pH-ului eluatelor fiecărei secvențe a testului pe probe monolit-DSLTL.

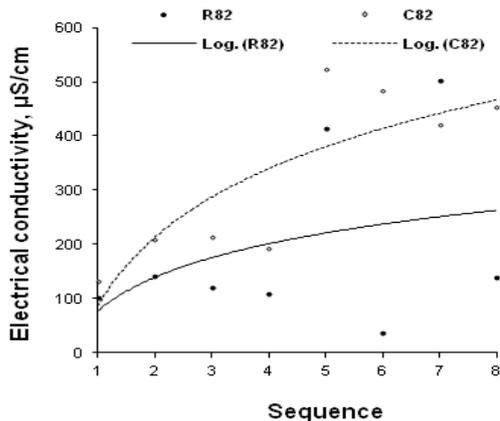


Fig. 2 - The electrical conductivity variation during tank test-DSLTL/Variația conductivității electrice a eluatelor fiecărei secvențe a testului pe probe monolit-DSLTL.

The time influence of heavy metals release from cement matrix of concrete, reported to the tank test specific working conditions is shown in Figure 3. The graphical representation of emission as function of time is presented, cumulatively, for Cr, Cu, Hg and Sb. The release of Cr follows a parabolic variation in relation with time, as a function of  $Y = f(t/2)$ , both in concrete R82 and C82. This mass transfer mode is considered of difuzional type [15]. The Hg and Sb release arising as a transport proportional function suggesting that leaching is controlled of solubilisation.

A comparison of release in  $mg/m^2$  observed in tank leach test at 64 days for investigated concretes, compared with the regulated values and other referential data are showed in Table 3.

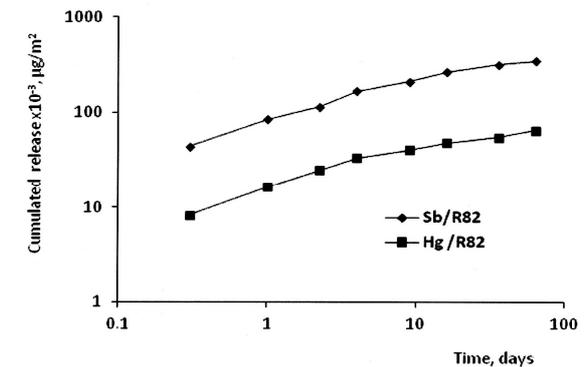
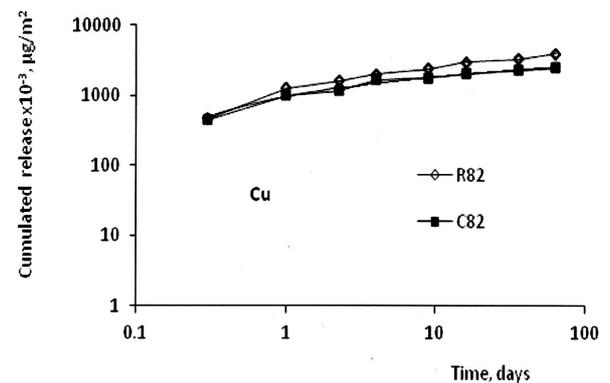
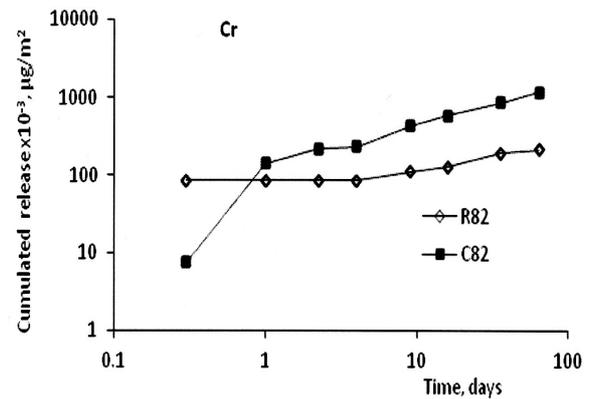


Fig 3 - Leaching behaviour of monolithic concrete sample: release of Cr, Cu, Hg and Sb as a function of time/Comportarea la lixiviere a probelor monolitice de beton: emisia de Cr, Cu, Hg și Sb în funcție de timp ( $mg/m^2$ ).

The Dutch Building Materials Decree (DMBD) [25] is the first regulation addressing environmental issues of construction materials. It is based on the laws for protection of soil and protection of surface water. So, this decree gives the quality criteria for the use of stone building materials and earth used as a construction material. There is no difference made between primary materials, secondary materials and use of waste materials. The Soil Quality Degree specifies slightly higher limit values [15].

The release values of the heavy metals from concretes determined in this work were well below of the DMBD regulated criteria, referring to category I of construction materials.

Table 3

Comparison of release in  $\text{mg/m}^2$  observed in tank leach test at 64 days for investigated concretes compared with ECRICEM values for mortars [15] and criteria of DBMD and for unrestricted use  
 Comparație a valorilor emisiilor în  $\text{mg/m}^2$  obținute prin testul de lixiviere pe probe monolit de beton la 64 de zile, cu valorile ECRICEM pentru mortare [15] și cu criteriile DBMD pentru categoria I de materiale de construcție

	Tank leach test –DSL/ Test de lixiviere pe probe monolit		ECRICEM ECN–E-11-020	DMBD Category I	SQD Category I
	Concrete with limestone filler R82/Beton cu filler de calcar R82	Concrete with fly ash C82/Beton cu cenușă de termocentrală C82			
As	0	0.001	0.75	41	3.8
Cd	0.002	0	0.44	1.1	60
Co	0.012	0.034	0.54	29	120
Cr	0.034	0.305	7.4	140	98
Cu	0.637	0.304	2.8	51	1.4
Hg	0.011	0.011	<0.3	0.43	
Mn	0.025	0.012	-	-	-
Ni	0.007	0.024	2.6	50	81
Pb	0	0	1.8	120	400
Sb	0.027	0	0.45	3.7	8.7
V	0	0	7.2	230	320

Table 4

The characteristics of the leachates/Caracteristicile eluatelor

Concrete sample / Probă beton	R82				C82			
	11.72				11.70			
Own pH after 3 hours/pH propriu								
Set point /initial pH /pH setat	4	5	7	10	4	5	7	10
Equilibrium /final pH after 3 h/pH final	5.51	5.55	7.57	10.16	4.88	6.55	7.47	9.95
Conductivity (ms/cm)/Conductivitate electrică	9.71	6.42	4.48	2.93	7.47	5.33	2.38	3.83
Volume of added 1M $\text{HNO}_3$ (ml)/Volum adăugat de soluție 1M $\text{HNO}_3$	89.32	54.01	34.41	21.36	63.42	49.02	36.14	29.22

Table 5

The extraction test leaching concentration of elements  
 Concentrația elementelor în eluatele obținute prin testul de extracție,  $\mu\text{g/l}$ 

	R82- Concrete with limestone filler/ pH of eluate/ Beton cu filler de calcar R82/pH eluat				C82-Concrete with fly ash/ pH of eluate/ Beton cu cenușă de termocentrală C82/ pH eluat			
	4	5	7	10	4	5	7	10
Hg	0.1	0.09	0.09	0.1	0.16	0.18	0.17	0.14
As	0.31	9.87	6.5	10.8	0.1	1.76	0.84	0.1
Cd	2.39	0.27	0	0	2.84	0.14	0.3	0.02
Co	16	38.4	4.6	3.5	19.1	11.7	5.31	5.22
Cr	40.7	42.5	115	80.4	1.13	0.32	11.5	16.1
Cu	22.4	12.8	1.24	1.23	38.1	1.48	3.31	1.55
Mn	640	143	4.38	0.42	558	44.7	23	0.66
Ni	74.8	101	61.2	12.3	65.8	133	10.4	7.25
Pb	9.3	10.8	7.9	9.4	12.9	7.31	8.88	7.51
Sb	8.68	11.1	12	11.9	7.69	7.36	7.69	7.59
Tl	0.14	0	0	0	0.05	0.06	0.04	0.04
V	3.87	21.7	53.1	42.6	0	18.3	25.4	36.9

### 3.2. The availability test

In this work was used the availability test as a modified procedure by extending of the pH area. So, the leachant pH was kept constant for three hours extraction time, at the set level of pH = 4, 5, 7 and 10 and corrected with 1 M  $\text{HNO}_3$  solution. The measured parameters were the final (of equilibrium) pH; the electrical conductivity and 1M

$\text{HNO}_3$  solution volume consumed and these are showed in the table 4.

The leachability of trace elements from cementitious materials depends on their speciation [17]. So, the cation solubility is limited by precipitation as hydroxides and solubility of oxyanionic speciation is limited by the formation of poorly soluble calcium salts. Also, the oxidation-state of the ionic species is influenced by the pH of

the aqueous environment e.g. (Cr (III) / Cr (VI) (As (III) / As (V)).

The eluate resulted after each extraction, at a some pH, was analyzed by atomic absorption spectral analysis (AAS) at As, Cd, Co, Cr, Cu, Mn, Ni, Sb, V, Hg concentration level of ( $\mu\text{g/L}$ ) and are gived in Table 5.

The leaching availability as maximum leachable fraction values and heavy metal emissions are depending on the pH of the leaching environment and of the concrete matrix type. Scenario is applied under  $\text{pH} = 4$ , to simulate the worst environmental feature e. g. acid rain or contact with acid water if is considered concrete as demolition waste.

The behavior at leaching of trace elements from cement matrix of the concrete is shown in Figures 4 and 5.

The transport of heavy metal compounds from concrete matrix into aqueous medium was significantly influenced by pH. Thus, increasing the leaching solution pH favored the emission of Cr, V, As and Sb both in concrete R82 and the C82 (Fig. 4). It is knows that leachability of oxyanions is maximal at  $\text{pH} 9 - 10$  [15, 17]. As according to van de Sloot et al [10, 15] all Cr leached at  $\text{pH} > 7$  is chromate. According to this, the higher alkalinity of eluate was favorable to leach of chromate ( $\text{CrO}_4^{2-}$ ), vanadate ( $\text{VO}_4^{3-}$ ), arseniate ( $\text{AsO}_4^{3-}$ ) and possibly as stibiate ( $\text{SbO}_4^{3-}$ ).

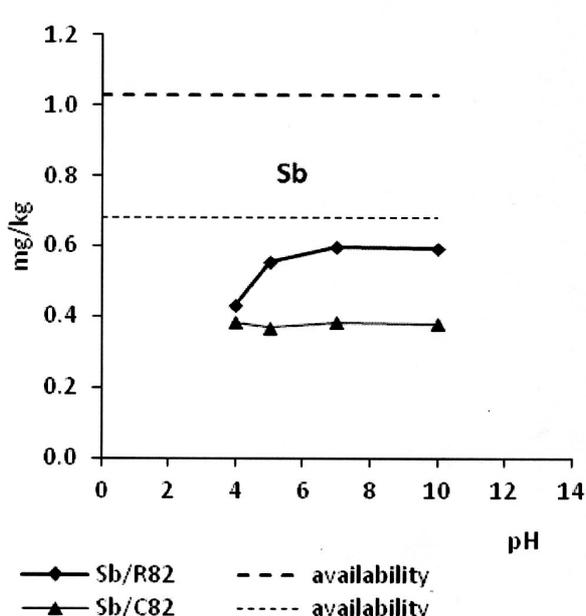
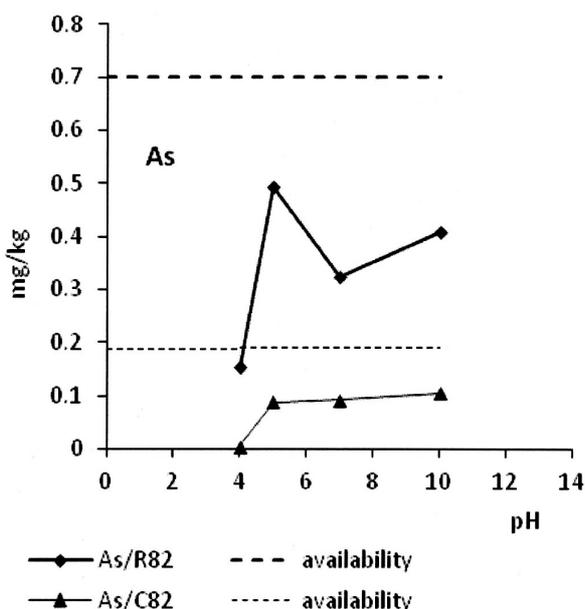
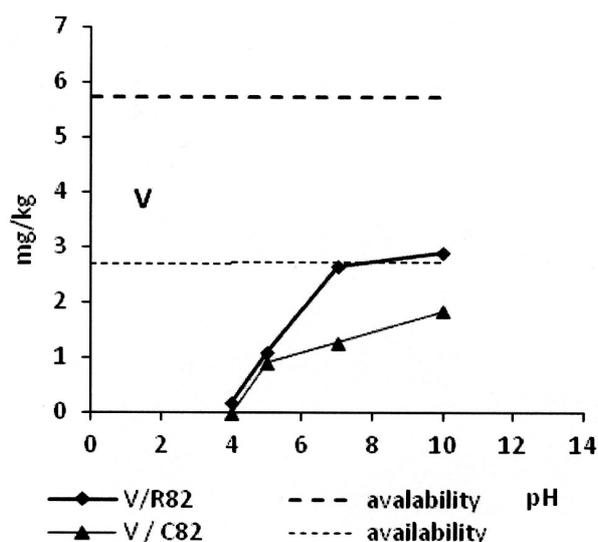
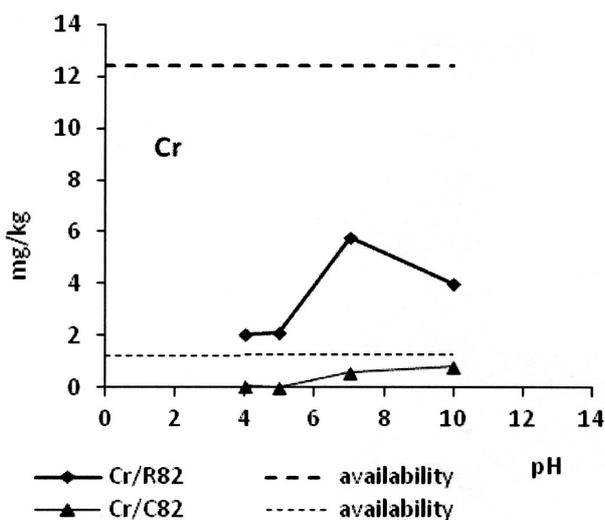


Fig. 4 - The leaching behavior of trace elements Cr, V, As and Sb, from cement concrete; the availability (at  $\text{pH} = 4$ ) and pH influence/Comportarea la lixiviere a elementelor minore din beton Cr, V, As și Sb; fracția maximă lixivibilă la  $\text{pH} = 4$  și influența  $\text{pH}$ -ului.

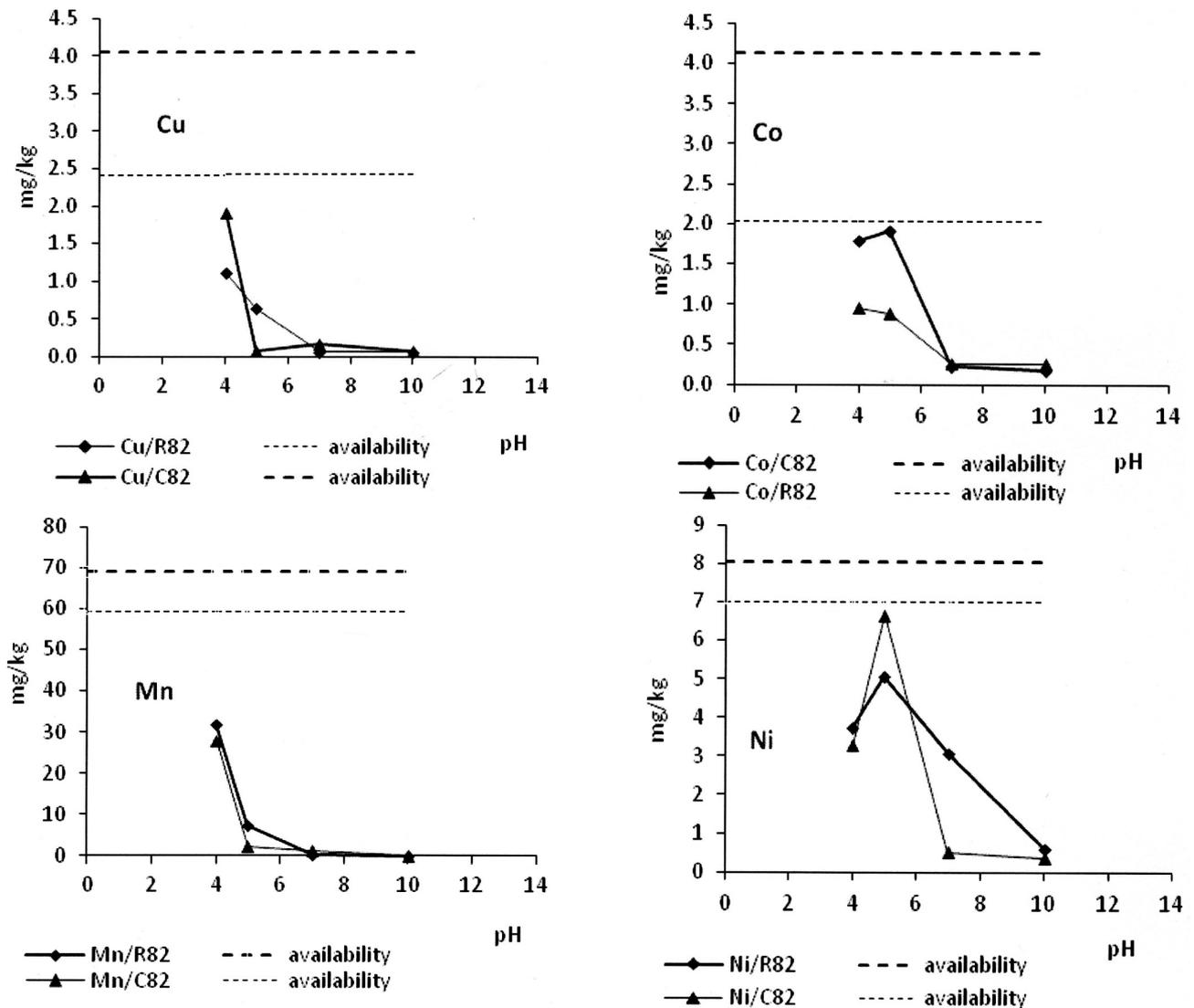


Fig. 5 - The leaching behavior of trace elements Cu, Co, Mn and Ni from cement concrete—the availability (at pH = 4) and pH influence/ Comportarea la lixiviere a elementelor minore din beton Cu, Co, Mn și Ni; fracția maximă lixivielabilă la pH = 4 și influența pH-ului.

Also, leaching of heavy metals cations of Mn, Ni, Co, and Cu was diminished with leaching solution pH increasing both from R82 and the C82 concrete (Fig. 5). So, the higher alkaline pH of the concrete pores aqueous solution has a positive role on fixing these heavy metals by precipitation as hydroxides [17, 25].

Therefore, the main influence factor of heavy metals release, qualitatively point of view, was the pH. From a quantitative perspective, concrete matrix with admixture of limestone filler or fly ash was slightly different. It seems to show a role on the Cr and V precipitation as calcium salts. The difference of As, Cr, V, Sb release may be explained based on a stronger carbonation of the fly ash admixture concrete and to concrete structure (open porosity, surface microcracks).

The experimental results values of the availability (maximum leachable fraction, MLF) at pH=4 and particle size of crushed concrete < 0.125mm, are showed, comparatively, with the ECRICEM median values [15] in the Table 6.

Generally, the obtained availability values are close to those of average reported by ECRICEM. The elements Cr, Cu, Mn, Ni and V show a rather high availability for leaching. The concentration values are higher than those median values of the different world cements as according to ECRICEM report.

Therefore, the results of the extraction leaching test showed that pH plays a crucial role in determining the solubility of the metals studied. According with that cationic contaminants (Mn, Ni, Co, and Cu) were leached at pH values < 7. The heavy metals leaching behaviour can be considered to be a pH-dependent process controlled by metal hydroxide solubility and, as consequence, the cationic concentrations in the extracts was controlled by metal hydroxide solubility. The leachate concentrations of trace-level metals have been found to exhibit equilibrium values that depend strongly on leachate pH [8, 10, 26]. Solubility of oxyanionic speciation Cr(VI), V(V) As(V) and Sb(V), is increased at pH values

Table 6. Availability as MLF values of the &lt;125 µm crushed concrete samples/Valorile fracției maxime lixiviabile determinate pe probe de beton mărunțit &lt;125 µm, mg/kg

Heavy metal/Metal greu	ECRICEM availability median values (nov. 2011)/Valori medii ale fracției maxime lixiviabile conform ECRICEM	Concrete with limestone filler (R82)/Beton cu filer de calcar (R82)	Concrete with fly ash (C82)/Beton cu cenușă de termocentrală (C82)
As	0.21	0.70	0.18
Cd	0.04	0.21	0.35
Co	18	4.14	2.03
Cr total	3.0	12.39	1.22
Cu	1.0	2.41	4.05
Hg	1.0	0.01	0.02
Mn	38	68.95	59.27
Ni	4	8.06	6.98
Pb	0.8	1.002	1.37
Sb	0.3	1.03	0.68
V	1.0	5.72	2.69

higher than 5 [15, 26]. The cations solubility is much lower than that of the oxyanionic in the alkaline aqueous medium of concrete pore with pH  $\geq 12.5$ .

#### 4. Conclusions

- The environmental properties of the cement concretes manufactured with alternative fuel were investigated considering two scenarios: the concrete as monolithic material in the construction application and the demolished concrete as granular waste, recyclable as aggregate.
- The tank leaching test on monolithic concrete sample, as dynamic surface leaching test (DSL) according to CEN/TC 351, was used for determination of surface dependent release of heavy metals from cement matrix of concrete.
- The release values of the heavy metals from concrete were well below of the DMBD regulated criteria, referring to categoria I of construction materials.
- The availability extraction test, NEN 7341, combined with pH control leaching, was used for investigation of the leaching properties of the concrete as demolished waste. The heavy metals release by extraction leaching test, from the crushed concrete cement matrix, depends, significantly, of the pH of the aqueous medium of contact. The cationic contaminants (Mn, Ni, Co, and Cu) were, significantly, leached at pH values < 7. The higher alkalinity of leachate was favorable to leach of oxyanions: Cr as chromate ( $\text{CrO}_4^{2-}$ ), V as vanadate ( $\text{VO}_4^{3-}$ ), As as arseniate ( $\text{AsO}_4^{3-}$ ) and Sb possibly as stibiate ( $\text{SbO}_4^{3-}$ ).
- Therefore the concrete with cement manufactured with alternative fuel studied in this work showed no leaching characteristics to prefigure a negative impact on the environment in the application as monolithic construction product or as waste granular material after demolition.

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## MANIFESTĂRI ȘTIINȚIFICE / SCIENTIFIC EVENTS

### International IABSE Conference, Rotterdam May 6 - 8, 2013

The Dutch and Belgian IABSE groups organise May 6 – 8, 2013 an International Conference dedicated to the very important topic for our society to keep our infrastructure in a good condition. IABSE, the International association for Bridge and Structural Engineering is a scientific / technical Association comprising about 4.000 members in 100 countries, counting 48 National Groups worldwide. Thus IABSE offers the platform for exchange of knowledge, opinions and ideas to explore new ways for this global problem.

#### **Assessment, Upgrading and Refurbishment of Infrastructures**

The co-operation between scientists and practitioners in IABSE proved to be an enrichment, leading to new ideas and initiatives for over 80 years now. In the field of construction materials and construction many changes can be noted. For progress nowadays working together in an International context is a must. In a fast changing world the considerable developments and challenges ask for a general and common approach and also bundling of forces. New material and construction types challenge the imagination of designing. The implementation of life cycle design is stimulated by ecological and economical constraints. Infrastructure is not only designed for safety and serviceability, but as well for flexibility and adaptability. Maintenance and upgrading of structures move to the centre of interest.

**Contact:** <http://www.iabse2013rotterdam.nl/index>

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