Performance study of cementitious systems containing zeolite and silica fume: effects of four metal nitrates on the setting time, strength and leaching characteristics

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Abstract

The aim of this study is to investigate the effect of four metal nitrate contaminants, namely chromium, manganese, lead and zinc on the mechanical and leaching characteristics of cement-based materials. For this purpose, three different matrices made of: (i) Portland cement, (ii) Portland cement and silica fume, and (iii) Portland cement and natural zeolite were studied. The effects of metals on the stabilised/solidified (S/S) product characteristics were monitored by measuring: (i) setting time, (ii) compressive strength, (iii) acid neutralisation capacity (ANC), and (iv) solubility of the metal contaminants as a function of pH.

The results of both mechanical and leaching tests showed the importance of the contaminant/matrix couple considered. Setting time was accelerated in presence of chromium, while in presence of manganese, lead and zinc it was delayed. However, for the last two contaminants, a 10% replacement of cement by silica fume and zeolite, markedly accelerated the setting time compared to the cement-only matrix. Although the early strength development was adversely affected in presence of all four contaminants, the long-term strength was less affected compared to the control materials. Although the ANC of the materials was not markedly affected by the presence of contaminants, the nature of the matrix did modify the ANC behaviour of the solidified materials. The increased strength and reduced ANC observed in the presence of silica fume are both due to pozzolanic reaction. The type of matrix used for solidification did not affect the solubility of the four metal contaminants. Overall, the results showed that the use of blended cements must be carried out with care and the performance assessment of waste-containing cement-based materials must take

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into consideration both the mechanical and leaching characteristics of the systems. © 2002 Elsevier Science B.V. All rights reserved.

1. Introduction

The disposal and recycling of industrial wastes is an important and complex issue. Hydraulic binders have been widely use to treat (stabilisation/solidification (S/S) process) or reuse (into concrete or road materials) waste materials. In theory, use of cementitious materials result in a durable matrix of low permeability and can significantly reduce the release of toxic species by providing an environment of high alkalinity, where many metal contaminants have a low solubility. In practice, however, there are complex interactions between waste components and binders that can lead to both short (delaying or inhibiting of the normal hydration reaction) and longer (release of heavy metals in groundwater) term difficulties. As an example, both lead and zinc salt additions are known to suppress cement hydration through the precipitation of protective coatings of gelatinous hydroxide, or related oxy-compounds, around the cement grains for as small amount as 0.15 wt.% [1,2]. Conversely, chromium has been found to accelerate the setting process [3].

Furthermore, blended cements have attracted intensive attention for their ability to enhance the physical and chemical properties of cement-based materials. Among them, materials possessing pozzolanic properties, such as silica fume and zeolite, play a special role. The reactive SiO_2 they contain reacts pozzolanically with the portlandite during the hydration of cement to form a stable cementitious compound, calcium silicate hydrate (CSH) [4]. Both materials, when substituted for cement at a level of around 10%, increase the compressive strength, decrease the pore size [5–7] and increase the corrosion resistance to acid solution [7,8]. Zeolites have been shown to increase the binding properties of cement-based materials for contaminants such as caesium [9]. Zeolite P, spontaneously formed in appropriate cement formulation, has been shown to have "reasonable" uptake characteristics for Pb^{2+} in a cement-like environment [10]. Addition of silica fume was shown to improve the leaching properties of solidified nuclear wastes [7] and produce more durable cement systems, owing to the changes caused by pozzolanic materials in the amount of the different hydrates, as well as in their chemical composition [4]. The substitution of silica fume, an expensive material, for natural zeolite and achieving the same environmental benefits would be of great interest.

Finally, the work presented here was aimed at obtaining a better understanding of the effect of industrial waste on cementitious systems. Previous work used foundry waste materials from industrial sources, which contained a range of metal contaminants [11]. Although it clearly appeared that the presence of wastes affected the mechanical and leaching characteristic of the materials, it was not apparent which contaminants were responsible for the effects. Therefore, the aim of this study was to examine the effect of four individual contaminants, namely chromium, manganese, lead and zinc, on the primary mechanical and leaching characteristics of cement-based materials. Three different matrices made of: (i) Portland cement, (ii) Portland cement and silica fume, and (iii) Portland cement and natural

zeolite, were investigated. The impact of the metal contaminants on the S/S materials characteristics was studied by measuring: (i) setting time, (ii) compressive strength, (iii) acid neutralisation capacity (ANC), and (iv) solubility of the pollutants of concern as a function of pH.

2. Materials and methods

2.1. Materials

Twelve cement-based waste materials were prepared for the study. The primary pollutants of concern, chromium, manganese, lead and zinc were introduced as metal nitrate salts. These elements were selected, as they are the main pollutants contained in industrial wastes that were previously studied [11]. Each metal was introduced to three different matrices (i.e. Portland cement, Portland cement and silica fume, and Portland cement and natural zeolite). The solidified waste materials were prepared with a water/binder ratio of 0.45, and a chromium, manganese, lead or zinc cation/binder mass ratio of 0.01. Silica fume and zeolite were substituted for Portland cement at a level of 10 wt.%. In addition, one control material was prepared for each of the three types of matrix. The calculated calcium/silica (C/S) ratio for the cement-only, cement/silica fume and cement/zeolite matrices were 3.23, 2.18 and 2.26, respectively.

The zeolite was size-reduced to 215 μm before the materials' preparation. The silica fume was provided as a slurry (50% water and 50% silica fume). The particle size and the specific surface were 0.1–0.2 μm and 15–20 m^2/g, respectively. The oxide composition and C/S ratio of the three binders are presented in Table 1. The composition of the zeolite was determined using X-ray fluorescence (XRF) and the manufacturers provided the silica fume and Portland cement compositions. The C/S ratio, which has an impact on the composition and amount of the main hydration products, was similar for the cement/silica fume and cement/zeolite matrices, while it was 30% higher for the cement-only matrix.

Materials were prepared according to the ASTM C305 method in 1982 for mixing paste, which had to be adapted to limit the formation of lumps. First, the metal nitrates were dissolved in de-mineralised water and the liquid phase placed in the mixing bowl. For the cement/silica fume matrix materials, the liquid phase consisted of both the metal solution and the silica fume slurry. The solid phase was poured onto the solution. For the cement/zeolite matrix materials, the solid phase consisted of a homogenised mix of cement and zeolite. After 30 s, the mixture was mixed at low speed (140 rpm) for 30 s. The mixer was then stopped to detach any paste from the sides of the bowl and the mixing started again at high

Table 1
Oxide composition (%) and C/S ratio of the Portland cement, silica fume and zeolite

Materials	SiO$_2$	Fe$_2$O$_3$	Na$_2$O	K$_2$O	CaO	MgO	Al$_2$O$_3$	SO$_3$	C/S
Cement	20.33	2.70	0.16	0.75	65.03	1.03	5.09	3.13	3.20
Zeolite	76.88	1.40	1.68	2.20	1.34	0.08	11.15	–	0.017
Silica fume	92	1.0	0.3	0.8	0.3	0.6	1.0	0.3	0.003

Table 2
Temperature and relative humidity recorded during the setting time tests

Type of matrix	Cement	Cement/silica fume	Cement/zeolite
Temperature (°C)	23	19	20
Relative humidity (%)	57	63	58

speed (285 rpm) for 1–3 min. This last period of mixing was variably increased, depending on the pollutant introduced and the type of matrix, to limit the amount and size of the lumps produced. Samples for compressive strength test were moulded in 50 mm cubes, vibrated for 15 s and stored at 100% relative humidity. After 1 or 3 days, depending on the setting time, the samples were removed from the moulds and stored at 100% relative humidity in sealed boxes until tested for compressive strength. Samples for other experiments were poured in plastic moulds kept sealed until the experiments were conducted. No carbonation, as checked with phenolphthalein, occurred before testing took place.

2.2. Micro-structural and mechanical characterisation of the materials

The setting time was determined according to ASTM C191-82 in 1982 by mean of a Vicat needle. All the materials were tested with the formulations described earlier rather than the standard consistency. It was considered advisable to proceed in this manner to obtain indications regarding the capability of the metal elements to accelerate or retard the cement setting time. The water/cement ratio adopted is rather high compared to that required when working at equivalent consistency, as envisaged by the standard. Nonetheless, this choice was prompted by the fact that certain pollutants, causing rapid solidification of the mix, would have interfered with the execution of the test. In addition, the temperature and relative humidity of the room where the test was conducted are recorded in Table 2 for the three different matrices. The initial setting time corresponds to the period of time between the preparation of the material and the moment when the needle penetrates the past by 25 mm. The final setting time is when the needle does not sink visibly into the paste.

Compressive strength was measured in triplicate on 5 cm cubes at 1 or 3, 7, 28, 56 and 90 days according to ASTM C109-87 in 1987. Depending on the strength of the material, a Denison or Farnell compression machine was used at a loading rate of 60 kN/min for samples tested at 1 and 3 days, 90 kN/min for samples tested at 7 days, 120 kN/min for samples tested at 28, 56 and 90 days.

2.3. Primary leaching characteristics of the materials

A batch equilibrium leaching test on size-reduced materials was used to determine: (i) acid neutralisation capacity of the material, and (ii) pollutant solubility as a function of pH. The test was conducted on all materials after 56 days of curing, according to the method developed at Environment Canada's Wastewater Technology Center [12].

Size reduction offsets long contact times and minimises the effect of mass transfer control on achievement of steady-state. All materials were ground to 150 μm, except for the cement/zeolite matrix materials, which were ground to 215 μm as the zeolite was reduced

to this size. The particle size difference should not affect the results as theoretical curves based on a diffusion model showed that 90% of steady-state concentration is reached after <24 h for a particle size of 300 μm [13]. Samples were crushed with a pneumatic drill until 100% of the material passed a 4 mm sieve. The crushed solid material was then ground in a ball mill, with seven 2 cm cylindrical sintered corundum balls, until greater then 95% of the initial sample mass had been size reduced to a maximum particle size of 150 and 215 μm for the cement/zeolite matrix materials. The grinding operation lasted for <2 h, which limited carbonation, recognised to greatly reduce the ANC of cement-based materials [14].

The ANC test involves contacting aliquots of finely crushed material with nitric acid solutions of varying concentrations to achieve a range of final pH values from 1 to 13. The liquid/solid (L/S) ratio is 6 ml/g of dry sample. After a contact time of 48 h under agitation, the leachates are filtered through 0.45 μm pore size polypropylene membranes and the leachate pH of each extract is measured. The filtered leachates are then preserved for chemical analysis, which was performed by ICP-AES.

3. Results and discussion

3.1. Effects of metal addition and type of matrix on materials' preparation

Generally, the consistency of the mixture was thickened in the presence of silica fume and zeolite by comparison to the cement matrix. This behaviour resulted from the high specific surface area of the silica fume and zeolite (15–20 m^2/g) compared to the cement (0.3–0.4 m^2/g), which increased water consumption. Although the zeolite and silica fume have comparable specific surface areas, the zeolite's is mainly represented by its high level of porosity where the silica fume's is mainly due to its very small particle size.

For all matrices, the presence of zinc highly increased the thickness of the mixture. Thomas et al. [2] and Arliguie and Grandet [15] measuring the heat of hydration, observed that the sharp highly exothermic peak deriving from the heat of wetting of the cement powder was increased in the presence of lead and zinc compared to control conditions. This increase was associated with the heat of formation of lead and zinc hydroxide gel. Therefore, the noticeable thickness of the mixture in the presence of zinc and not lead could be explained by the higher heat of zinc hydroxide formation (30% higher) compared to lead hydroxide. This hypothesis is simplified as it is based on phenomena observed with pure products, whereas in the case of lead, a rather variable mixture of lead hydroxide incorporating SO_4^{2-} and NO_3^- is formed [2]. Only, a detailed calorimetric investigation could explain this hypothesis. Although the mixing time was increased, small lumps were consistently observed for all the materials except for the controls.

3.2. Effects of metal addition and type of matrix on setting time

The effects of the contaminants and the type of matrix on the setting time were evaluated by comparing the initial setting time measured for each material, as illustrated in Fig. 1. The final setting time results are not considered here, as the results were varying too much. This is probably due to the fact that the test was run by different operators who can have a
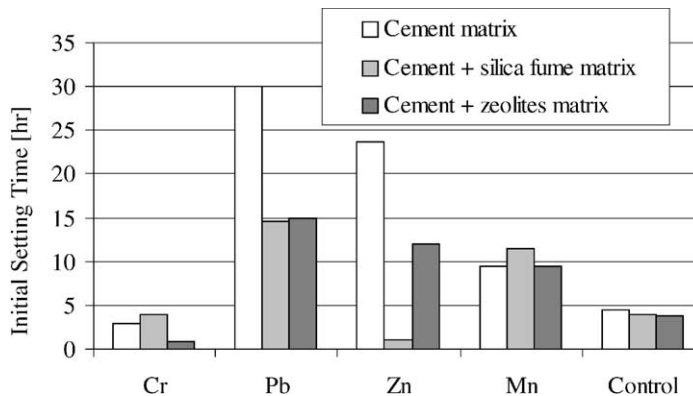
Fig. 1. Initial setting time for the solidified materials and control materials.

different perception about "the needle does not sink visibly into the paste", as described in the ASTM standard method. Initial setting time results are more consistent as they refer to a precise measurement (penetration of 25 mm).

The initial setting times of the control materials did not depend significantly on the type of matrix and vary between 3.7 and 4.5 h. Quality control data for the standard deviation was found to be 0.11 [16]. The same behaviour was observed in the presence of manganese, which nonetheless, increased the setting time by 100% compared to the controls. Conversely, in the presence of chromium, the setting was accelerated for the cement matrix, as already noticed by Ivey et al. [3] and even more for the cement/zeolite matrix. The effect of lead and zinc on the setting time is strongly dependent on the matrix type. For the cement matrix, both lead and zinc delayed the initial setting by 20–25 h. This result is consistent with the literature where both contaminants have been reported to suppress cement hydration through the precipitation of protective coatings of gelatinous hydroxide around the cement grains [1,2,17]. However, for the same contaminants, the setting took place 2–23 times more rapidly in the presence of zeolite and silica fume compared to the cement-only matrix. This is interesting behaviour as no noticeable difference was observed for the controls. Therefore, the mechanism by which both contaminants suppress cement hydration is affected by the presence of the two pozzolana studied. Arliguie and Grandet [18] and Thomas et al. [2] showed that the permeability of the coating formed on the cement grains depends on its colloidal fine structure and chemical composition. Any changes occurring in the colloidal structure of the coating, such as chemical conversion or crystallisation, could render it less effective after the initial retardation. The conversion of zinc hydroxide to crystalline calcium zincate in cement is an example of this effect. Therefore, the evolution of the hydroxide gel coating depends on different ions and their concentration in the pore solution. In the case of zinc-containing materials, the influence of the amount of C_3A and sulphate in the cement and concentration of Ca^{2+} and OH^- ions in the pore solution have been extensively studied [2,18,19]. In this study, the different species brought into solution by the silica fume and zeolite may form chemical compounds that could increase the permeability of the coating allowing the hydration of the cement particles. A study of the solution chemistry in the early

stage of hydration should be conducted to have a better understanding of the mechanism involved. Another hypothesis could be based on Lieber's work [20] who reported that for the same amount of admixture, the effectiveness of the coating depends on the surface area of the cement powder. Indeed, both silica fume and zeolite have a significantly higher specific surface area than that of the cement (up to 50 times higher [4]). Therefore, if we assume that the gel coating is going to form on those particles, this large amount of surface reduces the amount of coating on the cement grains and allows the cement hydration. Finally, one should note that the setting was most rapid for the zinc-containing cement/silica fume material. This result could be related to the thickening effect observed in presence of zinc during the materials' preparation and could well be a false set effect.

3.3. Effect of metal addition and type of matrix on compressive strength

Values of compressive strength for the control materials and pollutant containing materials are presented as a function of time in Fig. 2. Data are presented as the average of triplicates with error bar symbols to indicate the standard deviations. When comparing the compressive strength measured in the presence of pollutants, an important pollutant/matrix interaction is shown, as the strength behaviour of each couple pollutant/matrix was different from the one observed for the control materials. Indeed, the strength of the control materials was similar for the cement and cement/silica fume matrices, except at 1 and 90 days, and was constantly enhanced compared to the cement/zeolite matrix. In the presence of chromium, and taking into consideration the measurement ranges, the strengths of the materials were similar for the three different matrices, except at 1 and 90 days when the cement/silica fume matrix presented respectively a lower and higher strength compared to the two other matrices. In the presence of manganese and lead, the strength was the highest, at any time, for the cement/silica fume matrix. However, while in the presence of manganese the strength was higher for the cement matrix than for the cement/zeolite matrix, the opposite behaviour was observed in presence of lead. Finally, in the presence of zinc, after 56 days, the strength of the cement/silica fume matrix material became similar to the strength of the cement matrix material.

At 90 days, the presence of silica fume significantly enhanced the strength of all the materials, except in the presence of zinc. This observation suggests that the pozzolanic reaction, which consumes portlandite to form CSH, takes place over a long period [4]. Portlandite is a flake-like crystalline material, in which crystalline layers are bonded together by the very weak molecule chain bond force between layers and contributes little to the strength while the CSH gel-crystalline nets contribute to the strength. The contribution of zeolite to the strength was not shown for any of the materials, contrary to the information reported in the literature [6]. Firstly, this result could be due to the high water/binder ratio (0.45), which has been reported as a parameter decreasing the strength enhancement due to the presence of zeolites [6,21]. Secondly, the particle size of the zeolite used in this study (215 μm) decreased the specific surface area, which is known to play a role along with the chemical composition in the still imperfectly understood pozzolanicity mechanism [4].

One can notice that the zinc/cement/silica fume material presents a very low compressive strength, at 1 day, despite its very rapid setting, which could support the hypothesis of false
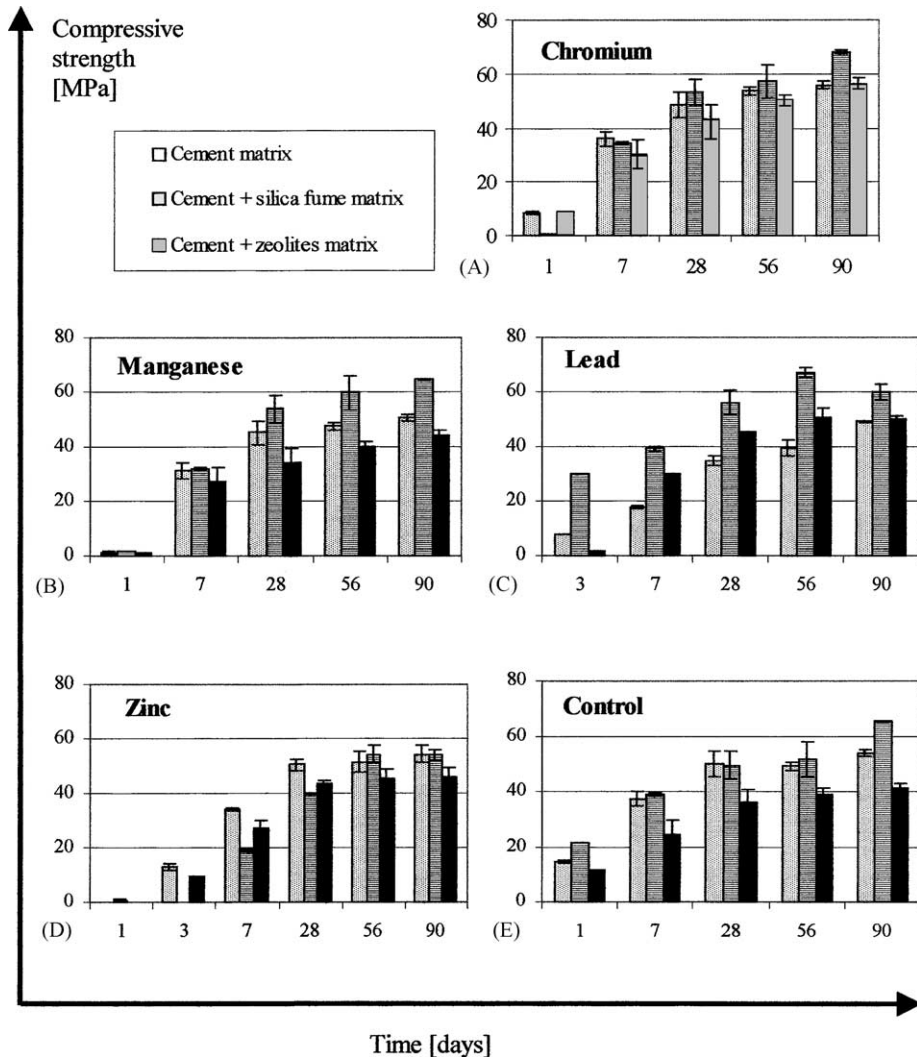
Fig. 2. Compressive strength at 1 or 3, 7, 28, 56, 90 days for the solidified materials in presence of (A) chromium, (B) manganese, (C) lead, (D) zinc, and (E) for the control materials.

set as previously suggested. It is only after 56 days that the strength of this material reached the same strength level as the cement-only matrix material.

When comparing, for each matrix, the strengths of the pollutant containing materials to those of their respective controls, general trends could be drawn (Fig. 3). At 1 day, for all matrices, the strength was drastically decreased in the presence of pollutants (by 40–100% compared to the control materials), except for the chromium/cement matrix material. However, for later measurements, most of the pollutant containing materials strengths ranged
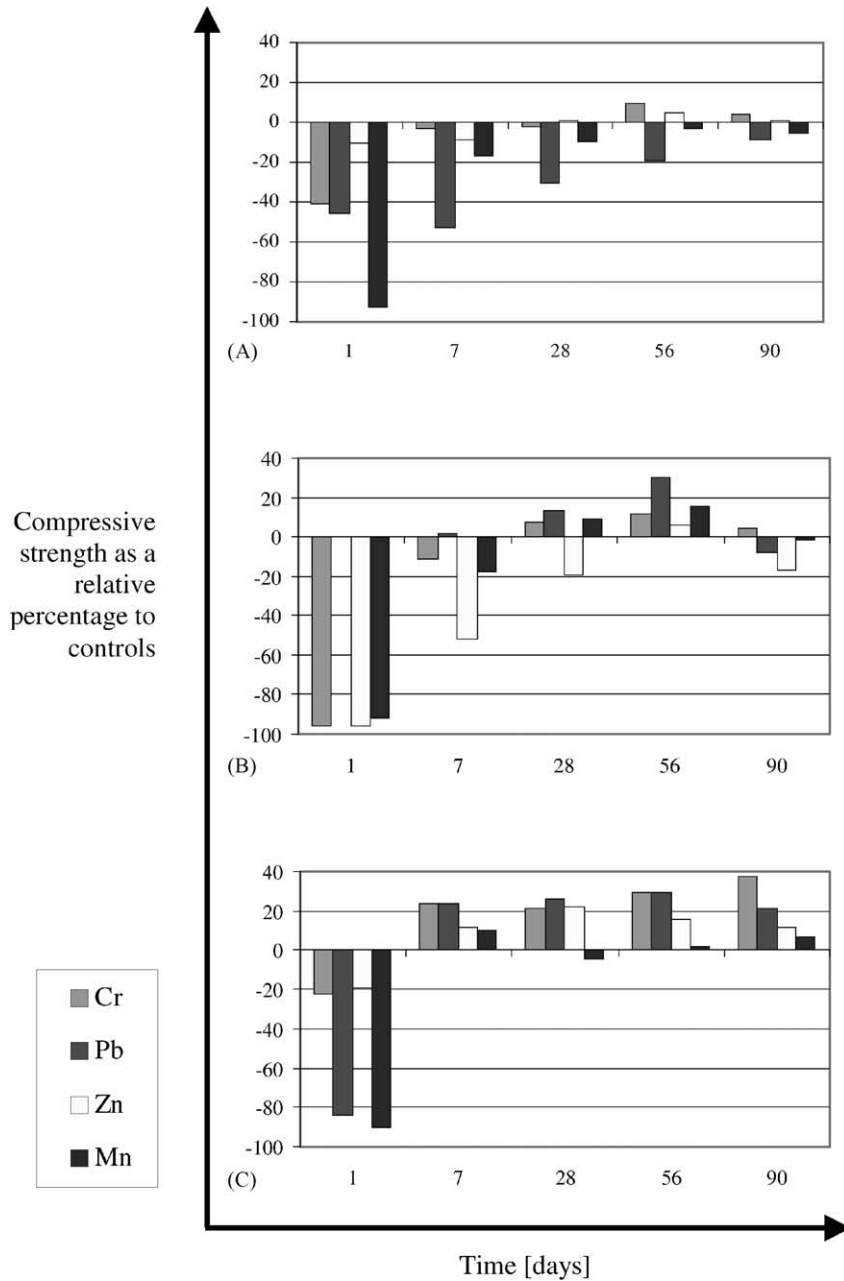
Fig. 3. Compressive strength as a relative percentage to controls at 1 or 3, 7, 28, 56, 90 days for (A) cement matrix, (B) cement/silica fume matrix, (C) cement/zeolite matrix.

±20% compared to the controls' strengths. Therefore, one can conclude that the presence of single metal pollutants at these levels seems to have little effect on the long-term strength development of cement-based materials.

3.4. Effect of metal addition and type of matrix on ANC

The acid neutralisation behaviour of the materials was evaluated by plotting the pH of each extract as a function of meq. of acid added per gram of dry solid as illustrated in Fig. 4.

It is clear from the ANC curves that 4 meq./g were needed for the cement and cement/zeolite matrices to reach pH 11.9, while <1 meq./g was necessary for the cement/silica fume matrix materials. It is noteworthy that pH 11.9 is the pH value corresponding to the total neutralisation of portlandite (i.e. the pH marking the transition between the couple $CSH/Ca(OH)_2$ and CSH rich in calcium and silica) [22]. This observation supports a higher rate of the pozzolanic reaction for the silica fume with the portlandite than for the zeolite. Indeed, Helland et al. [23] reported that for a 10% replacement of cement by silica fume, the portlandite content by weight of cement, after 52 days curing, was decreased to 18% compared to 30% measured for cement alone. In addition, Wu and Young [24] showed that, in C_3S–silica fume systems, the CSH formed directly by reaction between calcium hydroxide and silica fume had a slightly lower C/S ratio compared to the CSH formed by direct hydration of C_3S. However, from 8 meq./g onward, the ANC of the cement/zeolite matrix materials presented similar behaviour to that of the cement/silica fume matrix ones. For both matrices, the pH started to drop for 10 meq./g, while around 13 meq./g were necessary for the cement matrix materials (more specifically in the presence of chromium, the pH dropped from 12 and 14 meq./g for the control material). The beginning of the pH drop showed a decrease pH from 9.9 to 9, which has been identified as the threshold value for the stability of the major basic hydrates of a cement paste [25]. For lower pH, silica-rich CSH coexists with silica gel and a structurally stable matrix cannot exist [26]. Therefore, the cement/zeolite matrix materials presented an intermediate acid neutralisation behaviour, as for <8 meq./g, they showed a similar ANC to the cement matrix and confounding patterns with the cement/silica fume one when more acid was added. Based on the results obtained by Stegemann et al. [22], which illustrates the presence of different hydrates when the data are represented in a C/S versus pH graph, one can suggest that the cement/zeolite matrix materials could contain as much portlandite as the cement matrix material, but a lower amount of calcium-rich CSH. This result is supported by Atkins, who performed a series of experiments on $CaO–Al_2O_3–SiO_2–H_2O$ system and showed that zeolite P formed a stable assemblage with low ratio CSH [10].

For each type of matrix, comparison of the ANC curves of the pollutant-containing materials and the controls showed that the presence of contaminants slightly, but consistently decreased the buffering capacity of the materials when >8–10 meq./g of acid was added.

3.5. Effects of matrices on pollutant solubility as a function of pH

Chromium, manganese, lead and zinc concentrations of each extract were plotted as a function of the extract final pH to provide solubility or release as a function of pH (Fig. 5). The limits of detection of the instrument are represented by the dashed lines on the graphs.
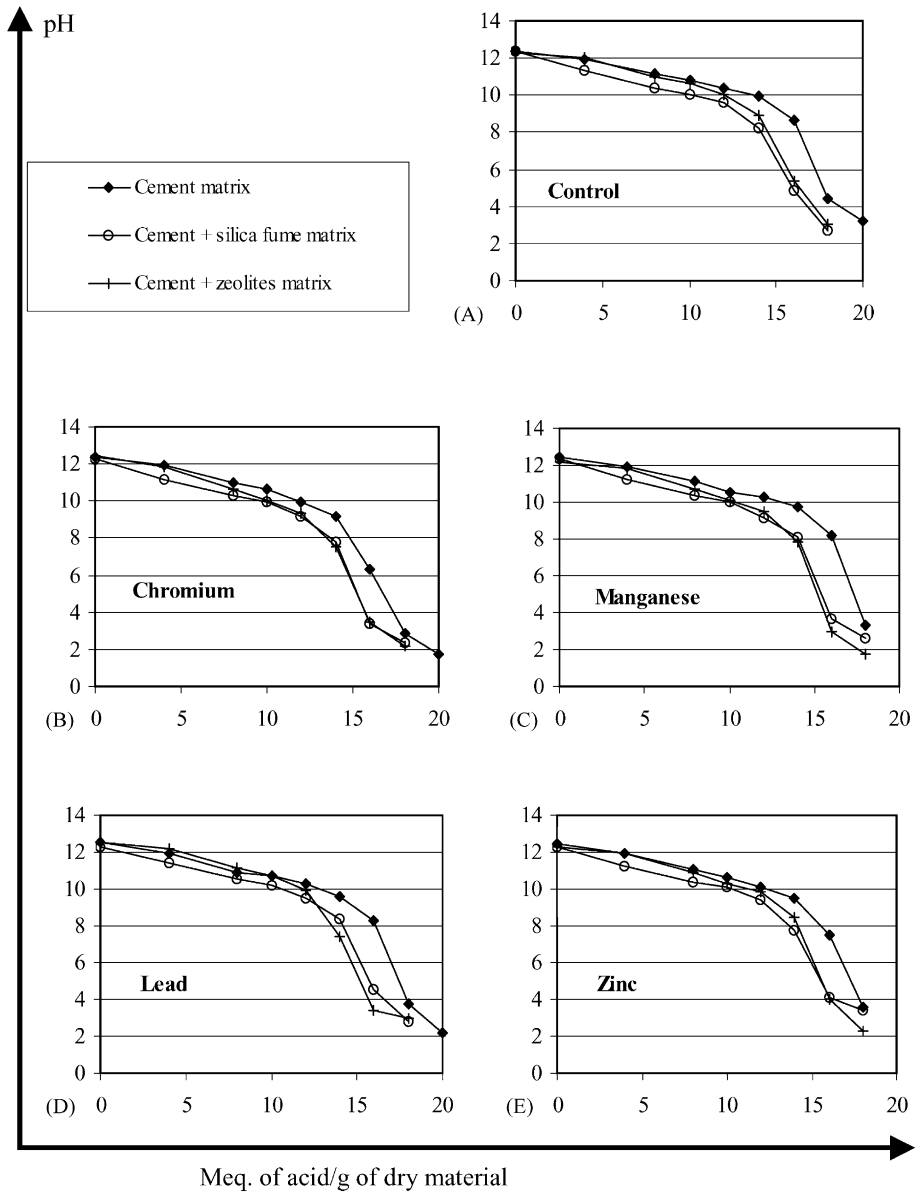
Fig. 4. Acid neutralisation capacity measured at 56 days for (A) control materials and for the solidified materials in presence of (B) chromium, (C) manganese, (D) lead, and (E) zinc.

The results showed that the pollutants' solubility was not affected by the type of matrices studied. Chromium solubility increased rapidly between pH 11 and 10, then remained constant pH from 10 to 5 (i.e. 10 mg/l), but increased again significantly for pH < 5. The amount of manganese in the leachates at pH > 8 was lower than the
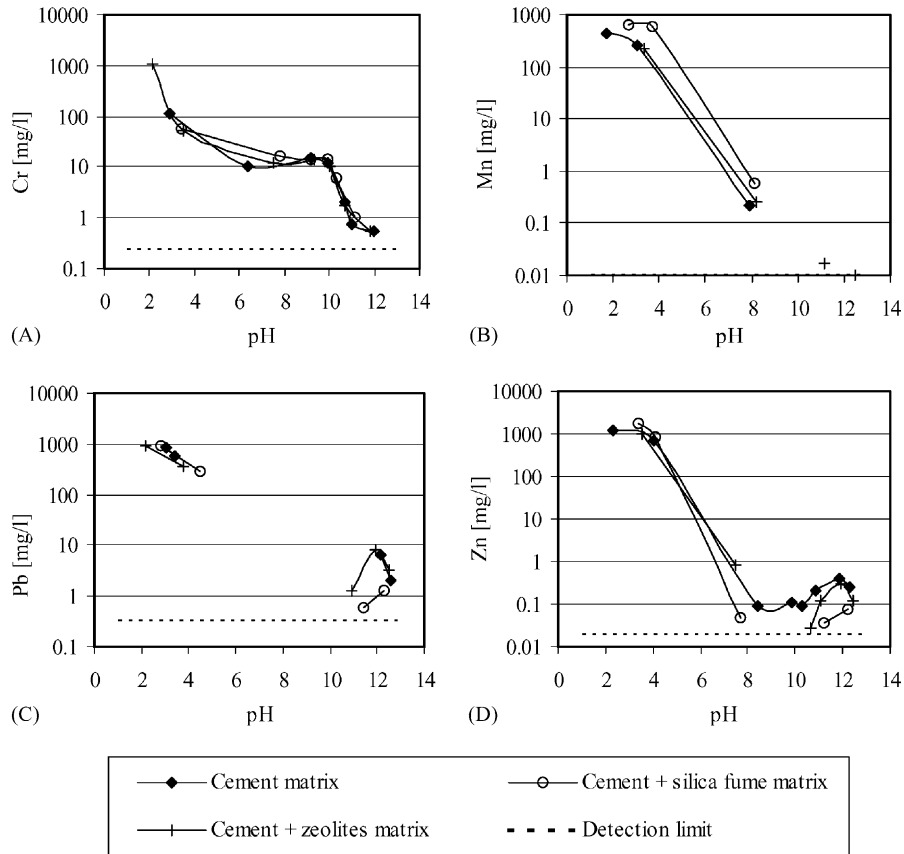
Fig. 5. Solubility as a function of pH of (A) chromium, (B) manganese, (C) lead, and (D) zinc, contained in the solidified materials.

detection limit (0.01 mg/l). For lower pH values, the solubility increased while the pH decreased. Lead and zinc presented classic amphoteric behaviour, with a minimum of solubility around pH 9–10 for the zinc systems. At very low pH, lead, zinc and manganese solubility seemed to be limited by the total content in the materials and did not reflect solubility.

No decrease of the pollutant solubility was observed for the cement/zeolite matrix despite the reported adsorption and ion-exchange properties of zeolite, which are used to remove heavy metals from contaminated effluents and soils [27]. Furthermore, as reported in the introduction, caesium-loaded ion exchange resins leachability has been successfully decreased using a zeolite/cement blend compared to cement alone [9]. In addition, an important and "reasonable" uptake capacity of, respectively, Cs and Pb by zeolite P in cement-like environment was reported [10]. Nonetheless, in this study, firstly, the zeolite and cement were added as an homogenised mixture to the polluted solution and most probably the Ca^{2+} ion liberated during hydration as well as readily soluble Na^+ and K^+ out-competed

the heavy metals for exchange sites within the zeolite structures [27]. Secondly, as Atkins et al. [10] pointed out, if zeolites become totally consumed through pozzolanic reaction, the compounds formed could have a much lower or even non-existent sorption efficiency. However, the former hypothesis is more probable than the later, as in this study, the strength of the materials prepared with zeolites was inferior to the cement-only materials suggesting that not all the zeolite have been consumed.

4. Conclusions

Analysis of the effects of four metal nitrate contaminants on the mechanical and leaching characteristics of three different type of matrices demonstrated the importance of the contaminant/matrix couple considered.

The effect on the initial setting time was found to depend on the pollutant considered, it was accelerated in the presence of chromium and was noticeably delayed in the presence of manganese, lead and zinc. In addition, in the presence of lead and zinc, the setting times of the materials prepared with zeolite and silica fume were noticeably lower compared to the cement-only matrix. Early strength development was found to be adversely affected in the presence of all four contaminants, however, the long-term strength appeared to be less affected in presence of the single metal contaminants studied. From 56 days of curing onwards, a 10% replacement of cement by silica fume was proven to increase the strength of all materials. Conversely, the zeolite replacement reduced the strength compared to the cement-only matrix which is could be due to the high water/binder ratio [6,14]. Whereas the ANC of the materials was not significantly affected by the presence of contaminants, the nature of the matrix did modify the acid neutralisation behaviour of the materials. Indeed, in the presence of both zeolite and silica fume, the ANC of the materials was decreased compared to the cement-only matrix. Materials prepared with zeolites exhibited a higher ANC than materials prepared with silica fume (Fig. 4A). The increased strength and reduced ANC, observed in presence of silica fume, are both due to the pozzolanic reaction, which consumes portlandite to form CSH. Indeed, owing to their composition and structure, portlandite contributes to ANC while CSH contributes to strength. The type of matrix did not affect the pollutant solubility.

In view of the reuse and disposal of waste containing materials, both strength and ANC are important parameters. This study showed that the effect of the matrix on the long-term behaviour, is predominant and can conversely affect strength and ANC as observed for the cement/silica fume matrix. Therefore, the use of blended cements must be carried out with care and the performance assessment of waste-containing cement systems must take into consideration both the mechanical and leaching characteristics of the final solidified products.

in Cement/Waste Systems (NNAPICS), involved seven other partners namely, Imperial College, University of Cantabria, University of Roma, Trinity College Dublin, British Nuclear Fuels Limited, Euroresiduos S.A. and GE-SE-NU.

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