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EFFECT OF UNIAXIALLY PRESSING ORDINARY PORTLAND CEMENT PASTES CONTAINING METAL HYDROXIDES ON POROSITY, DENSITY, AND LEACHING

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ABSTRACT

Synthetic metal hydroxide wastes containing Zn and Pb have been mixed with partially hydrated cement and uniaxially pressed. The effect on porosity, pore size distribution, and bulk and skeletal densities has been characterised using mercury intrusion porosimetry. $Ca(OH)_2$ formation has been determined using differential thermal analysis and metal leaching has been assessed in a series of static leach tests completed on monolithic samples. Pressed solidified materials have increased density, reduced porosity, and reduced $Ca(OH)_2$. They exhibit increased resistance to acid attack in terms of sample weight loss during leaching due to reduced release of alkalis. Leaching of Zn and Pb is primarily determined by pH. A peak observed in Zn leaching from pressed samples is due to the effect of changing leachate pH on the dominant Zn species present. *© 1998 Elsevier Science Ltd*

Introduction

Ordinary Portland cement (OPC) is used to solidify metal-containing industrial wastes, primarily to reduce leaching of hazardous components. The monolithic nature of the solidified material reduces the surface area available for leaching and the low permeability matrix effectively encapsulates the waste. Metal ion leachability is controlled by solubility, diffusion, and advection transport rates through connected porosity. Cement-solidified wastes also produce a pH environment in which some metals have low solubilities. Other interactions between wastes and cementitious binders, including metal sorption on silica-rich layers, are also reported to affect metal leaching rates (1).

Cementitious materials with reduced porosities have lower permeabilities and normally are more durable. It therefore seems probable that solidified wastes with reduced porosities also will show performance improvements. Cement paste porosity depends critically on the initial water-to-cement (w/c) ratio and the extent of cement hydration and the relationship between porosity and processing of cement pastes has been extensively investigated (2–10).

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Various processing techniques have been used to reduce total porosity and improve the properties of cement pastes. These have included high-pressure pressing, vibrational compaction, autoclaving, and hot pressing. Use of low w/c ratios combined with cement-dispersing organic compounds and high shear mixing techniques produced "macrodefect-free" cement that had extremely low porosities and high strengths (9). High-density, high-strength cement pastes also have been produced in DSP (densified with small particles) cements. These contain controlled additions of silica fume to produce high-strength, low-porosity materials (11–13).

High-pressure fluid extraction techniques have been applied to very young cement pastes to form "pore-reduced cements" (PRCs) (14–16). These can have compressive strengths approaching those of macrodefect-free cement. Microstructural development in PRC has been studied using scanning electron microscopy, x-ray diffraction, and mercury intrusion porosimetry (MIP). The mineralogy appears similar to that of unpressed cement, except they contain increased amounts of dense hydrate, which produces a shift to lower pore sizes. $Ca(OH)_2$ content in PRC is reported to be reduced relative to normal unpressed cement pastes, and ettringite does not form in significant quantities. Durability and microstructural studies show that PRC has improved resistance to chemical attack compared to normal cement pastes, except in sulfuric acid media, and PRC exhibits improved wet/dry cycling.

Processing cement-solidified wastes to produce more dense monolithic materials with reduced porosity is expected to improve long-term durability and leaching properties. Of the processing techniques used to improve cement pastes, uniaxial pressing is probably the most appropriate method to produce high-density, low-porosity, cement-solidified wastes.

In this work, processing techniques similar to those developed for PRC cements have been used on OPC containing synthetic Pb and Zn wastes. These particular metal wastes have been chosen because they have very different effects on cement hydration. The Zn waste causes severe retardation of hydration reactions, whereas the Pb waste causes a delayed hydration effect (17).

The effect of uniaxially pressing on leaching from these materials has been assessed by suspending monolithic samples in an acidic leachant. Leaching under these conditions has previously been described using a shrinking core/exposure model (18). Cement-solidified materials with cement-to-waste ratios of 10:1 have been uniaxially pressed, MIP has been used to characterise bulk and skeletal density, total porosity, and pore size distributions, and $Ca(OH)_2$ formation has been determined using differential thermal analysis (DTA).

Experimental

Materials

Two synthetic waste sludges were prepared by precipitating Pb or Zn from 1M metal nitrate solutions by adding 10M NaOH to pH 9.5 (SLR grade reagents, Fisons plc). The sludges were vacuum filtered through a No.1 filter paper (Whatman Ltd.), dried overnight at 105°C, ground to $<500\ \mu m$, and stored in sealed glass bottles. OPC supplied by Blue Circle Ltd. to BS12 class 42.5N was used in all the solidification experiments.

TABLE 1
Properties of pressed and unpressed cement solidified wastes.

Sample	Initial w/s	Pressing pressure (MPa)	Final w/s	Bulk density (g cm^{-3})	Skeletal density (g cm^{-3})	Porosity (%)
OPC	0.4	0	0.4	1.63	2.24	27.3
		16	0.21	2.02	2.43	16.7
		32	0.19	2.12	2.51	15.5
	0.5	0	0.5	1.46	2.21	33.9
		16	0.25	1.89	2.33	18.8
		32	0.22	1.95	2.41	18.8
OPC/Pb	0.4	0	0.4	1.70	2.28	25.6
		16	0.19	2.14	2.51	14.6
		32	0.17	2.21	2.55	13.3
	0.5	0	0.5	1.55	2.27	31.7
		16	0.22	2.03	2.42	15.8
		32	0.20	2.12	2.50	15.3
OPC/Zn	0.4	0	0.4	1.45	2.92	50.2
		16	0.19	2.03	2.55	20.5
		32	0.16	2.10	2.56	18.0
	0.5	0	0.5	1.33	2.87	57.2
		16	0.22	1.91	2.41	20.8
		32	0.19	1.99	2.43	18.1

Preparation of Uniaxially Pressed Solidified Wastes

The following procedure was used to form pressed cements containing Pb and Zn:

1. OPC was thoroughly mixed with water at w/c ratio of 0.44 and allowed to hydrate for 4 h. Mixes with a w/c of 0.55 also were prepared and hydrated for 6 h.
2. The synthetic Pb and Zn wastes were thoroughly mixed into the partially hydrated OPC pastes to give a cement-to-waste ratio of 10:1 and an initial water-to-solids (w/s) ratio of either 0.4 or 0.5.
3. For each mix formulation a constant mass was transferred to a cylindrical stainless steel dye (internal diameter 20 mm) and uniaxially pressed using a Wykham Farrance hand-operated compression apparatus, at either 16 or 32 MPa. The maximum load was retained on the sample for 1 min before being released, during which time a small amount of fluid was expelled from the sample.
4. Pressed samples were then weighed, wrapped in cling film, and left to hydrate in sealed plastic bags for 28 days. The change in weight during pressing due to fluid loss was used to determine the approximate final w/s ratio of pressed samples.
5. Pressed cement paste samples with initial w/c ratios of 0.4 and 0.5 that contained no waste additions were similarly prepared.

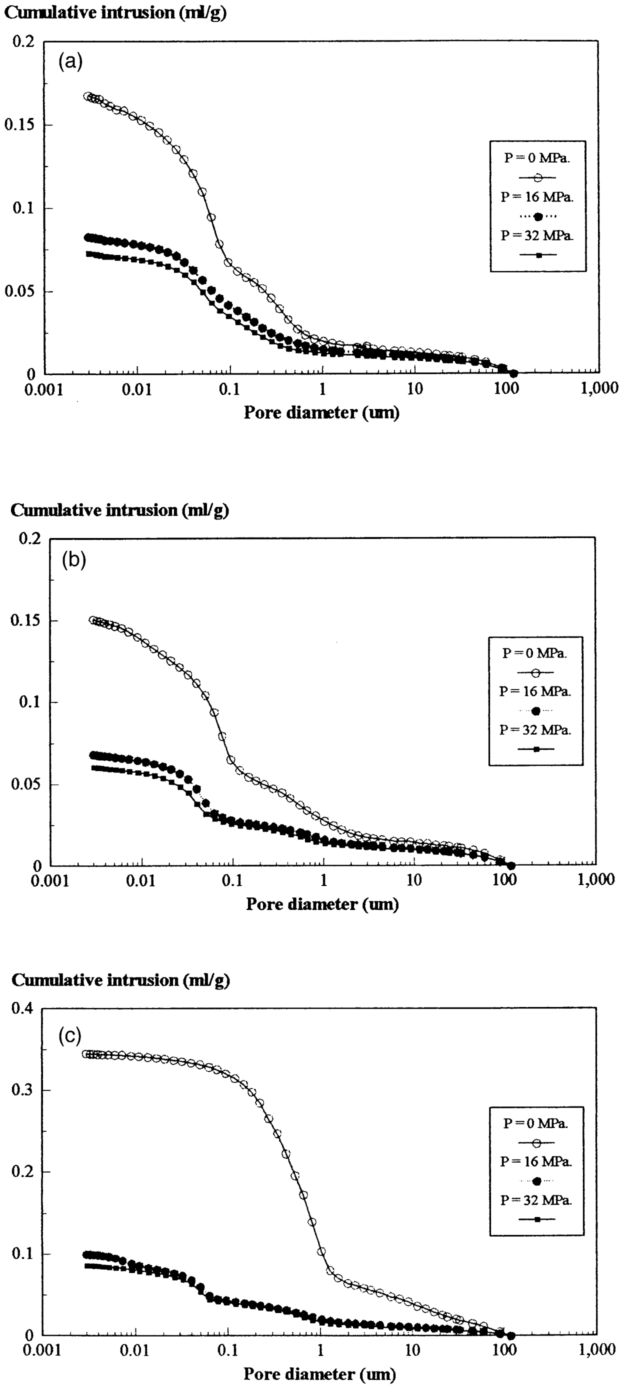

FIG. 1.

Cumulative intrusion volume versus pore diameter of samples (w/s = 0.4). (*a*) Control OPC; (*b*) OPC/Pb; (*c*) OPC/Zn.

TABLE 2
$Ca(OH)_2$ formation in pressed and unpressed solidified waste samples.

		% $Ca(OH)_2$	
		Initial w/s = 0.4	Initial w/s = 0.5
OPC	0	14.4	16.4
	16	10.4	11.1
	32	9.7	10.6
OPC/Pb	0	12.5	16.4
	16	6.4	9.5
	32	5.9	7.2
OPC/Zn	0	n.d.	n.d.
	16	3.8	4.7
	32	3.8	5.4

n.d. = not detected.

In addition, unpressed OPC and OPC solidified wastes with the same waste loadings as the pressed samples were prepared by mixing components to give samples with w/s ratios of 0.4 and 0.5.

The purpose of the prehydration in stage 1 was to allow the cement to partially hydrate under normal conditions and ensure uniform distribution of the excess mixing water. Within this range of prehydration periods it is reported that additional mixing does not significantly affect the final strength of cement pastes (19). Solution chemistry studies have shown that the soluble concentrations of Pb and Zn present in the mix water expelled during pressing would have negligible effect on the total metal content present in the solidified material (17).

MIP

After 28 days, hydration samples were crushed and sieved to give a granular material with particle sizes between 2 and 4 mm. This was dried overnight at 105°C and used to determine bulk density, skeletal density, and porosity levels using MIP (Micromeritics Autopore II 9220), where the skeletal density is defined as the ratio of sample mass to the volume actually occupied by that mass. The pore diameter was calculated from the applied pressure assuming cylindrical pores, to give cumulative mercury intrusion volume versus pore diameter data for meso pores (diameters 25 to 50 nm) and macro pores ($>$50 nm) with diameters up to 118 μm. A 0.392-mL stem volume calibrated penetrometer was used. The amount of sample for MIP analysis varied from 1.5 g for the pressed samples to 1.0 g for unpressed samples. Every fourth sample was analysed in duplicate to confirm reproducibility of the MIP data.

DTA

The development of $Ca(OH)_2$ in pressed and unpressed cement/metal waste samples hydrated for 28 days was investigated using DTA (Polymer Laboratories PL-STA) and

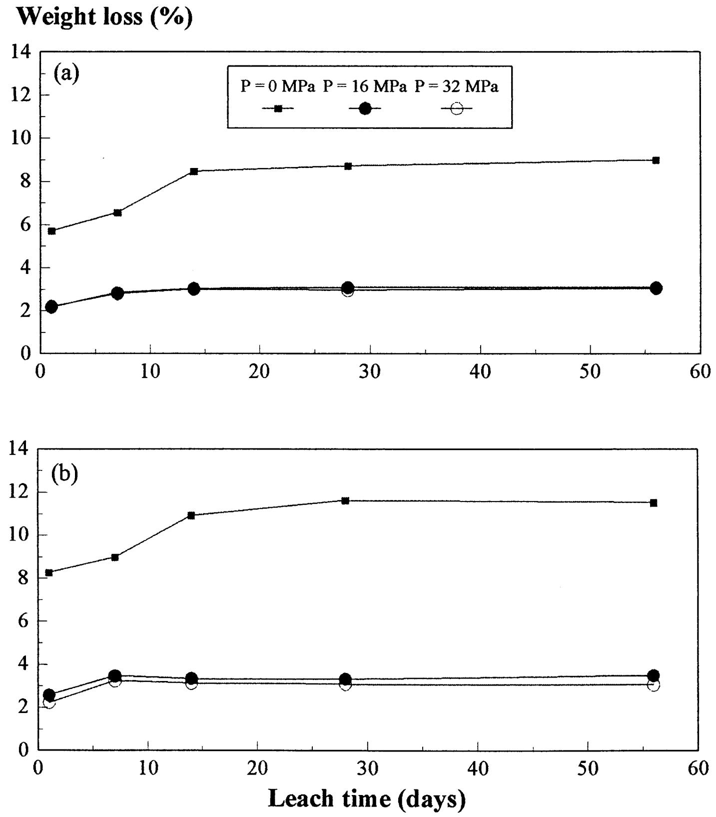

FIG. 2.

Weight loss for pressed and unpressed (*left*) OPC/Pb and (*right*) OPC/Zn samples. (*a*) Initial w/s = 0.4; (*b*) initial w/s = 0.5.

used to indicate the extent of hydration (17). Fifteen milligram subsamples, ground to <150 μm, were heated to 600°C in alumina crucibles under a N_2 atmosphere at a rate of 5° C min^{-1}. Under these conditions $Ca(OH)_2$ dehydrates at temperatures between approximately 370°C and 430°C. DTA data were calibrated by analysing varying amounts of analytical grade $Ca(OH)_2$ mixed with Al_2O_3, which gave a linear relationship between the area under the DTA peak and the amount of $Ca(OH)_2$ in the mix.

Leach Testing

The effect of uniaxially pressing solidified wastes on metal leaching has been assessed using a static leach test on OPC, OPC/Pb, and OPC/Zn samples hydrated for 28 days. Cylindrical samples were suspended in a plastic net in 0.05N acetic acid solution, at a leachant volume to surface area ratio of 20:1. The initial pH of the acetic acid leachant was 3.15 and the initial weight and dimensions of each sample were recorded.

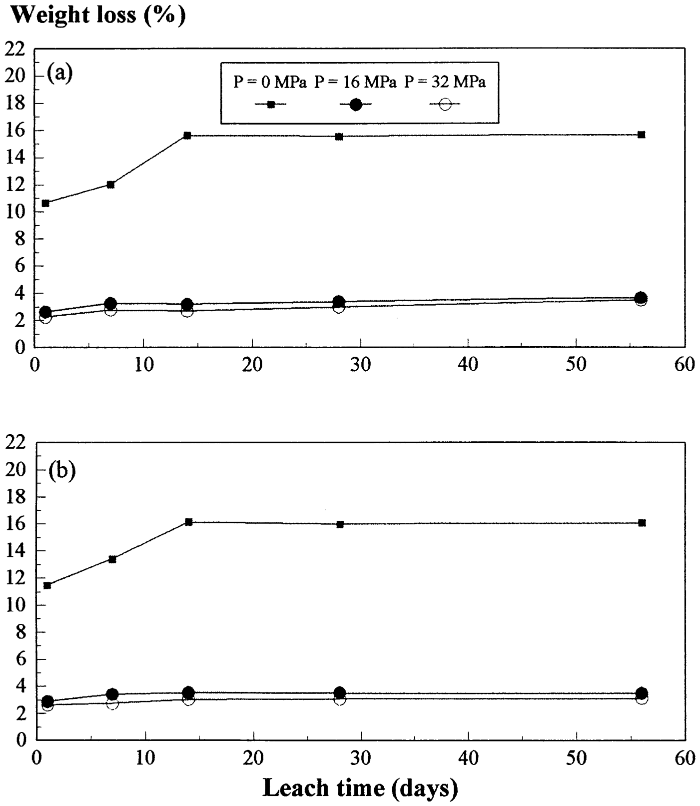

FIG. 2.
Continued

Samples were leached for 1, 7, 14, 28, or 56 days without renewing the leachant during the leaching period. Tests were completed in duplicate to confirm reproducability of the data. Leachates were then filtered through a 0.45-μm cellulose nitrate filter paper and the pH determined prior to acidification to 10% HNO_3 (Aristar, BDH) for metal analysis by ICP-AES. Leached samples were dried overnight in air and weighed.

Results

Effect of Pressing on Porosity

Bulk and skeletal density, porosity (%), and final w/s values of all samples are shown in Table 1. Pressing at 16 MPa reduced the w/s to less than half its initial value for samples prepared with initial w/s = 0.4 and 0.5. Increasing the pressing pressure to 32 MPa further reduced the final w/s ratio. Bulk densities of OPC samples and cement solidified wastes containing Pb increased significantly with increasing pressing pressure as the cement and waste become increasingly densely packed. This resulted in very significant reductions in porosity levels. Skeletal densities

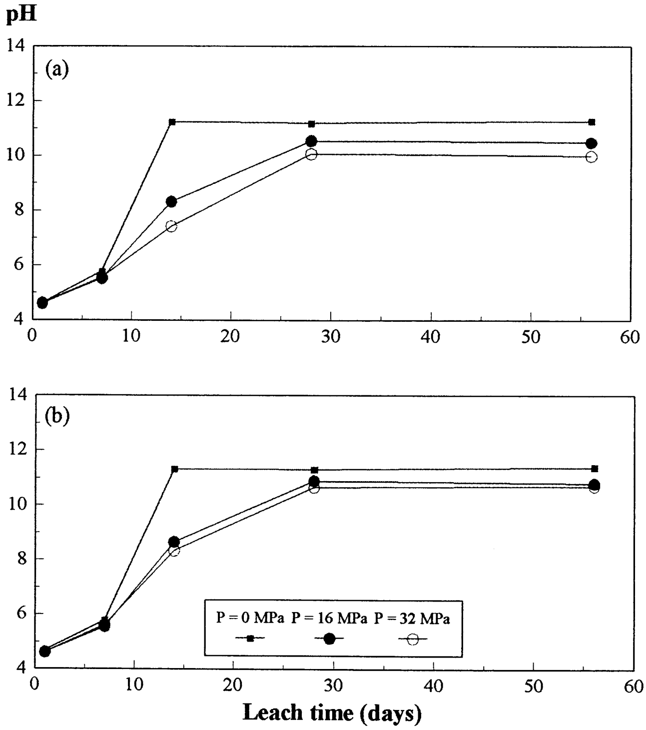

FIG. 3.
Leachate pH data for pressed and unpressed (*left*) OPC/Pb and (*right*) OPC/Zn samples. (*a*) Initial w/s = 0.4; (*b*) initial w/s = 0.5.

also increased as greater pressing pressures were applied, indicating either formation of dense hydration products or the presence of increased amounts of unreacted cement clinker phases.

Very different skeletal density results were obtained for the OPC/Zn samples. The Zn waste is known to cause severe retardation of OPC hydration. For the samples in which the waste was added at the start of cement hydration, i.e., the unpressed samples, the cement clinker particles remain largely unhydrated and form a highly porous structure, as limited hydration products are generated to fill the voids occupied by mix water. Skeletal densities were high because the solid volume consists primarily of high-density, unhydrated cement clinker particles.

For pressed OPC/Zn waste samples prepared with w/s = 0.4 and 0.5, the cement was allowed to prehydrate before the Zn waste was added, allowing limited formation of lower density hydration products. The result of subsequent pressing was significantly increased bulk density and reduced porosity but also reduced skeletal density due to initial cement hydration.

Pore Size Distribution

Figure 1 shows cumulative intrusion volume versus pore diameter data for meso- and macropores in pressed and unpressed OPC and OPC/Pb and OPC/Zn mixes. Data are

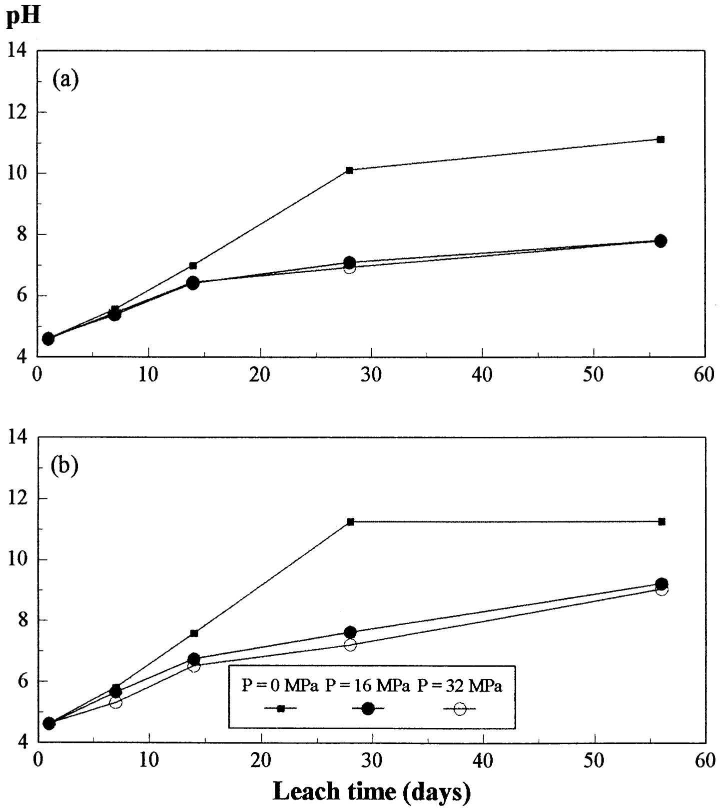

FIG. 3.
Continued

presented for samples with initial w/s = 0.4. Similar data were obtained for the w/s = 0.5 samples except that intruded volumes were marginally higher.

The results clearly show a significant reduction in cumulative intrusion volume in pressed compared to unpressed samples. There was a reduction in the volume of macropores in the pressed solidified wastes, particularly for the OPC/Zn samples. Increasing the pressing pressure from 16 to 32 MPa produced a small additional reduction in cumulative intrusion volume at a given pore diameter.

$Ca(OH)_2$ Formation

$Ca(OH)_2$ formation shown in Table 2 was reduced in the pressed OPC and OPC/Pb samples. This results from the low water content, which limits the extent of cement hydration, Ca depletion resulting from pressing, and the reduced void space in which $Ca(OH)_2$ crystals can form. The low $Ca(OH)_2$ levels in OPC/Zn are due to severe inhibition of cement hydration.

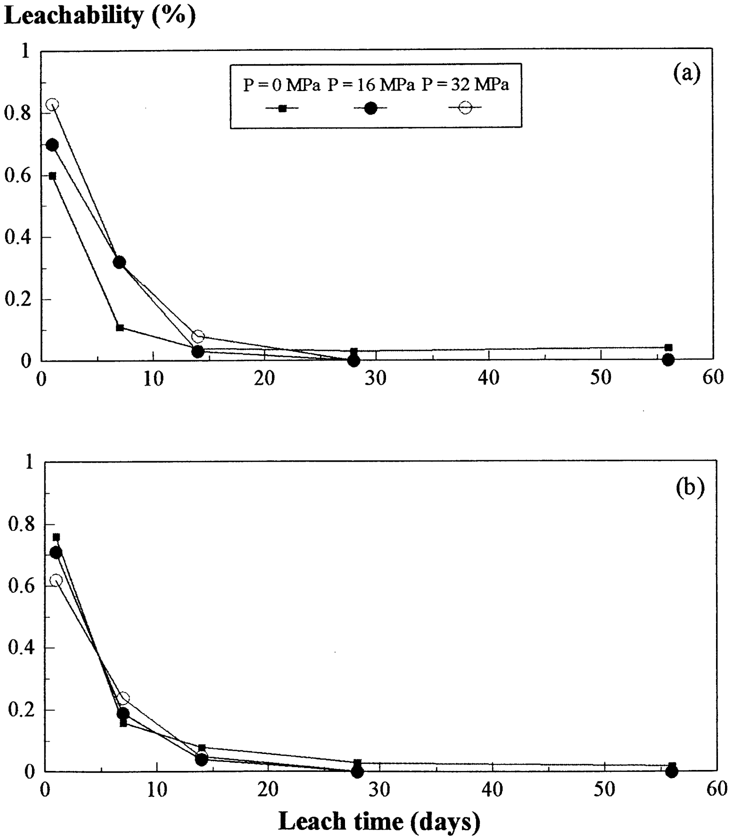

FIG. 4.
Leachability of Pb and Zn from pressed and unpressed (*left*) OPC/Pb and (*right*) OPC/Zn samples. (*a*) Initial w/s = 0.4; (*b*) initial w/s = 0.5.

Static Leach Test Results

Weight loss data during leaching for the OPC/Pb and OPC/Zn samples with initial w/s = 0.4 and 0.5 are presented in Figure 2. Weight losses for the 0.5 w/s samples tend to be slightly higher, especially for the unpressed samples. There is a reduction in weight loss from pressed compared to unpressed samples. Weight loss occurs primarily during the first few days in pressed samples whereas for the unpressed samples it continues over at least the first 14 days. Weight losses for unpressed OPC/Pb samples were significantly lower than those for unpressed OPC/Zn samples and all pressed samples showed similar weight losses.

Leachate pH data for OPC/Pb and OPC/Zn samples tested in the 0.05N acetic acid leachant are shown in Figure 3. The leachate pH of unpressed OPC/Pb samples increases rapidly so that, after 14 days, it had reached approximately 11. The leachate pH of pressed samples increases more slowly, and for OPC/Pb plateaued at values somewhat below 11 after 30 days. Similar results were obtained for pressed OPC. Samples containing Zn produced very

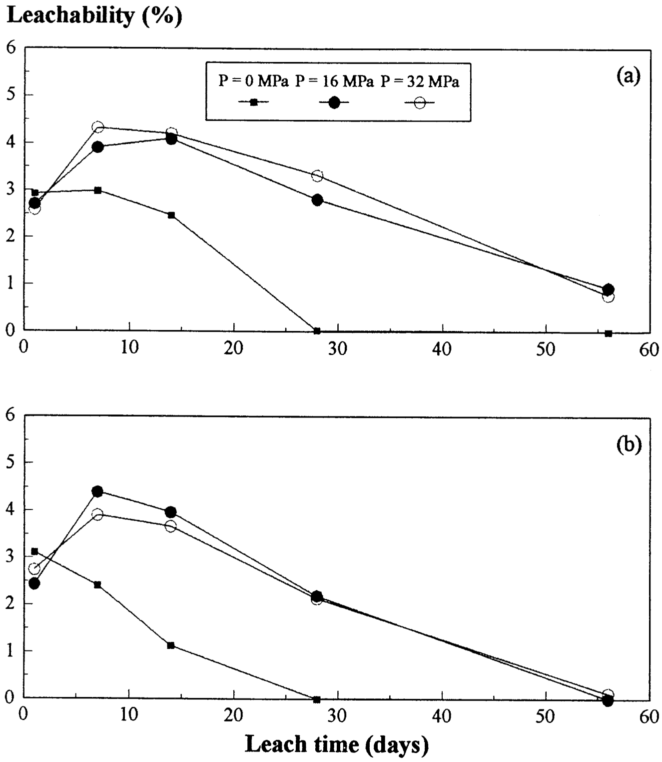

FIG. 4.
Continued

different leachate pH data. The increase in pH was retarded, particularly for pressed samples that only reached pH values of 7 to 8 after leaching for 56 days.

Pb and Zn leachability data, expressed as a percentage of the total metal present in the samples leached, are given in Figure 4. The percentage of Pb leaching is low and decreases throughout the duration of the test for all samples. Zn leaching from pressed samples shows an initial increase during the first 10 days but then decreases after longer exposure times. The leachability of Zn from the unpressed solidified waste gradually decreases to below detection levels after 28 days of testing.

Table 3 shows leachate concentrations of Ca, Na, and K determined by ICP-AES for pressed and unpressed OPC, OPC/Pb, and OPC/Zn samples. All other metals were at much lower concentrations. The data clearly indicate the increased release of these metals with leaching time and reduced release resulting from uniaxially pressed samples.

Discussion

The effect of uniaxial pressing during the formation of cement pastes containing metal hydroxide wastes on metal leaching has been investigated using a static leach test. The test

TABLE 3
Concentrations of Ca, Na, and K in leachates from pressed and unpressed samples leached in 0.05N HAC.

		Leach time (days)				
		1	7	14	28	56
Ca (mg/L)						
OPC	0 MPa	507	738	856	914	947
	16 MPa	494	721	843	900	937
	32 MPa	483	706	799	875	911
OPC/Pb	0 MPa	508	751	880	939	995
	16 MPa	501	736	845	915	945
	32 MPa	461	701	795	879	920
OPC/Zn	0 MPa	431	753	846	887	907
	16 MPa	452	729	806	837	869
	32 MPa	392	660	755	800	838
Na (mg/L)						
OPC	0 MPa	3.8	10.6	13.2	15.8	17.6
	16 MPa	3.1	5.7	8.1	9.2	10.5
	32 MPa	2.4	4.3	6.7	7.6	8.2
OPC/Pb	0 MPa	15.2	30.5	40.9	43.1	43.2
	16 MPa	6.9	14.0	17.5	20.9	24.3
	32 MPa	6.6	11.2	13.9	16.7	18.9
OPC/Zn	0 MPa	85.1	141.0	159.5	176.7	192.0
	16 MPa	40.9	65.1	78.7	99.0	113.2
	32 MPa	32.0	49.4	64.6	84.0	95.1
K (mg/L)						
OPC	0 MPa	32	65.7	73.2	84.8	93.2
	16 MPa	14.7	30.3	44.6	47.0	51.1
	32 MPa	11.7	24.0	31.3	33.0	38.5
OPC/Pb	0 MPa	43.3	80.0	85.7	90.1	98.0
	16 MPa	17.2	33.2	40.4	47.0	54.3
	32 MPa	14.6	27.2	30.9	36.0	42.8
OPC/Zn	0 MPa	46.4	72.3	82.5	90.7	99.6
	16 MPa	23.7	43.5	49.7	55.4	56.5
	32 MPa	19.0	33.56	43.7	50.0	53.6

allows monolithic samples to reach a steady state with a finite amount of acid solution, and the results illustrate the development of this steady state over time.

Metal release is determined by chemical interactions between the material and the leachant and by the microstructural properties of the material. The resistance of a cement matrix to acid attack depends on porosity, tortuosity, and the solubility and acid neutralisation capacity of the matrix components (20,21). In particular, acid neutralisation capacity is reported to have a major influence on the rate of contaminant leaching.

Uniaxially pressing partially hydrated cement with Pb and Zn wastes significantly increases the solidified waste bulk densities and reduces levels of meso- and macroporosity.

The pressing pressure forces the cement and waste together and physically reduces void space. The water content is reduced, and this reduces porosity, especially when hydration reactions are limited. In pressed samples the final w/s ratios were below the stoichiometric value required for complete hydration of the cement clinker and, therefore, reduced porosity primarily results from the effect of physical compaction. Uniaxially pressing also has the effect of reducing the amount of $Ca(OH)_2$ in hydrated samples.

OPC and OPC/Pb solidified wastes exhibited broadly similar behaviour in terms of density, porosity, and $Ca(OH)_2$ content for particular treatments and produced similar weight loss and leachate pH data.

Sample pressing had a significant effect on both microstructure and leaching properties. When solidified wastes are exposed to acid leachants, hydrogen ions diffuse into the capillary porosity. This changes the chemical equilibrium of the pore water, causing dissolution of available $Ca(OH)_2$, as seen in Table 3, and consumption of acid. The increased weight loss of unpressed samples can be attributed to increased $Ca(OH)_2$ dissolution and matrix decalcification (21). The lower levels of $Ca(OH)_2$ in pressed OPC and OPC/Pb explains the slower attainment of steady-state pH values and reduced weight loss.

Weight loss occurred predominantly during the first 7 to 14 days, which suggests a surface effect related more to the geometrical surface area of samples rather than to porosity. For unpressed samples, sufficient alkali material dissolved to completely neutralise the acetic acid leachant and the pH rapidly increased to steady-state levels. The rise in pH for the pressed samples continued beyond the initial weight loss period. In this pH range the titration curve for acetic acid is steep and, therefore, small diffusional releases of alkalis can produce significant changes in solution pH.

The leachate pH under steady-state conditions is a function of the leached alkali species and the surface chemistry of the exposed monolith matrix (21). The lower final pH of pressed samples may reflect the formation of C-S-H, with lower Ca/Si mole ratio resulting from Ca^{2+} losses during the pressing process (20).

Pressing also has the effect of increasing the concentration of metals per unit volume in the solidified waste samples, which may explain the higher initial metal concentrations at similar leachate pH values in pressed compared to unpressed samples. The solubility of Pb is a minimum at around pH 9.5; therefore, Pb leaching is drastically reduced in this pH range. As the pH continues to increase to around 11, Pb solubility increases as $Pb(OH)_3^-$ forms. It is not released back into solution because $Pb(OH)_3^-$ is believed to be sorbed onto C-S-H surfaces.

For OPC/Zn samples the porosity, particularly of samples that were not pressed, is considerably greater, resulting in high initial weight losses. The amount of cement hydration is reduced, as indicated by the high skeletal density and very low levels of $Ca(OH)_2$ formation. Low permeability $CaZn_2(OH)_6{\cdot}H_2O$ is reported to coat the C-S-H surfaces surrounding cement grains, which limits hydration (22).

During initial leaching the low pH acidic leachant causes dissolution of the Zn coating and Zn^{2+} ions are released into solution. This allows cement hydration to begin. Ca^{2+} and other alkalis diffuse into solution, causing the leachate pH to increase. As the pH increases the relative concentration of Zn^{2+} falls and $Zn(OH)_2^0$ becomes the dominant Zn species. Zinc hydroxide will either precipitate within the matrix, or within the bulk of the leachate. During the leaching period investigated the leachate pH did not rise to levels where $Zn(OH)_3^-$ is the dominant species (22).

Conclusions

The following conclusions can be drawn from this experimental study:

- Application of relatively low uniaxial pressing pressures (16 and 32 MPa) during the early stages of hydration of cement/metal waste mixes produces materials with increased density, reduced porosity, and reduced levels of $Ca(OH)_2$.
- Pressing reduces sample volume, which increases the amount of metal per unit volume.
- Pressing cement pastes containing metals reduces weight loss during exposure to 0.05N acetic acid leachant.
- In the leaching tests completed, leachability of Zn and Pb from pressed and unpressed solidified wastes samples is determined primarily by the rate at which leachate pH increases. Pressed samples reach a steady state at a slower rate and so have greater exposure to acidic conditions. Once equilibrium has been reached and alkali conditions exist, leaching of contaminants was low for all samples.
- Further experiments are required to fully assess whether application of uniaxial pressing pressures during hydration of cement solidified wastes produces materials with improved properties for disposal in the environment.

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