Development of process envelopes for cement-based stabilisation/solidification of metal treatment filtercakes

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Abstract

Treatment by stabilisation/solidification with cement-based binders (S/S) is an option for wastes that cannot be prevented or reduced, such as metal treatment sludges from a variety of industries. A laboratory research project at University College London (UCL) is being conducted to generate a coherent body of data regarding S/S of metal treatment filtercakes, with the intention of investigating relationships between engineering and leaching properties and stabilised/solidified (s/s) product composition.

This paper reports on the results from application of screening tests (consistence, bleeding, setting time, unconfined compressive strength (UCS), distilled water extraction, hydraulic conductivity) to more than 40 s/s products containing four different metal treatment sludges treated with four different binder systems. The threshold values of the key properties can be used to define process envelopes that describe the limits of applicability of S/S to the chosen waste types.

This investigation of metal treatment filtercakes is part of the ProCeSS project, which is being conducted by a consortium of five universities and 17 industrial partners, under the UK DIUS Technology Strategy Board (TP/3/WMM/6/I/ 15611).

Keywords: industrial waste treatment, testing, leaching.

1 Introduction

Treatment by stabilisation/solidification with cement-based binders (S/S) is often proposed for wastes from the chemical and metal industries that cannot be

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prevented or reduced. Waste treatment by S/S is complicated by the fact that the components of industrial wastes can interfere with cement hydration and final property development, potentially resulting in technology failure. Therefore, careful laboratory development and testing of S/S formulations are required prior to full-scale application. The ProCeSS project aims to support good practice in S/S by developing process envelopes for generic S/S of the most common UK residual waste types [1]. A series of laboratory research projects are being conducted to generate a coherent body of data, with the intention of investigating relationships between engineering and leaching properties and stabilised/solidified (s/s) product composition. Threshold values of the key properties can be used to define process envelopes that describe the limits of applicability of S/S treatment technology to the chosen waste types. This paper reports results from application of four blended binder systems to metal treatment filtercakes at University College London (UCL) in the first eighteen months of the project, which is on-going.

2 Materials and methods

2.1 Metal treatment filtercakes

S/s products were prepared using five batches of three metal treatment filtercakes from different sources. Each batch of filtercake was dried, ground and homogenised before determination of the properties summarised in Table 1. The homogenised filtercakes were then reconstituted to appropriate moisture contents for 24h before being used to prepare s/s products. X-ray diffraction analysis was performed for three of these filtercakes, and showed that calcium and magnesium carbonates predominate, with no evidence of crystalline minerals containing metal contaminants [2].

2.2 Binders

Samples of Portland cement (CEM I), pulverised fuel ash (pfa), ground granulated blast furnace slag (ggbs), hydrated lime (hlime) and silica fume (sf) were analysed for metals at the start of the project, and found to contain negligible trace quantities. Screening of 15 blended binder systems for setting time, 28d unconfined compressive strength (UCS) and initial pH resulted in selection of the following 4 binder formulations for s/s product preparation:

- CEM I:ggbs = 1:9
- hlime:ggbs = 1:9
- CEM I:pfa = 1:4
- CEM I:pfa:sf = 4:15:1.

2.3 Preparation and testing of stabilised/solidified products

Ninety s/s products containing metal treatment filtercakes were prepared for measurement of bleeding or consistence. Table 2 summarises the formulations for the 31 s/s products prepared for assessment using additional screening tests [3].

Table 1: Properties of metal treatment filtercakes.

Contaminant (mg/dry kg)	metal filtercake				
	BD1	ST1	ST3	TX1	TX2
Ag	2.1	140	140	650	16
Al	1200	8900	11000	37000	49000
Ba	110	140	110	430	380
Ca	44000	240000	240000	33000	230000
Cd	2	430	250	81	300
Co	260	8.2	7.0	1700	750
Cr(total)	11000	14000	17000	49000	29000
Cu	450	3100	1800	22000	9500
Fe	68000	7200	8500	45000	24000
Hg	1.5	13	8.9	45	2.0
K	460	1200	120	1100	170
Mg	15000	6800	7600	17000	1000
Mn	710	310	270	1400	1900
Mo	43	4.5	3.8	21	12
Na	330	1400	990	310	138
Ni	38	21000	25000	43000	49000
P	12000	6800	6700	39000	400
Pb	13	190	330	1300	410
Si	890	4000	3600	720	38000
Sn	1900	340	360	4000	4100
Sr	49	75	970	130	230
Zn	170000	5900	2700	61000	32000
VOCs*	<5	<5	<5	<5	***
TOC (%)**	***	4.4	***	1.6	0.86
moisture content (%)	67	60	***	82	78

* VOC = volatile organic carbon; **TOC = total organic carbon; *** = not measured.

Six screening tests were chosen for evaluation of key properties of the s/s products, with performance thresholds as indicated used to guide the experimental design [3]:

- mix consistence (flow table spread; BS EN 1015-3:1999) > 175 ± 10 mm
- bleeding (BS EN 480-4:2005) < 1% of total water
- setting time (BS EN 196-3:2005); 2 < initial setting time < 8 hours, and final setting time < 24 hours
- 7 and 28d UCS (before and after immersion, using BS EN 196-1:2005 with a 50 mm cube specimen); 28d UCS after immersion ≥ UCS before immersion > 1 MPa
- 3-point acid neutralisation capacity (ANC, e.g., prCEN/TS 15364:2005 with measurement of pH at acid additions of 0, 1 and 2 meq/g), at 7 and 28d; 12.2 ≥ 28d pH at 0 acid addition ≥ 11.9
- 28d hydraulic conductivity (ASTM D5084-03 method D) < 10^{-8} m/s.

Table 2: S/S product formulations and workability measurements.

MONOLITH2 Product ID	Waste Type	% dry mass Binder			Waste	Water	Flow (mm)
		CEM I	ggbs				
UCLP00020		*10.0*	*90.0*		*0.0*	*40.0*	
UCLP00779	ST3	6.5	58.7		34.8	34.8	166
UCLP00029	ST1	6.6	59.0		34.4	34.9	169
UCLP00030	ST1	7.3	65.0		27.3	32.4	141
UCLP01217	ST3	6.1	54.5		39.5	39.4	162
UCLP01114	ST3	8.5	76.2		15.3	35.6	217
UCLP01079	TX1	7.1	64.2		28.7	43.0	174
UCLP01297	TX1	6.0	54.2		39.7	48.6	209
UCLP01316	TX1	7.0	63.0		30.0	48.6	195
UCLP01303	BD1	7.6	68.3		24.1	36.1	171
UCLP01318	TX2	7.2	64.7		28.1	43.0	127
UCLP01323	TX2	9.0	81		10.0	43.0	247
UCLP01327	TX2	9.0	81		10.0	34.0	184
UCLP01461	TX2	7.2	64.7		28.1	40.8	120
		hlime	ggbs				
UCLP00016		*10.0*	*90.0*		*0.0*	*40.0*	
UCLP00891	ST3	6.6	59.4		33.9	33.9	177
UCLP01102	TX1	7.3	65.9		26.8	43.0	166
UCPL01298	TX1	6.0	54.2		39.7	48.6	190
		CEM I	pfa				
UCLP00636		*20.0*	*80.0*		*0.0*	*30.0*	
UCLP00042	$ST1_b$	13.1	52.2		34.4	39.7	179
UCLP01015	ST3	13.1	52.5		34.4	39.7	199
UCLP00041	$ST1_b$	14.5	58.2		27.3	37.5	180
UCLP00868	ST3	13.9	49.5		36.7	42.6	183
UCLP01218	ST3	12.8	45.7		41.6	47.0	155
UCLP00903	TX1	15.7	56.1		28.1	48.9	173
UCLP01309	BD1	15.6	55.7		28.8	49.8	242
UCLP01312	BD1	15.6	55.7		28.6	49.5	224
UCLP01328	TX2	14.4	57.5		28.1	55.5	165
UCLP01462	TX2	18	72		10.0	48.9	199
UCLP01466	TX2	18.3	72.5		9.2	44.9	189
UCLP01467	TX2	14.3	57.4		28.3	51.3	122
		CEM I	pfa	sf			
UCLP00649		*20.0*	*75.0*	*5.0*	*0.0*	*40.0*	
UCLP00761	ST3	13.8	46.2	3.4	36.6	42.1	182
UCLP01089	TX1	15.8	53.0	4.0	27.2	47.2	169
		CEM I					
UCPL01317	TX1	73.8			26.2	39.3	163

S/s product bulk density, moisture content and specific gravity are also being monitored for evidence of dimensional changes, drying, and changes to porosity. A full 10-point ANC with analysis of leachate concentrations of contaminants, and evaluation of contaminant mobility by diffusion in a monolithic leaching test (EA NEN 7375:2004) will be performed on selected s/s products in extended testing, which will also evaluate UCS after 56d and 90d.

2.4 Data collection in MONOLITH2

A database developed to collect data regarding cement-based products for a previous project [4] has been modified by Birkbeck University of London to collect data from the ProCeSS project, including the data for metal treatment filtercakes generated at UCL. The MONOLITH2 database is designed for use on-line, and has facilities easy data entry, browsing, search and output, as well as 3D plotting of s/s product properties as a function of composition [5].

3 Results and discussion

Selected results obtained from subjecting each of the s/s products containing metal treatment filtercake to the screening tests are summarised in Table 2 and Figures 1 to 6, plotted using MONOLITH2. It appears that:

- S/s product properties are dependent on the source of the filtercake, presumably because of their different contaminant concentrations; the maximum addition of highly contaminated filtercake to result in a s/s product of reasonable quality was 26%, whereas the maximum addition of filtercake with lower contamination was 34%. These waste addition levels are similar to those found in the literature [6].
- For filtercakes containing relatively low contaminant concentrations (e.g., ST), it is important to minimise the filtercake moisture content, as this is the limiting factor for filtercake addition to a s/s product; at higher levels of contamination (e.g., BD, TX), the contaminant concentration is the limiting factor for filtercake addition, so minimising the filtercake moisture content is less important.
- The water demand of a mix increases with the filtercake content, and is greater for formulations containing pfa than those containing ggbs.
- For a given waste content, the difference in mix water content between that required to compact the product into a mould and the bleed threshold is relatively small.
- Setting times of the s/s products containing filtercake BD (high Zn) were significantly retarded, and possibly unacceptable in an industrial setting; this is consistent with literature reports of effects of Zn on setting and early strength [7].
- Setting times of the remaining s/s products, containing filtercakes ST (intermediate contamination) and TX (high contamination; shown in Figure 1) were accelerated relative to those of the controls, also, in some cases, possibly to an industrially impractical extent; both filtercakes contained proportionally more Cr(III), a known accelerator of setting and hardening [7].
- Addition of metal treatment filtercake significantly decreases 7 and 28d UCS; a higher level of contamination clearly has a greater effect (illustrated for TX in Figure 2), and in some cases, the 28d UCS was below the proposed threshold of 1 MPa, indicating strong inhibition of binder hydration reactions.
- A high UCS corresponded with low porosity, as can be seen by comparing Figure 3 and Figure 4. The prominent peaks in Figure 3 (valleys in Figure 4),

correspond to s/s products containing low concentrations of plating sludge with a relatively low metal concentration, and the s/s product prepared with a CEM I-only binder.

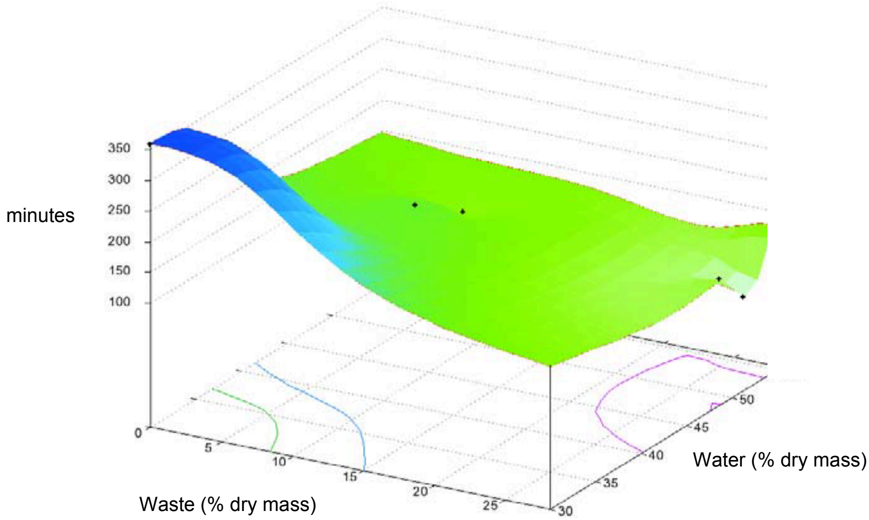

Figure 1: Initial setting time for filtercake with high metal contamination (TX), treated using CEM I:pfa = 1:4.

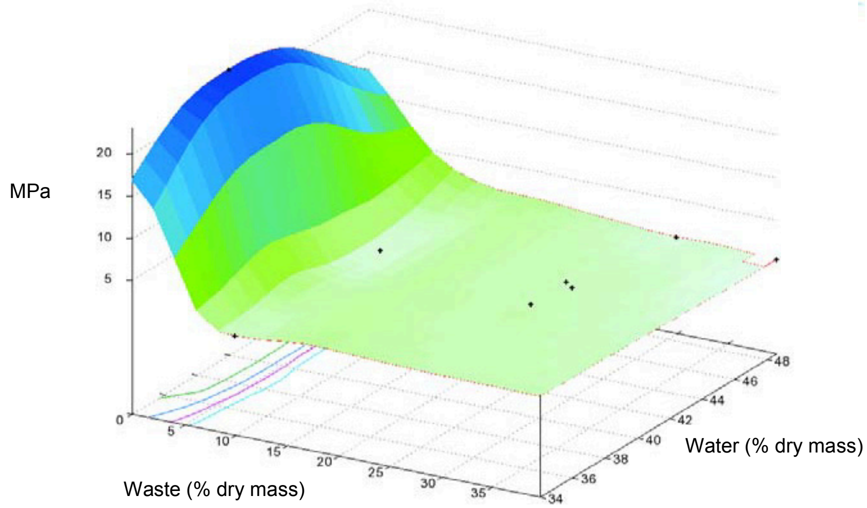

Figure 2: Unconfined compressive strengths measured at 28d for s/s products containing highly contaminated metal treatment filtercake (TX), treated with CEM I:ggbs=1:9.

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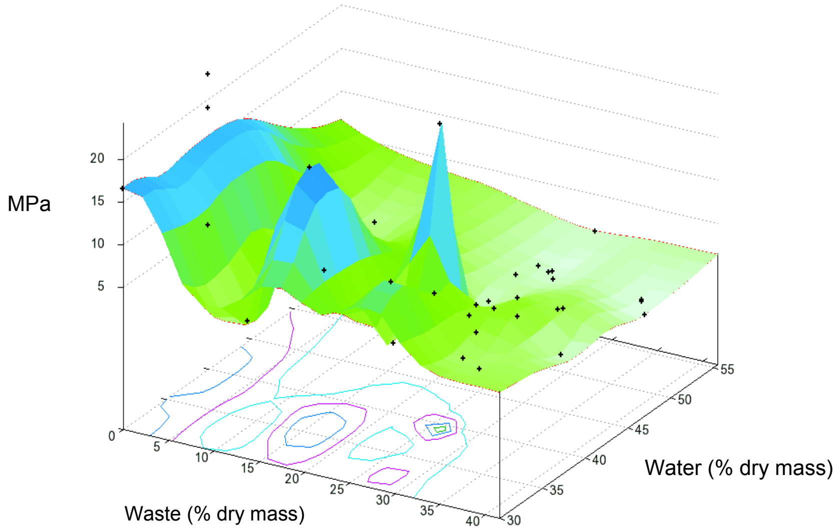

Figure 3: Unconfined compressive strengths measured at 28d for all s/s products.

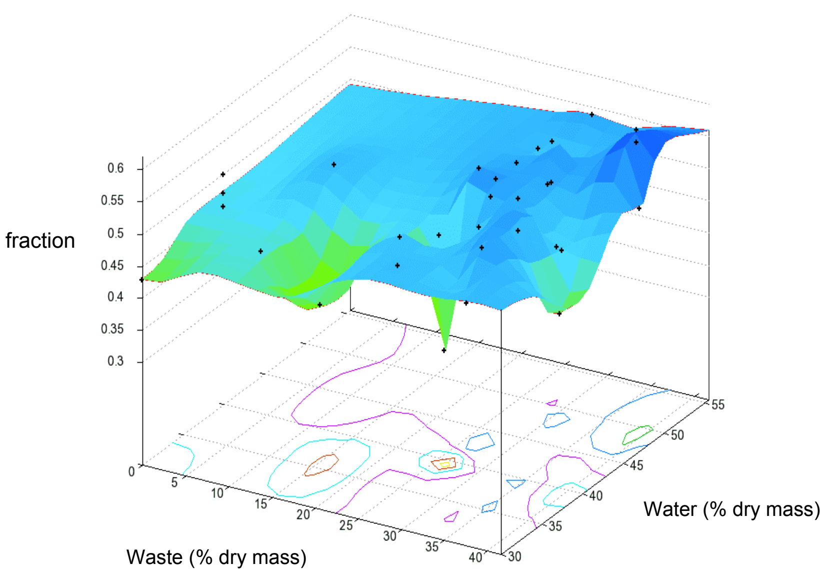

Figure 4: Porosities measured at 28d for all s/s products.

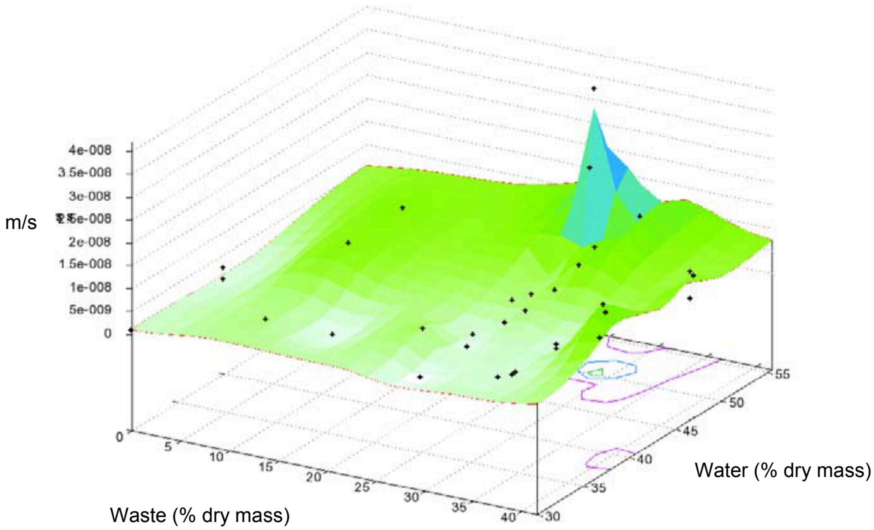

Figure 5: Hydraulic conductivities measured at 28d for all s/s products.

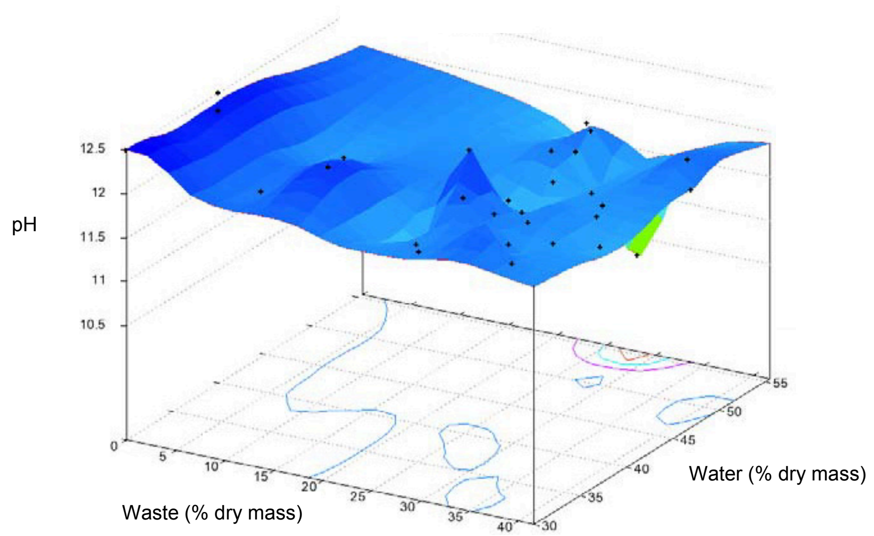

Figure 6: pH at 0 acid addition for all s/s products.

- The hydraulic conductivity of most formulations was below the proposed threshold of 1 x 10^{-8} m/s, as shown in Figure 5, but s/s products containing high Zn, and those approaching the bleed limit resulted in some higher hydraulic conductivities; as might be expected, it seems that hydraulic conductivity increases as a function of both water and waste content.

- S/s products with a hydraulic conductivity > 1 x 10^{-8} also had a UCS < 1 MPa, as can be seen in Figure 5 and Figure 3.
- The leachate pH of a L/S = 10:1 extraction with 0 acid addition was less than or equal to the proposed threshold of 12.2 in most cases after 28d;
- The leachate pH of a L/S = 10:1 extraction with 0 acid addition was less than 11.9 in most of the products with UCS < 1 MPa; since high quality calcium-silicate-hydrate can not exist at this pH, this observations substantiates concerns regarding inhibition of binder hydration. The relationship between UCS and initial pH can be observed by comparing Figure 3 with Figure 6.

4 Conclusions and on-going work

Results to-date show that the amount of metal treatment filtercake that can be incorporated in a s/s product is limited by both contaminant concentration and filtercake moisture content. Measurements of UCS, porosity, hydraulic conductivity, and leachate pH were found to be correlated, and are likely all indicative of the extent of binder hydration.

Development of process envelopes for metal treatment filtercakes is continuing with performance of a factorial design experiment, to attempt to quantify the different effects of individual waste components (matrix disruptors, accelerators and retarders) on s/s product properties, including metal leaching.

Parallel experimental programmes to develop process envelopes for air pollution control residues from waste incineration, electric arc furnace dust, and contaminated soil, are being undertaken by Imperial College London [8], University of Surrey and University of Cambridge, respectively.

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