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Optimization of solidification/stabilization treatment of ferro-alloy waste products through factorial design

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Abstract

Fine solid precipitates from lime neutralisation of liquid effluent streams from surface finishing operations in stainless-steel processing are treated by cement-based solidification. This process is examined using a modified factorial design technique, the Central Composite Rotational Design. The performance of these solidification/stabilization products was explored in terms of the effect of operational variables such as water-to-solids ratio, cement content and curing time which are deemed to be relevant in terms of engineering design practice. In particular, an attempt was made to quantify the interactions between those critical operational variables which dictate the suitability of the solidification method. Success criteria included leach resistance (for certain metal species) and mechanical strength of the composite products. It has been shown that the CCRD provides a rigorous account of solidified product behaviour and does so with a reduced requirement for a priori experimental testing. With the aid of this experimental design we have also shown an inverse relationship between strength and leach resistance of the critical constituents, here represented by chromium. © 1997 Elsevier Science B.V.

Keywords: Solidification/stabilization; Ferro-alloy; Factorial design; Leaching; Agglomerate strength; Chromium

1. Introduction

In recent years pressure on industry has been mounting to find effective technologies for the treatment of its waste products. Commonly encountered waste disposal scenarios

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include, where appropriate, incineration, physical, chemical or biological treatment, consignment to landfill and marine disposal [1]. When wastes are consigned to landfill, problems arise often as a result of long-term release of toxic constituents into the surrounding environment due to fluid percolation through the deposit. One group of technologies which aims to minimise both the release and mobility rates of such environmental pollutants is known as Solidification/Stabilization, or S/S [2]. In *Solidification* the waste is incorporated into a monolithic solid with a reduced surface area over which leaching can occur. Solidification processes do not necessarily imply that any form of chemical reaction has occurred. The term *Stabilization* on the other hand describes disposal technologies which chemically alter hazardous wastes to produce less toxic or mobile forms [3]. Many processes achieve immobilization by a combination of solidification and stabilization. A number of texts and papers are available in open literature which provide background discussions of S/S technologies and their implementations [3–5].

Portland cement, sometimes on its own or in combination with other treatment agents, is a popular S/S treatment agent due to its low cost, applicability to a wide variety of waste products and its relative ease of implementation. This paper focuses specifically on the use of Ordinary Portland Cement for solidification of fine solids which are produced as part of water treatment for liquid effluent from surface treatment of ferro-alloy products.

Performance of S/S systems is assessed traditionally using three parameters [3]. The extent of containment, as measured by **leaching behaviour**, is arguably the most significant. **Permeability** of the solidified mass will dictate the extent to which fluid can permeate through the waste form and access sites at which the contaminants are held. Finally, for handling and storage purposes it is desirable to produce a final product of sufficient **structural integrity** that it will not disintegrate when exposed to commonly expected stresses such as the weight of material placed above it in a landfill.

Due to the complexity of both cement setting and the wide variety of waste products produced by industry, each waste-binder combination must be assessed prior to use 'in the field'. A suitable treatment agent for the waste is identified by referring to available literature [4,6] and by conducting bench-scale compatibility tests. Once a suitable treatment agent has been found, it is desirable to optimise the amount of treatment agent required. A compromise needs to be reached between minimum implementation costs and maximum performance of the system as reflected by the quality parameters identified above. Laboratory-scale tests are used to assess the S/S process, but carrying out a large number of leaching, permeability and strength tests to find an optimum treatment formula can be time-consuming and sometimes expensive. It is therefore desirable to reduce the number of laboratory tests required.

The chemistry of S/S processes is complex, and understanding the interactions between the fundamental variables which define the quality of S/S products is a daunting task [4]. Whilst not detracting from the necessity to explore S/S product behaviour at this fundamental level, we have chosen to assess the potential of the factorial design approach to provide meaningful measures of the performance of S/S products based on simple characterisation tests. This is done in terms of the effects of aggregated variables which are easy for operators to comprehend and which relate directly to engineering practice. In the case of cement-based S/S, the amount of cement, the water-to-cement ratio and curing times are three of the variables which will be expected to have a significant effect on the product.

Other variables, not explored here, include the curing conditions, (ambient temperature and humidity), extent of mixing, extent to which air is removed from the mixture prior to setting, treatment of the waste prior to solidification and the incorporation of other additives in the mixture.

Common failures of S/S monolithic structures result from crack formation and propagation creating additional surface and accelerated leaching behaviour. The way in which process variables combine and interact to promote stability (or, conversely, failure via decrepitation) is complex. Physical models of species retention within solidified products are inadequate to provide sufficient understanding at a deterministic level to predict long-term stability. It can be said only that effects of the variables are not expected to be independent of each other [7].

The aim of laboratory experimentation is to determine how a response, for example one of the performance indicators as described above, is affected by a number of variables. A carefully planned experimental design, such as that obtained by factorial design, can minimise the number of tests required [8]. The output from a factorial design is a number of response surfaces which together describe the effect of all variables on the response, and provide an optimum operating regime in order to achieve a desired response.

This paper investigates the applicability of a commonly used statistical tool, the Central Composite Rotational Design (CCRD), to reducing the number of tests required for optimizing an S/S process in terms of three significant operational variables. The CCRD has been successfully used to model a number of minerals processing systems, including flotation operations [9] and hydrocyclones [10]. The authors have, however, found few references to its use in the design of waste-treatment technologies for minerals processing. Heimann et al. [11] have used an experimental design to optimize binder concentration and explore the effect of curing time on leaching from cements doped with various metal species.

Although factorial design provides a tool to optimize a system, it is recognised at the outset that in application to the S/S process it provides little to no information on containment mechanisms and mechanisms or kinetics of failure of the products.

2. The central composite rotational design

In determining experimentally the response of a system to changing input variables, it is desirable to minimise the number of tests required. Furthermore, where system response is dependent on a number of interacting variables, the nature and significance of these interactions needs to be determined. Factorial experiments in general are designed to achieve both these aims. They can reflect the effect of changing a variable independently of the other variables, as well as identifying any interactions which occur [12]. The number of experiments required for a factorial experimental design is

$$\text{Number of experiments} = (\text{model order} + 1)^{n} \qquad (1)$$

where n is the number of independent variables.

The model order is chosen based on the degree of interaction between the significant variables which is expected. A second-order model, for example, accounts for both first- and second-order influences of individual variables on a response, as well as the effect of first-order interactions between variables. A second-order model is usually sufficient to describe most systems. In such cases a three-level factorial design is often used—'three levels' implying that the effect of each variable is assessed at each of three levels—at the minimum in the range being considered, at some median value and at the maximum value. A disadvantage of such a design is that it requires a relatively large number of tests (3^n in this case).

An effective alternative to a standard three-level factorial design is the centrally composite rotational design (CCRD) [12]. This also produces a quadratic model, as does the three-level factorial design, but with considerably fewer experiments. Apart from determining the effect of a given variable on a result, the design also allows for the determination of statistical error in the results as a result of both systematic and random errors, as well as determining the significance of the interactions between variables. The latter comes about using a simple t-test at the desired significance level. The procedure for this significance testing can be found in texts such as [12].

The application of the CCRD proceeds as follows: the significant variables and high and low values for these variables are chosen by preliminary laboratory testing. The CCRD design procedure is used to calculate the composition of the samples which are required for the experimental programme [12]. The tests are performed and the responses determined. When testing is complete, a regression analysis is carried out to determine the coefficients of the response function (as given in Eq. (2)). Established relationships presented as part of the CCRD design procedure are used in the calculation of the coefficients of the model. For further details of these relationships, see Diamond [12]. An analysis of variance determines the accuracy of the model.

The general model for a CCRD includes three types of terms in addition to a constant a_0:

(i) Linear terms in each of the variables, $x_1, x_2, x_3, \ldots, x_n$.

(ii) Squared terms in each of the variables, $x_1^2, x_2^2, \ldots, x_n^2$.

(iii) First-order interaction terms for each paired combination, $x_i x_j$ where $i \neq j$.

For example, for a three-variable experimental design, the results of the experiments are reduced to a regressed function of the form:

$$y = a_0 + a_1x_1 + a_2x_2 + a_3x_3 + a_{11}x_1^2 + a_{22}x_2^2 + a_{33}x_3^2 + a_{12}x_1x_2 + a_{13}x_1x_3 + a_{23}x_2x_3 \quad (2)$$

where y is the response being estimated, a_i are the constants in the equation and x_i are the independent process variables.

In order to determine the variability in results, a number of repeat trials have to be carried out. Ideally, repeat testing should be carried out at each of the points in the design which will provide an accurate measure of the variability in the responses. Often, however, it is expensive, time-consuming and impractical to carry out such a large number of tests. If the variables are continuous, a reasonable estimate of the variance for

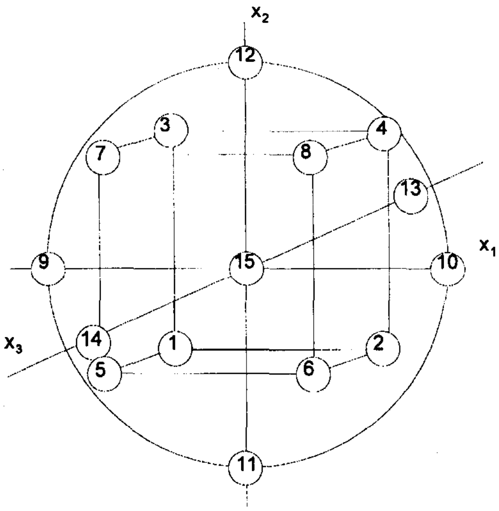

Fig. 1. Central composite rotational design.

the experimental set can be obtained by repeating the results at the centre of the system. For further details see Myers [8,12].

Three types of experimental trials define the CCRD; namely centre, factorial and axial trials. The location of each of these points relative to each other is illustrated in Fig. 1 for a design of three variables. Here the *centre point* is given by point 15, surrounded by *factorial trials* (1–8) and *axial trials* (9–14).

The experimental region of interest is defined by the maximum and minimum values that will be tested for each of the independent variables (process parameters) being investigated. These are the *axial* values. The *centre* of the design is equally spaced between the axial values. The CCRD lends itself to sequential experimentation in that, if a study is started with 2^n factorial experiments, and the first-order model is found to be inadequate, only the axial and centre point experiments need to be added for the second-order response surface to be estimated [8].

3. An experimental study of ferro-alloy wastes

Motivation for the experimental work carried out as part of this programme is provided by the following two conditions:

- Liquid effluent from surface treatment operations contains significant quantities of heavy metals. Whilst the decision to recover these for reprocessing is inevitably an economic one, due consideration must be given to final disposal strategies should such recovery not be warranted. Common practice suggests that acidic effluent be neutralized by a source of free lime. In this way metal species are recovered by ionic precipitation.
- Water quality for surface treatment of ferro-alloy products is of critical importance. Water reclamation and recycling options first require removal of heavy metals

Table 1
Compositions of filtrate solutions and filter cakes used in this study

Species	Wastewater stream composition			Filter cake Compositions	
	Stream 1 $(\mathrm{mg\,l^{-1}})$	Stream 2 $(\mathrm{mg\,l^{-1}})$		Cake 1 $(\mathrm{mg\,g^{-1}})$ (solid)	Cake 2 $(\mathrm{mg\,g^{-1}})$ (solid)
Fe 2+	369	332	Total Fe	13.2	13.3
Fe 3+	872	2190	Ni	1.19	0.98
Ni	176	359	Total Cr	3.6	2.7
Cr (III)	120	108	Mg	0.18	0.16
Cr (VI)	224	603	Ca	22.3	24
Na	1041	1059	Si	0.76	0.78
K	19	17			
Mg	188	183			
Ca	245	227			
SiO_2	51	49			
F	770	3077			
Cl	333	334			
SO_4	6174	5846			
NO_3	2464	9625			
CO_3	177	171			

followed by effective desalination. The ionic precipitation above is a necessary precursor.

We are concerned particularly with the fate of the fine metal hydroxide precipitates identified above.

Neutralization and precipitation of metal hydroxides from two different liquid effluent sources associated with ferro-alloy production were investigated. The primary aim of these treatment processes was the removal of chromium from the streams, and, due to its significance, chromium is used as an indicator of performance of the S/S products. Filter cake material was produced by controlled neutralization using calcium hydroxide which thus results in larger crystals within the precipitate, making the slurry easier to dewater [13]. Compositions of the liquid effluent and filter cake material are presented in Table 1.

4. Choice of relevant variables and responses

4.1. Variables

In the scope of this work three operational variables were chosen as being significant in determining the responses described below. The choice of the three variables were based on parallel studies [14,15].

(i) **Water-to-solids ratio** (w/s): The amount of water present will affect the extent to which hydration of cement occurs. In the case of the filter cake described in Section 3, the water content of the wet filter cake is determined. Past experience has shown that the water content of the filter cakes being used for this study is about 50%, obtained using a

Table 2
High and low values of variables in this study

Variable	Low	Mean	High
Water/solids	1.1	1.18	1.26
Cement content (%)	10	20	30
Curing time (days)	28	49	70

batch pressure filter. Water is then added to make the water-to-solids ratio up to the desired value.

(ii) **Cement content**: This will affect the strength and containment of the product. Cement content is presented in % given by:

$$\text{Cement}(\%) = \frac{\text{Cement}(\text{g})}{\text{Total solids}(\text{g})} \tag{3}$$

(iii) **Curing time**: The longer the sample has to cure, the greater the degree of cement setting and the greater the degree of physical and chemical containment.

As has already been mentioned, it is desirable to reduce the addition of treatment

Table 3
Experimental design for samples used in this study

Trial No.	Water/Solids	Cement (%)	Curing (days)
Factorial Trials			
1	1.23	14.1	37
2	1.23	25.9	37
3	1.23	25.9	61
4	1.13	25.9	61
5	1.23	14.1	61
6	1.13	25.9	37
7	1.13	14.1	61
8	1.13	14.1	37
Axial Trials			
9	1.10	20.0	49
10	1.26	20.0	49
11	1.18	10.0	49
12	1.18	30.0	49
13	1.18	20.0	28
14	1.18	20.0	70
Centre Trials			
15	1.18	20.0	49
16	1.18	20.0	49
17	1.18	20.0	49
18	1.18	20.0	49
19	1.18	20.0	49
20	1.18	20.0	49

agents to reduce both the costs of treatment and the volume increase of the final waste products. High and low levels of the variables used for the work presented here are detailed in Table 2 below. The values for the water-to-solids ratio and cement addition were chosen after a review of both cement and S/S literature and preliminary laboratory investigations. The minimum curing time was based on that required to obtain samples with sufficient structural integrity to survive subsequent testing. The high value for curing time of 70 days was suggested to be sufficient to ensure extensive hydration of the cement.

The resulting combinations of the operational variables, calculated using the factorial design procedure [12], appear in Table 3.

The filter cakes contain around 55% moisture (w/w) after filtration, which implies a water/solids ratio of 1.22. Upon addition of cement, this brings the water/solids ratio down to between 0.93 and 1.1. The water/solids ratio is brought up to its desired value using distilled water.

In industrial applications it would make little sense to dewater material and subsequently introduce more water. It would be more sensible to dewater to the optimal level, add cement and allow to set. In the case of this laboratory investigation dewatering was not controlled and thus additional water was required to achieve water-to-solids ratios in the regime which was being investigated.

4.2. Responses

The system responses which were investigated in this work are:

(i) Indirect Tensile Strength (ITS). Once the waste is placed into a landfill, it is important that it is able to support the weight of material placed above it without failing. The ITS tests are carried out using the procedure described in texts such as PCI [16]. The ITS is calculated from the load at fracture.

(ii) The leaching of constituents from the solidified body. The main aim of S/S is the immobilization of toxic constituents of a hazardous waste. Leaching tests will provide the best measure of effectiveness of containment.

The leaching of constituents from the material was determined using the standard US EPA testing procedure for alkaline materials, namely the Toxicity Characteristic Leaching Procedure (TCLP) [17]. The results from this test are widely used as a criterion for classifying wastes as being hazardous. The test comprises an 18 h batch test in which the material is mixed with an acetic acid solution prepared from glacial acetic acid. The leachate is filtered and analyzed for metals and organics. In this case leachates were tested for pH and Cr, Mg, Ca, Si, Na. Only results for Cr are presented here.

The TCLP has been the subject of much controversy in S/S literature since being developed as a waste classification tool [18]. The single batch test provides no indication of the rate at which toxic constituents will be leached from wastes nor the long-term stability of S/S wastes or the mechanisms by which containment is effected. It does, however, provide a convenient tool for comparing the relative amounts of contaminants leached from various S/S formulations. The TCLP is used as such in this work and its use here does not suggest that we advocate its use in providing a comprehensive indication of leach potential.

There is little open literature on the structure of S/S products made with solid waste materials—the majority of work has focused on solidification of liquid wastes and slurries. Using available literature on the structure of cement and concrete we have developed a physical picture of the cement-waste product. The physical structure of the end product is expected to be similar to a cement product made with fine aggregate, in which the solid particles are bound together by the products of cement hydration. The latter include fibrillar crystalline phases such as hydrated calcium silicate complexes (CS) and calcium sulpho aluminates. It is the CS phases which contribute primarily to the strength of the material [19], and the structural strength of the end product will therefore be dependent on the extent of cement bonding [20].

Drawing on literature of the S/S of liquid wastes, an understanding was developed of how different metal species interact with the cement hydration products [21–23]. This is presented in previous work by the authors of this paper. The retention of metals was identified to be dependent on the extent of cement setting. For example we postulate that chromium (III) is incorporated into cement hydration products. Therefore, as the crystalline silicate hydrate matrix evolves with cement curing we would anticipate greater retention of chromium (III) compounds. The retention of Cr(VI) is discussed further in our other work [24].

5. Preparation of S/S samples

The samples are prepared as follows:

(i) The water content of the filter cake is determined.

(ii) The required cement addition is calculated based on the dry mass of the filter cake used.

(iii) Distilled water is added to make the water content up to the desired water-to-solids ratio.

(iv) The contents are mixed in a paddle mixer. The moulded samples are vibrated on a vibrating table to remove entrapped air.

Samples are left to cure in moulds in a constant humidity and constant-temperature environment during the curing phase.

6. Indirect Tensile Strength (ITS) tests

ITS results for Filter Cake 2 appear in Table 4. The experimental design identifies the influence of all three variables (cement content, curing time and water-to-solids ratio) as being the significant variables in the determination of strength (at the 95% confidence limit), with the cement content being the most important variable. All three variables also had a second-order effect on strength, and the interaction between w/s and cement was identified as important, also at a 95% confidence limit. A plot of the response surface for strength vs. water-to-solids ratio and cement content generated using the model appears in Fig. 2 for a curing time of 49 days. The correlation between the experimental and predicted results gives a coefficient of correlation, $R^2 = 0.81$. Although this value is high, there is still some discrepancy between actual and predicted results.

Table 4
ITS and leachate compositions for Filter Cake 2

Trial	ITS (MPa)	pH	Cr	Mg	Ca	Si	Na
			(concentrations in ppm)				
Factorial Trials							
1	0.09	5.6	1.8	40.2	1751	44	46.2
2	0.04	7.5	4.6	29.8	1943	8.8	51.1
3	0.29	7.4	3.8	33.5	1843	14.9	108.8
4	0.35	7.5	4.8	31.8	1821	10.6	106.1
5	0.07	5.5	2.5	43.1	1505	63	111.4
6	0.01	7.9	6.2	28.7	1937	5.4	55.1
7	0.12	5.8	1.8	45.4	1476	50.9	118.6
8	0.08	5.7	2.9	41.5	1773	39.4	59.5
Axial Trials							
9	0.31	6.7	2.1	37.6	1833	26.4	106
10	0.22	6.4	1.8	38	1823	32.9	108.1
11	0.06	5.5	3.8	55.5	888	62.2	39.2
12	0.28	8.2	6.4	27.9	1793	6.9	105.6
13	0.18	6.9	2.2	49.5	1726	22.7	43.6
14	0.23	6.5	1.9	33.9	2192	29.9	79.2
Centre Trials							
15	0.26	6.7	2.1	37.6	1682	27.6	118.6
16	0.30	6.8	2.3	37.1	1726	24.5	98.1
17	0.29	6.7	2.0	38.4	1756	29.5	109.9
18	0.24	6.6	2.0	37.2	1671	28.2	106.2
19	0.30	6.5	2.3	37.3	1643	24.3	105.8
20	0.28	6.6	2.1	37.7	1716	26.8	106.3

The centre point trials serve to indicate the repeatability in results. In order to compare variability between the strength and leaching results, the coefficient of variation is used. The strength results show a coefficient of variation of 8.6%. The ITS test measures a macroscopic strength of a material. For this reason the variability in results is expected to be high. The variation in results would also account for the low correlation coefficient. Current work at University of Cape Town is looking at a fracture mechanics approach to provide a more sensitive and repeatable strength measurement. Although the latter tests provide more meaningful data in terms of bonding within the solid, they are time consuming. A correlation between ITS and fracture mechanics parameters will enable a sensitive strength characterisation via a more crude testing technique, namely the ITS.

7. Leach testing

Concentrations of the various elements of interest in the TCLP leachates, as well as leachate pHs appear for Filter Cake 2 in Table 4 alongside the ITS results. A plot of

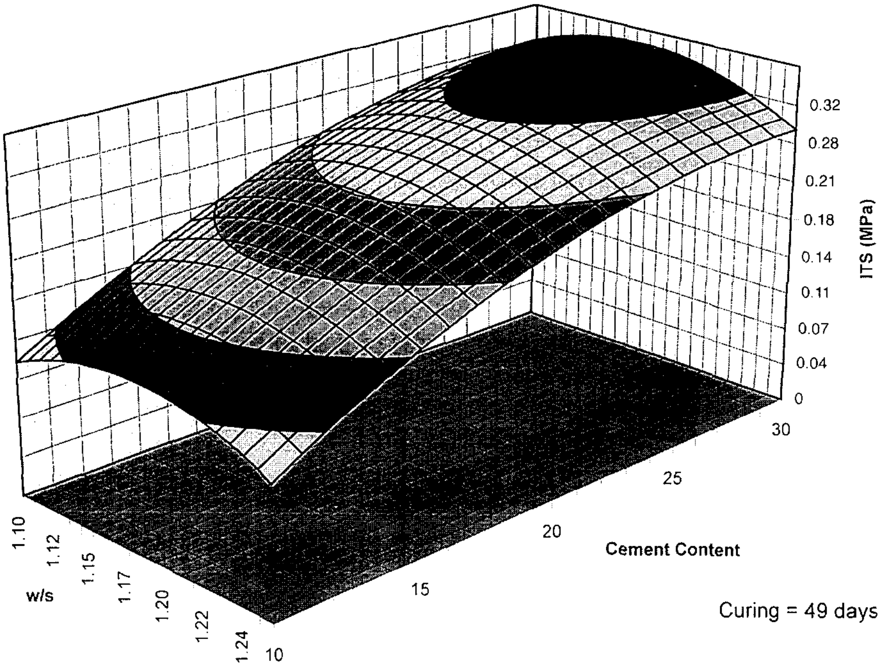

Fig. 2. Plot of strength vs. cement content and water to solids ratio for a curing time of 49 days.

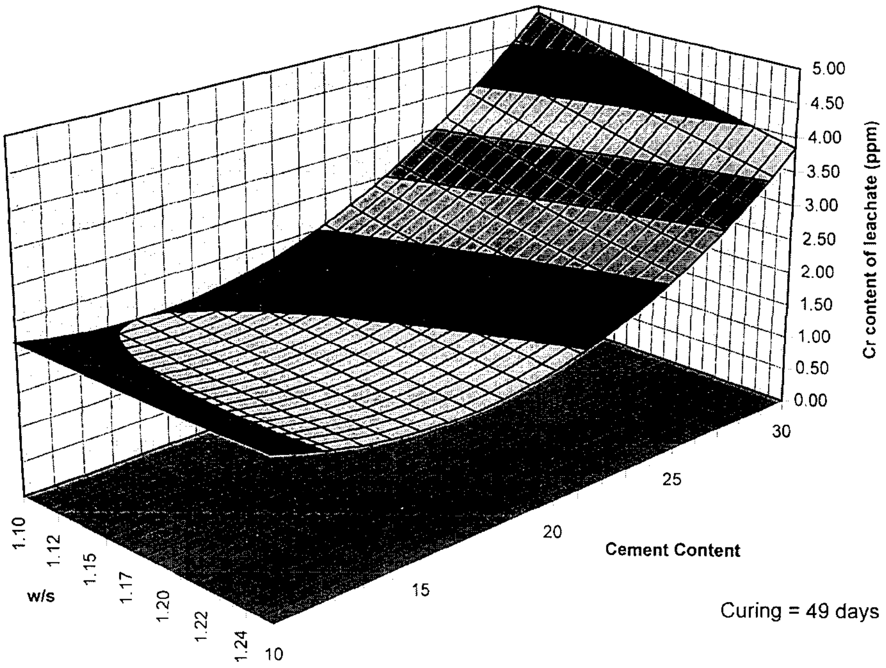

Fig. 3. Plot of chromium leaching vs. cement content and water to solids ratio for a curing time of 49 days.

chromium leaching as a function water-to-solids ratio and cement content for a curing time of 49 days is presented in Fig. 3. Predicted results show good correlation with the measured results, with $R^2 = 0.94$. This implies that the model predicts chromium leaching even more effectively than the strength behaviour. The coefficient of variation is lower than for the strength (5.9%). The experimental design indicates that all three variables are important in determining chromium leaching.

The leach test results also indicate that the pH values of the leachates from the TCLP are related to the cement content, with an increase in cement content causing an increase in pH of the leachate. This is to be expected due to the fact that cement contains high levels of free alkalis. This trend is validated by the model. The model also indicates that the interaction between curing time and cement content is important in determining the pH of the leachate at the end of the test period. In this specific case, the highly alkaline pH of the pore solution restricts the mobility of the metal cations due to the reduced solubility of the hydroxide species under this condition.

8. Optimization of the model

One of the aims of the laboratory work was to evaluate the optimum parameters for maximum strength and minimum leaching. By taking the partial derivative of Eq. (1) above with respect to the three variables (water-to-solids ratio, cement content and curing time), and solving for variable values with the partial derivative equal to zero (see Eq. (4)), the variable values for optimal response behaviour will be obtained. Not only will these optimal values be useful in designing S/S processes for implementation in the field, but they will also indicate whether or not the hypothesised interactions between strength and leaching discussed above do, in fact, exist.

$$\begin{aligned}\frac{\partial y}{\partial x_1} &= a_1 + 2a_{11}x_1 + a_{12}x_2 + a_{13}x_3\\ \frac{\partial y}{\partial x_2} &= \mathrm{a}_2 + 2\mathrm{a}_{22}\mathrm{x}_2 + \mathrm{a}_{12}\mathrm{x}_1 + \mathrm{a}_{23}\mathrm{x}_3\\ \frac{\partial y}{\partial x_3} &= a_3 + 2a_{33}x_3 + a_{13}x_1 + a_{23}x_2 \qquad (4)\end{aligned}$$

The optimal values for maximum strength and minimum chromium leaching as predicted by Eq. (4) appear in Table 5. It would appear that optimal response behaviour

Table 5
Optimal variable values for maximum strength and minimum leaching

Variable	Strength	Chromium leaching
Water/solids	1.1	1.2
Cement (%)	20	16.3
Curing time (days)	62	60

Table 6
Effect of changing operational variables on responses

Variable	Increase/decrease in variable value	Decrease in ITS	Increase in Cr leaching
Cement	5%	3–4%	13–18%
Water/solids ratio	0.05	0.1–3.8%	1.3–2.6%
Curing time	10 days	1.2–1.4%	1–2.5%

for the two responses can be achieved at similar variable values, providing some degree of support for our hypothesis of the relationship between strength and leaching behaviour [15,24].

In an industrial application it is difficult to control the operational variables to achieve the exact values presented in Table 5. It is significant at this point to identify the sensitivity of the system to variations in the values of the operational variables.

Table 6 shows the effect of a change in the operational variables on the responses. Strength is relatively insensitive to changing operational variables. Cr leaching is sensitive to a change in cement content, but is not as sensitive to the other variable values. Control of cement addition is therefore important, but fluctuations in the residual water content of the filter cakes will not have a significant influence on either Cr retention or product strength.

The model is capable of identifying optimal operating conditions for these specific S/S products based on laboratory-scale responses of leaching and strength. The challenge is to reconcile this 'bench-scale' assessment with full-scale engineering practice. The choice of operational variables was motivated by this very concern. As a demonstration of the validity of this work, we can point to the design and commissioning of an S/S product landfill deposit based on our experimental work [25].

9. Conclusions

This paper has shown the use of the CCRD in the modelling of the responses of a S/S system to the input variables. The polynomial model which is generated can be used to find optimum operating conditions.

Of the variables investigated here, cement content and curing appear to be the most important, with the water-to-solids ratio being less important in the ranges being investigated here. One trend which is highlighted for further work is that the strength decreases and leaching decreases as one goes past the optimal curing time. No explanation is presented for this trend here.

If the model for the strength behaviour is superimposed on that for chromium leaching, it is seen that minimum leaching is observed at a similar point to that of maximum strength (see Table 5). This indicates that the hypothesised relationship presented previously in this paper does in fact exist.

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