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Modeling the effect of sulphate on strength development of paste backfill and binder mixture optimization

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Abstract

The increasing use of paste backfill in underground mining makes it necessary to quantify the effect of sulphate on the strength development and economical performance of paste backfill. Therefore, the main objective of this study was to develop a methodological approach and a mathematical model to analyze and predict the strength development and cost of underground paste backfill containing different amounts of sulphate. This study demonstrates that the paste backfill can be defined as a "mixture system". We were able to show that a material model based on quadratic functions can be a suitable basis for the prediction of both the mechanical [uniaxial compressible strength (UCS)] and economic (binder cost) properties of paste backfill. The developed models allowed us to obtain valuable results about the relationship between the sulphate quantity and the mechanical performance of the backfill and the prediction of its strength at different curing times. This study has demonstrated that the sulphate significantly influences paste backfill strength. This influence is related to its concentration, the curing time and the amount and chemical composition of the cement.

Keywords: Paste backfill; Compressive strength; Sulphate; Cement; Modeling

1. Introduction

The use of paste technology for underground backfill has been accepted worldwide in the mining industry as a cost-effective alternative to rock and hydraulic backfill. Paste backfill is mainly used to fill the cavities created by mining to establish and maintain safe underground working conditions in an economic way. In addition, the underground disposal of tailings (mine waste), which are often potential pollutants (acid mine drainage, source of heavy metals), is a significant environmental benefit. Indeed, this can enable to reduce up to 60% of the mine wastes to be disposed on the surface.

Paste backfill is a mixture of water (fresh water and/or mine process water), tailings material (often a solid percentage between 70% and 85%) and hydraulic binders (usually ordinary Portland cement; used alone or blended with various binders, such as sulphate resistant Portland cement, blast furnace slag, fly ash, etc.). The main role of the binders is to give sufficient mechanical strength to the paste backfill created. Due to expense of the cement, the cost of binders can represent up to 75% of the cost of the backfill [1]. Therefore, an essential requirement for the cemented backfill mixture is that the binder content should be at the lowest level possible that does not affect the quality criteria of the paste backfill.

One of the most important quality criteria for the hardened paste backfill is mechanical stability. Indeed, the paste backfill must remain stable during the extraction of adjacent stopes to ensure the security of the mine workers. The uniaxial compressible strength (UCS) of the hardened backfill is often used in the practice to evaluate its stability because the test is relatively inexpensive and can be incorporated into routine quality control programs at the mine. The UCS value required for each underground mining operation varies, depending on the application or function of the backfill. For example, when the paste backfill is simply used to fill voids or for

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underground disposal, to eliminate the risk of liquefaction, strength values of between 150 and 300 kPa of the backfill are used as target values at several mines sites [2]. However, when the cemented backfill is used in open stoping operations, to maintain backfill stability, when free-standing wall faces are exposed during pillar recovery, strength values of up to 1 MPa are often required [1–4]. However, when the paste backfill is used for roof support, strength values higher than 4 MPa are required [4].

Generally, adding small percentages of ordinary Portland cement of between 3% and 7% by weight allows the development of sufficient strength to enable the backfill stability during mining operations. However, problems can arise when the paste backfill is rich in sulphates. The presence of sulphates often causes an internal sulphate attack. The latter can lead to strength loss of the backfill and its consequent failure.

Previous work [5–11] has already dealt with the strength loss of the cemented backfill due to sulphate attack. However, the mathematical approach for the analysis and modeling of the effects of sulphate on the strength of paste backfill has not been considered until now in this type of study, nor has the prediction of the mechanical strength and cost of the paste backfill under sulphate attack been done. Hence, modeling the time-dependent effects of sulphate presence and quantity on the strength of the paste backfill is necessary to obtain a safe mining work environment and to make cost effective paste backfill.

Considering the problems mentioned above and the fact that various underground mines in Canada are faced with the risk of failure of their paste backfill due to sulphate attack, the goal of the research was to develop an efficient approach and mathematical prediction model, which will allow us to do the following:

- find the relationship between the quantity of sulphate and the strength development of the paste backfill, i.e., to better understand the effect of sulphates on cemented backfill;
- predict the mechanical strength of the paste backfill under sulphate effect; and
- optimize the binder mixtures to produce a cost-effective backfill resistant to sulphate attack.

To reach these goals, the response surface method (RSM) was used for the modeling.

This paper is organized as follows. A review on sulphate effect on cemented material and description of the RSM are presented in the next section. This is followed by a presentation of the materials used and experimental tests done. After that, the proposed modeling approach and experimental design are presented. Then, the main results of the modeling and verification of the model are presented and discussed. Finally, we present our conclusions.

2. Geochemical and mathematical background

2.1. Geochemical background

2.1.1. Sulphate attack on cemented material

Sulphate attack has damaging effects on the quality (e.g., strength deterioration) of cement-based products (concrete, mortars, cemented paste backfill, etc.). While the sulphate attack on concrete and mortars is often external (external source of sulphate ions), the sulphate attack on cemented paste backfill is essentially internal.

Many studies done on sulphate effect on concrete and mortars have been concentrated on external sulphate attack [12–24]. According to Santhanam et al. [24], sulphate attack on cement mortars or concrete leads to the formation of minerals as ettringite, gypsum and other phases and also causes the degradation of the primary strength providing calcium silicate hydrate (C-S-H) gel. The formation of secondary ettringite and gypsum is common in cementitious material exposed to most types of sulphate solutions [24]. According to Bing and Cohen [25], sulphate attack on Portland cement concrete is often considered to arise from each of two major sulphate reactions: (1) The sulphate ions react with C_3A and its hydration products to produce ettringite with an increase in volume that results in expansion and subsequent cracking of the concrete and (2) the sulphate ions react with calcium hydroxide (CH) to produce gypsum [25].

The cations present in the sulphate solutions play also an important role in sulphate attack. In the case of sodium sulphate attack, gypsum and ettringite are formed by chemical reactions of sulphate and $Ca(OH)_2$, C_3A [12,14,26,27], while brucite, ettringite and gypsum are produced in the case of magnesium sulphate attack on cement-based materials [15]. The main damaging effect of magnesium sulphate solution is the decomposition of CSH gel to noncementitious MSH [28,29]. The mechanisms of sodium and magnesium sulphate attack are well described in following works [24,30].

According to Refs. [25,27], not only the ettringite formation resulting from sulphate attack can lead to the deterioration of the concrete or mortar, but also, the formation of gypsum plays a significant role in the deterioration of cemented material. Indeed, gypsum can cause softening of the material [28,31]. Additionally, it has been demonstrated that gypsum formation during sulphate attack may also cause expansion [25].

However, the importance of expansion in the deterioration of cemented material and the idea that gypsum formation leads to any expansion are still controversial and not currently accepted by all workers [32–34]. Indeed, there are works [20,35–37] contradicting the theory that gypsum formation is expansive, and those [15,38–42] supporting this theory.

Additionally, Metha [33] underlines that, in most cases, the loss of adhesion and strength of the cemented material,

Table 1
Main chemical elements in the binders used

Element unit	S (wt.%)	Ca (wt.%)	Si (wt.%)	Al (wt.%)	Mg (wt.%)	Fe (wt.%)	Si/Ca
Binder type							
PC I	1.5	44.9	8.4	2.4	1.6	1.9	0.2
PC V	1.2	45.5	9.3	2.0	0.9	2.7	0.2

not expansion and cracking, is the primary consequence of sulphate attack. Gollop and Taylor [43] suggested that the most likely reason for the cause of expansion is the absorption of water by the "cement gel".

Compared with the several studies about sulphate attack on concrete or mortars, only few studies [8–11] have dealt with sulphate effect on cemented paste backfill. These works [8–11] have experimentally demonstrated the damaging effect of sulphates within paste backfill on the strength development of the backfill. According to these authors, the deleterious effect of sulphate on paste backfill appears mainly as deterioration of the backfill strength over time. This deterioration is caused by internal sulphate attack and is mainly the result of the chemical interactions of the sulphate ions in the paste backfill with the Portland cement hydration products [8–11]. The most common products of these reactions are secondary ettringite, gypsum and monosulfoaluminate. The formation of these highly expansive products causes high internal pressure due to their expansion within the backfill, which often leads to the physical deterioration (strength loss) of the cemented backfill materials [8–11]. The level of these expansive pressures depends on the quantity of the sulphate in the backfill. The amounts of sulphate ions decrease as they react with the Portland cement hydrations products, and the rate of internal attack tends to slow with time [10]. However, to date, because of the complexity of the material paste backfill, the knowledge on sulphate effect on cemented paste backfill is quite limited. Indeed, the material cemented paste backfill is relatively new, and many differences exist between the behaviour of paste backfill materials and mortars/concrete materials [44].

2.1.2. Source of sulphate in the paste backfill

The primary source of sulphate in the paste backfill is the tailings material. The latter often contains low to high amounts (2% to 60%) of sulphides, especially in polymetallic mines tailings. Because of their reactivity, the sulphides are the most significant source of sulphate (Eq. (1)) in the tailings and, consequently, in the paste backfill. Pyrite (FeS_2) is the most common sulphide present in most tailings. The oxidation of pyrite has been studied by several authors [45–51], and the equation below (Eq. (1)) is generally accepted as the overall reaction for pyrite oxidation, although the reaction, in fact, consists of a series of complex subreactions with reaction mechanisms that, in some cases, are not full understood [52].

$$\mathrm{FeS}_{2(s)} + 7/2\,\mathrm{O}_{2(g)} + \mathrm{H_2O}_{(aq)} \rightarrow \mathrm{Fe}^{2+}(\mathrm{aq}) + 2\,\mathrm{SO}^{2-}_{4(aq)} + 2\mathrm{H}^{+}_{(aq)} \quad (1)$$

While sulphide oxidation is very active in the tailings, it is almost negligible within the paste backfill [44,53,54]. This is due to the fact that the paste backfill is saturated with water. This saturation limits oxygen availability as a direct oxidant of sulphide. Bacterial activity in the cemented paste backfill is impossible due to the high pH value (pH higher than 9) of the backfill.

However, the active oxidation of sulphides in the tailing is not the only way by which sulphates may be in the paste backfill. The destruction of cyanides, present in some mill tailings and/or mine process waters, using the method SO_2–air, can be also a significant source of sulphate in the tailings pore water and/or in the mixing water (if process waters are used for backfill mixtures). Another source of sulphate can be the used cement. The addition of gypsum ($CaSO_4.2H_2O$) or anhydrite ($CaSO_4$) in the clinker to control flash setting also adds a small amount of sulphate to the backfill.

2.2. Response surface method

Introduced by Box and Wilson [55], the RSM is a collection of statistical and mathematical techniques used to empirically relate an output variable (dependent), or response, to the levels of a number of input variables (independent). RSM provides a simple model spanning the entire design space (region of interest) for a complex model, for which no analytical solutions exists, by simplifying the relationships between the response and the independent input variables into a polynomial equation [56]. Usually, a low-order polynomial in some region of the independent variable (region of interest) is used. In this study, a second-order polynomial (Eq. (2)) was used. The latter is shown below

$$y = b_0 + \sum_{i=1}^{k} b_i x_i + \sum_{i=1}^{k} b_{ii} x_i^2 + \sum \sum_{i<j}^{k} b_{ii} x_i x_j + \varepsilon \quad (2)$$

where, y is the predicted response (UCS); b_0 is the intercept, b_i are constant regression coefficients for the linear terms, b_{ii} are constant regression coefficients for the pure quadratic

Table 2
Main chemical elements in the tap water

Element unit	Al (ppm)	As (ppm)	Ca (ppm)	Si (ppm)	Cu (ppm)	Fe (ppm)	Mg (ppm)	Na (ppm)	S (ppm)	SO_4^{2-} (ppm)
TW	0.01	< 0.03	46.60	0.64	0.21	0.03	2.57	–	22.10	66.20

terms; b_{ij} are constant regression coefficients for the cross-product terms. The x_i variables are the normalized values of each of the input variables that affect the responses; this normalization allows an increase in the computational accuracy in estimating the regression coefficients. The cross-term $x_i x_j$ represents two-parameter interactions and square terms, x_i^2 represents second-order nonlinearity. The interaction-terms $x_i x_j$ and the quadratic terms x_i^2 account for the curvature in the response surface. And finally, ε is the associated random error; it represents the combined effects of variables not included in the models. It is assumed that the additive error ε has a normal distribution with a mean 0 and a variance σ^2.

The regression coefficients for the second-order model (Eq. (2)) are estimated by using regression analysis applied to observations performed in experimental design. The experimental design constructed in this study was a central composite design (CCD) [57,58]. The replication of some observations in the center point of the region of interest gave the CCD orthogonal properties [57]. These orthogonal properties provided the minimum variance estimates of the model coefficients so that they are uncorrelated.

3. Materials and methods

3.1. Materials used

The used materials included binder reagents, waters and mine tailings.

3.1.1. Binder reagents

Type I Portland cement (PC I) with a specific gravity of 3.15 and Type V Portland cement (PC V) that met ASTM C 150 specifications was used in all mixtures. The main chemical elements in these two cements are shown in Table 1.

3.1.2. Waters

Tap water (TW) with very low sulphate content and waters (SW) with relatively high sulphate content (Table 2) were used as basic waters. The waters SW were made by adding a specific amount of sulphate concentrate to tap water. To simulate the effect of sulphate content on paste backfill, the creation of water with different sulphate concentrations was necessary. Thus, the mixing of specific amounts of TW and SW allowed creating water (W_1 to W_5) with different sulphate contents. Table 2 shows the chemical composition of TW, while the sulphate contents of the different waters are given in Table 3. The chemical compositions were determined by ICP-ES analysis.

Table 3
Sulphate content of the used waters

Water	TW	W_1	W_2	W_3	W_4	W_5
Sulphate (ppm)	66	320	1000	2000	3000	3680

Table 4
Main chemical element in the tailings

Element unit	Al (wt.%)	Ca (wt.%)	Si (wt.%)	Fe (wt.%)	Na (wt.%)	Pb (wt.%)	S (wt.%)	Zn (wt.%)
Tailing	3.52	1.01	13.09	29.47	0.91	0.26	26.08	0.71

3.1.3. Tailings

The tailing materials used were obtained from a polymetalic mine in eastern Canada. It contains about 30% sulphides (mostly pyrite; Table 4). The physical properties of the tailings material are given in Table 5. It can be noted that the material is quite dense and well graded (Table 5).

3.2. Specimen preparation

The received samples of tailings material were first mixed to create a homogeneous material and then distributed into five barrels. The tailings samples in each barrel had similar chemical and physical properties. Fig. 1 shows the grain size distribution of the tailings material from each of the barrels (TB_1, TB_2, TB_3, TB_4 and TB_5). All of the barrels were saturated with water to avoid atmospheric oxidation of the sulphides. The sulphate concentration in the tailings pore water of each barrel was higher than 3000 ppm.

The tailings samples were rinsed with tap water to obtain tailings material whose pore waters had specific sulphate concentrations. This allowed us to return the initial sulphate concentration of tailings pore water in the barrels TB_1, TB_2, TB_3 and TB_4, to 320, 1000, 2000 and 3000 ppm, respectively. However, the addition of specific amounts of synthetic sulphate solution to Barrel 5 and its homogenization allowed us to reach a sulphate concentration of 3680 ppm in the tailings pore water.

After eliminating the excess water in each of the barrels, the tailing materials, cement and water were mixed and homogenized in a mixer with double spiral for about 7 min to produce the desired paste backfill mixtures. Then, the consistency of the paste mixtures was measured by slump test according to ASTM C 143-90. The solid percentage of all of the mixes was maintained at approximately 80% (by weight). The produced paste backfill mixes were poured into curing cylinders, 10 cm in diameter and 20 cm high. The specimens were then sealed

Table 5
Physical properties of the tailings

Element unit	Sp (m^2/g)	Gs	D_{10} μm	D_{30} μm	D_{50} μm	D_{60} μm	D_{90} μm	C_u	Cc
Tailing	1.56	3.58	3.60	13.20	28.25	39.45	145.60	10.95	1.09

Sp: specific surface; Gs: specific gravity; Cu: Coefficient of uniformity; Cc: coefficient of curvature.

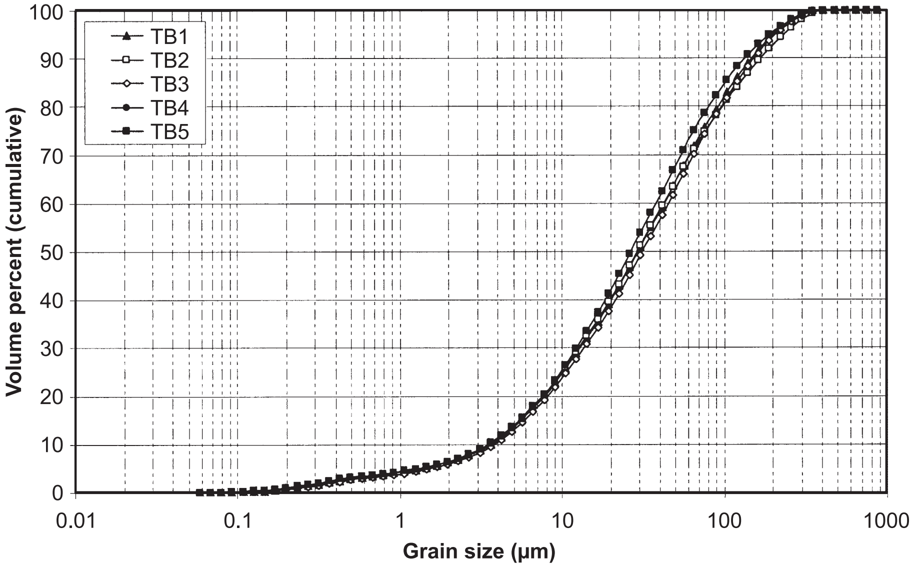

Fig. 1. Grain size distribution of the used tailing samples.

and cured in a humidity chamber maintained at approximately 80% humidity and 23 ± 2 °C for periods of 14, 28 and 56 days. This curing temperature and humidity simulated the normal curing conditions found in underground backfill operations in eastern Canada.

3.3. Testing of specimens

Compressible strength was measured after 14, 28 and 56 days curing period on each paste backfill specimen according to the ASTM standards. The compressive strength testing allowed us to determine the UCS of the tested samples.

The cost of the quantity of binder used in each backfill sample was evaluated. The latter was calculated from the mix proportions using the costs for each of the binder reagents from the eastern Canadian market in 2002.

4. Modeling approach and experimental design

4.1. Modeling approach

Because the sulphate exclusively affects the hydraulic binders, the chemistry of the cement, its proportion in the backfill and the chemistry (i.e., sulphate content) of mixing water (tailings pore water + added water) were considered to be the basis for the modeling. Thus, for the modeling, the paste backfill was idealized as a black box, whose mechanical (UCS_{ti}, UCS at different curing time) and economic (based on binder cost) properties depend on following three factors: percentage Portland Cement Type I (PC I: X_1), percentage Portland Cement Type V (PC V: X_2), sulphate concentration in the mixing water (sulphate: X_3). The physical properties of the tailings were maintained at constant levels. Fig. 2 shows a schematic repre-

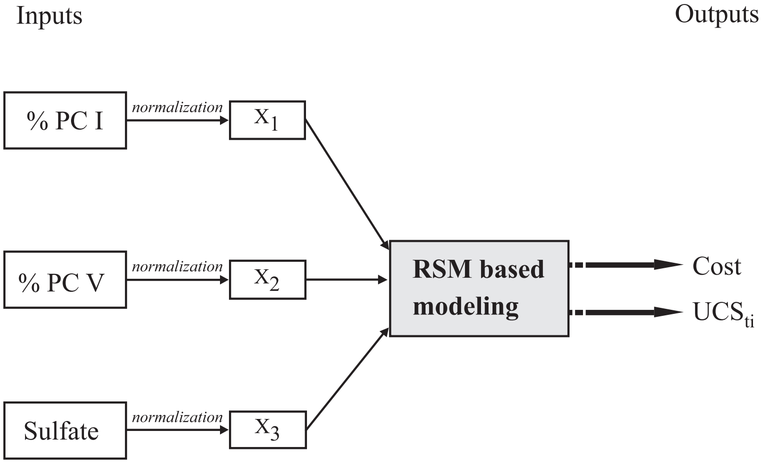

Fig. 2. Schematic representation of the used modeling approach.

Table 6
Design variable and normalization

Codes	– 1.68	– 1	0	1	1.68
Variables					
% PC I	0.45	1.40	2.80	4.20	5.15
% PC V	0.75	1.20	1.85	2.50	2.95
Sulphate	320	1000	2000	3000	3680

Sulphate: sulphate concentration of the mixing water (ppm).

sentation of the developed model and the main input and output parameters.

As seen in Fig. 2, the three factors (X_1, X_2 and X_3) were first normalized, and then their effects on the strength (UCS) and cost of the paste backfill were studied by means of the experimental design presented below. The results of this design were then analyzed. This allowed us to develop the predictive models for UCS and cost, and we gained valuable information by simulation about the effects of the model parameters on these properties. The developed models were then experimentally verified to test their accuracy.

4.2. Experimental design

A rotatable orthogonal CCD [57,58] was used to develop the material models for the paste backfill. The measured responses were the UCS after UCS 14 days, UCS 28 days and UCS 56 days curing time and the cost. The experiments were run in random order. Five levels of variables were used in the experimental design. Based on previous experimental study [59,60] and on economic reasons (binder costs), the ranges of these three factors were determined to be: 1.4% to 4.2% PC I content, 1.2% to 2.5% PC V content and 1000 to 3000 ppm of sulphate in the mixing water. Indeed, a binder proportion higher than 7% is not economically feasible in the mining industry in Canada. The variables and their levels, both normalized and actual units, selected for this study are shown in Table 6. The correspondence between the normalized and actual values can be obtained using Eq. (3).

$$x = \frac{X - X_0}{\Delta X} \tag{3}$$

where x is the normalized value, X is the corresponding actual value, X_0 is the actual value in the center of the domain and ΔX is the increment of X corresponding to 1 unit of x.

Table 7 shows the experimental design carried out for developing the models.

Table 7
Performed experimental design

Exp. *N*°	PC I	PC V	Sulphate	Tailings	Water	% Cement
1	– 1	– 1	– 1	TB_2	W_2	2.6
2	– 1	– 1	1	TB_4	W_4	2.6
3	– 1	1	– 1	TB_2	W_2	3.9
4	– 1	1	1	TB_4	W_4	3.9
5	1	– 1	– 1	TB_2	W_2	5.4
6	1	– 1	1	TB_4	W_4	5.4
7	1	1	– 1	TB_2	W_2	6.7
8	1	1	1	TB_4	W_4	6.7
9	0	0	0	TB_3	W_3	4.7
10	0	0	0	TB_3	W_3	4.7
11	0	0	0	TB_3	W_3	4.7
12	0	0	0	TB_3	W_3	4.7
13	– 1.68	0	0	TB_3	W_3	2.3
14	1.68	0	0	TB_3	W_3	7.0
15	0	– 1.68	0	TB_3	W_3	3.6
16	0	1.68	0	TB_3	W_3	5.7
17	0	0	– 1.68	TB_1	W_1	4.7
18	0	0	1.68	TB_5	W_5	4.7
19	0	0	0	TB_3	W_3	4.7
20	0	0	0	TB_3	W_3	4.7

PC I, PC V and sulphate represent normalized values of percent PC I, percent PC V and concentration of sulphate of the mixing water, respectively.

5. Results and discussions

5.1. Model development

Following the construction of the CCD, as described above, Eq. (1) was used to fit the data of this experimental design. All of the data were then analyzed using standard statistical software, which estimates average effects, statistical significance and regression coefficients for all of the variables and their interactions. To identify the most important b terms to include in the developed equations, t tests were conducted. Thus, the square and interactions terms that were below 95% confidence levels were discarded from the models through stepwise regression. This allowed the development of three models for predicting the UCS after 14, 28 and 56 days of curing (UCS 14 days, UCS 28 days and UCS 56 days) and one model for predicting the cost (Cost) of paste backfill. Analysis of lack of fit was then performed to test if the developed second-order models adequately describe the behavior of the responses (UCS, Cost) in the design space before using the models for simulation. The test of lack-of-fit was undertaken by using the ANOVA. The JMP software was used [61].

The main results of the statistical analysis are shown in Tables 8 and 9 and in Fall and Benzaazoua [62]. The performed statistical analysis has shown that PC I, PC V and sulphate were all significant factors for the strength development, and cement content was the most significant factor. The interaction between PC I, PC V and sulphate concentration was significant, indicating a synergism between PC V and sulphate and an antagonistic effect between

Table 8
Main results of the analyses of variance and lack-of-fit

Models	Analyses of variance		Analyses of lack-of-fit	
	F	Prob>*P*	*F*	Prob>*P*
UCS 14 days	434	< .0001	1.90	.39
UCS 28 days	128	< .0001	0.75	.66
UCS 56 days	285	< .0001	0.63	.71

Table 9
Coefficient of determination of models

Models	UCS 14 days	UCS 28 days	UCS 56 days	Cost
r^2	.99	.99	.97	.99
r^2 Adjusted	.99	.99	.97	.99

PC I and sulphate after 28 days of curing. This confirms mathematically the nonresistant character of PC I and the resistance of PC V to sulphate attack. As we expected, cost is controlled by the proportions of PC I and V. Table 8 shows the results of the analysis of variance for the different models. It can be noted that all models have very high *F* values. The developed models are significant ($P < .0001$). The obtained coefficients of determination (Table 9) are very close to 1.0 for all models. Table 9 shows that the fitted models accounted for over 97% to 99% of the variation in UCS and cost. This indicates that the models are able to represent the real relationships among the selected model parameters (percent PC I, percent PC V, sulphate). The insignificant lack-of-fit (Table 8) also indicates that the developed models are able to represent the experimental data.

5.2. Effect of model parameters on its responses

The effects of the proportion of PC I, PC V and sulphate on the strength development and cost of the paste backfill were simulated based on the developed predictive models (UCS 14 days, UCS 28 days and UCS 56 days).

Fig. 3 shows, as expected, that the UCS of the paste backfill increases with the cement content. It can be also observed that among the model parameters, PC I and V have the largest effect on the strength development. The contribution of PC I to early strength gain (up to 28 days of curing) of the backfill is more significant than one of PC V. However, the effect of PC V on the strength gain increases with curing time and sulphate content (Fig. 3). This effect is stronger after 56 days of curing. This difference in the contribution of PC I and V to strength acquisition of the backfill depending on curing time and sulphate content may be attributed to the higher proportions of C_3S in PC I and to the vulnerability of PC I to sulphate attack. The C_3S is largely responsible for early strength of Portland cement [63–66]. The influence of sulphate on the UCS depends on its concentration, the curing time, the cement content and ratio. For a proportion of PC I = 2.8% and PC V = 1.9%, increasing of the sulphate content up to around 2000 ppm leads to higher UCS 14-day and UCS 28-day strength. This may be attributed to the precipitation of secondary hydrated minerals (as gypsum, ettringite and brucite) within the paste backfill. This contributes to the hardening mechanism of paste backfill. Indeed, in addition to the porosity reduction due to the process of cement hydration, these precipitates contribute to fill the void spaces or micropores within the

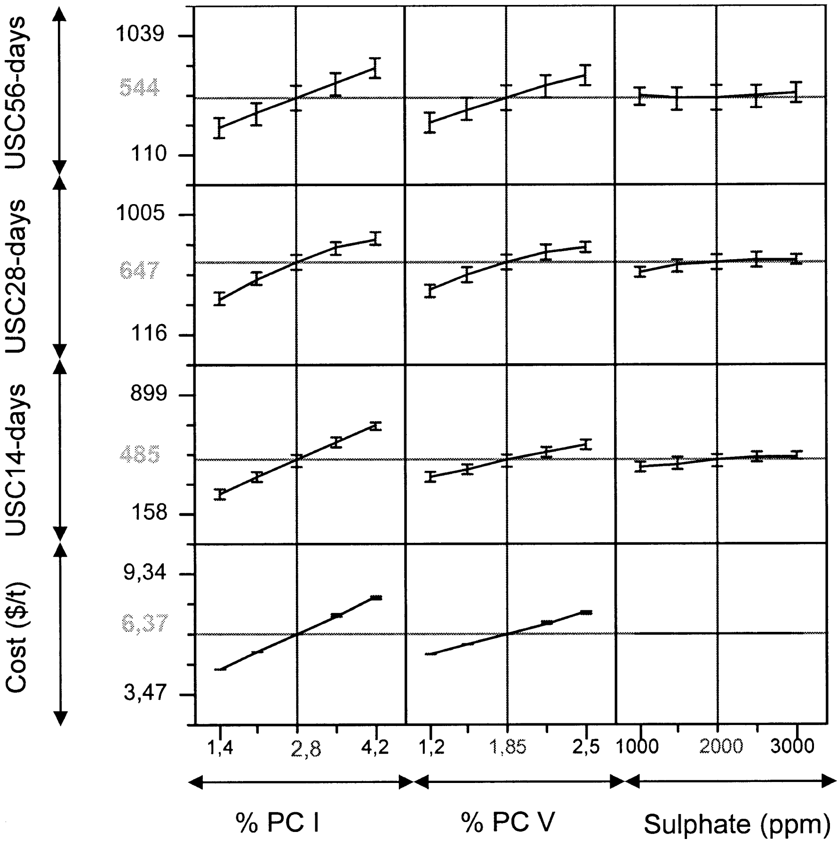

Fig. 3. Effect of PC I, PC V and sulphate (2000 ppm) on the strength development and cost of the paste backfill; UCS in kPa.

paste backfill. This also leads to a decrease in its internal porosity. The latter increases the backfill strength. These results are in substantial agreement with earlier experimental investigations performed by Anderson [45] and Benzaazoua et al. [67] and Belem et al. [68].

The SEM observations (Fig. 4) of a paste backfill sample after 28 days curing, done by Benzaazoua et al. [67], also support these modeling results. It can be observed that the pores within the paste backfill are filled with secondary gypsum. However, this increase in the early backfill strength with the sulphate content depends on the sulphate concentration. As shown in Fig. 3, elevated levels of sulphate in the mixing water significantly retard the hydration of the cement until 28 days of curing time. This may be caused by the strong inhibition of the hydration of C_3A by the sulphate [63–65,69]. On the other hand, for a proportion of PC I = 2.8% and PC V = 1.9%, it can be observed in Fig. 3 that, after 56 days curing (for sulphate = 2000 ppm), the backfill strength decreases. This can be attributed to the combined effect of the mineral phases (ettringite, gypsum and brucite) resulting from the reactions of sulphate with the cement hydration products. The formation of the mineral phase ettringite, which has high molar volumes, might have produced expansive pressures that has damaged the backfill. The formation of gypsum should also be taken into consideration as a source of this backfill strength deterioration. Indeed, the CH calcium hydroxide (CH) generated during the hydration of Portland cement may consequently permit the production of a large amount of gypsum within the backfill material. The gypsum is also capable of deteriorating the backfill material through a process, leading to the reduction of strength; this is followed by expansion and cracking. The SEM observations of paste backfill samples, published elsewhere [67], showed that the predominant mineral phases formed due to the sulphate effect was gypsum. Ettringite was formed in smaller amount. Additionally, although the used mixing waters contains only small amounts of magnesium ions (Mg^{2+}), the damaging effect of magnesium sulphate solution should be considered as an additional possible cause of the cemented backfill strength deterioration [24,28,29,39,43].

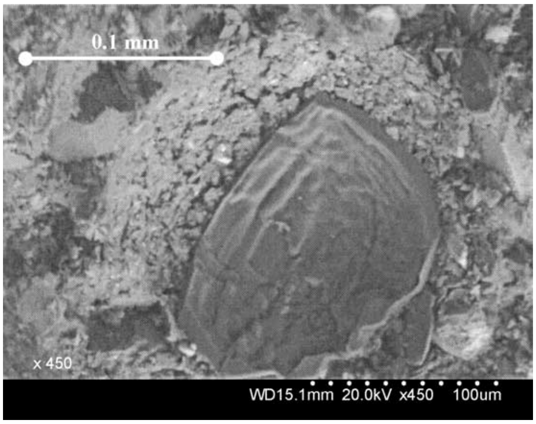

Fig. 4. SEM image showing paste backfill pore filled with secondary gypsum.

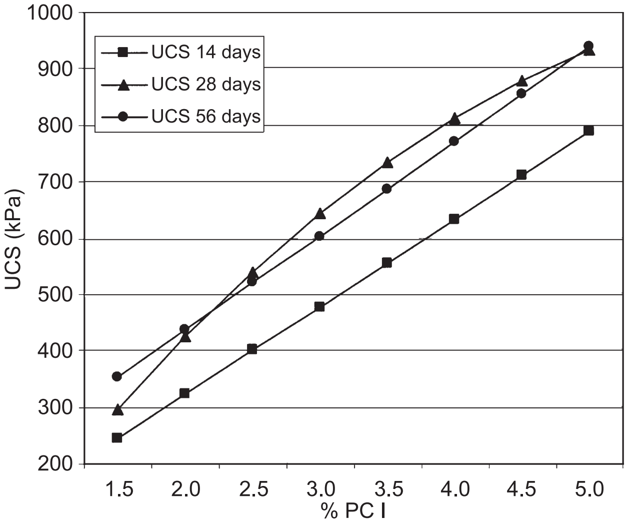

Fig. 5. UCS vs. percent PC I for sulphate concentration equal to 1000 ppm.

Figs. 5 and 6 summarize the results of the strength development simulation of paste backfill related to the proportion of PC I and initial sulphate concentration at fixed content of PC V (1.9%), while Figs. 7 and 8 illustrate the UCS development related to contents in PC V and sulphate at a PC I proportion fixed at 2.8%. These figures give a good comprehensive indication of the interaction between the amounts of PC I or V and the sulphate concentration and its effect on the strength gain or loss in backfill. From these prediction profiles, it is clear that the severity of the backfill strength deterioration due to damaging effect of sulphate increases with the sulphate concentration. It can also be noted that not only increased sulphate concentrations, but also the relative proportion of PC I to sulphate play a decisive role in the sulphate resistance of the hardened backfill. Fig. 5 indicates that, in the presence of

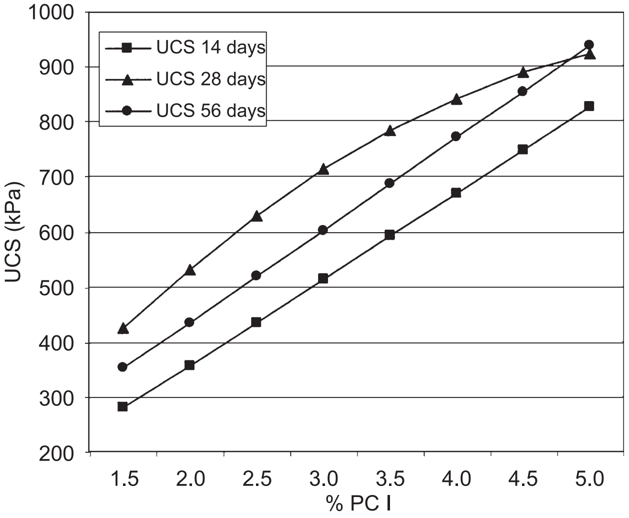

Fig. 6. UCS vs. percent PC I for sulphate concentration equal to 2000 ppm.

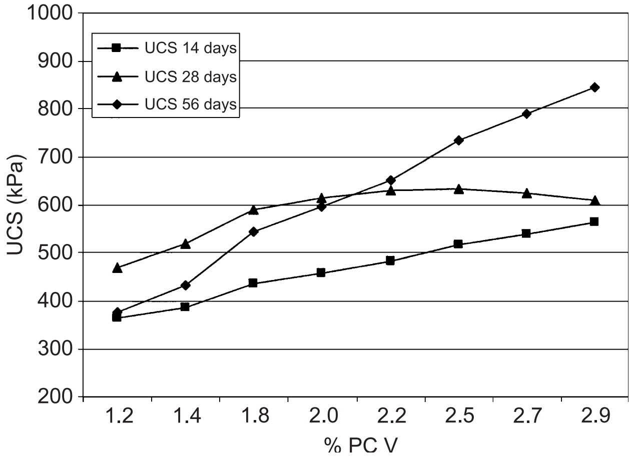

Fig. 7. UCS vs. percent PC V for sulphate concentration equal to 1000 ppm.

1000 ppm sulphate, the UCS 56 days starts to be lower than the UCS 28 days (increased strength loss) after about 2% of PC I. This strength deterioration with increasing PC I content gradually continues up to a proportion of 4.5% PC I. The maximum strength loss is reached at a PC I content of approximately 3.5%. With a PC I content of 4.5%, a gradual increase of UCS 56 days with the PC I proportion was observed. These observations can be attributed to the relative proportions of cement and sulphate within the backfill, to the amount of C_3A available and also to the amount of CH generated during the hydration of Portland cement. Thus, increasing the amount of PC I led to an increase in C_3A and in hydration products CH available to form expansive minerals, such as gypsum and/or ettringite or other phases, and this consequently causes a more severe sulphate attack. However, the severity is weakened with a certain proportion of PC I. This may be due to the fact that when the PC I content becomes elevated compared with the sulphate content, its positive effect on the resistance prevails over the negative effect of the sulphate, and this significantly reduces the rate and severity of sulphate attack.

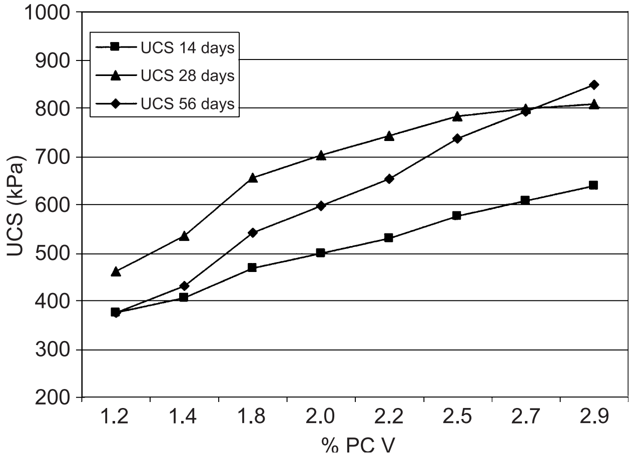

Fig. 8. UCS vs. percetnage PC V for sulphate concentration equal to 2000 ppm.

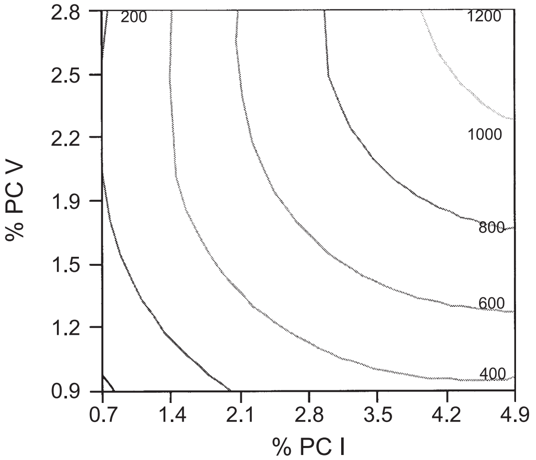

Fig. 9. Contour plot for UCS 28-day strength (in kPa) vs. percent PC I and V.

On the other hand, compared with the cement PC I, increased proportions of PC V almost always lead to strength gain and an improvement in the sulphate resistance of the backfill (Figs. 7 and 8). This is attributable to the reduction of the availability of C_3A susceptible to sulphate attack. However, Figs. 6 and 8 show that higher PC V proportions (compared with PC I proportions) cannot always prevent damage due to sulphate attack (strength loss of the backfill material after 56 days curing). This can be attributed to the sulphate attack on the CH component resulting from the hydration of C_3S and C_2S. This leads to the formation of gypsum, which is harmful. This is concordance with following works [70,71]. The latter have shown that, in some field cases and laboratory, the use of Type V cement, with the amount of C_3A less than 5%, may not prevent damage due to sulphate attack [71,72].

Additionally, higher PC V proportions significantly affect the early strength gain and cost of the backfill. This makes evident the importance of optimization of the binder ratio (PC I/PC V) for the design of cost-effective and sulphate-resistant paste backfill.

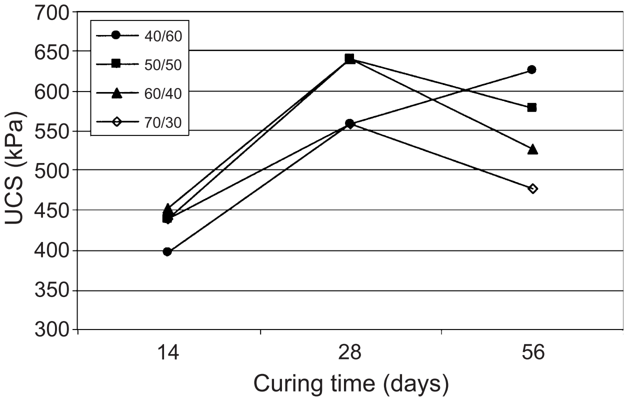

Fig. 10. Strength development of the paste backfill related to the binder ratio for a mixing water containing 2000 ppm sulphate (cement content: 4.5%).

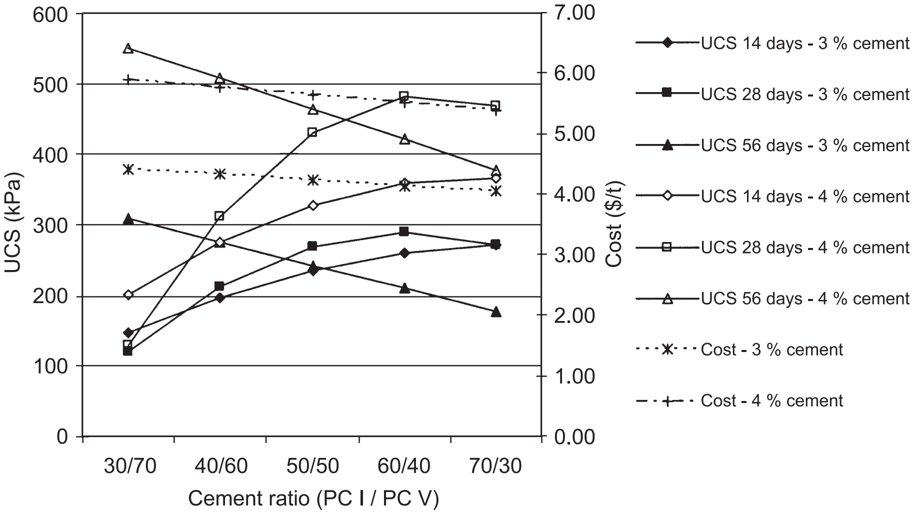

Fig. 11. UCS vs. cement ratio as a function of curing time and cement content, at a sulphate concentration = 1000 ppm.

5.3. Effect of binder ratio on sulphate resistance and cost of the paste backfill

Fig. 9 shows the contour plots for UCS 28 days of the backfill versus the mass proportion of PC I and V at a fixed sulphate concentration of the mixing water (2000 ppm). It confirms that not only is the proportion of used binder important for strength development of paste backfill, but the binder ratio (PC I/PC V) also plays a decisive role, as illustrated in Fig. 10. The latter presents the evolution of the backfill strength as related to the binder ratio. The binder mass proportion was fixed at 4.5%. The sulphate concentration was equal to 2000 ppm. It can be observed that for all of the binder ratios, the backfill strength increases up to 28 days of curing. The ratios 50/50 and 60/40 give the cemented backfill better strength at 28 days of curing (Fig. 10). This is due to the difference in the rate of strength gain between PC I and V, and also to the amount of C_3A and CH (resulting from cement hydration) available susceptible to sulphate attack.

Indeed, for the major constituents in Portland cement, the hydration of the C_3A and C_3S compounds occurs most rapidly [63–65]. Portland cements with higher proportions of these compounds (this means higher PC I/PC V ratios) generate higher early strength gains. However, high amounts of C_3A and/or C_3S lead to a decrease in the sulphate resistance of the cemented material. Therefore, the ratio 70/30 produces lower strength at 28 days curing than 60/40 or 50/50 does. However, at 56 days of curing, an inverse trend was observed. Lower ratios of PC I/PC V generate higher strength. The ratio 40/60 gave the highest strength to the backfill at 56 days of curing time. This might have resulted from the low C_3A content in PC V, which improved the ability of the binder to resist the negative effects of the sulphate. It is also of interest to note that the backfill strength loss was eliminated with a PC I/PC V blend ratio of 40/60 (Fig. 10).

Figs. 11 and 12 show the backfill strength after 14, 28 and 56 days of curing time for different binder contents and

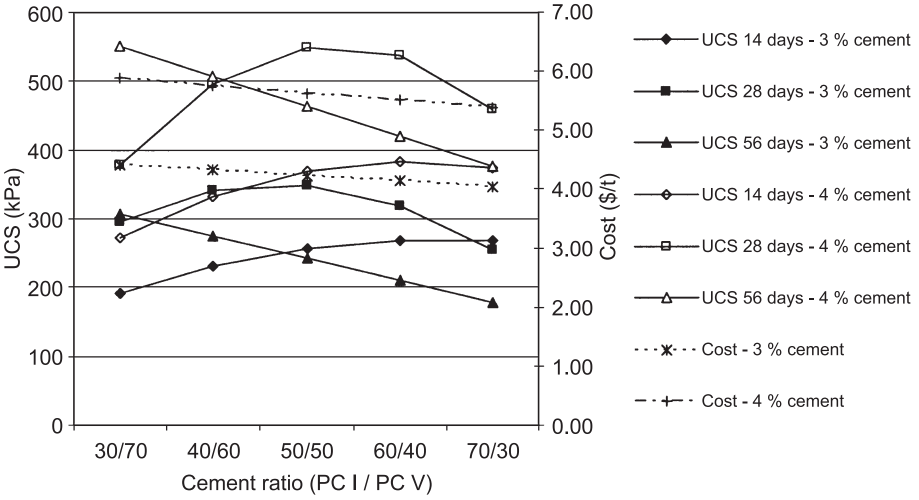

Fig. 12. UCS vs. cement ratio as a function of curing time and cement content, at a sulphate concentration = 2000 ppm.

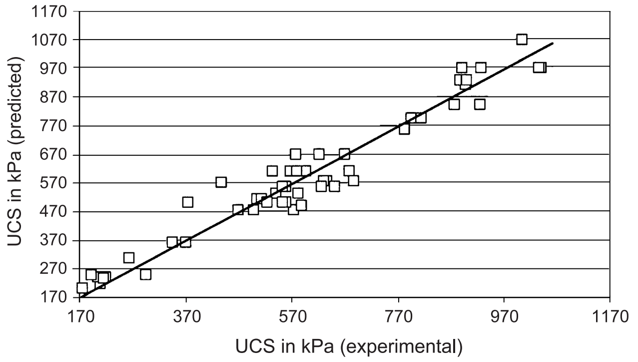

Fig. 13. Comparison of predicted UCS with those based on experimental tests.

ratios, depending on the sulphate concentrations in mixing water. They indicate that high cement content significantly reduces the severity of sulphate attack. A cost-effective cement ratio depends on the sulphate concentration and the cement content. It can be noted, e.g., that for a sulphate concentration lower than 1000 ppm and cement content of 4%, the ratio 50/50 is more effective than the 60/40 (Fig. 11). On the other hand, for a sulphate concentration of 2000 ppm and cement content of 4%, the most cost-effective cement ratio is a ratio of 40/60 (Fig. 12).

5.4. *Verification of the accuracy of the models*

The developed models were experimentally validated by conducting several tests. Figs. 13 and 14 show the comparison between the predicted UCS and cost values with those from experimental testing. All of the experimental results fit into the predicted result ranges, verifying that the model was valid. It can be seen that the model predictions and experimental values agree. Table 10 summarizes the comparison between values of UCS and cost obtained by doing additional experimental tests and with those predicted by using the developed models. It confirms the agreement between the measured and predicted values. This suggests that the model is able to describe the effects of PC I, PC V and sulphate concentration on backfill strength development and cost within the experimental domain. Globally, the results of verification of the validity of the developed models showed that the models permit us to accurately predict the UCS and the cost of the backfill under sulphate influence.

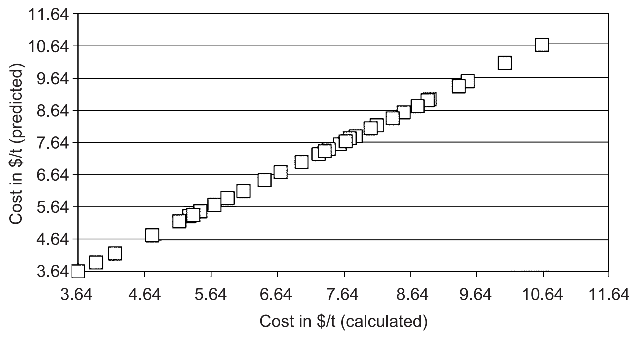

Fig. 14. Comparison of predicted paste backfill costs with those calculated (based on laboratory price evaluation).

Table 10
Selected results from experimental verification

	PC I (%)	PC V (%)	Sulphate (ppm)	Expe. values	Pred. values	Err. (%)
UCS 14 days (kPa)	4.2	1.2	1000	505	514	0.9
	4.2	2.5	1000	795	791	1.6
	4.2	1.2	3000	540	532	5.3
	1.4	2.5	1000	221	239	8.5
	2.8	1.85	2000	470	475	0.6
				average error = 3%		
UCS 28 days (kPa)	1.4	1.2	1000	177	202	14.3
	4.2	2.5	1000	901	926	2.8
	2.3	2.3	1000	686	646	1.4
	2.8	1.85	320	506	520	2.7
	2.8	4.2	2000	877	839	4.3
				average error = 7%		
UCS 56 days (kPa)	1.4	1.2	1000	163	143	12.1
	4.2	1.2	1000	567	610	7.5
	4.2	2.5	1000	927	968	4.4
	1.4	1.2	3000	170	140	15.7
	1.4	2.5	1000	553	502	9.3
				average error = 10%		
Cost (US$)/*t*	1.4	1.2	1000	3.634	3.64	0.2
	4.2	2.5	1000	9.175	9.16	0.1
	4.2	1.2	3000	7.134	7.12	0.2
	2.8	1.85	2000	6.405	6.4	0.1
	2.8	4.2	2000	10.110	10.10	0.1
				average error = 0.1%		

Err.: error; Expe.: experimental; Pred.: predicted.

6. Summary and conclusions

The objective of this study was to develop a method and a mathematical model to analyze and predict the effects of sulphates within the backfill on its mechanical and economic performance. RSM models that allow the prediction of the UCS at different curing times (14, 28 and 56 days) and cost of paste backfill have been developed. Based on these models, simulations were done to illustrate the strength evolution of the hardened backfill under sulphate influence, to evaluate the interactions between the different components of the models (CP10, CP50 and sulphate) and their effect on backfill sulphate resistance, as well as on its cost. It has been demonstrated that the RSM represents a reliable technique to model the properties (UCS and cost) of cemented backfill. It was also proven that a model based on quadratic functions form a good basis for the prediction of the UCS and cost of the cemented backfill that contains sulphates. The validation tests done showed good agreement between the experimental and modeling results.

From this study, it is clear that the sulphate significantly influences paste backfill strength. This influence is related to its concentration, the curing time and the amount and chemical composition of the cement. At concentrations lower than 2000 ppm, the sulphate contributes to the early strength gain of the backfill through a decrease of the internal porosity. This is due to the filling of pores within the backfill with hydrated cement products and by the precipitation of hydrated sulphates as gypsum and/or ettringite or other phases within the voids and micropores. These precipitates modify the microstructure of the paste backfill. However, for curing times greater than 28 days (in dependence of the sulphate concentration and cement proportion), this positive effect of the sulphate on UCS becomes a negative effect. Indeed, with increasing time, the hardened backfill becomes progressively weaker due to the destructive action of these expansive minerals that form in the backfill pores. Depending on the cement content and ratio, high sulphate concentration can slow the early strength gain of the backfill by inhibition of cement hydration.

The study has shown that the most effective cement ratio for avoiding strength loss due to sulphate attack is dependant on the sulphate concentration in the mixing water and the cement proportions.

The results of the models in this study agree with the experimental work on sulphate effect on cemented backfill done by previous authors [8–11,60,72].

Despite the results obtained in this study, the developed models do not include the effects of changing the physical properties of the tailings (particularly the tailings particles size) on the sulphate resistance of the paste backfill. A variation of the particles size might have a significant effect on the porosity of the paste backfill and, consequently, on the void space or micropores that can be filled with gypsum, ettringite and other phases. Therefore, further studies are necessary to build models that will account for change in the physical properties of the tailings and curing times of up to 120 days. It will be also interesting and useful in the future to carry out detailed mineralogical investigations to determine all the mineral phases produced by the effect of sulphate on cemented paste backfill.

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