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Response surface methodology optimization of hydraulic conductivity change by microbially induced $CaCO_3$ precipitation

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Abstract

In columns packed with glass beads in the range of 0.25 mm to 3 mm in diameter, the effect of the number of cells deposited in hydraulic conductivity of porous media using $CaCO_3$ precipitation produced by *Sporosarcina pasteurii* (ATCC 11859) was investigated in the present study. Following the introduction of resting *Sporosarcina pasteurii* cells into the columns, a precipitation solution containing 500 mM $CaCl_2$ and 500 mM urea was injected into the columns under continuous flow conditions. To investigate the relation between deposited cell number and hydraulic conductivity, Response surface methodology was applied. The results indicated that each response has been affected by factors in a different scale. By creating models, experimental design dimension can be declined.

Introduction

Many researchers have studied microbially induced calcium carbonate (MICP) to develop biotechnology methods such as improving construction materials [1], reducing hydraulic conductivity of porous media [2,3], changing soil behaviors [4,5], sealing cracks [6] using ureolytic bacteria. The precipitates can fill the pores within the soil matrix and bridge the loose sand particles, enhancing the soil's strength and stiffness while decreasing its permeability. The formation of calcium carbonate crystals from urea hydrolysis catalyzed by urease can be characterized as follows [2]:

$$NH_2CONH_2 + H_2O \xrightarrow{urease} 2NH_3 + CO_2 \quad (1)$$
$$2NH_3 + 2H_2O \rightarrow 2NH_4^{\ +} + 2OH^- \quad (2)$$
$$CO_2 + OH^- \rightarrow HCO_3^{\ -} \quad (3)$$
$$CaCl_2 \rightarrow Ca^{2+} + 2Cl^- \quad (4)$$
$$Ca^{2+} + HCO_3^{\ -} + OH^- \rightarrow CaCO_3(\downarrow) + H_2O \quad (5)$$

Urea is hydrolyzed to ammonia and carbon dioxide by ureolytic bacteria, and the generation of ammonia causes a rise in pH in the environment, which causes calcite precipitation when the environment contains calcium ions [2]. The effectiveness of the MICP process is affected by physiochemical and biological parameters such as pH, temperature, the presence of nutrients, their concentration, the concentration of precipitation reagents, and oxygen availability [7,8]. The density of cells, which is linked to the urease enzyme, is one of these factors.

In the present study, the experiments were conducted using columns packed with glass beads to investigate the effect of cell density on reduction in hydraulic conductivity. The relation between decrease in hydraulic conductivity and cell density was also investigated used design of experiment approach. Response surface methodology was selected as an appropriate method for this study.

Material and Method

Sporosarcina pasteurii [American Type Culture Collection (ATCC) 11859] was used in the experiments to obtain calcium carbonate precipitation. Resting cells of *Sporosarcina pasteurii* were provided by inoculating cells in Tris-YE medium which consisted of Tris buffer (130 mM, pH 9.0), ammonium sulphate (10 g/L) and yeast extract (20 g/L), overnight at 30°C with 120 rpm agitation. The optical densities (OD) of 0.15, 0.75, and 2.25 (abbreviated as OD_{600} 0.15, 0.75, and 2.25) at 600 nm was obtained using distilled water after harvesting the cells to investigate to the relationship between cell concentration and hydraulic conductivity.

In the experiments, 50 mL plastic syringes were utilized as columns, with an inner diameter of 3 cm and a height of 10 cm. For each experiment, the glass beads with an average diameter of 0.25 mm, 0.50 mm, 1.0 mm, 2.0 mm, and 3.0 mm were employed and saturated conditions were provided. The precipitation solution which contained 500 mM $CaCl_2$ and 500 mM urea was prepared aseptically.

First, cell suspension was introduced to the column. Then, the samples were collected from the effluent to calculate the number of cells deposited in the columns. Finally, the precipitation solution was introduced into the column until the hydraulic conductivity became stable. Manometer was employed to measure the hydraulic conductivity of the columns during the experiments.

Table 1 presents the factors and their design combinations showing the levels.

Table 1. Factors and levels of experiment

Factor Name	Units	L1	L2	L3	L4	L5
OD_{600}	-	0.15	0.75	2.25	*	*
Glass Bead Size	mm	0.25	0.5	1	2	3

Design of experiment

The goal of Design of Experiment (DoE) is to prepare and run experiments in order to collect data from appropriately specified experiments. It is feasible to gather a big quantity of information from smaller trials while using a predefined chart for a group of experiments and reviewing the results utilizing various methods. Because more than one factor can be investigated at once, the experiment becomes less expensive [9].

Response surface methodology

RSM is a modeling technique that employs mathematical and statistical methods [10]. The link between some input variables and one or more output variables, which is a response, is investigated. This process entails optimizing data gathered through preset experiments using polynomials fitted to the data. The variables that should be greater than one is represented by x_i in this approach, and the response is expressed as a value that needs to be optimized by y_i. x_i and y_i have a relationship. ε represents the error or noise in the response y_i, and x_i represents the independent variable – factor [11].

$$y_i = f(x_1, x_2, x_3, \ldots, x_i, \ldots, x_n) + \varepsilon \qquad (6)$$

Results and Discussion

In the present study, in a random order, 15 experimental trials were completed in keeping with the optimal (custom) design. Design-Expert 11.0.5 was used to get the results. The operating factors were optical density OD_{600} (A), and glass bead size (B). The responses were hydraulic conductivity (K) value (Y_1), deposited cell no (Y_2) and average amount of $CaCO_3$ (Y_3) were considered as the dependent factors. The performance of the design was evaluated by analyzing the minimization of the K value. The results of the experiment are given in Table 2.

The ANOVA results for the current investigation at a 95% confidence interval are summarized in Fig. 1. If the p-value is less than 0.05, this indicates that the model terms are significant. In the present study, for both response of hydraulic conductivity and deposited cell no; A, B and AB are significant model terms and for response of average amount of $CaCO_3$; A, B, AB, A^2 and B^2 are significant model terms.

The actual mathematical model for Y_1 is as follows:

$$K(cm/s) = 0.0351 - 0.0170 * OD_{600} + 0.2546 * \text{Glass Bead Size} - 0.0578 * OD_{600} * \text{Glass Bead Size}$$

The actual mathematical model for Y_2 is as follows:

$$\text{Depozite Cell No} = 3.0789E{+}08 + 1.6391E{+}09 * OD_{600} - 1.27754E{+}08 * \text{Glass Bead Size} - 5{,}8293E{+}08 * OD_{600} * \text{Glass Bead Size}$$

The actual mathematical model for Y_3 is as follows:

Average Amount of $CaCO_3$ (g)=-0.000393+ 0.000799 * OD_{600} + 0.014305 * Glass Bead Size – 7.99002E 06 * OD_{600} * Glass Bead Size -0,000288 * OD_{600}^2

For each model of Y_1, Y_2 and Y_3, R^2 and R^2 adjusted (abbreviated as R^2 adj) values was calculated. Response Y_1 has 0.9423 of R^2 value and 0.9423 of R^2 adj value. Response Y_2 has 0,9126 of R^2 value and 0.8888 of R^2 adj value. Response Y_3 has 0.6752 of R^2 value and 0.4947of R^2 adj value. As seen in Fig. 2, there are nonlinear relationship between OD_{600} and Glass Beads Size for all response types. A three-dimensional response surface plot is represented.

Table 2. Results of experiment with actual value

	Factor 1	Factor 2	Response 1	Response 2	Response 3
Run	**A: OD_{600}**	**B: Glass Bead Size**	**K (cm/s)**	**Deposited cell No**	**Average Amount of CaCO3 (g)**
3	2,25	1	0,191396	3,23874E+09	0,006
8	2,25	3	0,369667	9,37517E+07	0,00152753
12	2,25	2	0,222667	1,81727E+08	0,0123517
13	2,25	0,25	0,0108472	3,49731E+09	0,0020502
14	2,25	0,5	0,0271667	3,4482E+09	0,00950438
1	0,75	2	0,428667	7,37517E+07	0,0128517
4	0,75	3	0,632667	5,37517E+07	0,00158753
5	0,75	1	0,366396	7,81727E+08	0,0062
10	0,75	0,5	0,0871667	9,81727E+08	0,00910438
11	0,75	0,25	0,0348472	1,29731E+09	0,0022502
2	0,15	0,25	0,0424847	8,81727E+08	0,0026402
6	0,15	3	0,722667	2,37517E+07	0,00146875
7	0,15	1	0,366396	2,85727E+08	0,00624
9	0,15	2	0,568667	3,22517E+07	0,0118517
15	0,15	0,5	0,113485	5,81727E+08	0,00820438

ANOVA for 2FI model

Response 1: K (cm/s)

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	0,7328	3	0,2443	59,87	< 0.0001	significant
A-OD 600	0,1230	1	0,1230	30,15	0,0002	
B-Glass Bead Size	0,6023	1	0,6023	147,63	< 0.0001	
AB	0,0406	1	0,0406	9,95	0,0092	
Residual	0,0449	11	0,0041			
Cor Total	0,7776	14				

ANOVA for 2FI model

Response 2: Deposited Cell No

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	2,117E+19	3	7,057E+18	38,29	< 0.0001	significant
A-OD 600	6,698E+18	1	6,698E+18	36,34	< 0.0001	
B-Glass Bead Size	7,854E+18	1	7,854E+18	42,61	< 0.0001	
AB	4,135E+18	1	4,135E+18	22,43	0,0006	
Residual	2,027E+18	11	1,843E+17			
Cor Total	2,320E+19	14				

ANOVA for Quadratic model

Response 3: Average Amount of CaCO3 (g)

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	0,0002	5	0,0000	3,74	0,0414	significant
A-OD 600	2,342E-07	1	2,342E-07	0,0261	0,8752	
B-Glass Bead Size	7,401E-06	1	7,401E-06	0,8256	0,3872	
AB	7,768E-10	1	7,768E-10	0,0001	0,9928	
A^2	2,107E-07	1	2,107E-07	0,0235	0,8815	
B^2	0,0002	1	0,0002	18,48	0,0020	
Residual	0,0001	9	8,964E-06			
Cor Total	0,0002	14				

Figure 1. ANOVA for each response's model by using Type III – partial sum of squares

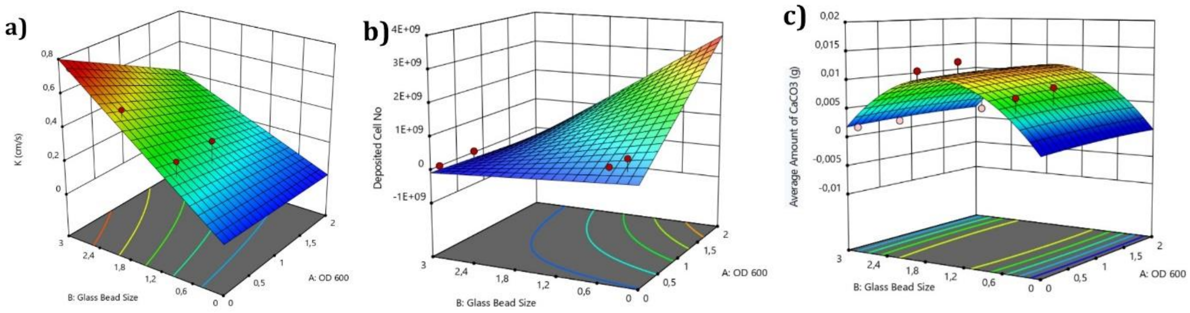

Figure 2. Surface plot of (a) response Y_1, (b) response Y_2, (c) response Y_3

Conclusion

The effect of the number of cells deposited on the decrease in hydraulic conductivity of porous media utilizing $CaCO_3$ precipitation produced by *Sporosarcina pasteurii* (ATCC 11859) was examined in columns packed with glass beads in the range of 0.25 mm to 3 mm in diameter. A precipitation solution containing 500 mM $CaCl_2$ and 500 mM urea was fed into the columns under continuous flow conditions after the resting *Sporosarcina pasteurii* cells were introduced into the columns. Response Surface Methodology was used to study the relationship between deposited cell number and hydraulic conductivity. The findings revealed that factors on a different scale influenced each response. The experimental design dimension can be reduced by creating models. Based on the results of the experiments with various factors and levels, it can be stated that a determined prediction model produced with RSM is effective for analyzing the optimum points and values of preset factors while remaining flexible.

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