Sustainable Chemical Engineering
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Research Article

Ratio Optimization of Magnesium Oxychloride Cement and Improvement of Its Water Resistance Based on Response Surface Methodology

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Received: 27 December 2023; **Revised:** 9 January 2024; **Accepted:** 12 January 2024

Abstract: Magnesium oxychloride has excellent early strength, lightweight and environmentally friendly properties, and excellent application value. However, insufficient water resistance affects its engineering application. This paper uses fly ash to improve the water resistance of magnesium oxychloride cement (MOC). The response surface method was used to optimize the ratio of magnesium oxychloride cement. The influence of fly ash content, magnesium oxychloride cement ratio, and their interaction on the water resistance of magnesium oxychloride cement was studied using a response surface experiment. The fly ash content and magnesium oxychloride cement ratio were optimized to form magnesium oxychloride cement with good water-resistance. The experimental results show that the fitting efficiency of the response surface model is $R^2 = 0.9951$; the model reflects the relationship between the factors of magnesium oxychloride cement and water resistance well. The magnesium oxychloride cement has a maximum softening coefficient of 0.898 when the amount of fly ash added is 21.94%, and the molar ratio of magnesium oxychloride cement is 11.49 : 1 : 11.77. The magnesium oxychloride cement has good water resistance by optimizing the ratio based on external fly ash and response surface methodology. The study provides a reference for improving the water resistance of magnesium oxychloride cement.

***Keywords*:** magnesium oxychloride cement, fly ash, water resistance, response surface methodology, optimization

1. Introduction

With the rapid development of materials science, magnesium-based cementitious materials have become an indispensable primary material in the building materials industry in recent years.[1,2] Magnesium cement is an air-hardening cementitious material mainly made of MgO, with a simple production process, many excellent physical and mechanical properties, and unique engineering properties.[3,4] Magnesium oxychloride cement is a mixture of $MgCl_2$, MgO, and H_2O in a certain proportion, which is fully hydrated to form an air-hardening cementitious material mainly composed of a $Mg(OH)_2 \cdot b\ MgCl_2 \cdot c\ H_2O$.[5] Russia produces brick products and masonry wall materials using magnesium oxychloride cement. At the same time, Austria has invested in many magnesium cement particle board production lines using magnesium oxychloride cement as the main component. Eitomation in the Netherlands, Weiler

DOI: https://doi.org/10.37256/sce.5120244166

in Germany, and Herrando Industrial S. A in Spain all use magnesium oxychloride products as their main products. Magnesium oxychloride cement has high fire resistance, high mechanical strength, and is environmentally friendly. It has exceptionally high application value in building decoration and special environmental conditions.[6,7] Magnesium oxychloride cement has excellent early strength performance. However, it still suffers from defects such as poor water resistance and unstable volume, which seriously limits the engineering application of magnesium oxychloride cement.[8,9] The performance of magnesium oxychloride cement is influenced by various factors, including external temperature and environmental humidity, the raw material ratio, and additives. Li et al.[10] studied the effect of raw material composition on the performance of magnesium oxychloride cement and found that magnesium oxychloride cement performs better at a better molar ratio of $MgO/MgCl_2$ and $H_2O/MgCl_2$. With the change in ratio, the mechanical properties of the cement slurry undergo significant changes. Therefore, it is necessary to optimize its ratio design to improve the performance of magnesium oxychloride cement. Poor water resistance is one of the main reasons limiting the widespread application of magnesium oxychloride cement. Therefore, many scholars have conducted extensive research on improving the water resistance of magnesium oxychloride cement, mainly focusing on exploring the reasons for poor water resistance, modification measures, and modification mechanisms. Xu et al.[11] studied the use of hydrophobic admixtures to modify the pore structure of MOC. They found that the hydrophobic mixture and gel substances prevented water erosion and improved water resistance. Huang et al.[12] studied using silica fume to improve the water-resistance of magnesium oxychloride cement. They concluded that the formation of magnesium chloride hydrate gel and the densification of microstructure contributes to the significant improvement of the water resistance of MOC. Abdulsada et al. and Huang et al.[13,14] studied the effect of fly ash on the performance of magnesium oxychloride cement and found that fly ash can improve the water resistance of MOC. However, the early strength of magnesium oxychloride cement decreased by adding fly ash. Pivak et al.[15] investigated the effect of fly ash and other additives on magnesium oxychloride cement, and the results showed that the water resistance of modified MOC slurry was significantly improved in MOC containing fly ash and phosphoric acid. Li et al.[16] optimized the ratio of magnesium oxychloride raw materials using response surface methodology (RSM), significantly improving the strength of magnesium oxychloride cement. Research has found that fly ash can effectively improve the water-resistance of magnesium oxychloride cement. However, current research on using fly ash to improve the performance of magnesium oxychloride is mostly based on the control variable method. When fly ash is added to magnesium oxychloride cement, it will interact with the components of magnesium oxychloride cement. There is relatively little research on the interaction between fly ash and magnesium oxychloride cement. Using a single additive to improve the water-resistance of magnesium oxychloride cement has a poor effect, and improving the water resistance of magnesium oxychloride cement still requires scientific mixed design methods.

The response surface methodology has extremely high feasibility in process and ratio optimization. This article studies the method of improving the water resistance of magnesium oxychloride cement with fly ash. It optimizes magnesium oxychloride's mix ratio and fly ash content using response surface methodology. The effect of fly ash and its interaction with magnesium oxychloride cement components on the water resistance of magnesium oxychloride cement was studied through response surface analysis. The magnesium oxychloride cement with good water resistance was formed by optimizing the mix ratio. This study provides a reference for using scientific methods to improve the water-resistance of magnesium oxychloride cement.

2. Materials and methods

2.1 *Materials*

In the experiment, industrial grade lightly burned magnesium oxide with a content of 85.6% was used as the main material, purchased from Yingkou, Liaoning. The composition of magnesium oxide is shown in Table 1. Industrial-grade hexahydrate magnesium chloride with a purity of 45.42%, purchased from a chemical plant in Shandong, is used as a regulator. The chemical composition of magnesium chloride is shown in Table 2. Use grade II fly ash as the admixture.

Table 1. Chemical composition of lightly burned magnesium oxide

Component	MgO	SiO_2	CaO	Fe_2O_3	Al_2O_3	Ignition loss
Content/wt %	85.8	6.45	1.08	0.24	0.39	7.03

Table 2. Chemical composition of hexahydrate magnesium chloride

Component	$MgCl_2$	NaCl	$CaCl_2$	KCl	SO_4^{2-}
Content/wt %	45.42	0.55	0.17	0.36	1.76

2.2 *Method*

2.2.1 *Sample preparation*

The weighed water and hexahydrate magnesium chloride were added to the mixer according to the designed proportion of magnesium oxychloride raw materials (molar ratio) and quickly stirred for 2 minutes (TG-3060A, Shenyang Taige Petroleum Instrument Equipment Co., Ltd. China). Lightly burned magnesium oxide was added and stirred slowly for 1 minute. Additives were added to the obtained mixture according to the mass fraction of magnesium oxide, and the mixture was stirred slowly for 60 seconds to form a magnesium oxychloride cement sample.

The MOC cement was poured at a standard mold with a diameter of 50.8 mm × 50.8 mm × 50.8 mm, and then it was placed in a constant temperature and humidity box for continuous curing. After curing to the specified age, take samples out and conduct sample performance tests.

2.2.2 *Box behnken experimental design*

The molar ratio of $MgO/MgCl_2$ (A) was selected at three levels of 6, 9, and 12, the molar ratio of $H_2O/MgCl_2$ (B) was selected at three levels of 8, 11, and 14, and the fly ash (C) content was selected at three levels of 10%, 20%, and 30% of magnesium oxide mass, a three-factor and three-level experiment was designed based on the Box Behnken module with the cement softening coefficient (R1) as the response value, the influence of each experimental factor and its interaction on the softening coefficient of magnesium oxychloride cement was studied with a total of 15 sample experiments.

The codes 1, 0, and -1 were used to explain a single factor's upper, middle, and lower levels. The experimental factor design and level coding are shown in Table 3.

Table 3. Variable encoding and level

Actual variable	Code	Code level		
		-1	0	1
$MgO/MgCl_2$ (mol)	A	6	9	12
$H_2O/MgCl_2$ (mol)	B	8	11	14
Fly ash (wt %)	C	10	20	30

2.2.3 *Compressive strength test*

The cement sample formed after curing was taken out, placed in the center of the hydraulic pressure testing machine (HY-200 universal mechanical testing machine, Shanghai Hengyi Precision Instrument Co., Ltd, China), and uniformly loaded at a constant loading rate of 1.2 kN/S until the sample was damaged and the maximum load did not change. The data was recorded.

The softening coefficient characterizes the water resistance of magnesium oxychloride cement and is another important indicator for evaluating product quality. The size of the magnesium oxychloride cement specimen used in this study to determine the softening coefficient is 50.8 mm × 50.8 mm × 50.8 mm, with a curing period of 28 days. The measurement method is as follows:[17,18] The compressive strength of the cement cured for 28 days and the cement was immersed in distilled water and placed in a constant temperature environment of 25 °C for 7 days were measured, and the softening coefficient R_f was calculated according to equation.

$$R_f = \frac{R_{(w,7)}}{R_{(a,28)}} \tag{1}$$

In the formula: $R_{(w,\,7)}$- The compressive strength of the sample soaked in water for 7 days; $R_{(a,\,28)}$- The compressive strength of a sample cured in air for 28 days.

Table 4. Box behnken experimental design and results

No.	Code			Factor			Softening coefficient	
	A	B	C	$MgO/MgCl_2$ (mol)	$H_2O/MgCl_2$ (mol)	Fly ash (wt %)	Actual value	Predictive value
1	0	-1	-1	9	8	10	0.77	0.765
2	0	-1	1	9	8	30	0.82	0.822
3	1	1	0	12	14	20	0.83	0.831
4	0	1	-1	9	14	10	0.72	0.717
5	-1	1	0	6	14	20	0.69	0.686
6	1	0	-1	12	11	10	0.85	0.851
7	0	0	0	9	11	20	0.9	0.89
8	0	1	1	9	14	30	0.75	0.755
9	0	0	0	9	11	20	0.88	0.89
10	-1	-1	0	6	8	20	0.76	0.758
11	1	0	1	12	11	30	0.87	0.863
12	-1	0	-1	6	11	10	0.68	0.686
13	-1	0	1	6	11	30	0.77	0.768
14	1	-1	0	12	8	20	0.87	0.873
15	0	0	0	9	11	20	0.89	0.89

3. Results and discussion

3.1 *Establishment of multiple quadratic regression equations*

In order to make the magnesium oxychloride cement have good long-term stability in the working environment, it requires good water resistance. The experimental design scheme and measured results of the response surface are shown in Table 4.

The binomial prediction model of the water resistance of the magnesium oxychloride cement and three factors obtained through experimental data fitting is as follows:

$$R1 = 0.89 + 0.065A - 0.0288B + 0.0238C + 0.0075AB - 0.0175AC - 0.005BC - 0.0375A^2 - 0.065B^2 - 0.06C^2 \quad (2)$$

In the formula, R1 represents the softening coefficient (water resistance index) of magnesium oxychloride cement; A represents the molar ratio of $MgO/MgCl_2$; B represents the molar ratio of $H_2O/MgCl_2$; C represents the amount of fly ash added; AB represents the interaction between $MgO/MgCl_2$ and $H_2O/MgCl_2$; AC represents the interaction between $MgO/MgCl_2$ and fly ash; BC represents the interaction between $H_2O/MgCl_2$ and fly ash.

This binomial equation considers the influence coefficients of factors on the water resistance of magnesium oxychloride cement, reflecting the influence of single and double-factor interactions on the water resistance of magnesium oxychloride cement.[19]

3.2 *Significance test*

The analysis of variance (ANOVA) was used to analyze variance on the binomial equation, and significance testing was conducted on the sources of error in the binomial model. The results of the analysis of variance are shown in Table 5.

Table 5. Analysis of variance for binomial equations

Source	Sum of squares	Df	Mean square	F-value	P-value	Significance
Model	0.0764	9	0.0085	113.12	< 0.0001	Significant
A-$MgO/MgCl_2$	0.0338	1	0.0338	450.67	< 0.0001	-
B-$H_2O/MgCl_2$	0.0066	1	0.0066	88.17	0.0002	-
C-Fly ash	0.0045	1	0.0045	60.17	0.0006	-
AB	0.0002	1	0.0002	3.00	0.1438	-
AC	0.0012	1	0.0012	16.33	0.0099	-
BC	0.0001	1	0.0001	1.33	0.3004	-
A^2	0.0052	1	0.0052	69.23	0.0004	-
B^2	0.0156	1	0.0156	208.00	< 0.0001	-
C^2	0.0133	1	0.0133	177.23	< 0.0001	-
Residual	0.0004	5	0.0001	-	-	-
Lack of fit	0.0002	3	0.0001	0.5833	0.6812	Not significant
Pure error	0.0002	2	0.0001	-	-	-
Cor total	0.0767	14	-	-	-	-

In Table 5, the binomial regression model for the softening coefficient of magnesium oxychloride cement has an F-value of 113.12 and a P-value of < 0.05, indicating significant significance at the 95% confidence level. The P-value of the mismatch term 0.5833 is much greater than 0.05, which means the experimental data has a high degree of fitting, and the mismatch term is not significant. Figure 1 shows that the predicted values of the prediction model fit well with the actual values. The complex correlation coefficient R^2 of the model is 0.9951, the corrected correlation coefficient R_{adj}^2 is 0.9863, and the coefficient of variation (CV) is 1.08% < 10%, indicating that the selected model has high fitting accuracy and strong reliability.[20,21] The P-values of independent variables A, B, and C are less than 0.05. Independent variables A, B, and C significantly impact the corresponding value R1, with a significance order of A > B > C. The significant order of interaction between factors is AC > AB > BC.

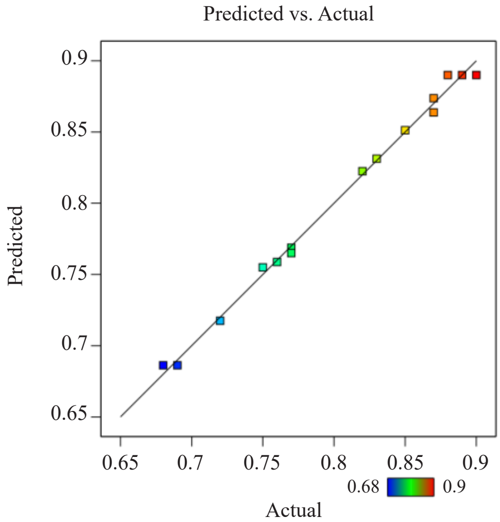

Figure 1. Comparison between predicted and actual values of binomial model

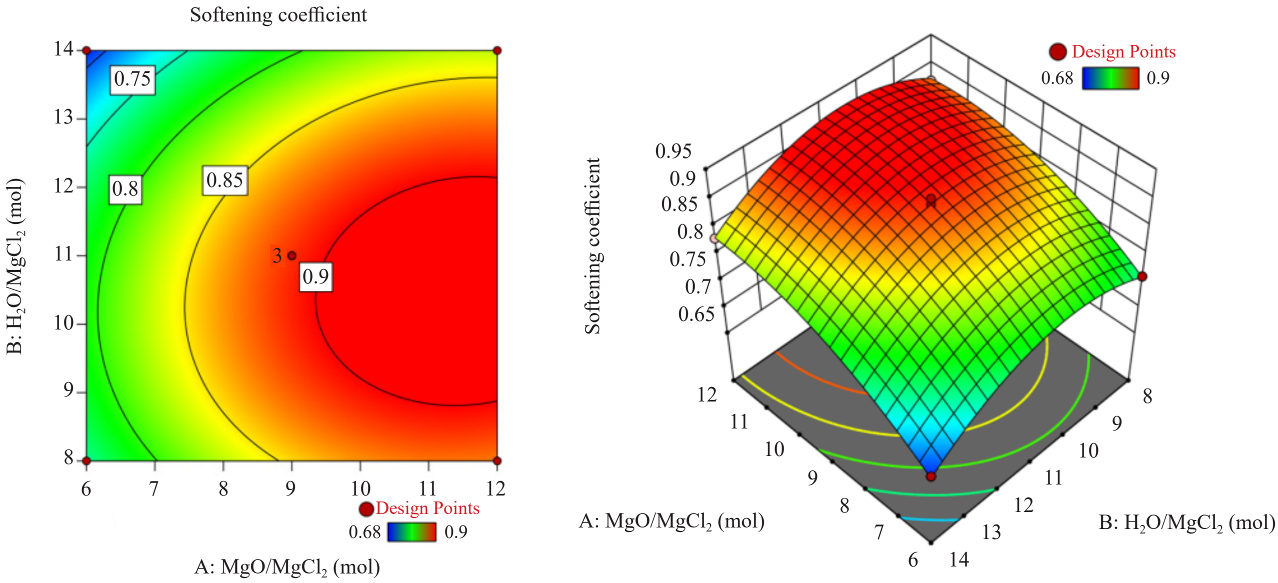

Figure 2. Interactive effects of $MgO/MgCl_2$ and $H_2O/MgCl_2$ on the softening coefficient

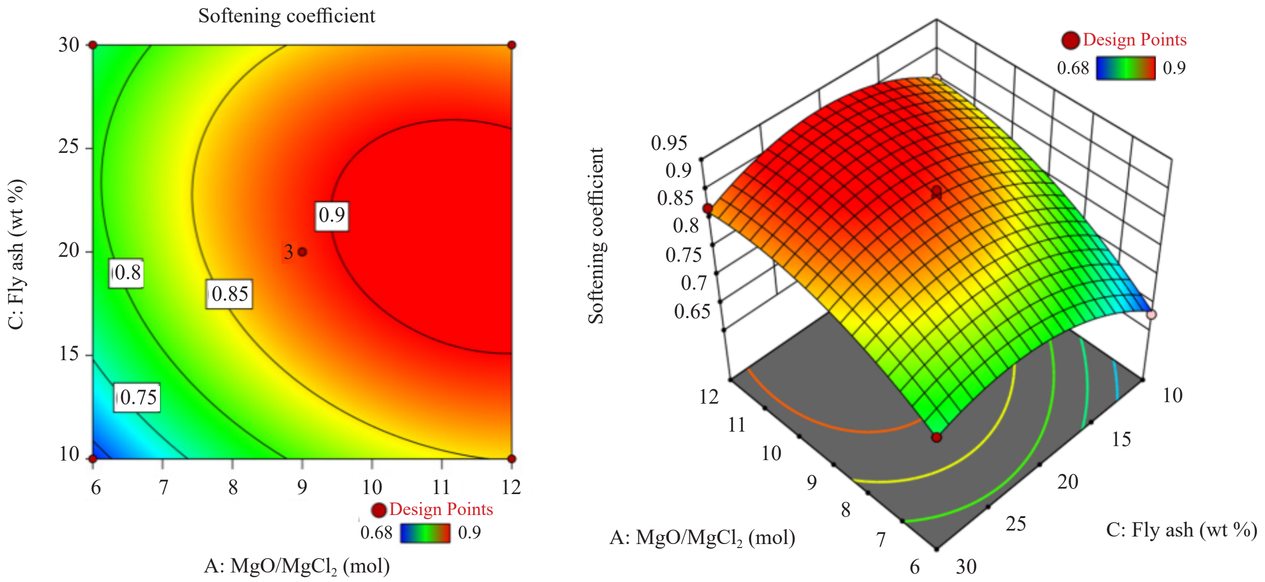

Figure 3. Interactive effect of MgO/MgCl$_2$ and fly ash on the softening coefficient

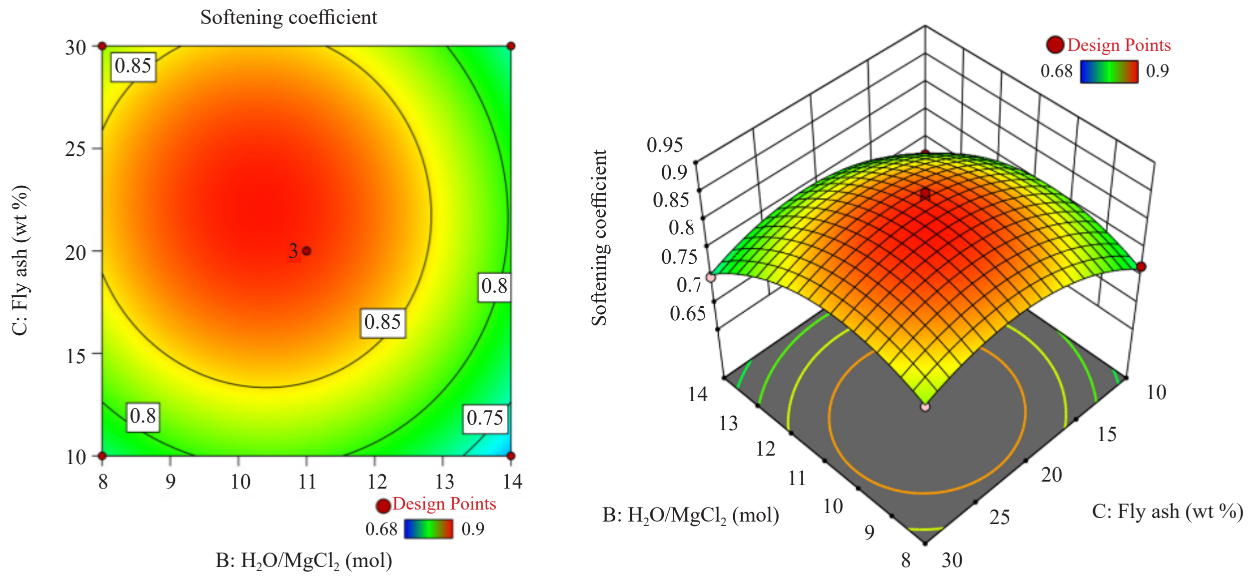

Figure 4. Interactive effect of H$_2$O/MgCl$_2$ and fly ash on the softening coefficient

3.3 *Analysis of factor interaction*

In order to visually observe the effect of the interaction between MgO/MgCl$_2$, H$_2$O/MgCl$_2$, and fly ash content on the softening coefficient of magnesium oxychloride cement, a three-dimensional response surface graph was constructed using the molar ratio of MgO/MgCl$_2$ (A), H$_2$O/MgCl$_2$ (B), and fly ash content (C) as the horizontal and vertical coordinates, and the softening coefficient of magnesium oxychloride cement (R1) as the Z-axis coordinate, as shown in Figures 2-4.

Figure 2 shows the contour and response surfaces of the interaction between MgO/MgCl$_2$ and H$_2$O/MgCl$_2$ on the softening coefficient of magnesium oxychloride cement. The response surface is a parabolic surface with an open downward ridge. The surface of MgO/MgCl$_2$ is steep, while the surface of H$_2$O/MgCl$_2$ is gentle, indicating that MgO/

$MgCl_2$ significantly impacts the softening coefficient of magnesium oxychloride cement. With the increase of $MgO/MgCl_2$, the softening coefficient of magnesium oxychloride cement first significantly increases, then slightly decreases. When the proportion of magnesium oxide increases, magnesium oxychloride cement has more solid phase support strength, which can form more stable crystal phases, resulting in a dense structure of the cement stone, low permeability, and significantly increased water resistance. As the proportion of magnesium oxide continues to increase, a significant amount of unhydrated magnesium oxide remains in magnesium oxychloride cement, resulting in a more porous structure of the cement stone, higher permeability, and a decline in water resistance. As the proportion of water increases, the degree of hydration of magnesium oxychloride cement increases to a certain extent, the structure becomes denser, and the water resistance increases. As the water ratio is too high, the structure of magnesium oxychloride cement becomes looser, and the water resistance decreases.[22] Under appropriate proportions, magnesium oxychloride cement has a higher and better water resistance coefficient.

Figures 3 and 4 illustrate the interaction between fly ash and $MgO/MgCl_2$ and between fly ash and $H_2O/MgCl_2$ on the softening coefficient of magnesium oxychloride cement. The results show that with the increase in fly ash dosage, the water resistance coefficient of magnesium oxychloride cement first increases and then decreases. Under a certain fly ash dosage, the water resistance of magnesium oxychloride cement can be significantly improved. Fly ash is weakly alkaline and can promote the hydration of cement in magnesium oxychloride cement, resulting in a denser cement structure. Meanwhile, fly ash is filled in magnesium oxychloride cement, with lower porosity and improved water resistance. When the amount of fly ash added is too large, it destroys the complete structure of magnesium oxychloride cement, leading to loose structure and poor water resistance.[23] The interaction between fly ash and $H_2O/MgCl_2$ on the softening coefficient of magnesium oxychloride cement is significant. When fly ash is added to magnesium oxychloride cement, it affects the fluidity of magnesium oxychloride cement. At an appropriate $H_2O/MgCl_2$ ratio, it is beneficial for the hydration of magnesium oxychloride cement, improves its structural stability, and thus improves its water resistance. It indicates that under appropriate fly ash dosage and $H_2O/MgCl_2$ ratio, magnesium oxychloride cement has a higher water resistance coefficient and better water resistance.

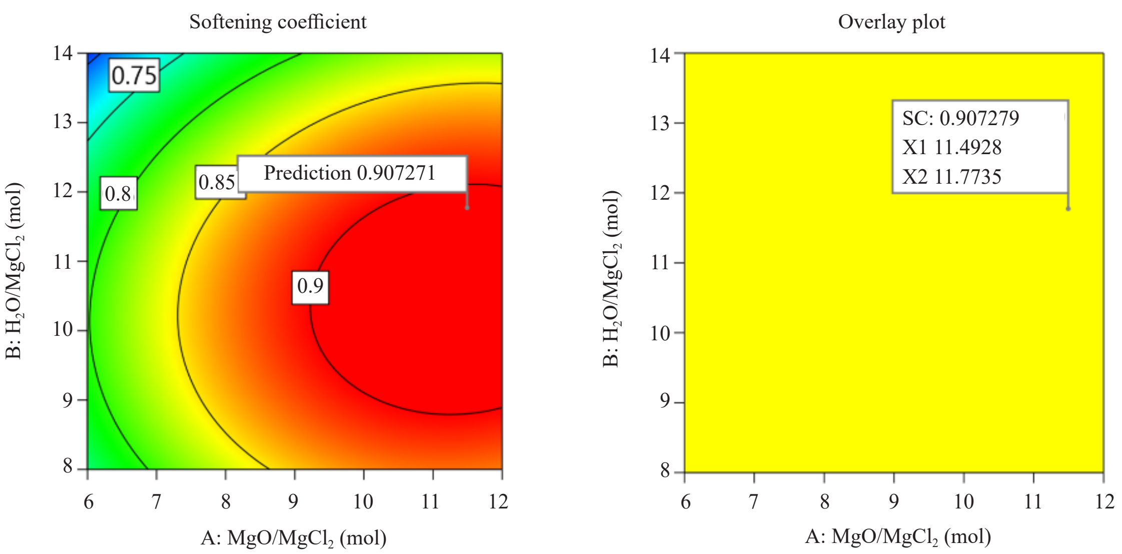

Figure 5. Prediction of softening coefficient of magnesium oxychloride cement

3.4 *Optimization and verification*

After analyzing the influence of $MgO/MgCl_2$, $H_2O/MgCl_2$, and fly ash on the water resistance coefficient of magnesium oxychloride cement through response surface methodology, the optimal material ratio for constructing magnesium oxychloride cement with good milk water properties was determined to be: magnesium oxide: magnesium chloride: water ratio of 11.49 : 1 : 11.77; The amount of fly ash added is 21.94% of the mass of magnesium oxide. The predicted softening coefficient is shown in Figure 5.

The softening coefficients of magnesium oxychloride cement were repeated three times under these conditions, with an average value of 0.898. The sample of magnesium oxychloride cement after adding fly ash is shown in Figure 6. The sample of magnesium oxychloride cement added with fly ash has a relatively dense structure, and its fracture surface is intact and smooth after fracturing, indicating that fly ash can effectively fill the pores of magnesium oxychloride cement, which is also one of the reasons for improving the water resistance of magnesium oxychloride cement. The predicted softening coefficient of magnesium oxychloride cement obtained through the established binomial prediction model was 0.907, with an average error of 1.00%. The magnesium oxychloride cement constructed based on the response surface method and Box-Behnken design with the optimal ratio has good water resistance performance. This result is reliable according to the analysis results of the response model.

(a) (b)

Figure 6. Magnesium oxychloride cement sample with added fly ash (a: Complete sample; b: Fracture surface)

4. Conclusion

(1) The water resistance of magnesium oxychloride cement is significantly affected by its composition and fly ash, which can be improved by improving the raw material ratio and adding fly ash. The interaction between fly ash and $H_2O/MgCl_2$ is significant.

(2) When the molar ratio of $MgO:H_2O:MgCl_2$ in magnesium oxychloride cement is 11.49 : 1 : 11.77 and 21.94% fly ash is added, magnesium oxychloride cement has the best water resistance performance.

(3) The application of response surface methodology can effectively optimize the ratio of magnesium chloride cement and fly ash and improve the water resistance of magnesium chloride cement.

Conflict of interest

The authors declare no competing financial interest.

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