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Optimization of sludge dewatering process by inorganic conditioners under mild thermal treatment

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ABSTRACT

Conditioning sewage sludge with $FeCl_3$ and steel slag under mild temperature has been proved to be an effective mean to enhancing the dewaterability. The Box–Behnken experimental design and response surface methodology (RSM) were chosen to evaluate the combined effects of the three independent variables. Capillary suction time (CST) was used to characterize sludge dewatering. Zeta potential, Fourier transformed infrared spectroscopy, and scanning electronic microscopy (SEM) were employed to explore influencing mechanisms. The results show that after RSM optimization, CST could drop to 7.8 ± 0.9 s at $FeCl_3 = 5.89$ mg/g, steel slag = 48.25 mg/g, and $T = 52.06$℃. An increase in temperature combined with conditioners led to decrease in zeta potential. The variation in functional groups implied that conditioners interacted with the component of sludge during the hydrothermal treatment, SEM images revealed the rupture of sludge flocs, and formation of block structures as temperature increases. This work offered a new understanding to the role of temperature and conditioners in improving sludge dewaterability.

Keywords: Sewage sludge; Dewatering; Response surface methodology; Steel slag; $FeCl_3$; Mild thermal treatment

1. Introduction

Sewage sludge production is increasing every year, but the treatment and disposal of sludge is still a problem to be solved in most countries. In China, with the development of industrialization and urbanization, large amount of sewage has been generated [1]. As to sludge production, its total amount increased by an average annual growth of 13% from 2007 to 2013 in China [2]. Dewatering is a key process in sludge volume reduction, transport, and ultimate disposal. Such sludge, however, is often difficult to be dewatered due to the strong hydrophilicity. Nowadays, a variety of approaches have been developed to improve the sludge dewaterability, such as thermal treatment, ultrasonication [3], electro-dewatering [4]. Studies about thermal treatment have been reported earlier. Microbial flocs dissolution, cell rupture, and organic compound hydrolysis reduced the binding effect of sticky granular sludge on water, which has fundamentally changed water characteristic in the sludge, thus improving the dewaterability of sludge

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[5]. Neyens and Baeyens summarized some previous studies [6]. In these studies, some chemicals were introduced to cooperate with the thermal treatment to get a better dewaterability, while the temperature usually exceed 90℃. The high temperature means large amount energy-consuming, few researchs are found to promote sludge dewatering at a lower temperature of hydrothermal treatment. The addition of Fe(II)-$S_2O_8^{2-}$ reverses the dewaterability deterioration caused by mild thermal treatment for improvement [7]. Guan et al. found $CaCl_2$ could improve the sludge dewatering performance under 50–90℃, as thermal treatment could thus strengthen the bridging between Ca^{2+} and the flocs [8], while the higher temperature is better [9]. For the thermal treatment, there is still a room to lower the temperature and meanwhile improve the dewatering efficiency. Ferric chloride as a common conditioner has been used in research [10]. Sewage sludge was conditioned with skeleton builders, i.e. fly ash and lime combined with ferric chloride for the purpose of improving the dewatering efficiency, and the dewatered sewage sludge was then directly reused as landfill cover materials [11]. However, little research has focused on mild thermal treatment to enhance the effect of ferric chloride and steel slag, so we aim to improve the efficiency of dehydration as well as to reduce energy consumption. In this study, Ferric chloride, inorganic non-metallic material steel slag, and thermal temperature are determined as main factors through a series of single-factor preliminary tests. The interactions between each other have been analyzed through response surface methodology (RSM). Zeta potential, Fourier transformed infrared (FTIR) and SEM of sludge are tested to help explain the mechanism.

2. Materials and methods

2.1. Raw sewage sludge

The raw sewage sludge used in the study was a mixture of primary and secondary sludge from Sanjintan Sewage Treatment Plant in Wuhan, China. The capacity of this municipal wastewater treatment plant is 3×10^5 m^3/d. Raw sludge samples were collected in polypropylene containers and stored at 4℃ in a refrigerator. The period of preservation shouldn't exceed 4 d [12,13]. Before experiment, the raw sludge was taken out of the refrigerator and put in environmental condition until the temperature raised to 20℃. The characteristics of raw sludge were tested according to the standard methods (US EPA1995). The results are shown in Table 1.

2.2. Inorganic conditioners

In this study, steel slag and ferric chloride solution were used as inorganic conditioners. The steel slag was obtained from Wuhan Iron and Steel Corporation, China. It was crushed to less than 80 μm by grinding in a laboratory ball mill for 30 min. The main chemical compositions are listed in Table 2.

2.3. Experimental procedures

Raw sludge was stirred rapidly for 30 min to make sure the sludge particles evenly mixed with water, and then sieved through a 1.2 mm screen to eliminate the suspended residues and impurities. The heating process was proceeded in a double-jacketed glass reactor equipped with a thermometer, electric stirrer, and glass condenser. In each experiment, ferric chloride solution and solid powder steel slag were added into 500 ml pretreated sludge, respectively (the dosage was calculated by dry solid content). First, sludge was conditioned with ferric chloride solution at 270 rpm for 3 min, then conditioned with solid powder steel slag at 60 rpm for 5 min. The conditioned sludge was then heated up to an expected temperature by using hot-water bath in this process with continuous stirring at 45 rpm. Then the sample was taken for analysis after the temperature cooled to 25℃.

2.4. Experimental design

The Box–Behnken experimental design and RSM [14] were chosen to evaluate the combined effects of the three independent variables at three levels. Ferric chloride solution, solid powder steel slag, and hydrothermal temperature were main factors based on preliminary tests; capillary suction time (CST) was taken as response (Y); experimental range and levels are shown in Table 3. In this study, 17 groups tests were implemented according to the Box–Behnken design, and the experimental data were analyzed by design expert 9.0.5, when it fitted to an empirical quadratic model. The system is explained by the following quadratic equation as Eq. (1):

$$Y = \beta_0 + \sum_{i=1}^{m} \beta_i x_i + \sum_{i=1}^{m} \beta_{ii} x_i^2 + \sum_{i=1}^{m} \sum_{j=1}^{m} \beta_{ij} x_i x_j \quad (1)$$

where Y is the process response or output (dependent variable), m is the number of the patterns, i and j are the index numbers for pattern, β_0 is the free or offset

Table 1
Basic characteristics of the raw sludge

Parameters	Moisture (%)	pH	Organic content (%)	CST (s)
Raw sludge	79.3 ± 2.8	7.18 ± 0.14	40.45 ± 0.21	412

Table 2
Main inorganic chemical compositions of steel slag (wt%)

Constituents	MgO	Al_2O_3	SiO_2	P_2O_5	SO_3	CaO	TiO_2	MnO	Fe_2O_3
Percentage	5.28	3.19	18.67	1.26	0.53	40.77	0.39	1.37	12.01

Table 3
Experimental range and levels of independent variables

Variable	Symbols Coded	Range and levels		
		−1	0	1
$FeCl_3$ dosage (mg/g)	X_1	4	5.5	7
Slag dosage (mg/g)	X_2	25	37.5	50
T (℃)	X_3	40	60	80

term called intercept term, x_1, x_2, …, x_k are the coded independent variables, and β_i is the first order (linear) [15,16].

2.5. Analytical methods

The CST was measured by a 304 M CST equipment (Triton, UK) at 25℃, enough sludge was poured into a 1.8 cm inner diameter funnel and the time was recorded automatically. The zeta potential was tested using a Malvern Zetasizer Nano ZS (Malvern Instruments Ltd, UK) by collecting the supernatant of sludge after centrifuging at 4,500 rpm for 5 min before the supernatant was collected and mixed with the sludge at a ratio of 9:1. 50 mL of sludge sample was filtrated to remove excess water. The filter cake was dried at 105℃ for 24 h, then grounded into powder, and dried again under the same conditions. The FTIR spectra were recorded using an ALPHA FTIR spectrometer (Bruker Optics, Germany) in the range of 4,000–400 cm^{-1}. The dewatered sludge samples were collected and dried in an oven at 40℃, then characterized by scanning electron microscope (SEM, Quanta 200).

3. Results and discussion

3.1. RSM analysis

By applying multiple regression analysis on the design matrix and the responses, the following second-order polynomial equation Eq. (2) in coded form was established to help find the optimal conditions and minimum CST:

$$\begin{aligned} Y = {} & 37.19236 - 1.48889\,x_1 - 0.55400\,x_2 - 0.39708\,x_3 \\ & - 0.018\,x_1 x_2 - 0.012917\,x_1 x_3 + 0.00455\,x_2 x_3 \\ & + 0.25278\,x_1^2 + 0.0038\,x_2^2 + 0.00248438\,x_3^2 \end{aligned} \quad (2)$$

where Y is the predicted CST, and x_1, x_2, and x_3 are the coded terms for three independent test variables, ferric chloride dosage, steel slag dosage, and T ℃, respectively.

Table 4 illustrates the analysis of variance (ANOVA) of the regression model. The model F-value of 37.38 showed that the quadratic model was highly significant. There was only a 0.01% chance that a large F-value could occur due to noise. Values of "Prob. > F" less than 0.0001 indicate that model terms are significant. The "R^2" was 0.9796 and the "Pred. R^2" of 0.7739 was in reasonable agreement with the "Adj. R^2" of 0.9534, which indicated a good consistency between experimental and predicted values [17].

Table 4
ANOVA of the response surface model

Source	Sum of Squares	DF	F-value	Prob. (p) > F
Quadratic	25.71	9	37.38	<0.0001
A-$FeCl_3$	0.45	1	5.90	0.0454
B-M	11.28	1	147.61	<0.0001
C-T	1.250E-003	1	0.016	0.9018
AB	0.46	1	5.96	0.0447
AC	0.60	1	7.86	0.0264
BC	5.18	1	67.72	<0.0001
A^2	1.36	1	17.82	0.0039
B^2	1.48	1	19.42	0.0031
C^2	4.16	1	54.40	0.0002

"Adeq Precision" measures the signal to noise ratio. A ratio greater than four is desirable. The ratio of 21.931 indicates an adequate signal and the model can be used to navigate the design space. The *F*-value denotes the significance in the model equation [18,19]. The regression and corresponding value of "Prob. > *F*" less than 0.0500 indicate that model terms are significant, while value greater than 0.1000 indicates the opposite. In this case, A, B, AB, AC, BC, A^2, B^2, C^2 are significant model terms.

The three-dimensional response surfaces and their corresponding two-dimensional circular contour plots were generated by design expert (Fig. 1). When interactions of two variables were discussed, the third factor was kept at zero level. 3D response surfaces and their corresponding contour plots can facilitate the straightforward examination of the effects of the experimental variables on the responses [20]. S-1 reveals that an increase in steel slag dosage has a positive effect on CST reduction. At steel slag dosage of 30–50%, little increase in ferric chloride dosage has a positive effect, but continuous increase has slight effect. It can be seen from S-2 that temperature and steel slag dosage have strong synergistic effect on CST reduction. At low dosage of steel slag, CST was reduced with the increase in temperature, after 55℃, the effect was not significant. At high dosage of steel slag, increase in temperature has small effect on result, moreover, high temperature caused a negative impact. S-3 depicts the interactive influence between temperature and ferric chloride dosage. The lowest point appears in middle of the graph, suggesting CST was affected negatively when variables are at low or high levels. Design expert software and response surface analysis were used to determine optimum conditions of the operating variables in the composite conditioning [16]. On the basis of the calculation steps defined for the optimization algorithm, the optimal values of the test variables were found as $FeCl_3 = 5.89$ mg/g, steel slag = 48.25 mg/g, $T = 52.06$℃, with CST = 7.7 s.

3.2. Validation of the regression model

To verify the validity of the proposed model, several additional batch experiments were carried out in the experimental area of the Box–Behnken design, and each experimental response was compared with the predicted one. These extra experiments were chosen randomly in the experimental domain. The solutions of process variables and experimental results are listed in Table 5. The replicate experiments were in close agreement with the model prediction.

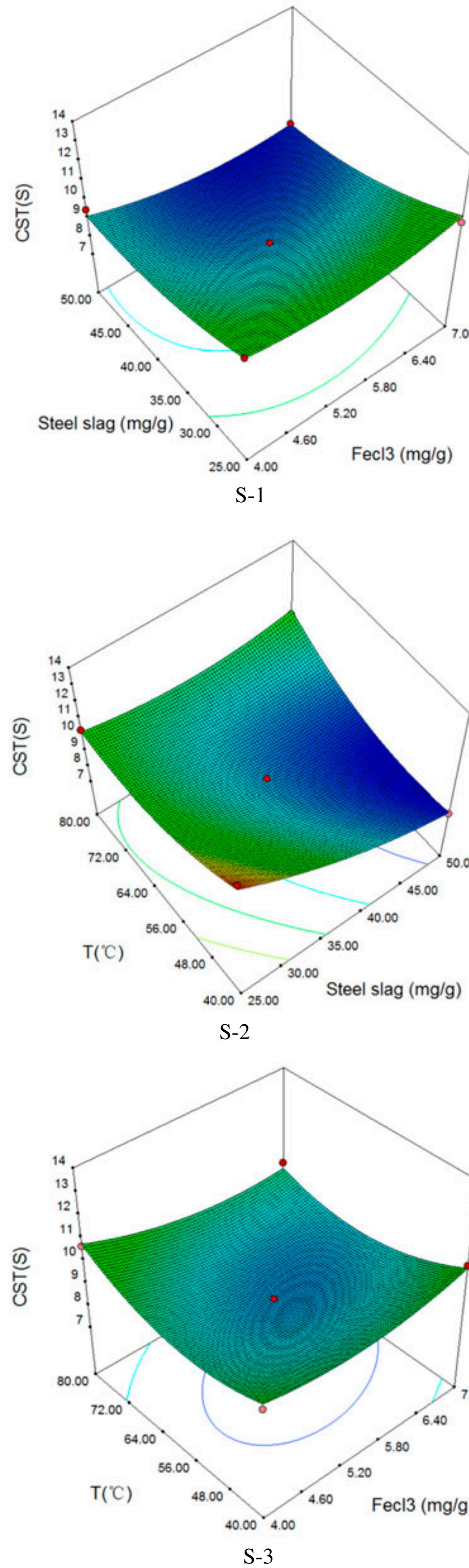

Fig. 1. Response surface plots: S-1 dosage of steel slag and $FeCl_3$; S-2 dosage of steel slag and temperature; S-3 dosage of $FeCl_3$ and temperature.

3.3. Enhanced sludge dewatering under mild thermal treatment

The temperature of thermal treatment ranges from 40 to 80℃ in this study and it can be seen from Fig. 2 that increase in temperature have a significant enhancement in dewaterability of sludge conditioned by inorganic composite conditioners. At 55℃, when the steel slag dosage elevated from 25 to 50 mg/g DS, a sharp decrease in CST was observed at low $FeCl_3$ dosage (4 mg/g DS); the CST varied a little with increased dosage of $FeCl_3$ (from 4 to 7 mg/g DS) at low steel slag dosage (25 mg/g DS).

Extracellular polymers are thought to be of considerable importance in bioflocculation, settling, and dewatering of activated sludge [21,22]. Zeta potential was significantly related to extracellular polymers, the decrease in sludge negative charge could reduce the repulsion forces and enhance the compaction of sludge flocs [23], Zhang et al. demonstrated a strong correlation existed between SRF and zeta potential ($R^2 = 0.99$, $p < 0.01$) [24], therefore, zeta potential could partly reflect the sludge dewatering performance. As the Fig. 3 shows, zeta potential in blank sludge test of 25, 55℃ around −20 mv accordingly, showed modifiers can reduce the zeta potential of sludge obviously. At 25℃, by adding 50% steel slag and 5.5% $FeCl_3$, respectively, zeta potential was reduced to −12.1 and −11.3 mv; at 55℃, by adding 50% steel slag and 5.5% $FeCl_3$, respectively, zeta potential was reduced to −5.9 and −8.2 mv. By comparing with the condition at 25℃, the zeta potential at55℃ was reduced by 6.2 and 3.1 mv, showing that the rise in temperature could reduce the zeta potential obviously, especially when adding 50% steel slag. Under the condition of 25 and 55℃, the mixing investment of steel slag and $FeCl_3$ was more effective in reducing the zeta potential than adding, respectively. The zeta potential of sludge without modifier could not reduce when the temperature rised, showing that rising the temperature independently will not promote the sludge dewatering performance, which matched the previous test.

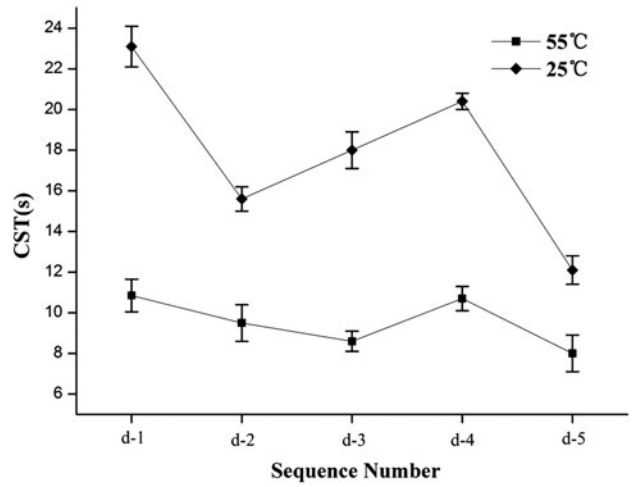

Fig. 2. CST evolution of sludge as a function of temperature and dosages (d-1:4 mg/g DS $FeCl_3$ and 25 mg/g DS Steel slag; d-2:4 mg/g DS $FeCl_3$ and 50 mg/g DS Steel slag; d-3:5.5 mg/g DS $FeCl_3$ and 37.5 mg/g DS Steel slag; d-4:7 mg/g DS $FeCl_3$ and 25 mg/g DS Steel slag; d-5:7 mg/g DS $FeCl_3$ and 50 mg/g DS Steel slag).

The FTIR analysis of dried sludge is given in Fig. 4, the band at 3,400 cm^{-1} is the O–H stretching vibration [25]; the bands at 2,920 and 2,851 cm^{-1} are asymmetric and symmetric vibration of CH_2 of aliphatic structures and lipids, respectively [26,27]; two broad bands at 1,540 and 1,654 cm^{-1} are of protein origin (Amide II and Amide I, correspondingly) [27,28]; the band of 1,437 cm^{-1} may corresponds to the C=C band of aromatic rings polarized by oxygen atoms bound near one of the C atoms and suggesting the presence of basic oxygen-containing functionalities such as chromene structures, diketones, or quinone groups and pyrone-like groups [29]. The band at 1,426 cm^{-1} correcponds to phenolic O–H and C–O stretching [27]. According to Fig. 4, some transformation of bands could be observed with test groups, and the details of the shifts of characteristic bands are listed in Table 6. By comparing with group c, there

Table 5
Comparison of predicted and experimental values

	$FeCl_3$ dosage (mg/g)	Steel slag dosage (mg/g)	T (℃)	CST (s)	
				Predicted	Experimental
1	5.89	48.25	52.06	7.7	7.8 ± 0.9
2	4.50	30.00	45.00	10.7	11.3 ± 0.6
3	6.00	46.50	65.50	8.2	8.4 ± 0.8
4	6.60	27.80	72.50	9.9	10.0 ± 0.7

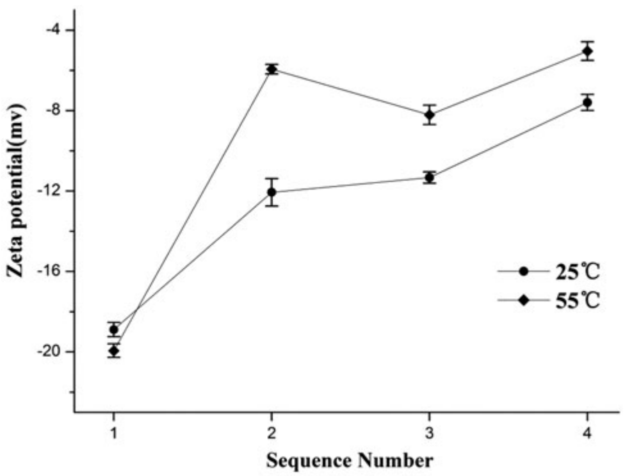

Fig. 3. Zeta potential evolution of sludge as a function of dosage under mild thermal treatment at 25 and 55℃ (1–0; 2–50 mg/g DS Steel slag; 3–5.5 mg/g DS $FeCl_3$; 4–5.5 mg/g DS $FeCl_3$ and 50 mg/g DS Steel slag).

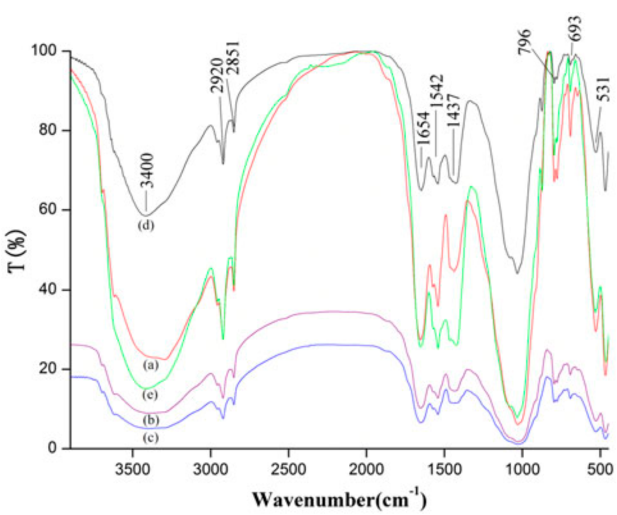

Fig. 4. FT-IR spectra of the sludge with different dosages and temperatures (a) 0; (b) 50 mg/g Steel slag; (c) 5.5 mg/g DS $FeCl_3$; (d) 5.5 mg/g DS $FeCl_3$ and 50 mg/g DS Steel slag; (e) 5.5 mg/g DS $FeCl_3$ and 50 mg/g DS Steel slag; (a), (b), (c), (d) under 55℃; (e) under 25℃).

were more shifts of bands with group b, meanwhile, considering the change of zeta potential, it could be speculated that the strong interation between steel slag and the functional groups at 55℃ diminished the surface charge of flocs. The obvious shifts of protein bands with group d indicate the degradation and removal of protein-like substances.

The direct examination of flocs by scanning electron microscopy (SEM) showed raw and conditioned sludge structure The raw sludge seems to be colloidal in structure less porosity; When the temperature increased without modifier, there was no obvious change with sludge granular structure, it was still colloid, but porosity increased; Upon adding $FeCl_3$, the colloidal structure changed to larger flocs structure and the porosity increased; the sludge appears irregular block structure after adding steel slag, meanwhile, a large number of pores can be found between the irregular block structure; After the composite conditioning with ferric chloride and steel slag, the original dense layer disappeared, instead there were lots of random block structures and pores. From Fig. 5(e) and (f), the hydrothermal treatment could strengthen the impact, the block structures turned smaller, and there was a significant increase in porosity. So, it could be concluded that the hydrothermal treatment plays an important role in conditioning.

3.4. Mechanism analysis

Raw sludge was the mixture of organic matter, protein, carbohydrate, micro-organism, and mineral composition. There are some tiny soluble polymers existed around the staggered overlapping hybrid flocs, with a negative charge in surface. The role of ferric chloride mainly attribute to two aspects. On one hand, ferric chloride was capable of removing most of the solution protein and some of the solution polysaccharide [10], and neutralizing a part of the surface charge. Consequenlty, it could reduce the bind between moisture and sludge particles. Moreover, the hydrolysis of ferric chloride could reduce the repulsion between floc particles via neutralizing the negative surface charges; on the other hand, the tiny soluble polymers could bind together by flocculation and form larger particle structures, which indirectly reduced the surface area of solid particles and released the free energy of the water [30]. After adding steel slag powder, parts of the cells ruptured with the conditioning of alkaline component. Metal cations neutralizes the surface charge of flocs markedly by interacting with some functional groups (Figs. 3 and 4), thus hybrid flocs were disintegrated and regrouped. In this process, the inorganic mineral composition of steel slag scattered between flocs, as a carrier; flocs attaches around the mineral phase and form random block structure (Fig. 3). The mild thermal treatment enhanced hydrolysis of $FeCl_3$ and dispersion of inorganic particles in flocs, promoting the dewaterability of sludge. From zeta potential, FTIR analysis, and SEM images, the steel slag plays a more significant role in the sludge dewatering under mild thermal treatment.

Table 6
Main shifts of infrared absorption bands of sludge

Band					
a	*b*	*c*	*d*	*e*	Assignment
3,297.2	3,390.0	3,400.1	3,413.8	3,419.5	O–H stretching vibration
1,655.2	1,653.9	1,653.9	1,647.7	1,653.7	Amide I (H-bonded C=O carbonyl stretch)
1,542.4	1,542.7	1,542.4	1,546.1	1,541.8	Amide II
1,437.4	1,435.2	1,437.3	1,429.6	1,433.9	O–H and C–O stretching of phenols

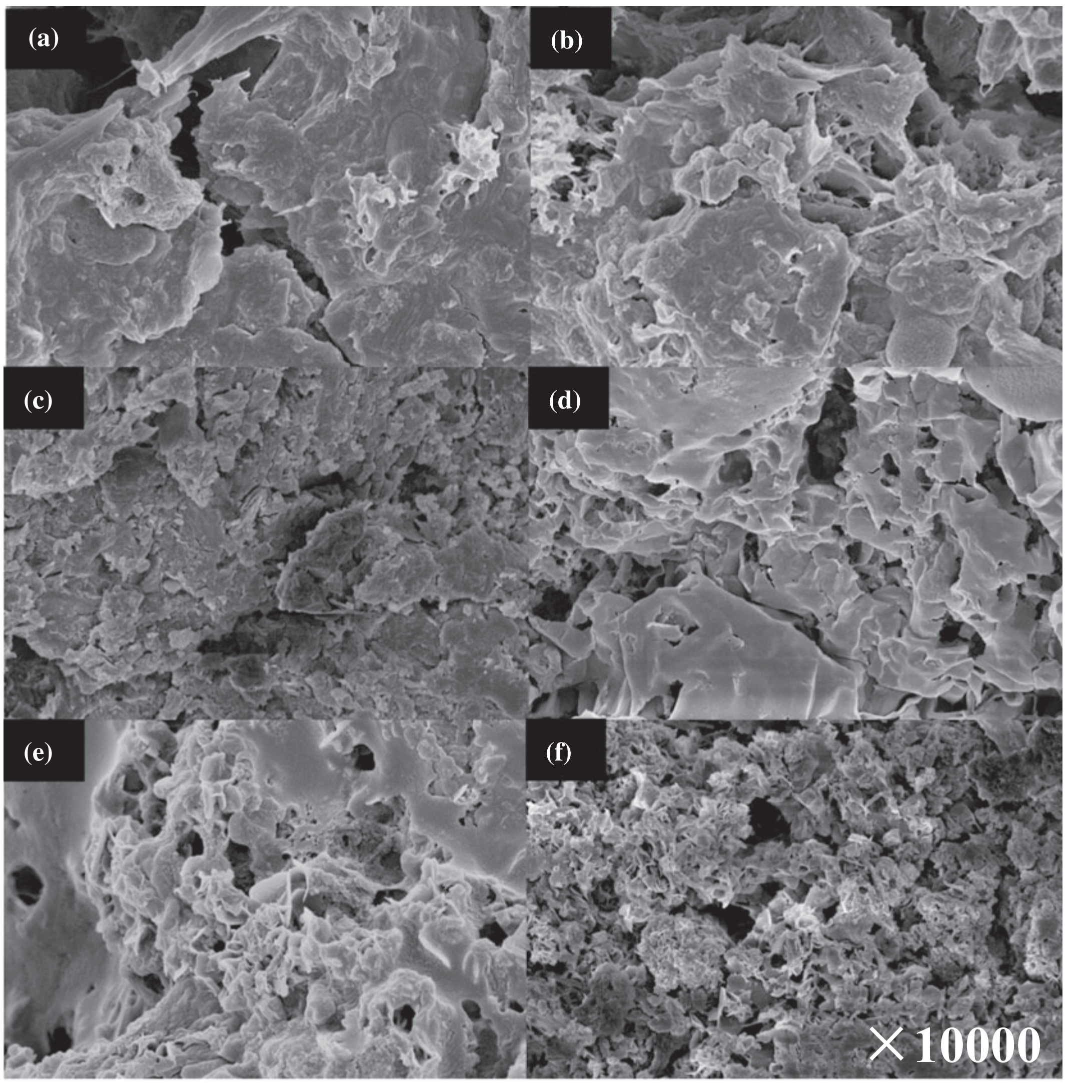

Fig. 5. SEM images of (a), (b) raw sludge; (c) conditioned by 5.5% $FeCl_3$; (d) conditioned by 50% Steel slag; (e), (f) conditioned by 5.5% $FeCl_3$ and 50% Steel slag; (a), (e) under 25℃; (b), (c), (d), (f) under 55℃.

4. Conclusion

(1) The experimental results show that $FeCl_3$, steel slag, and temperature have significant impact on the performance of sludge dewatering. After RSM optimization, the CST could be dropped to 7.8 ± 0.9 s under the conditions comprising $FeCl_3$, Steel slag dosages of 5.89, 48.25 mg/g, and temperature of 52.06℃, respectively.

(2) Hydrothermal treatment has a positive effect on inorganic conditioners, which enhanced hydrolysis of $FeCl_3$ and dispersion of inorganic particles in flocs. The pretreatment of thermal and conditioners contributes to reduce the surface charge of sludge. From FTIR test, it could be speculated that the strong interation between steel slag and the functional groups at 55℃ diminished the surface charge of flocs; the obvious shifts of protein bands indicate the degradation and removal of protein-like substances. The SEM test indicates that the structure of flocs changed to small irregular blocks, with more micropores.

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