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Optimization of thermo-alkaline pretreatment on municipal sludge and enhanced subsequent anaerobic digestion

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ABSTRACT

The municipal sludge cause heavy contamination. The hydrolysis process is one of the rate-limiting steps in the anaerobic digestion of municipal sludge. Thermo-alkaline pretreatment can accelerate the hydrolysis process and enhance the subsequent anaerobic digestion of methane production. To further optimize the process parameters of the municipal sludge by thermal-alkali pretreatment, the effect of thermo-alkali pretreatment on the sludge disintegration and subsequent anaerobic digestion were investigated using response surface method (RSM). Results showed that the effect on sludge decomposition is decreasing from pH over temperature to reaction time. The Box–Behnken design model was applied for the predicting of the sludge disintegration. The regression analysis determined the coefficient of $R^2 = 0.9741$ with $P < 0.0001$, indicating the model was effective and fitting in a high degree as well. The optimal temperature, pH and reaction time were 90°C, 104 min; pH = 12, respectively. Taking these values into account, the predicted degree of sludge disintegration (DD_{COD}) for RSM was 47.32% and 46.45% for the measured values. Results showed that the methane production rate of the sludge anaerobic digestion was 145 mL/g VS, which was 79% higher than that of the original sludge. Furthermore, the methane yield and anaerobic digestion efficiency were increased.

Keywords: Degree of sludge disintegration; Thermal-alkaline treatment; Response surface methodology; Anaerobic digestion

1. Introduction

In recent years, together with the increasing of waste water treatment, the production of municipal sludge (MS) is also increasing dramatically. In 2016, the annual production of MS is up to 7,997,232 ton (dry weight) in China [1]. With such a big amount, inappropriate disposal and treatment of MS can cause serious secondary contamination, due to the high concentrations of pathogens and organic materials [2,3]. There are multiple methods for the treatment of MS, like composting technology [4–6], and anaerobic digestion [2,3]. Anaerobic digestion is one of the most widely used methods for MS treatment with the purposes of mass reduction, organics stabilization, harmlessness and energy (biogas) production. The treatment follows four fundamental steps: hydrolysis, acidogenesis, acetogenesis, and methanogenesis The first step, hydrolysis, which is widely regarded as the limiting step as the high molecular weight compounds can be broken down into monomers and other smaller molecules in this step. However, the complex floc structure of MS and hard cell wall lead to long Hydraulic Retention Time (HRT), which means large reactor volume is needed during the application [8,10]. In order to improve the hydrolysis efficiency, pretreatment, including chemical, thermal, mechanical, and biological methods, can be used for accelerating the dissolution of intracellular substances [8–10]. These pretreatments destroy the cell wall, facilitating the release of extracellular or intracellular constituents, and making them more accessible to subsequent microbial actions [7]. Compared with other

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methods, chemical pretreatment has received considerable research globally due to its high efficiency. Furthermore, new chemical methods are also tried to apply in waste activated sludge treatment [7–9].

In recent years, combination pretreatment technologies are more and more popular, the relative researches have increased to quadruple from 2010 to 2016 [7]. The most common pre-treatment combinations are thermo-chemical and physical-chemical [7]. The combination of thermo-chemical pretreatment technologies shows easier operation, higher efficiency, and improved application prospect [7,12]. There are two main thermal pretreatments, high-temperature treatment (100–210°C) and Low-temperature treatment (<100°C) [13,14]. The main mechanism of high temperature lysis is the physical disruption leads to thermal solubilization of organic matter. The low temperature hydrolysis includes stimulation of thermophilic bacteria and solubilization by hydrolytic enzymes beside thermal solubilization. Not only temperature, other chemical conditions can also promote the cell membrane rupture. For example, in the high pH condition, the saponification of lipid with solubilization of cell membrane may lead to the disruption of cells and the inner matters can be released [15]. In addition, studies also reported that strong chemical degradation and ionization of the hydroxyl groups (-OH→-O) can also lead to extensive swelling and subsequent solubilization [16]. Furthermore, different alkali can also affect the efficiency of the pretreatment, the increase of soluble COD was quite similar between NaOH (39.8%) and KOH (36.6%), but significant higher than and $Mg(OH)_2$ (10.8%) and $Ca(OH)_2$ (15.3%) [17]. However, the methane production and the metabolic pathway of anaerobic microflora can be affected by the high concentration of Na^+ [18]. By comparing with a separate thermal or alkali treatment, the combination of thermo-chemical pretreatment shows a better solubilization of the intracellular substance and a higher improvement biogas yield. Xu et al. (2014) achieved up to 44% COD solubilization by using a thermo-alkaline pre-treatment at 90°C and pH 11 for 10 h, similar to the efficiency of single alkaline pretreatment in 8 d [19].

Many studies also reported that the sludge crack by thermo-alkaline treatment can be significantly affected by different factors, such as temperature [19,20], pH value [19,20], time [19], alkaline type [17]. For example, different pH and temperature were studied for the effect of sludge crack, and the results showed at pH 11 and in 90°C, the soluble COD was 70 mg/l and the methane production rate reached 280 ml CH_4/g VSS within ten hours [20].

However, the study of the sludge soluble cell has been largely limited to single and double factors and has been poorly understood to optimize the pretreatment conditions, which also include multiple factors, such as the temperature, time, and pH.

In this study, the main multiple factors, including temperature, time and pH (adjusted by KOH), were considered for the improvement of the thermal-alkaline treatment.

Optimization of the process variables during MS treatment can be achieved using response surface methodology (RSM) which makes treatment process modeling simple and efficient [21]. The response surface analysis based on multiple quadratic regressions is applied for fitting the function relationship between independent variables. The response was analyzed through a 3D surface figure which can be continuously applied for each level of the experiment [22]. Box–Behnken design, which is widely used in laboratory for data evaluation, includes the fact that only 17 experimental runs, instead of having 27 experimental points if the run was done in 3^3 complete factorials [22,23].

The aims of this study are: (1) to optimize the decoding conditions of the sludge soluble cells by applying a three-factor, three-level Box–Behnken experimental design combining with RSM and quadratic program-ming for maximizing the DD_{COD}; (2) to identify the effects of three independent variables (temperature, time and pH) and their interactions on the DD_{COD} ; (3) to validate the proposed model by additional batch experiments conducted in the experimental area of the Box–Behnken design, and then obtaining the optimum technological parameters; (4) to verify the methane increment under the best conditions of the pretreated.

2. Materials and methods

2.1. Experimental materials

The MS was obtained from a municipal sewage treatment plant in Nanning, Guangxi, China. Before using, the collected MS was stored in 4ºC for 24 h in a refrigerator. The total solid (TS) concentration was adjusted to about 5% before the experiments. The anaerobic inoculation sludge was obtained from the experiments in Guangxi Sugar Industry Group. The basic characteristics of the MS and inoculum are given in Table 1.

2.2. Experiment methods

2.2.1. Response curve optimization experiment

A three factor, three-level Box–Behnken experimental design contained 17 batch experiments which were conducted in triplicate. In accordance with the principle of Box–Behnken design (BBD), the effect of temperature (A), time (B), and pH value (C) were investigated . Each of independent variables was consecutively coded as A, B and C at three levels: −1, 0 and 1. The experimental range and levels of independent variables considered in this study are presented in Table 2. The central values chosen for the experimental design were temperature (A) = 80, time (B) = 100 min and pH value (C) = 11 in uncoded form. The DD_{COD} (Y) was used as the response value. The fitting formula is as follows:

$$Y=a_0+a_1A+a_2B+a_3C+a_{12}AB+a_{13}AC+a_{23}BC+a_{11}A^2+a_{22}B^2+a_{33}C^2 \quad (1)$$

Table 1
Basic characteristics of the sludge used in the experiments

Parameters	TS %	VS %	SCOD ($mg·L^{-1}$)	TCOD ($mg·L^{-1}$)	pH
MS	5.85	2.79	920.9	29424.3	6.7
Inoculum	6.48	4.30	552.5	–	7.1

Table 2
Design of the level of experiment factors

Factor	Values of each encoding level		
	–1	0	1
Temperature (°C)	70	80	90
Time (min)	80	100	120
pH	10	11	12

Type: Y denotes the response value; A, B, and C are values for the independent variable coding; a_0 indicates the constant; a_1, a_2, and a_3 represent the linear coefficients; a_{12}, a_{13}, and a_{23} refer to the interaction coefficients; and a_{11}, a_{22}, and a_{33} stand for the quadratic term coefficients.

2.2.2. The thermal alkali pretreatment

Besides the control, 17 batch experiments were conducted in a water bath by thermal alkali pretreatment in triplicate. The pH of 100 mL sludge in different 250 mL conical flasks was adjusted to the experimental setting values (pH = 10, 11, 12) by KOH (4 mol·L^{-1}) in water bath at the corresponding temperatures (70°C, 80°C, 90°C) as hot basic treatment. The conical flasks were cooled to room temperature after a certain time (80 min, 100 min, 120 min) of the hot alkali treatment.

2.2.3. Batch anaerobic digestion

In accordance with the treatment conditions of thermal alkali obtained after the response surface optimization, first, the thermal-alkali pretreatment sludge and the original sludge 30 g-TS were added to 1 L conical bottles. Second, the anaerobic sludge was inoculated with 15 g-TS. Third, the distilled water was fixed to 900 mL, and then, the nitrogen was used for 2 min. Finally, the static fermentation for 20 d in the thermostatic water bath was conducted with the rubber plug seal, and the methane production was measured by the drainage method with 3% NaOH solution.

2.3. Analytical methods

Standard methods [24,25] were used to determine TCOD, SCOD, TS, VS, pH. The 50 mL sample were centrifuged for 10 min at the speed of 6000 rpm. The potassium dichromate method was used for the determination the supernatant's COD after filtration [24]. DD_{COD} is the indicator for the cracking degree of the sludge. The formula for the DD_{COD} calculation is as follows [26,27]:

$$DD_{COD} = (SCOD_t\text{-}SCOD_0)/(TCOD\text{-}SCOD_0) \quad (2)$$

In the formula, $SCOD_t$ is the dissolved chemical oxygen demand of the sludge by thermo-alkali pretreatment, and $SCOD_0$ and TCOD denote the dissolved and total chemical oxygen demands of the original sludge, respectively. TS and VS are measured by the gravimetric method [28].

2.4. Statistical analysis

The Design Expert 11.0 software and Origin 9.0 were used for regression and graphical analyses of the data obtained.

3. Results and discussion

3.1. Secondary model establishment and significance analysis

The results of BBD response surface experiments are summarized in Table 3. The effects of single factors, interaction terms, and quadratic terms on the sludge crack were obtained, and the quadratic polynomial regression equation could be expressed as:

$$Y= 38.34+2.63A+1.19B+7.32C-0.072AB+0.66AC+0.68BC +1.23A^2-4.38B^2-3.04C^2 \quad (3)$$

The results of the variance analysis and regression significance test which performed on the quadratic polynomial model were summarized in Table 4. The F-value indicates the significance of the interaction between variables. A higher F-value suggests the more significant influence of the corresponding factor on the response value. In p-value analysis, $P < 0.01$, $P < 0.05$ and $P > 0.1$ indicate the influence of the factor on the response value is considerably significant, significant, non-significant respectively. Therefore, the results of the significance analysis of various factors in the model show that the linear effects of A and C on sludge cracking are significant, and the quadratic terms B^2 and C^2 are also considerably significant. According to the F-value, the influence of the independent variable on the response value is in the order: pH > temperature > time. By regression analysis, the F-value of DD_{COD} is 29.3 with $P < 0.0001$, which indicates that the model is considerably significant. In addition, the relationship between the three factors described in the regression equation and DD_{COD} is statistically significant. Furthermore, the decision coefficient ($R^2 = 0.9741$) of the regression model indicates that the model can explain the 97.41% change of the response value. The correction determining factor is Adj-$R^2 = 0.9409$, which is close to R^2, and the model fitting degree is high. The coefficient of variation often reflects the reliability and accuracy of the model [21,22]. In the experiment, the CV= value (4.36%) lower than 10%, indicating that the model has a high reliability and accuracy. The model signal-to-noise ratio is 20.323, which is higher than 4, indicating the true degree of the model. The P value of the missing item is 0.567 which is significant higher than 0.05. The almost identical values of the obtained equation and the actual fitting with the small uncertainty, indicating that the model can be used to analyze and predict the cracking effect of MS by thermo-alkali pretreatment.

3.2. Analysis of response surface graph

The size of the response value of the sludge was evaluated in order to provide more accurate interaction effects of temperature, time, and pH. The interaction of the element response surface map of the various factors was shown in Fig. 1. Fig. 1a illustrates that DD_{COD} has a slowly rising

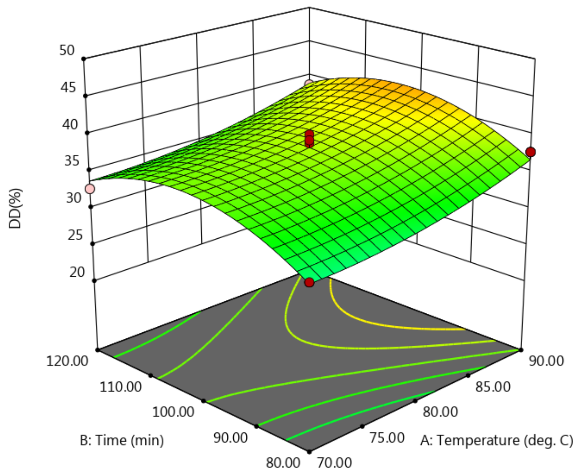
Table 3
Experimental design and results of response surface

Serial number	Temperature (°C)	Time (min)	pH value	DD_{COD}(%)		Relative error (%)
				Measured values	Predictive values	
1	–1	–1	0	31.51	31.29	0.70
2	1	–1	0	37.76	36.70	2.81
3	–1	1	0	32.77	33.83	–3.23
4	1	1	0	38.73	38.94	–0.57
5	–1	0	–1	28.08	27.24	2.99
6	1	0	–1	31.18	31.18	0.01
7	–1	0	1	40.55	40.56	–0.01
8	1	0	1	46.29	47.14	–1.81
9	0	–1	–1	22.03	23.09	–4.81
10	0	1	–1	24.33	24.11	0.90
11	0	–1	1	36.15	36.36	–0.61
12	0	1	1	41.18	40.12	2.57
13	0	0	0	38.92	38.34	1.52
14	0	0	0	36.12	38.34	–6.12
15	0	0	0	40.08	38.34	4.37
16	0	0	0	39.33	38.34	2.54
17	0	0	0	37.22	38.34	–3.01

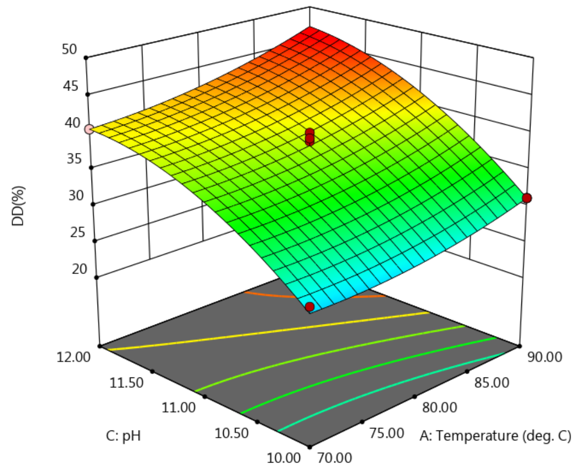
Table 4
Variance analysis of regression model

Source	Sum of squares	df	Mean square	F-value	p-value	
Model	627.79	9	69.75	29.3	<0.0001	Considerably significant
A-Temperature	55.4	1	55.4	23.27	0.0019	
B-Time	11.42	1	11.42	4.8	0.0647	
C-pH	428.66	1	428.66	180.04	<0.0001	
AB	0.0207	1	0.0207	0.0087	0.9283	
AC	1.74	1	1.74	0.7299	0.4212	
BC	1.87	1	1.87	0.7842	0.4052	
A^2	6.39	1	6.39	2.68	0.1454	
B^2	80.68	1	80.68	33.89	0.0006	
C^2	38.91	1	38.91	16.34	0.0049	
Residual	16.67	7	2.38			
Lack of fit	6.11	3	2.04	0.772	0.567	Insignificant
Pure error	10.55	4	2.64			
Cor total	644.46	16				

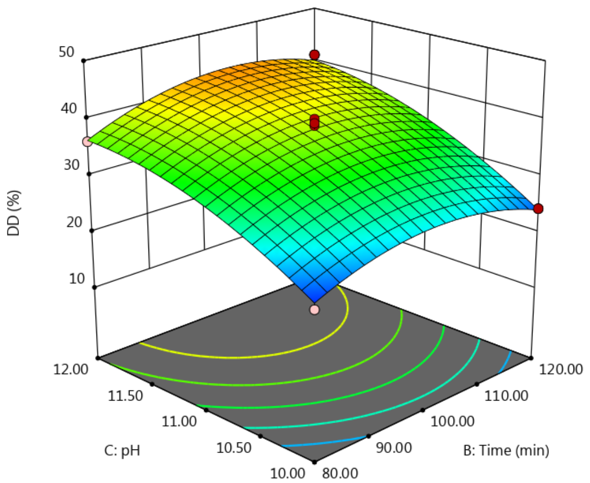
trend under the interaction of temperature and time, but the overall increment is small. Thus, the interaction of temperature and time has no significant promotion effect on DD_{COD} and is insignificant for the sludge cracking effect, which in agreement with the conclusion of variance analysis. Fig. 1b shows the change trend of DD_{COD} after sludge cracking under the interaction of temperature and pH value. It exhibits the increasing of DD_{COD} accompany with the obvious increase of temperature and pH. The reason is that the alkaline condition reduces the temperature resistance of the cell wall, as the high pH can damage the cell structure leading to the continuous dissolution of the sludge intracellular material [28,29]. The higher surface slope of the 3D graph and, the greater influence of the interaction effect of the factors, indicates that the pH playing a leading role in the influence of sludge cracking. Fig. 1c presents the interaction effects of time and pH. When the pH keeps a certain value, the DD_{COD} has insignificant effect on react time, but DD_{COD} increases continuously with increasing pH value. The surface diagram (Fig. 1) shows that the temperature, time, and pH can all affect the efficiency of thermo-alkaline treatment. The order of each factor affecting on the pretreatment is as follows: pH > temperature > time, which is in agreement with the F-value analysis.



a. The interaction between temperature and time.



b. The interaction between temperature and pH value.



c. The interaction between time and pH value.

Fig. 1. Effects of interaction of various factors on DD_{COD}.

Previous study also reported the higher effect of pH compare to the temperature [19]. Increasing the pretreatment time is also not a sufficient way to improve the efficiency as the COD concentration was almost the maximum COD concentration of initial MS. Further, increasing the pretreatment time would lead to higher cost, and bigger tank volumes as well [19,20].

3.3. Parameter optimization and model validation

Based on solving the quadratic polynomial regression equation analysis, the best process conditions for the treatment of the sludge were obtained as: the treatment temperature was 90°C, the processing time was 104 min, and the pH value was 12. This research result is consistent with the study of Vlysside, who reported pH = 11 with a temperature of 90°C are the best process conditions and the predicted extent of DD_{COD} is 47.32% [19]. In order to validate the accuracy and reliability of the model, triplicate experiments were conducted under the optimum process conditions (90°C, 104 min, pH 12). The degrees of sludge disintegrations are 47.11%, 46.63%, and 45.62% respectively. The variability of the predicted values and the experimental values is less than 5%, indicating that the actual and predicted values fit well.

3.4. Effect of thermo-alkali optimization process on anaerobic digestion

Fig. 2 shows the variation of cumulative methane during anaerobic digestion. The results show that the methane production of the original sludge and thermal-alkali treatment group both significantly increased during the anaerobic digestion in the first 3 d, but gradually decreased from 4 to 10 d, and slightly increased again 10 d. The maximum methane production of the thermo-alkali group is significantly higher than that of the original sludge group, i.e., 145 and 81 $mLCH_4/VS_{add}$, respectively. Because in the pretreatment group, the soluble protein and polysaccharide in the cell can be dissolved after the cell wall is ruptured [15]. The dissolved protein and polysaccharide can be easily used by the micro-

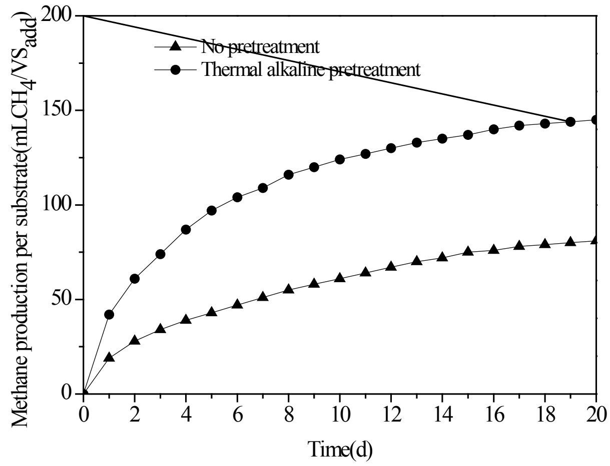


Fig. 2. Variations of methane production per substrate.

organisms for the production of methane. However, the fast consumption of the dissolved compounds in the first 3 d leads to the production of methane slower down during the 4–10 d. The methane production has been greatly reduced as the organic matter is consumed more or less after 10 d. Fig. 3 presents the changes of SCOD in the anaerobic digestion process. The initial SCOD concentration of the thermo-alkaline pretreatment group was as high as 6142.7 mg·L^{-1}, as the release of the organic matter was accelerated by the pretreatment of thermo-alkali. In the early stage of digestion, SCOD was rapidly decreased, indicating that the dissolution rate of the sludge extracellular and intracellular polymer was slower than the consumption rate of methane-producing bacteria. The SCOD removal rate in 9 d was more than 79%, and the removal rate of SCOD after anaerobic digestion was 90.2%. The low initial concentration of SCOD and the original mud group was 617.7 mg·L^{-1}, SCOD removal rate was only 64.7%, after the digestion, which indicates the dissolution of organic matter in the cell was not complete and the microbial hydrolysis rate was slow. Therefore, pretreatment groups compared with the controls help to improve the anaerobic digestion performance.

3.5. Comparative verification test

Table 5 presents the anaerobic digestion properties of thermo-alkali pretreatment and control. It shows that the cumulative methane production of the sludge processed by the thermo-alkali treatment was significantly improved (3434 mL) compare to the control group (1927 mL) after anaerobic digestion. It also exhibits that the methane production rate of sludge treated with thermo-alkali increased by 79%, reaching 145 mL·g VS^{-1}, and the SCOD and VS removal rates increased by 39% and 71%, respectively. The VS removal rate was higher than the previously reported value (43.15%) [30]. Besides the increase of methane production, the removal of SCOD and VS in the pretreatment group, the T80 was 8 d which was significantly shorted compare to the 11 days of the unpretreated group.

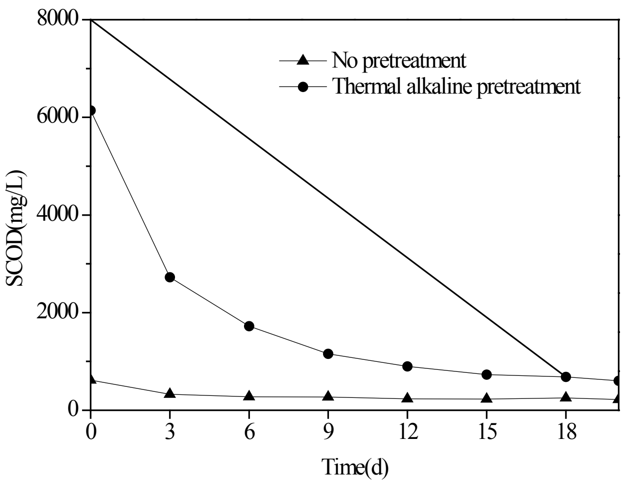


Fig. 3. Variations of SCOD in the anaerobic digestion process.

4. Conclusion

(1) Base on the BBD response surface analysis method, the regression model of the influence of the temperature, time, and pH value on DD_{COD} was established, and the variance analysis was conducted. The model reached a significant level, and the fitting degree was high in the whole regression region of the study, with a high degree of credibility and accuracy.

(2) The heat response in the face of alkali treatment process was optimized, and the optimal temperature, pH and reaction time for hot alkali treatment sludge are temperature, 90°C, 12 and 104 min, respectively. The DD_{COD} values in the three validation tests under the optimum technological conditions were 47.11%, 46.63%, and 45.62%, respectively. The relative error between the measured value and predicted values was less than 5%.

(3) Response surface analysis showed that the effect on sludge decomposition is decreasing from pH over temperature to reaction time. The pH value and temperature influence reached a considerably significant level.

(4) By comparing with unpretreated MS, under optimal anaerobic digestion conditions, the ratio of methane yield, SCOD removal rate, and VS removal rate increased by 79%, 39%, and 71%, respectively. The anaerobic digestion time T80 shortened by 3 d. The pretreatment of sludge by thermo-alkali optimization process could not only improve the anaerobic digestion performance but also improve the removal rate of organic matter.

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Table 5
The anaerobic digestion properties of thermo-alkali pretreatment group and the control group

Group	Cumulative methane (mL)	Methane production rate (mL·gVS^{-1})	SCOD removal rate (%)	VS removal rate (%)	T80 time (d)
Control	1927	81	64.7	10.2	11
Thermo-alkali treatment	3434	145	90.2	17.4	8

Note: T80 is 80% of the amount of methane accumulation in an anaerobic digestion cycle.

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