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Promotion of Sugar Extraction from Sewage Sludge by Microwave Combined with Thermal-Alkaline Pretreatment

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Abstract: A large amount of sludge is produced in the process of municipal sewage treatment. The recovery and utilization of large amounts of sugar, protein, lipids and other organic matter from sewage sludge (SS) is of great significance for reducing environmental pressure and producing clean energy. In this study, microwave combined with thermal-alkaline pretreatment was used to accelerate the dissolution of primary sedimentation sludge and the release of intracellular substances, and to promote the extraction of sugar from SS. The results showed that the yield of crude sugar and the extraction efficiency of pure sugar increased with the increase in NaOH dosage. The extraction of crude sugar reached the equilibrium at about 30 min. During the response surface analysis, the optimal pretreatment conditions were determined as follows: the dosage of NaOH was 9.93 mL, and the leaching time and the microwave time were 27.65 min and 33.2 s, respectively. The crude sugar yield and extraction efficiency obtained under this condition were 39.80 ± 3.57% and 89.74 ± 3.61%, respectively. The pretreated sludge and crude sugar were characterized with scanning electron microscopy and Fourier transform infrared spectroscopy. The results showed that the combined use of thermal-alkaline and microwave effectively destroyed the structure of the sludge and increased the yield of crude sugar.

Keywords: sewage sludge; crude sugar; microwave; thermal-alkaline

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1. Introduction

A large amount of sludge is produced during the operation of wastewater treatment plants. Sewage sludge (SS) contains a large amount of carbohydrates, proteins, lipids and other organic matter content [1], which will pollute the environment and waste resources if not treated properly. With the increase in sewage treatment facilities and the increasingly stringent discharge standards, the cost of SS treatment and disposal may be as high as 60% of the total operation costs of wastewater treatment plants [2,3]. At the same time, organic matter content in sludge can reach 50% of dry weight, which provides conditions for its resource recovery and utilization [4].

The extraction of proteins and lipids from SS has recently gained more attention with a view to their utilization for animal feed and biodiesel production, respectively [5,6]. Otherwise, research on recovery from SS is rare. As one of the important organic components in sludge, sugar can also be extracted, which is beneficial for converting organic biomass into value-added products and renewable energy [7]. Some studies have been conducted to recover sugar from pineapple leaves and achieve the production of bioethanol [8]. Recovery of sugars from sludge cannot only achieve sludge reduction but also prepare fuel ethanol to reduce dependence on fossil resources and alleviate energy shortages [9,10].

Pretreatments are often required to disrupt the complex sludge structures, solubilize organic components and improve the efficiency of sludge disintegration [11,12]. Common pretreatment methods include ultrasonic pretreatment [13], microwave [14], ozone oxidation [2], Fenton oxidation [15] and acid/alkali hydrothermal pretreatment [16,17]. Acid and alkali pretreatments have the advantages of convenient operation, simple equipment and high efficiency. Compared with acid treatment, alkali treatment uses hydroxyl to decompose and destroy the floc structure and converts insoluble organic matter content in SS into soluble matter content. Extracellular polymers (EPSs) can be loosened and promote the dissolution of polysaccharides [11,18].

Microwave pretreatment was found to be most effective in inducing biomass disintegration. Short irradiation time, rapid heating and non-contact heating are the advantages. Microwave pretreatment used to destroy the structure of sludge flocs not only has the characteristics of fast heating speed, high thermal efficiency and no secondary pollution but also can further improve the inactivation rate of pathogens in sludge and the dissolution rate of total suspended solids and volatile suspended solids [14,19]. However, this pretreatment consumes more energy, which makes it unattractive for scaling up. As a solution, combining it with another pretreatment may synergistically promote solubilization and reduce microwave power consumption and treatment time [20]. Microwave combined with thermal-alkaline pretreatment of sludge was applied in this study to enhance the extraction of crude sugar from SS. The combined pretreatment could effectively inhibit the adaptability of microorganisms to temperature at high pH and increase the dissolution efficiency of organic matter content, due to its shorter treatment duration than microwave or thermal-alkaline pretreatment alone.

This study used microwave combined with thermal-alkaline pretreatment to promote the extraction of sugars from SS. The main objects of this study were: (1) to determine the effects of NaOH dosage, alkali leaching time and microwave pretreatment time on the extraction of crude sugar; (2) to optimize the pretreatment conditions for extracting crude sugar based on response surface optimization analysis; (3) to characterize the extracted crude sugar with scanning electron microscopy and Fourier transform infrared spectroscopy (FTIR). Results from this study can offer a better understanding of resource recovery as an alternative approach for the utilization of SS.

2. Materials and Methods

2.1. Materials

The sewage sludge used in the experiment was taken from the primary sedimentation tank of a wastewater treatment plant in Nanyang, China. It was recovered and allowed to stand for 72 h. Then, the supernatant was discarded and stored in a 4 °C refrigerator. The contents of sugar, crude protein and crude fat in the raw sludge were determined with the DuBois method, Kjeldahl method and Soxhlet extraction method, respectively [21,22]. The ash content was measured by burning at 550 °C for 4 h in a muffle furnace. The main properties of the sludge are shown in Table 1.

Table 1. Characteristics of raw sludge.

Moisture (wt.%)	Ash (wt.% dry)	Elemental Compositions (wt.%) [1]					HHVs [2] (MJ/kg)	Organic Compositions (wt.% daf.)			
		C	H	O [1]	N	S		Carbohydrates	Proteins	Lipids	Others
97.98 ± 1.2	39.75 ± 0.6	29.54 ± 0.2	5.61 ± 0.3	18.84 ± 0.5	5.26 ± 0.4	0.79 ± 0.1	14.77 ± 0.5	16.04 ± 0.8	44.08 ± 1.1	17.37 ± 0.7	22.51 ± 0.9

Notes: [1] Calculated by difference. [2] Higher heating values (HHVs) calculated by the Dulong Formula, i.e., HHVs (MJ/kg) = 0.3393 C + 1.443 (H − O/8) + 0.0927 S + 0.01494 N [22].

The glucose, anhydrous ethanol, hexane, methanol, sodium hydroxide (NaOH), acetone, phenol, concentrated sulfuric acid and Coomassie brilliant blue G-250 powder were sourced from Tianjin Kemiou Chemical Reagent Co., LTD, Tianjin, China.

2.2. Experimental Procedure and Analysis

2.2.1. Sludge Pretreatment Test

To determine the effect of the NaOH dosage, 0, 2, 4, 6, 8 and 10 mL of 1 mol/L NaOH solution were added to 50 mL of raw sludge, respectively, and the mixture was shaken for 30 min at a controlled temperature of 45 °C. After cooling, the sugar was extracted from the mixture.

To study the effect of NaOH leaching time, 50 mL of raw sludge was oscillated for 0 min, 15 min, 30 min, 45 min, 60 min and 75 min at a constant temperature under the dosage of 10 mL NaOH.

In the test using microwave combined with thermal-alkaline pretreatment, conical bottles with 50 mL of raw sludge were sealed with sealing film, subjected to 850 W microwave irradiation for 0 s, 15 s, 30 s, 45 s, 60 s and 75 s, respectively, and cooled to room temperature naturally. Then, the crude sugar was extracted after pretreatment with the dosage of 10 mL NaOH and a leaching time of 30 min.

2.2.2. Extraction of Crude Sugar

After the sludge pretreatment mixture was centrifuged, the supernatant was placed in a beaker and heated to concentrate to 1/4 of the volume. After concentration and cooling, 95% ethanol was added to the beaker and precipitated for 12 h. The supernatant was centrifuged, and the precipitate was washed with 3 mL anhydrous ethanol and 2 mL acetone. Then, the mixture was recentrifuged, and the precipitate was washed with ultrapure water. Finally, the washing was freeze-dried to obtain crude sugar. All experiments were repeated three times to obtain the mean value.

2.2.3. Purity of Crude Sugar

The purity of crude sugar was calculated with the DuBois method with d-glucose as the standard [23,24], as shown in Equation (1).

$$y = 0.0141\,x - 0.0029\ (R^2 = 0.999) \tag{1}$$

2.2.4. Response Surface Analysis

Since all three pretreatment conditions may affect the results of sugar extraction, multivariate statistical models were used to study the weight of any variable and its role for optimizing the interaction between different variables. The response surface methodology (RSM) uses a series of designed experiments to estimate the multivariate polynomial fitted with the independent variables [8,9]. The independent variables were alkali leaching time (0–75 min), alkali solution addition (0–10 mL) and microwave time (0–75 s) in the optimization of crude sugar yield. Finally, experimental verification was carried out according to the optimized sludge pretreatment conditions.

2.2.5. Characterization

Fourier transform infrared spectroscopy (FTIR, ALPHA II, Bruker, Billerica, MA, USA) was used to characterize the functional groups of the extracted crude sugar and the pretreated sewage sludge. Scanning electron microscopy equipped with an energy dispersive spectrometer (EDS) (SEM-EDS, JSM-7900) was used to analyze the surface morphology of the sludge after different pretreatment methods, and the composition of the crude sugar.

2.3. Data Definition

The yield of crude sugars was obtained as the quotient of the mass of extracted crude sugars and the mass of organic matter in the raw sludge, as presented in Equation (2):

$$\text{Yield of crude sugars}(\%) = \frac{\text{Extracted crude sugars}}{\text{Dry sludge organic matter}} \times 100\% \tag{2}$$

The purity of crude sugars was obtained as the quotient of the mass of pure sugar and the mass of the crude sugars extracted from the raw sludge, as presented in Equation (3):

$$\text{Purity of crude sugars}(\%) = \frac{\text{Extracted pure sugars}}{\text{Extracted crude sugars}} \times 100\% \tag{3}$$

The extraction efficiency was obtained as the quotient of the mass of pure sugar extracted and the total sugar in the raw sludge, as presented in Equation (4):

$$\text{Extraction efficiency}(\%) = \frac{\text{Extracted pure sugars}}{\text{Total sugars in raw SS}} \times 100\% \quad (4)$$

3. Results and Discussion

3.1. Effects of Pretreatment Conditions on Sugar Extraction

3.1.1. Effect of NaOH Dosage

It can be seen from Figure 1 that both the crude sugar yield and sugar extraction efficiency increase with the increase in NaOH dosage. The crude sugar yield and extraction efficiency are only 2.60% and 3.23%, respectively, with no alkali added, but both reach the maximum values of 19.91% and 70.35%, respectively, with the NaOH dosage of 10 mL. This is because the larger the dosage of NaOH, the stronger the alkalinity and the greater the damage to the cell structure in the sludge. Alkali can enhance the hydrolysis of bio-polymers, improving the solubility and extraction efficiency of sugar monomers [6]. Better EPS cracking effects and dissolution of more sugars were thus achieved. Due to a higher amount of alkali, the economic cost increases, and a digestion and degreasing reaction may also occur in the sludge [16], resulting in the structural damage of sugars, leading to a decrease in extracted yields. Therefore, the NaOH dosage of 10 mL is set as the maximum.

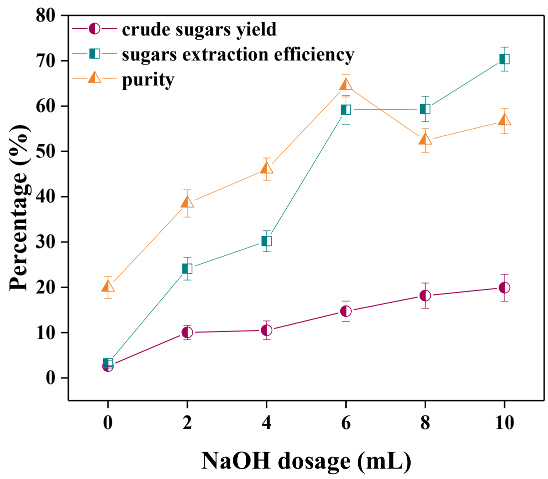

Figure 1. Effects of NaOH dosage on crude sugar yield, crude purity and sugar extraction efficiency.

As shown in Figure 1, the purity of crude sugar is only 19.94% with no alkali used. The purity of crude sugar increases with the increase in the dosage of NaOH, and peaks at 64.47% with the dosage of NaOH of 6 mL. When the NaOH dosage continues to increase, the purity of crude sugar decreases. In the absence of alkali, the sludge cells are not broken, the crude sugar is rarely dissolved and the extracted crude sugar contains more inorganic particles. However, when the dosage of NaOH is higher, lipids and proteins can also be dissolved and extracted with sugars by thermal treatment, resulting in a lower purity.

3.1.2. Effect of Alkali Extraction Time

Figure 2 shows the effect of leaching time on sugar extraction. In the early stage of alkali treatment, the yield of crude sugar increased rapidly with time, and reached the maximum (29.72%) at 30 min. With the increase in leaching time, the yield of crude sugar decreased slowly. The variation trend of crude sugar purity and sugar extraction efficiency was similar to that of crude sugar yield, reaching the maxima at 45 min, which were 48.58% and 89.23%, respectively.

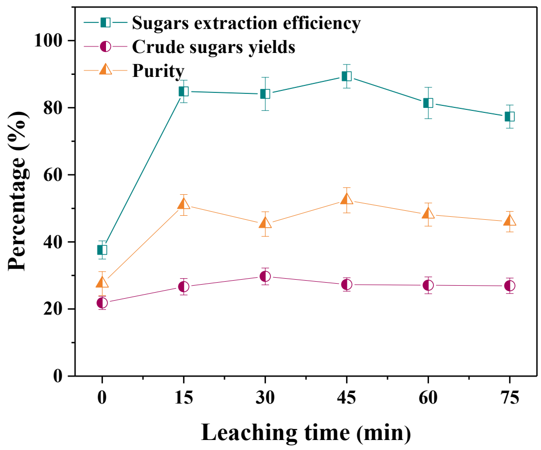

Figure 2. Effects of leaching time on crude sugar yield, purity and sugar extraction efficiency.

It is speculated that the extraction of crude sugar from sludge may reach the equilibrium in about 30 min. Shorter extraction time may result in incomplete extraction of crude sugar due to insufficient reaction. When the equilibrium point is exceeded, the leaching treatment may lead to degradation of the extracted sugars. It is well known that sugars can be easily hydrolyzed into smaller molecules such as organic acids and furfural under thermal conditions. Meanwhile, polyphenol synthesis can arise between reducing sugars [25]. Thanks to these processes, the maximum leaching time of crude sugar yield is 30 min with the addition of 10 mL NaOH.

3.1.3. Effect of Microwave Time on Sugar Extraction

As shown in Figure 3, the yield of crude sugar gradually increases when the microwave exposure time is from 0 to 45 s, and reaches the maximum of 39.38% at 45 s. However, with the increase in microwave time, the yield of crude sugar decreases obviously. The trend of sugar extraction efficiency is consistent with that of crude sugar yield, reaching the maximum of 88.62% at 45 s. This may be because the cracking effect of sludge cells gradually increases in the first 45 s of treatment. After 45 s, part of the organic matter content in the sludge decomposes due to excessive microwave exposure time [14,26], resulting in the reduction in crude sugar yield.

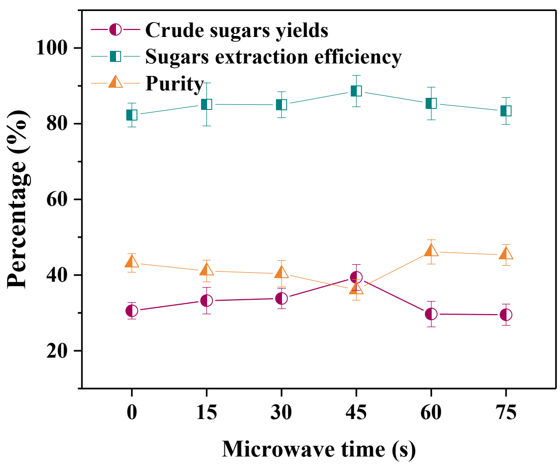

Figure 3. Effects of microwave time on crude sugar yield, purity and sugar extraction efficiency.

When the microwave treatment time was less than 45 s, the purity of crude sugar decreased with the increase in time, down to 36.1% at 45 s. When the microwave time increased to 60 s, the purity of crude sugar increased rapidly to the maximum value of

46.12%. The maximum yield of crude sugar was obtained at a microwave time of 45 s with the lowest purity. This might be because the cracking effect of sludge promoted the dissolution of other organic matter, resulting in a low purity of crude sugar [27].

3.2. Response Surface Analysis

According to the RSM design method and Box–Behnken design principle, the pretreatment conditions affecting the yield of crude sugar were optimized, including NaOH dosage, NaOH leaching time and microwave treatment time. The experimental design and results are shown in Table 2.

Table 2. Crude sugar yield response surface design and results.

Number	NaOH Addition (mL)	Leaching Time (min)	Microwave Time(s)	Crude Sugar Yield (%)
1	0	30	0	2.60
2	2	30	0	10.04
3	4	30	0	10.52
4	6	30	0	14.72
5	8	30	0	18.16
6	10	30	0	19.91
7	10	0	0	21.82
8	10	15	0	26.65
9	10	45	0	27.31
10	10	60	0	27.07
11	10	75	0	26.92
12	10	30	0	30.55
13	10	30	15	33.24
14	10	30	30	33.79
15	10	30	45	39.38
16	10	30	60	29.68
17	10	30	75	29.50

Taking the yield of crude sugar as the response value, the quadratic multinomial regression equation obtained by the RSM test is shown as Equation (5), where A, B and C are NaOH dosage, NaOH leaching time and microwave treatment time, respectively. The variance analysis of the RSM results is shown in Table 3.

$$\text{Crude sugar yield (\%)} = 23.90 + 11.10\ A + 2.12\ B + 1.02\ C + 1.53\ A^2 - 1.90\ B^2 - 8.92\ C^2 \quad (5)$$

Table 3. Results of ANOVA analysis of the regression model. Parameters with p-value ≤ 0.001 are considered significant.

Source	Sum of Squares	df	Mean Square	F-Value	p-Value	
Model	1395.73	6	232.62	19.84	0.0001	Significant
A	465.68	1	465.68	39.72	0.0001	
B	12.69	1	12.69	1.08	0.3253	
C	4.16	1	4.16	0.3544	0.5663	
A^2	2.69	1	2.69	0.2296	0.6433	
B^2	4.13	1	4.13	0.3524	0.5674	
C^2	101.52	1	101.52	8.66	0.0164	
Residual	105.52	9	11.72			
Lack of Fit	48.92	8	6.11	0.1080	0.9840	Not Significant
Pure Error	56.60	1	56.60			
Cor. Total	1501.26	15				

It can be seen from Table 3 that the p values of the model are less than 0.001 (not significant), indicating the effective model simulation. The yield of crude sugar was significantly affected by the dosage of NaOH, leaching time and microwave time. Based on the response surface optimization analysis, the optimum pretreatment conditions (crude sugar yield = 39.41%) were NaOH dosage of 9.93 mL, leaching time of 27.65 min and microwave time of 33.2 s. The crude sugar yield obtained with experiments under this condition was 39.80 $\pm$ 3.57%. The crude sugar yield and extraction efficiency obtained in the experiment under this condition were 39.80 $\pm$ 3.57% and 89.74 $\pm$ 3.61%, respectively. The measured value of the crude sugar yield was close to the predicted value, indicating that the model could predict the crude sugar yield well.

Figure 4 depicts the interactive effects of each of the two sludge pretreatment parameters on crude sugar yield. As can be seen in Figure 4a, when the dosage of NaOH is 10 mL and the leaching time is 15–45 min, there is a good yield of crude sugar. The effect of NaOH dosage on the yield of crude sugar is greater than that of leaching time. Figure 4b shows a gentle response surface contour slope, indicating that the interaction between NaOH dosage and leaching time has little effect on the yield of crude sugar.

Figure 4c shows that the yield of crude sugar is better when the NaOH dosage is 8–10 mL and the microwave time is 15–60 s. The effect of microwave time on the yield of crude sugar is greater than that of NaOH dosage. The response surface contour slope is slow (Figure 4d), indicating that the interaction between the two has little effect on the crude sugar yield.

Figure 4e shows that when the microwave time is 15–60 s and the extraction time is 15–75 min, the crude sugar yield is higher. The influence of microwave time on the yield of crude sugar is greater than that of extraction time. Figure 4f shows that the response surface contour is relatively oval, indicating that the interaction between the two has a greater impact on the yield of crude sugar. In this response surface model, the optimal value of crude sugar yield is 39.41%, with NaOH dosage of 9.93 mL, leaching time of 27.65 min and microwave time of 33.2 s. In the experiments conducted under the optimal conditions, the crude sugar yield reaches 39.80 $\pm$ 3.57%. The integrated process considerably diminishes the time and specific energy required for biomass disintegration, which makes the process more attractive for practical applications. Therefore, energy analysis and technical–economic analysis will be further employed to evaluate the scaling up of the process.

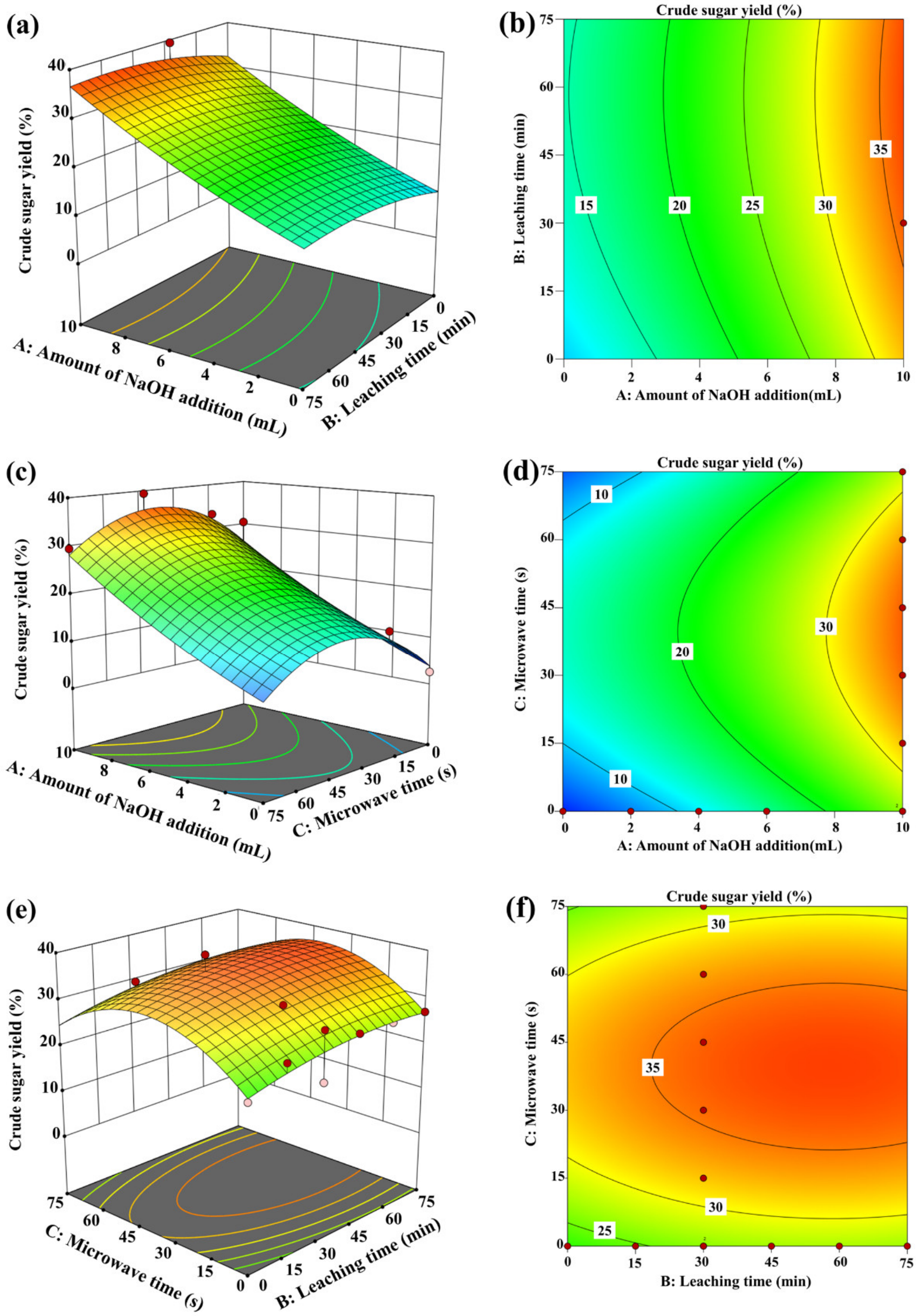

Figure 4. Response surface plots and contour plots of crude sugar yield based on the effect of leaching time and amount of NaOH addition (**a**,**b**), amount of NaOH addition and microwave time (**c**,**d**), microwave time and leaching time (**e**,**f**). The circles represent the crude sugar yield obtained in the experiment, which are red if they are above the predicted value and pink if they are below the predicted value.

3.3. Characterization of Sludge and Crude Sugar

3.3.1. Fourier Transform Infrared Spectra

The Fourier transform infrared spectra of sludge and extracted crude sugar are shown in Figure 5. It can be seen from Figure 5a that the absorption peaks of raw sludge, thermal-alkaline pretreated sludge and thermal-alkaline combined with microwave pretreated sludge all appeared at the positions of 3432, 1596, 1357 and 1040 cm^{-1}. The absorption peak at 3432 cm^{-1} was broad and strong, which was due to the stretching vibration peak of O–H in the hydrogen bond [4]. The C=O vibration peak at 1596 cm^{-1} could be attributed to the carboxylate [9]. The peak at 1357 cm^{-1} was associated with the N–O vibration peak [28]. The strong absorption peak at 1040 cm^{-1} was ascribed to the C–O stretching vibration, which was also the characteristic peak of a pyran glycosidic bond, indicating that a pyran glycosidic bond might exist in the sludge [29]. The pretreated sludge showed a weak absorption peak at 2928 cm^{-1}, which could be attributed to the stretching vibration of C–H in carbohydrate [30], while the raw sludge showed no absorption peak at this point, indicating that the alkali extraction and microwave treatment destroyed the sludge structure and thus made the sugar dissolve out [31].

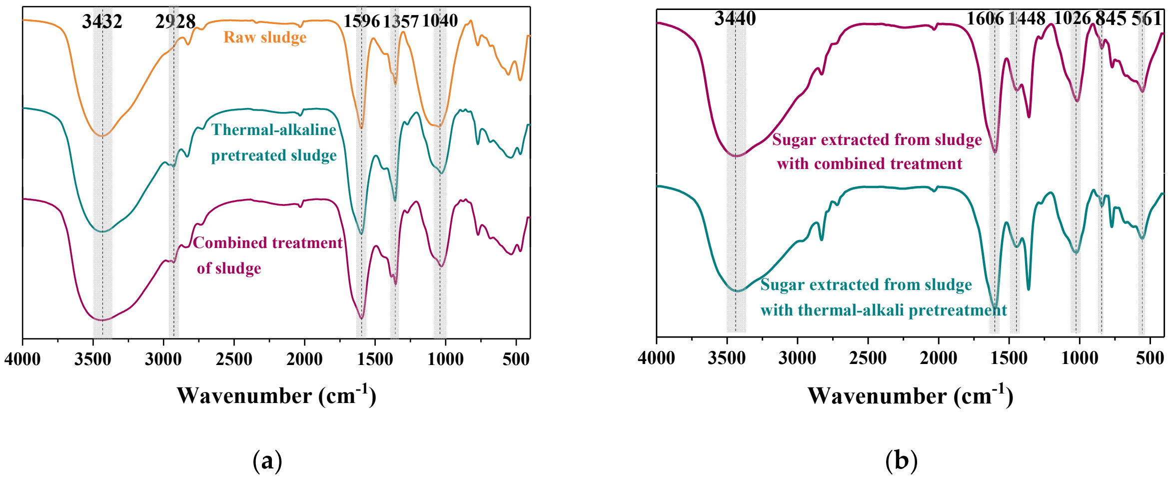

Figure 5. FTIR spectra of (**a**) raw sludge, thermal-alkaline pretreated sludge and sludge with microwave combined with thermal-alkaline pretreatment; (**b**) extracted sugars from sludge with combined pretreatment and thermal-alkaline pretreatment.

It can be seen from Figure 5b that the crude sugar extracted by microwave combined with thermal-alkaline and thermal-alkaline pretreatments alone showed wide and strong absorption peaks at 3440 cm^{-1}, which were due to the stretching vibration peak of O–H in the hydrogen bond [4]. The vibration of C=O at 1606 cm^{-1} may have been caused by amides in carboxylic acids and derivatives [9]. The peak at 1362cm^{-1} was associated with the N–O vibration peak [28]. The absorption peaks at 1026 cm^{-1} and 845 cm^{-1} were attributed to the polysaccharide and glycoconjugates containing galactose and mannose [27,28], which further confirms the successful extraction of crude sugar.

3.3.2. Sludge Surface Morphology

The micromorphology of raw sludge, thermal-alkaline treated sludge and sludge with microwave combined thermal-alkaline treatment is shown in Figure 6. It can be seen that the raw sludge in Figure 6a has a cluster-like porous structure, in which the sludge is closely combined into a cluster and distributed with several pores of different sizes. The raw sludge in Figure 6b has a multi-prism block structure, and the surface of the block protrudes with a plurality of irregular edges.

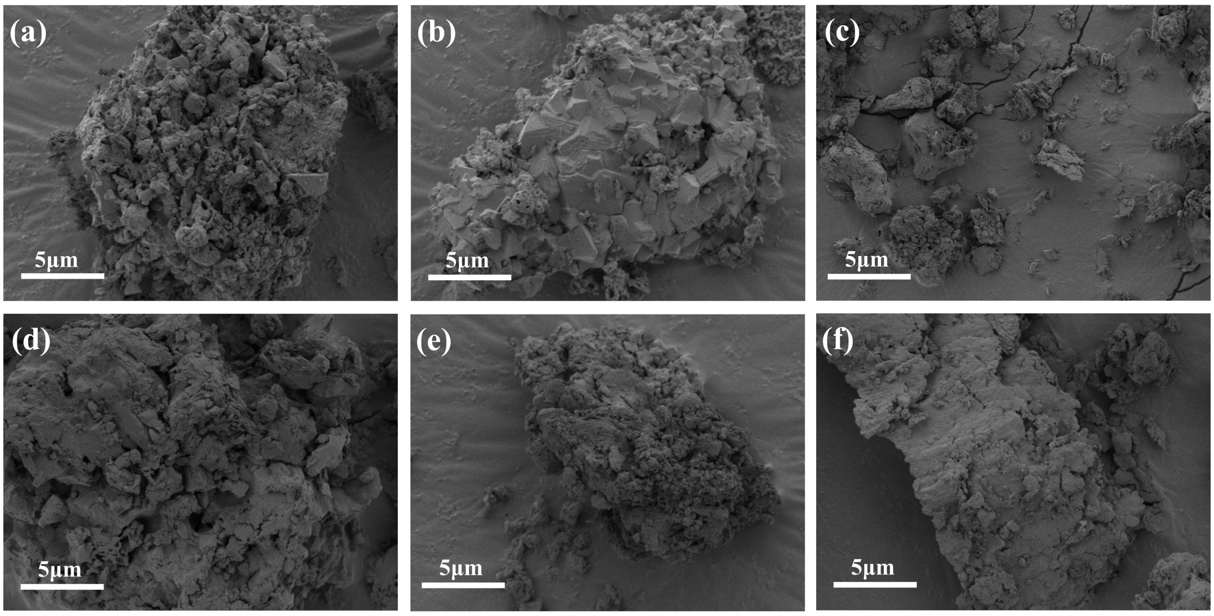

Figure 6. SEM images of raw sludge (**a**,**b**), thermal-alkaline pretreated sludge (**c**,**d**), and sludge with microwave combined with thermal-alkaline pretreatment (**e**,**f**).

The microscopic shape of the sludge after 30 min of thermal-alkaline treatment is shown in Figure 6c,d. Compared with the raw sludge, the sludge after thermal-alkaline treatment has a greatly changed microstructure. In Figure 6c, the agglomerated porous structure becomes loose, changing from agglomerates to many small, scattered groups. Pores can still be observed on the surface of small groups, indicating that although alkali treatment has a certain crushing effect on the agglomerated porous structure of the sludge, it does not change the porous characteristics of its surface. It can be seen from Figure 6d that the multi-surface protrusions of the raw sludge after thermal-alkaline treatment are destroyed, and the porous morphology on the surface appears. This may be because alkali leaching has a crushing effect on the convex structure of the sludge surface but fails to break its large, combined structure.

The morphology of the sludge after microwave combined with thermal-alkaline pretreatment is shown in Figure 6e,f. Compared with the raw sludge, the change of the aggregate porous structure of the sludge after the thermal-alkaline combined microwave treatment is not obvious (Figure 6e), indicating that the microwave treatment has no significant effect on the aggregate porous structure of the sludge. The raw sludge with a polygonal block structure changes obviously after thermo-alkali combined with microwave pretreatment. The convex prismatic structure on the surface is destroyed, while the block structure becomes relatively loose, and a layered structure with smooth surface and pores appears (Figure 6f). This shows that microwave treatment can destroy the surface structure of the sludge and reduce the tightness of the sludge structure.

3.3.3. Crude Sugar Surface Morphology and Composition

Figure 7 presents the morphology and elemental composition of crude sugar extracted by microwave combined with thermal-alkaline pretreatment. It can be seen from Figure 7a that the overall structure of the crude sugar is flocculent, and a large number of tiny voids are distributed on the surface. As presented in Figure 7b, the proportions of O, C, N, S, K and Na in crude sugar are 47.4%, 31.6%, 3.7%, 1.6%, 0.6% and 15.2%, respectively. Common sugars and their derivatives mainly contain C, H, O, N and other elements, but the proportion of Na in the crude sugar obtained in this experiment is 15.2%, which can reduce the purity of sugar. This phenomenon is consistent with the fact that the purity of crude sugar extracted by microwave combined with thermal-alkaline pretreatment in

Figure 3 is lower than 50%, indicating that a high NaOH dosage affects the purity of crude sugar.

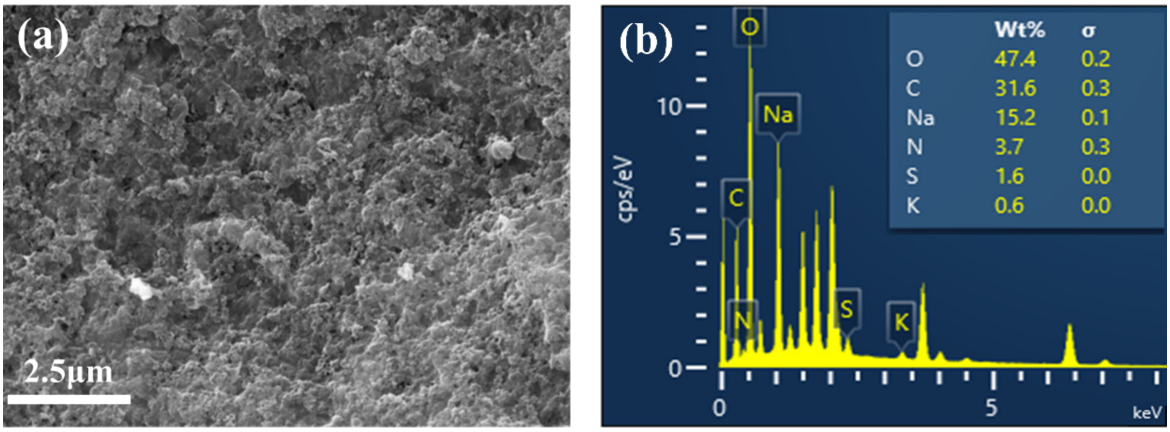

Figure 7. SEM image (**a**) and EDS spectra (**b**) of the extracted crude sugar.

4. Conclusions

The extraction efficiency of sugars from SS was evaluated under different microwave pretreatment combined with thermal-alkaline conditions. The yield of crude sugar and the extraction efficiency increased with the increase in NaOH dosage, but the purity decreased when the NaOH dosage was too high. The extraction of crude sugar from sludge reached the equilibrium at about 30 min. Microwave treatment for 45 s could increase the yield of crude sugar to 39.38%, but decreased the purity of the crude sugar.

Based on the response surface optimization analysis, the optimal pretreatment process conditions with a crude sugar yield of 39.41% were obtained as follows: the dosage of NaOH was 9.93 mL, the leaching time was 27.65 min and the microwave time was 33.2 s. The crude sugar yield and extraction efficiency obtained in the experiment under this condition were 39.80 ± 3.57% and 89.74 ± 3.61%, respectively. Higher NaOH dosage, longer leaching time and stronger microwave conditions decreased sugar yields due to the hydrolysis of soluble sugars.

Author Contributions: Conceptualization, Y.F.; methodology, Y.F.; formal analysis, Y.L. and P.C.; investigation, P.C. and J.L.; writing—original draft preparation, P.C.; writing—review and editing, L.Y.; visualization, Y.L.; funding acquisition, Y.F. All authors have read and agreed to the published version of the manuscript.

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Data Availability Statement: Data will be made available on request.

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Conflicts of Interest: The authors declare no conflict of interest.

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