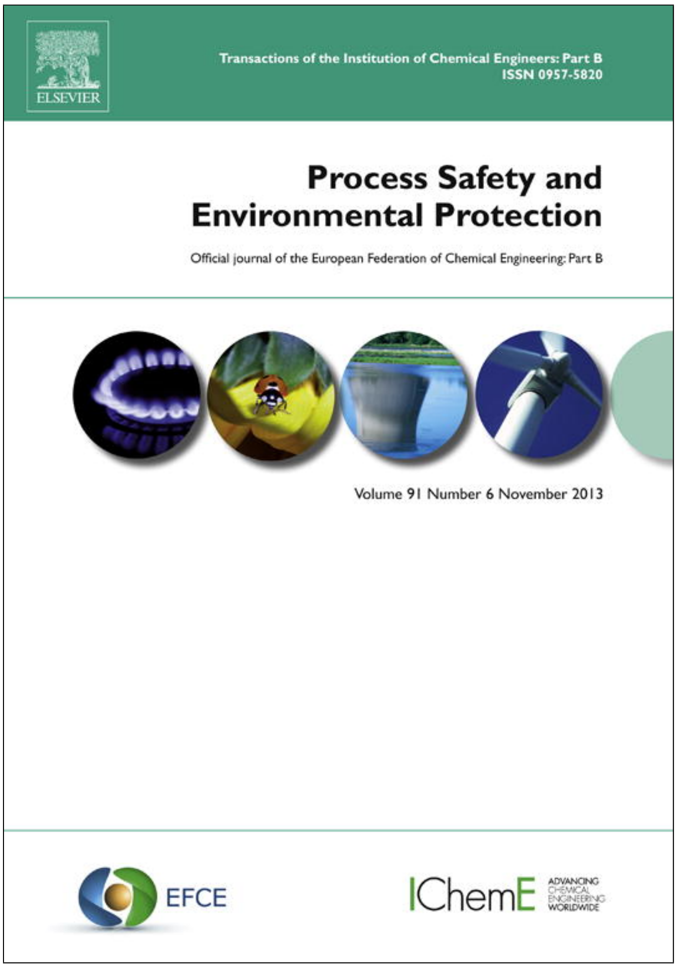
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Improving disintegration and acidification of waste activated sludge by combined alkaline and microwave pretreatment

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ABSTRACT

The individual alkaline or microwave pretreatment has been proved to be effective in disintegration and acidification of waste activated sludge (WAS). In this study, the effects of combined alkaline and microwave pretreatment at different pH and specific energy input (Es) on WAS disintegration were investigated using response surface methodology (RSM). Combined pretreatment achieved disintegration degree (DD) of 65.87% at Es of 38,400 kJ/kg TS and pH 11.0. The ANOVA further demonstrated that pH showed more significant effect on DD than Es. Anaerobic batch experiment results showed that combined pretreatment not only significantly improved volatile fatty acids (VFAs) accumulation but also shortened the time for the highest VFAs accumulation. The maximal VFAs accumulation (1500 mg COD/L) obtained at Es of 28,800 kJ/kg TS and fermentation time of 72 h, which was about two times that of the treatment without microwave (850 mg COD/L) at 96 h. The analysis of VFAs composition showed that the VFAs mainly consisted of acetic and iso-valeric acids, accounting for 57.3–70.1% of total VFAs.

Keywords: Acidification; Alkaline-microwave pretreatment; Disintegration; Response surface methodology; Waste activated sludge

1. Introduction

In China, the application of activated sludge process in wastewater treatment plants (WWTPs) has improved the quality of stream and river water, meanwhile a large amount of waste activated sludge (WAS) are generated during this process. Because of its pathogen content and its unstable, decomposable nature, WAS would pose a threat to the environment if not properly treated and/or disposed. The treatment of WAS represents 30–40% of the capital cost and about 50% of the operating cost of many wastewater treatment plants (Vlyssides and Karlis, 2004).

Anaerobic digestion (AD) is thought to be the most traditional, cost-effective sludge treatment method because it can reduce the volume of WAS, transform organic matters into biogas, generate substantial nutrient final products, destroy pathogens in the sludge and limit odor problems simultaneously (Mata-Alvarez et al., 2000; Toreci et al., 2009). However, its application has often been limited by long retention times (20–50 days) and low overall degradation efficiency (20–50%), which are generally associated with the hydrolysis stage of WAS (Kim et al., 2010). As WAS is mainly composed of microbial cells, and cell walls form physical and chemical barriers to prevent intracellular organics to be easily biodegraded through digestion. Therefore, slow and incomplete hydrolysis of extracellular polymeric substances (EPS) and microbial biomass together limit the rate and extent of degradation in anaerobic digestion (Higgins and Novak, 1997). In order to disrupt sludge flocs and bacteria cells, release cellular components and accelerate subsequent acidification, the sludge

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disintegration methods such as mechanical (Kampas et al., 2007), thermal (Camacho et al., 2002), chemical (Lin et al., 1997) and biological treatments or combination of any two of these methods are necessary before AD (Tyagi and Lo, 2012).

Compared with other sludge pretreatment methods, alkaline can effectively solubilize particulate organic matter in the sludge and improve its digestibility in a simple device (Jin et al., 2009; Li et al., 2008). Especially, alkaline treatment combined with other disintegration methods such as thermal, ultrasonic, and microwave treatments shows high efficient to sludge disintegration. Liu et al. (2008) investigated thermo-acid, thermo-alkaline, ultrasonic-alkaline and ultrasonic-acid, on solubilization and subsequent acidification efficiency of WAS and found that both thermo-alkaline and ultrasonic-alkaline significantly improved the solubilization of WAS with 60.2–61.6% of volatile solids (VS) solubilization. It was also demonstrated that they had similar effects on the acidification of WAS.

During the past several years, many studies have been focused on employing microwave (MW) as an alternative to conventional heating for environmental engineering applications, due to the facts that MW heating can reach the desired temperature more rapidly, consume less energy and has a smaller potential hazardous emission (Chang et al., 2011). Pino-Jelcic et al. (2006) found that at similar temperatures MW irradiated as sludge thermal pretreatment led to more COD solubilization and biogas production compared to conventional heating. Moreover digested sludge pretreated by MW showed a better dewaterability compared to conventionally heated and non-pretreated digested sludge.

Since individual alkaline or MW pretreatment method has been proven to be highly effective to sludge disintegration, their combination maybe produce synergistic action and even more efficient method can be obtained. However, in most previous studies, they were examined separately or sequentially to enhance the sludge disintegration (Chang et al., 2011; Doğan and Sanin, 2009), few studies had been conducted to evaluate the combined alkaline and microwave pretreatment on sludge disintegration and subsequent acidification. Response surface methodology (RSM) provides a systematic and efficient research strategy for studying the interaction of various parameters effect using statistical methods. Therefore the main objective of this study was to investigate the combined influence of pH and specific energy input on WAS disintegration using RSM. To show the comprehensive efficiency of the combined pretreatment method on subsequent sludge acidification, the accumulation and composition of VFAs was examined.

2. Materials and methods

2.1. WAS

Sewage sludge for this study was obtained from the secondary sedimentation tank of the second WWTPs in Changsha, China. Fresh sludge was filtered through a 0.71-mm metal sieve and then refrigerated at 4 °C for 24 h before use. The characteristics of sludge were as followings: pH 6.68 ± 0.15, total chemical oxygen demand (TCOD) 10,000 ± 200 mg/L, soluble chemical oxygen demand (SCOD) 220 ± 10 mg/L, total solid (TS) 12,490 ± 250 mg/L, volatile suspended solids (VSS) 6840 ± 80 mg/L, carbohydrate 45.65 mg/L ± 2 mg/L, protein 450.58 ± 25 mg/L.

2.2. *Sludge disintegration*

The combined alkaline and microwave disintegration of WAS was conducted as follows: The pH value of 100 mL sewage sludge was firstly adjusted to 8.0, 9.0, 10.0, 11.0 and 12.0, respectively, by adding 1 M sodium hydroxide (NaOH) or 1 M hydrochloric (HCl), and the desired pH values were kept with ± 0.1 unit fluctuations.

And then the alkaline sludge in a quartz reactor was immediately heated in MW heating apparatus (LWMC-205, Nanjing Robiot Co. Ltd., China, 0-750W, maximum temperature: 180 °C) with a microwave power of 300 W. And the specific energy input (Es) was set as 9600, 19,200, 28,800, 38,400, 48,000 kJ/kg TS by altering the irradiation time. The Es was defined as a function of microwave power (*P*), microwave time (*t*), sludge sample volume (*V*), and initial total solid (*TS*):

$$\mathrm{Es} = \frac{P \times t}{V \times TS} \tag{1}$$

In order to minimize random error, each sample was analyzed in triplicate and the average values were determined for each set.

2.3. *Response surface methodology (RSM)*

The purpose of using RSM as a sensitivity analysis tool is to improve the analyst's understanding of the sensitivity between the output of the model (response) and its associated input variables and then use this information to improve the decision model (Bauer et al., 1999). Based on quantitative data from appropriate experiments, the regression model equations and operating conditions determined by RSM are useful for developing, improving and optimizing processes.

In this study, the experimental design was carried out using a central composite design (CCD) with the Design-Expert 8.0.6 (Stat-Ease Inc., USA) software, which was employed to investigate the simultaneous effect of two independent variables: pH and Es on WAS disintegration. The target response was the disintegration degree (DD) of WAS, which was calculated as in Eq. (2) according to Zhang et al. (2007).

$$\text{Disintegration degree (\%)} = \frac{\mathrm{SCOD} - \mathrm{SCOD_0}}{\mathrm{TCOD_0} - \mathrm{SCOD_0}} \times 100 \tag{2}$$

In which SCOD is the SCOD of the pretreated sludge, and $SCOD_0$, $TCOD_0$ is the SCOD, TCOD of the untreated sludge, respectively.

These two variables, pH (x_1) and Es (x_2), together with their respective ranges were chosen based on previous studies as given in Table 1. A binary quadratic equation was afforded to correlate the response to the independent variables.

The experimental data obtained by CCD were analyzed by RSM using a second-degree polynomial equation as given by Eq. (3).

$$Y = \beta_0 + \sum_{i=1}^{2}\beta_i x_i + \sum_{i=1}^{2}\beta_{ii} x_i^2 + \sum_{i=1}^{1}\sum_{j=i+1}^{2}\beta_{ij} x_i x_j \tag{3}$$

where Y is the response variable, x_i and x_j are the coded variables, β_0 is the constant coefficient, β_i is the linear coefficients, β_{ii} is the quadratic coefficients, β_{ij} is the interaction coefficients.

2.4. Anaerobic batch test

Anaerobic batch tests were carried out in five glass flasks with an effective volume of 0.5 L each. The seed sludge was also taken from the secondary sedimentation tank of the second WWTPs in Changsha, China. Every reactor contained a volume of 100 mL sludge and the pH value in reactors was adjusted to 11.0 by 1 N NaOH or 1 N hydrochloric (HCl). One reactor was operated as the control test and fed with untreated sludge, while the others fed with sludge pretreated by fixed microwave power of 300 W and different Es (9600, 19,200, 28,800, and 38,400 kJ/kg TS, respectively). Before fermentation, in order to maintain strict anaerobic condition, all reactors were capped with rubber stoppers and oxygen was removed from the headspace by nitrogen gas sparging. The reactors were placed in water-bath shakers (100 rpm) operated at 35 °C (±2 °C) for 7days.

2.5. Analytical methods

Sludge samples from the reactors were firstly filtered through Whatmann GF/C glass microfibre filter (0.45 μm pore size). Filtrate was used for analysis of the soluble substances including SCOD, VFAs, soluble proteins and soluble carbohydrates and the filter residue was assayed for TS, VS and so on. TCOD, SCOD, pH, TS, and VS were determined according to the Standard Methods (APHA, 1995). Soluble protein was determined by the Lowry–Folin method with bovine serum albumin (BSA) as standard (Lowry et al., 1951). Carbohydrate was measured by the phenol sulfuric method with glucose as the standard (Herbert et al., 1971).

The quantification of VFAs was determined according to the method described in previous publication (Luo et al., 2011). The total VFAs were recorded as the sum of measured acetic acid, pro-pionic acid, *n*-butyric acid, iso-butyric acid, *n*-valeric acid and iso-valeric acid.

3. Results and discussion

3.1. Optimization of combined alkaline and microwave pretreatment

3.1.1. Development of regression model equation

The complete design matrixes are shown in Table 1, together with the response values. Higher DD was found to range from 50 to 65% by combined pretreatment. According to the sequential model sum of squares, the models were selected based on the highest order polynomials where the additional terms

Table 1 – Experimental design matrix for combined alkaline and microwave pretreatment.

Run	Coded variables		Experimental variables		DD (%)
	x_1	x_2	x_1	x_2 (kJ/kg TS)	
1	0	0	10.00	28,800	41.76
2	−2	0	8.00	28,800	16.84
3	0	2	10.00	48,000	50.15
4	−1	1	9.00	38,400	19.87
5	1	1	11.00	38,400	65.87
6	0	0	10.00	28,800	41.65
7	2	0	12.00	28,800	62.50
8	0	−2	10.00	9600	18.63
9	1	−1	11.00	19,200	51.25
10	−1	−1	9.00	19,200	16.02

were significant and the models were not aliased. The corresponding second-order polynomial equation after analysis for the regression had been given by Eq. (4):

$$Y = -100.98 + 14.88x_1 - 1.00 \times 10^{-3}x_2 + 2.80 \times 10^{-4}x_1 \times x_2 - 0.43x_1^2 - 1.90 \times 10^{-8}x_2^2 \quad (4)$$

The quality of the developed model was evaluated using the correlation coefficients, R^2 value. In fact, the models at low standard deviation and high R^2 statistics seem to be the best, it indicates a high correlation between the predicted value and the actual value in the response (Ahmad et al., 2009). In this experiment, the standard deviation for the model was 7.78, which demonstrated the predicted values would be more accurate and closer to its actual value. The correlation coefficients, R^2 was 0.9280, which implies that 92.80% of the experimental data confirm compatibility with the data predicted by the model. Therefore, this empirical model can be used to predict the DD of WAS.

3.1.2. Analysis of variance

The adequacy of the model was further justified through analysis of variance (ANOVA). The model with calculated F-value and a very low *p*-value imply that the model is highly significant. The ANOVA for the quadratic model for DD of WAS is listed in Table 2. The model F-value of 10.30 and Prob. > F of less than 0.05 indicated model terms were significant. In this case, x_1, x_2 were significant model terms whereas x_1x_2, x_1^2, x_2^2 were all not significant, which implied that pH and Es had a significant effect on improving the DD. However, pH showed more significant effect on DD than the Es according to the F-value and prob. > F. In literature, Li et al. (2010) had inferred from the kinetics model that pH had the most significant effect on the sludge disintegration in combined (alkaline + ultrasonic) pretreatment. The "lack-of-fit F-value" of 13338.63 showed that the lack-of-fit was significant relative to the pure error. There was only a 0.64% chance that "lack-of-fit F-value" could occur due to noise.

3.1.3. Response surface plots

The 3D response surfaces and 2D contours plot describing the tendency of DD with respect to pH and Es levels is shown in Fig. 1. It was observed that the pH and Es had significantly effect on the DD. The response surface of DD gradually increased with increasing pH. This was similar to the findings of other studies, one of which reported that when WAS was pretreated by MW + pH 10, MW + pH 11, MW + pH 12 and MW + pH 12.5, respectively, SCOD/TCOD ratio was increased from 0.005 (MW only), 0.18, 0.27, 0.34 and 0.37 (Doğăn and Sanin, 2009). This might be explained that increase in pH resulted in more solubilization of organics for combined pretreatments.

Fig. 1 illustrates that the DD goes through an initial rise, a mid peak (38,400 kJ/kg TS), and a final slightly fall as Es rises from 19,200 to 48,000 kJ/kg TS. It had reported that higher Es could result in higher DD (Yu et al., 2010), which can be concluded that microwave irradiation is a useful tool for enhancing sludge disintegration. However, in the experiments of sludge disintegration by combined (alkaline + ultrasonic) pretreatment, Kim et al. (2010) observed that there existed a peak point (20,000 kJ/kg TS) at which further energy input increase can no longer improve disintegration rate of sludge.

Table 2 – ANOVA for a quadratic response surface model.

Source	Sum of squares	Degree of freedom (DF)	Mean square	*F* value	prob. > *F*
Model	3118.23	5	623.65	10.30	0.0211
x_1–X_1	2481.13	1	2481.13	40.99	0.0031
x_2–X_2	553.66	1	553.66	9.15	0.0390
x_1x_2	29.00	1	29.00	0.48	0.5269
x_1^2	3.06	1	3.06	0.05	0.8332
x_2^2	50.76	1	50.76	0.84	0.4116
Lack-of-fit $R^2 = 0.9280$	242.10	3	80.70	13338.63	0.0064

Similarly, the peak point also existed in combined alkaline-MW pretreatment and could be determined by Eq. (4) in this study, which should be helpful for avoiding wasteful energy consumption in microwave pretreatment from an economic point of view.

As seen from Table 1 and Fig. 1, it clearly showed that preconditioning of sludge at high pH levels played a crucial role in enhancing the disintegration efficiency of microwave pretreatment. For example, 16.84% of DD was obtained when 28,800 kJ/kg TS of Es was applied to the alkali-pretreated sludge at pH 8, but it was enhanced to 41.65 and 62.50% when the same Es was applied to the alkali-pretreated sludge at pH 10 and pH 12, respectively. Contrarily, when Es was above 28,800 kJ/kg TS, 41.65% of DD at pH 10 had no evident difference under different MW conditions, which was 41.76 and 50.15% at Es of 28,800 and 48,800 kJ/kg TS, respectively.

3.1.4. *Process optimization*

The optimum condition for DD of WAS was predicted using the "Point Optimization" tool of the Design Expert Version 8.0.5 (STAT-EASE Inc., Minneapolis, USA). The optimal values obtained for the test variables for maximum DD were as follows: Es 38,400 kJ/kg TS and pH 11.

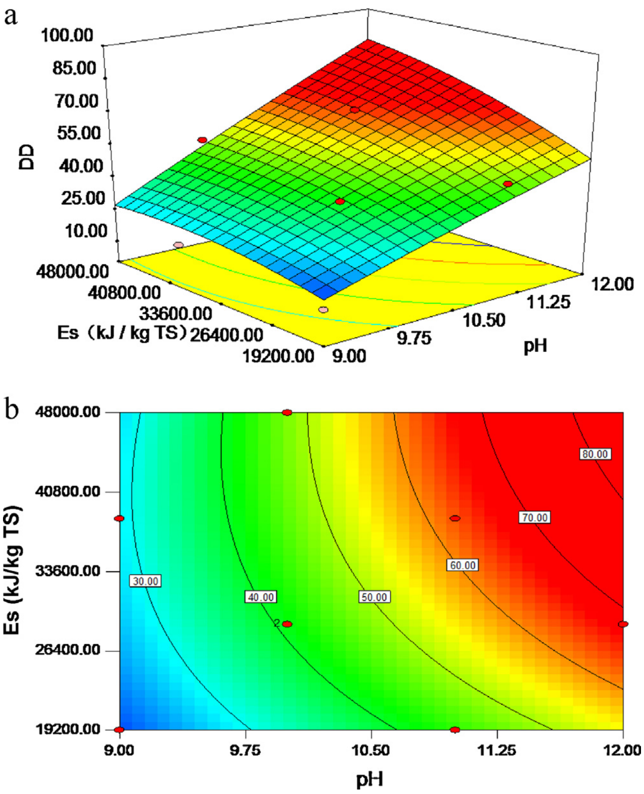

Fig. 1 – 3D response surface and contour plots: interactive effects of pH and Es on DD.

The experimental DD of 65.87% and the predicted value of 62.75% corresponded well. The high DD of WAS achieved clearly indicated that the optimum condition derived by RSM was favorable for the sludge disintegration by combined alkaline-MW pretreatment.

3.2. *Anaerobic digestion*

3.2.1. *WAS hydrolysis*

It is well-known that protein and carbohydrate are the main constituents of WAS, so the sludge proteins and carbohydrates were respectively converted to soluble proteins and carbohydrates in hydrolysis and acidification process of sludge. The effects of Es on the concentrations of soluble protein and carbohydrate at pH 11.0 are shown in Fig. 2.

It was observed that the concentrations of these two products had a similar variation trend during fermentation process at all Es investigated. Obviously, the higher were the Es, in response to the more proteins and carbohydrates. For example, the proteins concentration increased from 1255.37 to

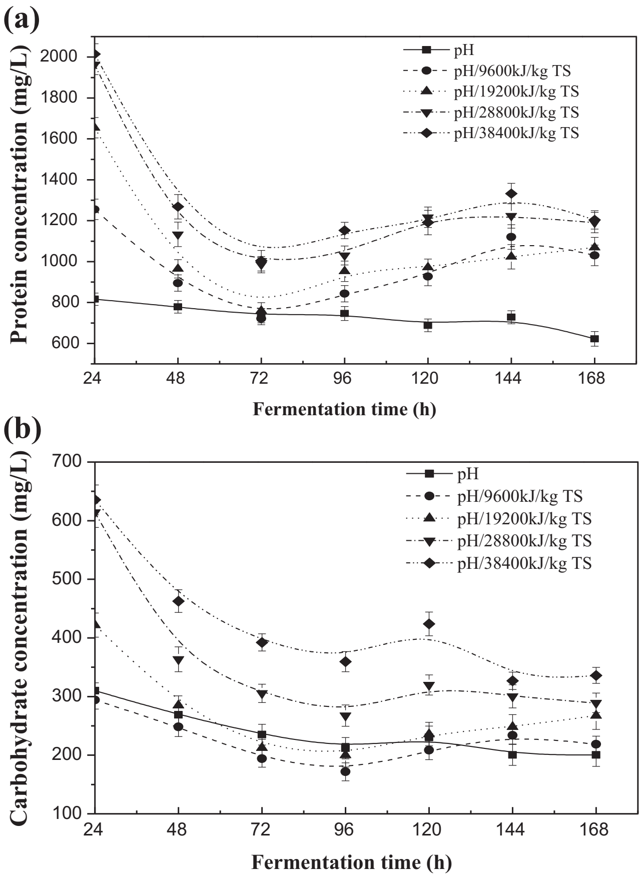

Fig. 2 – Effects of Es on (a) soluble proteins concentration and (b) soluble carbohydrates concentration during hydrolysis and acidification period at pH 11.0.

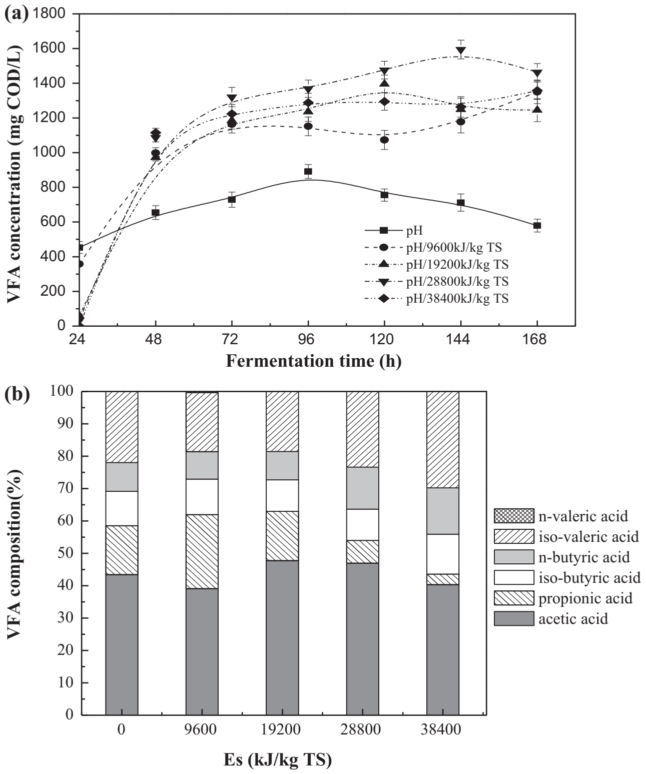

Fig. 3 – Effects of Es on (a) the VFAs accumulation and (b) the VFAs composition at pH 11.0.

2015.02 mg/L as the Es increased from 9600 to 38,400 kJ/kg TS, and the corresponding concentration of carbohydrates increased from 294.23 to 635.45 mg/L. However it was also observed that the concentration of soluble proteins and carbohydrates gradually reduced with the extension of fermentation time, and the protein showed higher removal rate than those of the carbohydrate, which might be correlated with two points. On one hand, the sludge hydrolysis process was accelerated at higher temperature and alkaline condition as a consequence of the combined alkaline-MW pretreatment. On the other hand, the MW pretreatment resulted in the disintegration of the complex and hardly biodegradable compounds (EPS and some colloidal material) to simpler ones that can be more readily biodegradable (Hong et al., 2006). The DD of WAS had a direct relationship to the energy input. Thus, higher concentrations of proteins and carbohydrates were observed at higher Es. As the acidification reaction continued, soluble proteins and carbohydrates were consumed as substrates to produce short chain fatty acids (SCFAs) (Kang et al., 2011).

It can also be seen in Fig. 2 that the concentrations of proteins and carbohydrates decreased dramatically at the beginning of 72 h and increased slowly over 3 days. It was suggesting that the soluble organic matters in the first 3 days were mainly released by microwave and consumed quickly by acidogenics. As the fermentation time exceeded 3 days, the overdose of alkaline began to give benefits on WAS hydrolysis with more soluble organic matters released into the fermentation liquid.

3.2.2. VFAs accumulation and composition

During the hydrolysis and acidification process, the effect of various Es and fermentation times on total VFAs production at pH 11.0 is shown in Fig. 3(a). It was observed that VFAs production increased with Es between 0 and 28,800 kJ/kg TS and decreased when Es was 28,800 kJ/kg TS at a given fermentation time. The maximal accumulation of the VFAs concentration was about 1500 mg COD/L. The VFAs accumulation decreased as Es exceeded 28,800 kJ/kg TS, which was due to the fact that most microbial cells were destroyed and sludge activity decreased drastically. The result was in good agreement with that in the literature (Ahn et al., 2009). Also, when the fermentation time was not more than 72 h, the VFAs concentration increased with fermentation time at any given Es, and no significant increase of VFAs was observed after 72 h. The reason for this might be that the release rates of fermentative soluble protein and carbohydrate apparently slowed down in the latter stage of fermentation when the fermentation time exceeded 72 h, the rates of release and degradation could not maintained a dynamic balance. Apparently, Es of 28,800 kJ/kg TS and fermentation time of 72 h could be regarded as the suitable fermentation conditions for maximal VFAs accumulation.

As discussed above, the more soluble protein and carbohydrate were correspondence with higher VFAs production. As Fig. 3(a) illustrated, the maximal VFAs production without microwave pretreatment (96 h) was much longer than that when WAS was pretreated by microwave (72 h), which implied that the combined alkaline-microwave pretreatment not only significantly enhanced VFAs accumulation but also shorten the fermentation time for the highest VFAs accumulation.

As is well known, the type of VFAs in wastewater always influences the removal efficiency of nutrients such as nitrogen and phosphorus during biological wastewater treatment (Moser-Engele et al., 1998). So the distribution of VFA is crucial when the hydrolysate of WAS is used as external carbon source to biological nutrient removal. The distribution of individual VFA at different Es at the fermentation time of 72 h was shown in Fig. 3(b). As familiar to the previous literatures (Moser-Engele et al., 1998; Oehmen et al., 2004), acetic acid formed the major product, whose percentage accounting for total VFAs was above 40%. In most cases of this study, propionic acid was the second major product. However, VFAs distribution had a slightly variation after microwave irradiation. When Es increased from 9600 to 38,400 kJ/kg TS, the propionic percentage accounting for 22.91% of the total VFAs declined to 3.25%, whereas that of iso-valeric increased from 18.22 to 29.76%. The data in Fig. 3(b) showed that iso-butyric and *n*-butyric percentages accounting for total VFAs were about 10.0% with only a few variations as Es changed between 9600 and 38,400 kJ/kg TS. The *n*-valeric acid percentage was no more than 0.5%, and only appeared at 9600 kJ/kg TS.

4. Conclusion

The experiments demonstrated that combined alkaline-microwave pretreatment had an important effect on the disintegration and acidification of WAS. The optimal values obtained using RSM for the maximum DD of 65.87% were Es of 38,400 kJ/kg TS and pH 11.0. WAS hydrolysis and VFA accumulation at pH 11.0 improved significantly after microwave irradiation. The optimum conditions for VFA accumulation were Es of 28,800 kJ/kg TS and fermentation time of 72 h. The acetic acid, iso-valeric acid and propionic acids were the three main components of VFAs with the fraction of 46.97%, 23.38% and 7.02%, respectively.

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