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Optimizing food waste hydrothermal parameters to reduce Maillard reaction and increase volatile fatty acid production

Jiaze Liu[1,2,3], *Jun Yin*[1,2,3,*], *Xiaozhen He*[1,2,3], *Ting Chen*[1,2,3], *Dongsheng Shen*[1,2,3]

[1] *School of Environmental Science and Engineering, Zhejiang Gongshang University, Hangzhou 310012, P.R. China*
[2] *Zhejiang Provincial Key Laboratory of Solid Waste Treatment and Recycling, Zhejiang Gongshang University, Hangzhou 310012, P.R. China*
[3] *Instrumental Analysis Center of Zhejiang Gongshang University, Hangzhou 310018, China*

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ABSTRACT

The occurrence of the Maillard reaction and melanoidins formation during the hydrothermal treatment of food waste can reduce the yield of volatile fatty acids (VFA); however, few studies have investigated the adverse effects of the Maillard reaction. This study identified the impact of hydrothermal treatment parameters on hydrolysis and melanoidins formation and optimized the hydrothermal treatment conditions to enhance VFA production by minimizing the impact of the Maillard reaction. A response surface methodology was employed to optimize the hydrothermal treatment parameters and VFA production was evaluated. Results showed that temperature, reaction time, and pH were significant interacting factors with respect to hydrolysis and melanoidins formation while the C/N ratio and moisture content of food waste had little impact. The optimal conditions for hydrothermal treatment (temperature of 132 °C, reaction time of 27 min, and a pH of 5.6) enhanced VFA production by 22.1%. Under optimal hydrothermal treatment conditions, a higher initial C/N ratio further increased VFA production.

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Introduction

The disposal of vast quantities of food waste in China has resulted in serious environmental pollution and expense. However, food waste can be regarded as a valuable resource because it contains large quantities of organic matter which can be converted into energy (Jiang et al., 2014). Hence, anaerobic fermentation has been adopted as a relatively cost-effective waste treatment for renewable energy production and volume reduction of this high-moisture and energy-rich material. However, the hydrolysis acts as a barrier, limiting the effectiveness of biological processes, such as anaerobic fermentation, due to the complex chemical and physical structure of the raw materials (Pellera and Gidarakos, 2017). Therefore, to accelerate the hydrolysis of biomass, a suitable pretreatment prior to anaerobic digestion is a viable strategy to enhance its conversion to volatile fatty acids (VFA) and biogas, such as acid/alkaline, sulfite, nitrite, ultrasonic and hydrothermal treatments (Bougrier et al., 2006; Liu et al., 2020a, b). To address this, hydrothermal treatment (HT) has been studied

* Corresponding author.
E-mail: jyin@zjgsu.edu.cn (J. Yin).

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to promote the hydrolysis process by the solubilization of organic compounds to increase the bioenergy yield (Li et al., 2016; Xue et al., 2015).

While the enhancement of organic solubilization can be achieved, HT has also been found to promote the Maillard reaction (MR) and the formation of melanoidins (Li et al., 2017). Furthermore, the MR is accompanied by a reduction in the nutrient substrates (sugar or protein loss) (Martins and Van Boekel, 2005). As the final product of the MR, melanoidins have been found to have negative biological impacts and cytotoxic effect on microbial growth in high doses (Chandra et al., 2008; Wang et al., 2011). Consequently, it is necessary to eliminate the side-effects of the MR to optimize the utilization of the substrate. Nevertheless, previous studies have mainly focused on the bio-resource yield of HT, and the inhibitive impacts of MR have largely been ignored. Yin et al. (2014b) reported that the color of food waste changed to brown after HT at 180 °C and VFA production was lower compared to HT at 160 °C. Furthermore, kitchen waste showed a 7.9% decline in anaerobic biodegradability due to the production of melanoidins (Liu et al., 2012). Therefore, minimizing the influence of MR on VFA production needs to be further studied.

Previous studies have shown that temperature, pH, and reaction time are common parameters that impact the hydrolysis of substrates and melanoidins formation (Ariunbaatar et al., 2014; Cai et al., 2016). However, most of these studies focused on the dissolution of organic compounds and melanoidins formation under specific conditions and did not consider the interactive effects of these parameters. The carbon-nitrogen ratio (C/N) and moisture content are key properties of food waste. Thus, the impact of these two parameters on melanoidins formation also needs to be considered. However, the optimization of a single variable cannot effectively evaluate the interactive impacts of different factors on the targets (Elksibi et al., 2014). Comparative experiments are also time consuming and labor intensive, especially when difficult operations are involved (e.g., anaerobic fermentation). These problems could be overcome by employing the response surface methodology (RSM), a traditional technique used to optimize the performance parameters (Amado et al., 2014).

The objective of this study was to obtain a better understanding regarding the influence of hydrothermal parameters on hydrolysis efficiency and melanoidins formation. The optimal conditions for HT using the RSM were also explored. Finally, the feasibility of reducing the MR to accelerate VFA accumulation was verified under optimal hydrothermal conditions.

Table 1 – Main characteristics of food waste and inoculated sludge (based on % dry matter).

Parameter	Food waste	Anaerobic sludge
pH	6.1	/
TS (%)	20.5	10.1
VS (%)	97.8	86.8
Total carbohydrate (%)	51.2	13.8
Total nitrogen (%)	2.4	5.0
TCOD (%)	121.4	119.8
C/N	19.1	9.0

Table 2 – Coded variable levels used for process optimization.

Coding	Operating variables				
	Temperature (°C)	Reaction time (min)	pH	C/N	Moisture content (%)
−2.38	106	3.1	2.9	12.9	68.1
−1	120	10.0	5.0	17.0	75.0
0	130	15.0	6.5	20.0	80.0
1	140	20.0	8.0	23.0	85.0
2.38	154	26.9	10.1	27.1	91.9

1. Materials and methods

1.1. Raw materials

The characteristics of food waste were closely related to the local canteen at Zhejiang Gongshang University (Hangzhou, China), and the major components included rice (44%), noodles (16%), vegetables (23%), meat (6%), and tofu (11%), which was prepared as described in our previous study (Liu et al., 2018; Yin et al., 2019). Five materials were collected from the same vendor at Cui Yuan Market (Hangzhou, China). The food waste was crushed, mixed with a mangler, and stored frozen at −18 °C prior to use. Anaerobic granular sludge collected from an up-flow anaerobic sludge blanket at the Xihu Brewery (Hangzhou, China) was used as inoculum. The characteristics of the food waste and the inoculum sludge are listed in Table 1.

1.2. Hydrothermal treatment

The HT of food waste was described in our previous study (Liu et al., 2018). Briefly, air-tight pressure vessels with a volume of 80 mL were used for the HT. During the experiment, different hydrothermal conditions (including the initial substrate conditions) designed using Design Expert software (Version 8.0, Stat-Ease Inc., USA) were evaluated (Table 2). The reaction time was measured from the time at which the oil bath reached the set temperature.

1.3. Three-dimensional excitation and emission fluorescence analysis

The three-dimensional excitation and emission fluorescence with parallel factor analysis (3DEEM-PARAFAC) method was used (Liu et al., 2018; Yin et al., 2019). In brief, the exported emission excitation matrices (EEMs) were used to evaluate the dissolved organic matter of food waste. The 3DEEM spectra were generated over excitation wavelengths of 200–500 nm and emission wavelengths of 250–500 nm. Milli-Q water blanks were subtracted to remove the effect of Raman scattering (Murphy et al., 2010). Exported EEMs were then normalized by the Raman area. The PARAFAC method can decompose EEMs into individual components. PARAFAC models were computed, and the optimal component number was selected. The maximum fluorescence intensity (F_{max}, R.U.) represented the concentration scores of each component.

The EEM spectra from hydrothermally treated food waste were determined using the 2-component model and fluorescent components were defined as components one (C_1) and two (C_2) (Appendix A Fig. S1). C_1 had a fluorescence peak at an E_x/E_m wavelength of 285/354 nm, associated with soluble microbial products and defined as carbohydrates and proteinaceous material which could be biodegradable (Le and Stuckey, 2017; Li et al., 2018). With an E_x/E_m peak of 340/424 nm, C_2 was confirmed as melanoidins (Liu et al., 2018). The $F_{max}(C_2)/F_{max}(C_1)$ was employed as an indicator to demonstrate the hydrothermal modification of food waste.

1.4. *Response surface methodology*

A central-composite design (CCD) was used in this study and the $F_{max}(C_2)/F_{max}(C_1)$ value was measured as the response. The RSM experiments were carried out using Design Expert software (Version 8.0, Stat-Ease Inc., USA). A five-level-five-factor consisting of 50 experiments with 32 factorial and 10 axial points, and 8 replicates at the center point was required for this procedure. Table 2 shows the ranges and levels of the coded independent variables. The experimental data obtained from the design were analyzed following the methods described by Chen and Wu (2010). The results obtained by CCD were further analyzed using Design Expert software and a second-order polynomial model for $F_{max}(C_2)/F_{max}(C_1)$ fitted in terms of the coded parameters was suggested to analyze the experimental data. Analysis of variance (ANOVA) was used for statistical analysis and the regression coefficients were significant when the value of $P < 0.05$ (Montgomery, 2017). Three-dimensional (3D) surface plots and two-dimensional contours were adopted to visualize the individual and interactive effects of factors on the response. The RSM was employed to optimize the level of each factor for the best fit response.

1.5. *Acidogenic fermentation*

Fermentation experiments were carried out in identical amber wide-mouth bottles with a working volume of 500 mL. The fermented materials consisted of a mixture of 28 g hydrothermally treated food waste and 7 g inoculum (dry weight). The experimental design for the acidogenic fermentation of food waste (different hydrothermal conditions) is shown in Appendix A Table S1. In one group, the maximum VFA production from pretreated food waste under optimal HT (OHT) conditions was determined, and food waste without HT (non-HT) was used as a control. The VFA production from food waste treated under extreme HT conditions was also evaluated to compare with OHT conditions. The code=−2.38 HT conditions and code=2.38 HT conditions listed in Table 2, representing the situation of insufficient HT and melanoidins formation, were adopted. For the other group, the maximum VFA accumulation was also determined with different C/N (C/N = 27 and 40) under OHT conditions to verify if the higher initial C/N ratio could increase VFA production. The same initial C/N of food waste under OHT conditions were used for comparison.

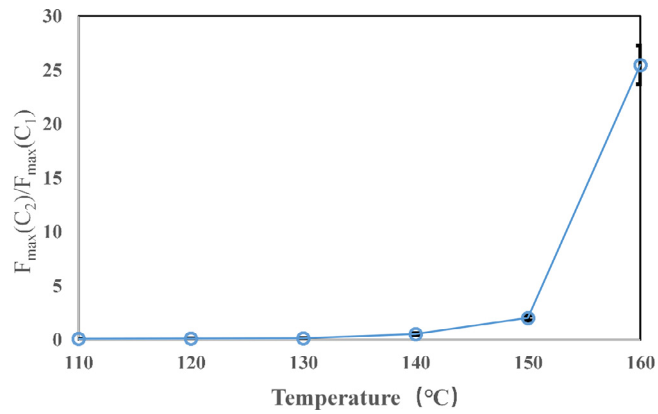

Fig. 1 – Effect of temperature on $F_{max}(C_2)/F_{max}(C_1)$ value of food waste.

ORP levels were stabilized between −100 mV and −200 mV under limited aeration condition. Other fermentation conditions were similar to our previous study (Yin et al., 2014). Each reactor was duplicated and run for 21 days.

1.6. *Analytical methods*

First, the samples taken from the reactors were diluted with deionized water (solid to water ratio of 1:10 W/V). Then, the mixtures were shaken for 40 min in a horizontal shaker at 30 ± 2 °C and centrifuged at 12,000 r/min for 5 min. Finally, the supernatants were filtered using a microfiltration filter (0.45 µm) for the measurement of VFAs (acetic, propionic, and butyric acids). VFAs were determined as previously described by high-performance liquid chromatography (Waters Corp., USA) using a C-18 column (Yin et al., 2019). Total solids (TS), volatile solids (VS), pH, total COD (TCOD) were all determined using standard methods (APHA, 1998).

2. Results and discussion

2.1. *Characterization of solubilized organics and melanoidins under different temperatures*

Significant color variations in hydrothermal food waste indicated the formation of melanoidins, depending on the reaction temperature. Therefore, the influence of temperature on $F_{max}(C_2)/F_{max}(C_1)$ was determined (Fig. 1). Food waste was treated under high pressure for 30 min at 110, 120, 130, 140, 150, and 160 °C. The $F_{max}(C_2)/F_{max}(C_1)$ was significantly elevated when the temperature exceeded 140 °C, indicating that the promotional effect of organic solubilization decreased with melanoidins formation. Accordingly, $F_{max}(C_2)/F_{max}(C_1)$ was applied as an indicator for the characterization of organic solubilization and melanoidins formation.

2.2. *Modeling for hydrothermal treatment based on influencing factors*

The experimental and predicted data for $F_{max}(C_2)/F_{max}(C_1)$ based on the CCD modeling technique with a five-level-five-

Table 3 – Fitting and ANOVA results for the experimental models.

Source	Sum of squares	df	Mean square	F-value	P-value Prob>F
Model	7.53	20	0.38	9.41	<0.0001
A-Temperature	1.43	1	1.43	35.8	<0.0001
B-Reaction time	0.37	1	0.37	9.23	0.0050
C-pH	3.00	1	3.00	74.9	<0.0001
D-C/N	0.026	1	0.026	0.64	0.4295
E-Moisture content	0.16	1	0.16	3.97	0.0559
AB	0.19	1	0.19	4.69	0.0387
AC	0.31	1	0.31	7.84	0.0090
BC	0.21	1	0.21	5.31	0.0285
A^2	0.28	1	0.28	7.10	0.0125
C^2	1.31	1	1.31	32.8	<0.0001
Residual	1.16	29	0.040		
Lack of fit	1.15	22	0.052	35.8	<0.0001
Pure error	0.010	7	1.460E-003		

R^2=89.64%; Adeq Precision=14.23.

factor design are summarized in Table S2. The ANOVA results for the response ($F_{max}(C_2)/F_{max}(C_1)$) showed an F-value of 9.41 with a low probability ($P < 0.0001$), indicating that the model was significant (Table 3). Consequently, a mathematical regression model (Eq. (1)) based on the coded factors with the determined coefficient for response was developed:

$$Y = 0.24 + 0.18A + 0.092B + 0.26C + 0.024D - 0.061E + 0.077AB + 0.0099AC + 0.082BC + 0.071A^2 + 0.15C^2 \quad (1)$$

where Y is the predicted response ($F_{max}(C_2)/F_{max}(C_1)$) and A, B, C, D, and E are temperature, reaction time, pH, C/N, and moisture content, respectively. According to previous studies, a negative sign in front of the term represents an antagonistic response to an increase, while positive sign demonstrates a synergistic response (Shuit et al., 2010). Therefore, we conclude that A, B, C, D, AB, AC, BC, A^2, and C^2 all had a favorable impact on the response value.

The P-value was used as a tool to identify the statistical significance of the model terms. A, B, C, AB, AC, BC, A^2, and C^2 were all significant model terms ($P < 0.05$), while D and E were not significant ($P > 0.05$) (Table 3). Table 3 also shows that a smaller P-value corresponded to a larger model coefficient term, indicating that temperature, reaction time, and pH can significantly impact the $F_{max}(C_2)/F_{max}(C_1)$ in the following order: pH > temperature > reaction time. Conversely, the influence of C/N and moisture content can be ignored. The coefficient of determination (R^2=0.8964) serves as a parameter to identify the model fit, indicating that 89.64% of the variability in the response can be explained by the model.

2.3. Analysis of response surface

The impact of temperature, reaction time, and pH on $F_{max}(C_2)/F_{max}(C_1)$ was explored. The response of a variable was determined against any two independent variables, while keeping other variables at their zero levels (Fig. 2). Fig. 2a and b shows the interactive effects of temperature and reaction time on the response when pH was maintained at 6.5. It can be seen that an abrupt increase in the response occurred with a mutual interaction between temperature and reaction time. When the temperature exceeded 125 °C, extending the reaction time could promote an increase in $F_{max}(C_2)/F_{max}(C_1)$. This may be due to the fact that a higher reaction temperature helps to speed up the MR between carbohydrates and proteins, causing the reduction of the biodegradable organic substrate to exceed the promotion of dissolved organics (Barber, 2016). The 3D response plot also revealed that temperature was a more important parameter than reaction time for $F_{max}(C_2)/F_{max}(C_1)$ according to their degree of variation. This result is consistent with Eq. (1), where the absolute value of the temperature coefficient (0.18) was approximately twice that of the reaction time (0.092). Fig. 2c and d shows the simultaneous effects of temperature and pH on the transformation of $F_{max}(C_2)/F_{max}(C_1)$. The influence of pH and temperature was not substantial below 134 °C and 6.9, respectively, because the conditions were not sufficient to promote melanoidins formation. Conversely, increasing temperature and pH created favorable conditions for melanoidins formation, resulting in a high response value. This is in accordance with our findings that the effect of pH on $F_{max}(C_2)/F_{max}(C_1)$ was more powerful compared to temperature (0.26 > 0.18). Furthermore, this observation is consistent with the report of Jiang et al. (2017), who found that an increase in pH led to an increase in the rate constant for the MR. Fig. 2e and f shows the 3D plot and the impact of pH and reaction time on the response value. At a lower pH, $F_{max}(C_2)/F_{max}(C_1)$ increased slightly with reaction time; however, as the initial pH increased, the $F_{max}(C_2)/F_{max}(C_1)$ had an obvious increase with reaction time. When pH exceeded 6.9, the reaction time resulted in a linear increase of $F_{max}(C_2)/F_{max}(C_1)$. Furthermore, the impact of pH on response declined from the high to the low temperature region. Therefore, a substantial change is expected to occur beyond a specific range for these three factors.

In addition to the significant terms, the 3D response surface graphs related to C/N and moisture content (insignificant terms) are shown in Fig. 3. The $F_{max}(C_2)/F_{max}(C_1)$ remained constant at approximately zero irrespective as to how these parameters changed. Furthermore, other 3D graphs (Appendix A Fig. S2) showed that while C/N had a small influence under low pH, temperature and reaction time, this influence was negligible at high pH, temperature and reaction time. This indicated that the variation of C/N merely changed the substrate type and did not accelerate the MR. A similar trend was also found for moisture content; hence, its effect on the $F_{max}(C_2)/F_{max}(C_1)$ ratio can be ignored (Appendix A Fig. S2). To summarize, pH, temperature, and reaction time are the key parameters to optimize during the HT process to reduce the MR and maintain hydrolysis efficiency.

2.4. Optimization of hydrothermal treatment conditions based on the model

The "Point Optimization" tool of the Design Expert software was employed to further predict the optimal conditions for $F_{max}(C_2)/F_{max}(C_1)$. The $F_{max}(C_2)/F_{max}(C_1)$ value obtained in the raw material was 0.05. If this value increases, then more melanoidins have formed (inhibition effect) compared to the

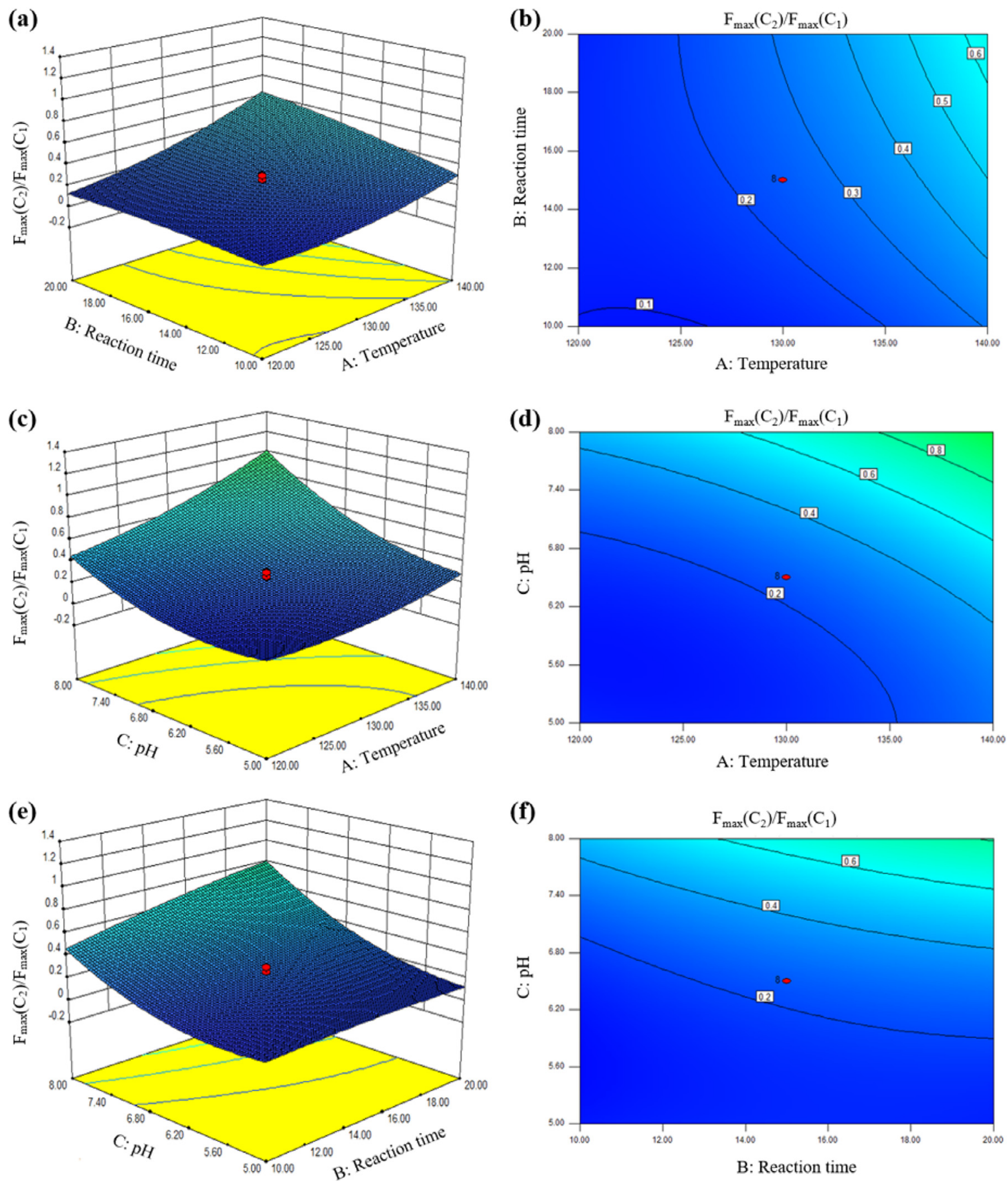

Fig. 2 – Three dimensional (3D) response surface and contour plots showing the impact of (a) and (b) temperature and reaction time, (c) and (d) pH and temperature, and (e) and (f) pH and reaction time when all other variables are set to zero.

solubilization of biodegradable organics. Therefore, optimized conditions correspond to a lower $F_{max}(C_2)/F_{max}(C_1)$ value. Based on our studies, there was no need to consider the impact of C/N and moisture content on the $F_{max}(C_2)/F_{max}(C_1)$. The optimum variables for best response were the temperature of 132.23 °C, reaction time of 26.88 min, and pH of 5.58. Under these conditions, the predicted $F_{max}(C_2)/F_{max}(C_1)$ (0.012) was approximately four times lower compared to the raw material, and the experimental value (0.011) corresponded well with this. With respect to C/N and moisture content, choosing the conditions which best fitted with acidogenic fermentation or HT could promote VFA production. In the hydrothermal reaction, water mainly plays two roles: one is to provide solution environment for chemical reaction of various substances; the other is to accelerate the speed of mass transfer and heat transfer. Ren et al. (2006) found when

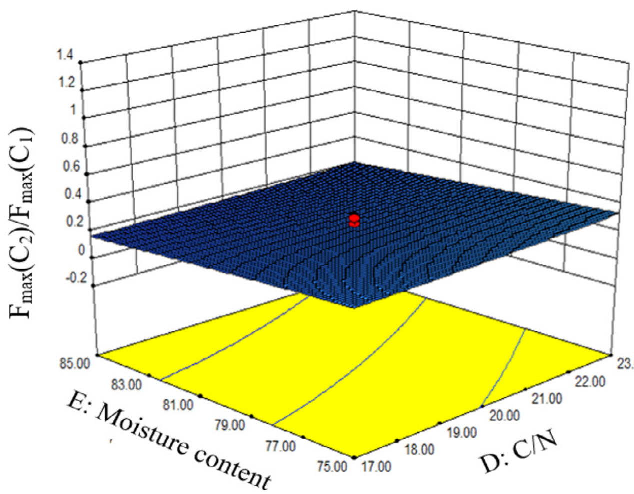

Fig. 3 – 3D response surface graph showing the impact of C/N and moisture content.

the moisture content is more than 85%, the heat transfer rate does not increase significantly. If the water content continues to increase, the heating rate will not increase significantly, and more energy for heat will be consumed. However, the influence of initial C/N on HT and VFA production requires further investigation.

2.5. VFA production under different hydrothermal conditions

To identify the impact of optimal HT conditions on VFA production, extreme conditions were used for comparison purposes. Fig. 4a shows that the highest VFA production was 376 ± 11 mg/g VS under OHT conditions, followed by 312 ± 3 mg/g VS in the high code (2.38) HT reactors, 308 ± 5 mg/g VS in the control reactors, and 297 ± 7 mg/g VS in the low code (−2.38) HT reactors. Accordingly, VFA production was promoted under optimal hydrothermal conditions (22.1%). This result implies that insufficient HT (the low code HT group) and melanoidins formation (high code HT group) cannot accelerate VFA production. Therefore, minimizing the occurrence of the MR can effectively optimize the hydrothermal parameters and improve acidogenic fermentation.

Adding high carbon content materials to adjust C/N ratio of the feedstock can improve the fermentation performance and VFA accumulation of solid waste (Yen and Brune, 2007). Hence, rice was used as an added carbon source to improve the C/N ratio, and VFA production under different initial C/N conditions was investigated. Fig. 4b shows that the maximum VFA yield increased with the initial C/N ratio. This illustrates that high C/N is beneficial to VFA accumulation, attributed to increased butyric acid production (Appendix A Fig. S3). This is consistent with our previous result (Yin et al., 2016), which was that butyric acid was a major contributor to VFAs produced from carbohydrates. Fig. 4b also shows that VFA production from food waste with a high C/N was significantly improved by HT. Following treatment under optimal hydrothermal conditions, the maximum VFA production of C/N = 40 group (458 ± 6 mg/g VS) and C/N = 27 group (405 ± 1 mg/g VS) was 13.1% and 28.6%, respectively, higher than untreated groups, indicating that HT could further enhance VFA production from food waste with higher C/N ratio. Consequently, elevating the C/N of food waste could serve as an effective way to optimize HT and accelerate VFA production.

3. Conclusions

In this study, hydrolysis effect and melanoidins formation ($F_{max}(C_2)/F_{max}(C_1)$) were successfully optimized using RSM, and a quadratic model was suggested to predict the response. The ANOVA result indicated that pH, temperature and reaction time were important factors that significantly impacted the response, while C/N and moisture content had a limited impact. The best fit conditions derived via RSM for optimal response were temperature of 132.23 °C, reaction time of 26.88 min, and pH of 5.58. Under these conditions, the predicted $F_{max}(C_2)/F_{max}(C_1)$ value was four times lower than the initial value, in good agreement with the experimental result. A 22.1% increase in VFA production from food waste was

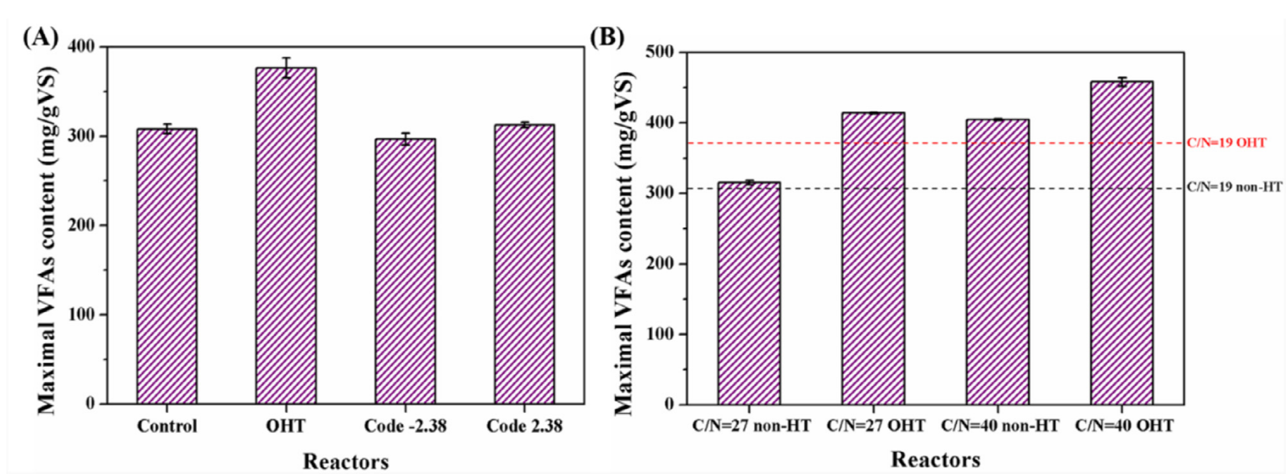

Fig. 4 – Maximal VFAs yields under different HT conditions and different initial C/N conditions.

achieved following optimal HT, and a further elevated VFA production could be obtained by using a higher initial C/N. The HT and acidogenic fermentation processes were effectively optimized through minimizing the MR.

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Appendix A Supplementary data

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.jes.2020.09.032.

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