Archives of Environmental Protection
Vol. 48 no. 3 pp. 70–79

PL ISSN 2083-4772
DOI 10.24425/aep.2022.142691

Application of response surface methodology to improve methane production from jerusalem artichoke straw

Yan Meng, Yi Li, Laisheng Chen, Rui Han*

Qinghai Key Laboratory of Vegetable Genetics and Physiology,
Academy of Agriculture and Forestry Sciences, Qinghai University, Xining, Qinghai 810016, China

*Corresponding author's e-mail: hanrui11473@163.com

Keywords: Jerusalem artichoke straw, anaerobic digestion, response surface methodology, methane production

Abstract: Anaerobic digestion (AD) converts organic matter and biomass waste into biogas, making it an environmentally friendly technology to improve energy resources for a wide range of applications. Jerusalem artichoke straw (JAS) has an enriched content of cellulose and exhibits a high potential for methane production. AD-based production of methane can effectively utilize waste JAS. This study investigated the AD performance of JAS to explore the enhancement of methane yields by employing a Box-Behnken experimental design (BBD) of response surface methodology (RSM). The overall goal was to identify the optimal levels of pretreatment factors, including HCl concentration, pretreatment time, and pretreatment temperature, for producing optimal biomethane yields from JAS. The highest value of methane production achieved was 256.33 mL g^{-1}VS by using an optimal concentration of HCl as 0.25 M, a pretreatment time of 10 h, and a pretreatment temperature of 25°C. These results inform the future application of JAS in enhanced methane production.

Introduction

The global demand for energy has increased with the development of societies and economies, yet traditional fossil energy falls far short of meeting contemporary human requirements (Kozłowski et al. 2019). Thus, exploration of clean and renewable energy sources is urgently needed. Biomethane is one of the most important renewable clean energy fuels that are produced from biomass wastes and that are derived from anaerobic digestion (AD) processes (Wu et al. 2021, Pokój et al. 2014). The lignocellulosic material used as feedstock for such wastes are low cost and abundantly available, since they are widely found in various crop straws (Tian et al. 2021). Thus, the extraction of clean bioenergy from lignocellulosic biomass through AD is a feasible method with high potential of application.

Jerusalem artichoke (JA) is an important alternative energy crop because it is tolerant to several environmental stresses, including drought, salinity, plant diseases, and pests, and thereby sustaining its strong growth in marginal ecosystems to reach high biomass yields (Long et al. 2016). JA has been widely cultivated following introduction into China in the 17th century. The Qinghai-Tibet Plateau is one of the earliest areas of China to conduct large-scale planting and processing of JA (Yang et al. 2019). Indeed, the annual JA plantation area within the Qinghai Province is about 1,400 ha and plants can grow to a height of 2–4 m, achieving Jerusalem artichoke straw (JAS) yields of over 70 tons per hectare based on fresh biomass weight. Although JA tubers have been investigated for their potential in inulin extraction (Li et al. 2015) and biofuel production (Pfariso et al. 2021), fewer studies have been focused on JAS. Only a small component of JAS is used as feed, while most of it is burned or discarded, leading to resource wastes. The discharge of the waste can constitute environmental problems. In contrast, if these wastes can be used as the source of clean energy, the local environment can be protected at the same time (Oyekanmi et al. 2021a, Oyekanmi et al. 2021b). In addition, the cellulose yield of JAS is over twice that of corn straw, rice straw, bagasse, and wheat straw, thus rendering it a high-potential raw material for biofuel research (Gunnarsson et al. 2014, Ciccoli et al. 2018). Nevertheless, JAS consists of rigid cellulose structures (like other lignocellulosic resources), in combination with amorphous hemicellulose and lignin cross-linked structures (Gnansounou and Dauriat 2010). This inherent chemical complexity leads to resistance to enzymatic digestion during the generation of fermentable sugars. Thus, pretreatment is necessary to convert the complex lignocellulosic structures into more enzymatically digestible forms (Kim et al. 2013).

Common pretreatment methods include physical, chemical, and biological methods. Physical methods include grinding, extrusion, and cavitation, all of which can effectively

reduce the crystallinity of lignocelluloses. However, these methods are difficult to widely implement due to their high energy consumption. Biological pretreatments are also difficult to implement due to the need for long pretreatment cycles, sugar consumption, or the need for efficient microbial agents (Hossain et al. 2019). Thus, chemical pretreatment is comparatively ideal, because it can effectively destroy lignocellulosic structures and internal chemical bonds, while also requiring much lower energy consumption (Paudel et al. 2017). The widely used chemical pretreatment agents include hydrochloric acid, sulphuric acid, phosphoric acid, calcium hydroxide, potassium hydroxide, and sodium hydroxide (Zhang et al. 2018b, Khalid et al. 2019). Suitable reagents are often selected at appropriate concentrations based on substrate characteristics. Several acid-base pretreatments have been implemented in our previous studies, leading to the observation that low-concentrations of HCl (0.2–0.4 M) are most suitable for JAS pretreatment. In addition, pretreatment time and temperature also affect pretreatment outcomes, necessitating further optimization of JAS pretreatment conditions.

Response surface methodology (RSM) is an effective and reliable method to model the effects of several variables and is highly applicable to AD studies, along with many other fields (Cai et al. 2021, Khalid et al. 2019). RSM comprises a set of mathematical methods that can effectively describe the relationships of numerous individual variables with one or more response values (Adeleke et al. 2017). Consequently, the objectives of this study were to maximize the methane production from JAS using RSM to optimize HCl concentrations, pretreatment time, and treatment temperature.

Materials and methods

Substrates and inoculum

JAS were obtained from the Horticultural Innovation Base of the Academy of Agriculture and Forestry Sciences at Qinghai University. JAS were sectioned into small 1–2 cm pieces using a shredding machine. Inoculated sludge was obtained from the agricultural biogas digester of Qinghai Zhiyuan Characteristic Agriculture Co., Ltd. that used cow dung as the raw material and which was operating stably. To reduce endogenous gas production, inoculated sludge was anaerobically preincubated for seven days. The basic characteristics of the inoculated sludge and JAS are shown in Table 1.

Experimental procedure

A three-level-three-factor Box-Behnken design (BBD) was used to evaluate the effects of three independent variables in addition to their interactive effects on the response variable. The variables included HCl concentration (0.2, 0.3, and 0.4 M), pretreatment temperature (20, 25, and 30°C), and pretreatment time (6, 12, and 18 h), which were coded as X_1, X_2, and X_3, respectively. The experimental design was established with the Design Expert program (Version 12.0.0 Stat-Ease Inc., Minneapolis, MN, USA), and the real operating variable values are summarized in Table 2.

According to the pretreatment conditions set in Table 2, a mass of 10 g of raw JAS was added to the respective solutions for pretreatment. The moisture content of each pretreatment vessel was 70%. After the pretreatment, the straws for anaerobic digestion were rinsed with distilled water to reach the neutral pH value. After that, all the pretreated samples were dried at 40°C for 12 h for the subsequent experiment.

Anaerobic digestion tests were performed using an automatic methane potential test system (AMPTA, MultiTalent 203) under mesophilic (35 ± 0.5°C) conditions. Trials were conducted in 500 mL reactors; the VS based inoculum to substrate ratio was set at 2:1, and the organic loading was 8.05 gVS L^{-1}. To eliminate background gas production derived from the inoculum, the reactors containing only inoculum (i.e., without substrates) were included as blanks. A positive control which consisted of pure cellulose was established to test the activity of the inoculum and to evaluate the experimental protocol. Raw JAS was also used as another experimental control, to compare the difference of methane production before and after pretreatment. All reactors were prepared in triplicate to facilitate statistical analysis.

Analytical methods

The TS and VS contents of prepared samples were measured in triplicate by using standard methods (APHA. 2005). Sample pH was measured with a pH meter (pHS-2F, Shanghai INESA Scientific Instrument Co., Ltd, Shanghai, China). Total alkalinity (TA) was determined with an automatic potentiometric Titroline instrument (ZDJ-4A, Shanghai INESA Scientific Instrument Co., Ltd, Shanghai, China). Total ammonia nitrogen (TAN) and total volatile fatty acid (TVFA) concentrations were determined by the application of colorimetry using a SPECORD 210 UV-Vis spectrophotometer (Jena Analytical Instruments AG, Germany). Hemicellulose, cellulose, and lignin contents were determined by using neutral detergent fiber (NDF), acid detergent fiber, and lignin (ADF/ADL) analyses, as described by Van Sowest et al (1991). Structural changes of JAS were evaluated with Fourier transform infrared spectroscopy (FTIR-8400S, SHIMADZU). Scanning was conducted between 4,000 and 400 cm^{-1} at a resolution of 4 cm^{-1}, with 40 scans being recorded.

Cumulative methane production and kinetic modeling

The cumulative methane production for each run was calculated by subtracting the cumulative methane production

Table 1. Substrate and inoculum characteristics

Component	JAS	Inoculum
TS (%)[a]	95.37 ± 0.13	3.74 ± 0.21
VS (%)[b]	89.67 ± 0.81	1.62 ± 0.06
Cellulose (%)[b]	46.36 ± 1.27	n/a
Hemicellulose (%)[b]	7.68 ± 1.33	n/a
Lignin (%)[b]	12.03 ± 0.36	n/a
pH	n/a	7.59 ± 0.02
TVFA (mg/L)	n/a	408.33 ± 18.38
TA (mg $CaCO_3$/L)	n/a	6,994.49 ± 162.43
TAN (mg/L)	n/a	461.10 ± 23.06

TS: total solid; VS:volatile solid; TVFA: total volatile fatty acid; TA: total alkalinity; TAN: total ammonia nitrogen;
[a] based on fresh matter ; [b] based on TS; n/a, not applicable

of the blank and dividing by the amount of organic matter (based on VS) added to each reactor. The specific calculation formula (Eq. (1)) is as follows:

$$CMP = \frac{V_S - V_B \frac{m_{IS}}{m_{IB}}}{m_{VS}} \quad (1)$$

where:
CMP refers to the cumulative methane production (mL g^{-1}VS);
V_S, the cumulative ultimate methane production of the substrate (i.e., material and inoculum), mL;
V_B, the cumulative ultimate methane production of the inoculum (i.e., blank), mL;
m_{IS}, the total mass of the inoculum in the experimental sample, g;
m_{IB}, the total mass of inoculum in blank group, g;
m_{VS}, the mass of organic matter (based on VS) in the experimental bottle, g.

Modified Gompertz models were used to evaluate JAS digestion performance, as shown in Eq (2):

$$P = P_{\max} \cdot \exp\left\{-\exp\left(\frac{R_{\max} \cdot e}{P_{\max}} \cdot (\lambda - t) + 1\right)\right\} \quad (2)$$

In this equation, P is the cumulative methane production (mL g^{-1}VS) for time t; P_{max} is the maximum methane potential (mL g^{-1}VS) at the end of digestion; R_{max} is the maximum methane production rate (mL g^{-1}VS d^{-1}); λ is the lag phase (d); t is time (d); and e is exp (1), i.e., 2.71828.

Results and discussion

Methane production among different treatments

Among the 17 runs with test data based on RSM modeling, the lowest observed cumulative methane production was 192.70 mL g^{-1}VS, while the highest was 264.88 mL g^{-1}VS (Table 2). Further, the actual methane values were closer to the predicted values for all 17 runs, implying that RSM is a suitable method for optimizing these factors during AD.

The cellulose control showed a methane production of 370.29 mL g^{-1}VS (Figure 1a), 89.23% of the theoretical methane yield of cellulose (415 mL g^{-1}VS) which is the same range as those reported in other studies (Nges et al. 2016, Kreuger et al. 2011). It was therefore assumed that the inoculum had an ideal activity and was suitable for the batch experiment. As illustrated in Figure 1, the methane production trend of pretreated JAS and raw JAS both increased at first and gradually plateaued; the cumulative methane production of JAS after pretreatment was significantly higher than that of the raw JAS. This indicated that HCl pretreatment can effectively improve the methane production of JAS. Consistently, a plethora of studies have shown that methane production of biomass waste can be enhanced after applying hydrochloric acid pretreatment, such as dairy cow manure, microalgae, and *Miscanthus lutarioriparius* (Passos et al. 2017, Passos et al. 2016, Nges et al. 2016). This phenomenon can be explained by the fact that acid pretreatments promote organic solubilization and increase the surface area available for enzymatic accessibility (Song et al. 2014).

Table 2. Response surface design and results

Runs		Process variables					Response	
		Coded		Actual				
	X_1	X_2	X_3	HCl	Time	Temperature	Actual methane production (mL g^{-1}VS)	Predicted methane production (mL g^{-1}VS)
Run 1	−1	−1	0	0.2	6	25	241.48	248.36
Run 2	1	−1	0	0.4	6	25	211.90	212.51
Run 3	−1	1	0	0.2	18	25	209.21	208.59
Run 4	1	1	0	0.4	18	25	227.18	220.30
Run 5	−1	0	−1	0.2	12	20	227.21	226.77
Run 6	1	0	−1	0.4	12	20	202.22	208.04
Run 7	−1	0	1	0.2	12	30	235.07	229.24
Run 8	1	0	1	0.4	12	30	223.39	223.83
Run 9	0	−1	−1	0.3	6	20	218.18	211.74
Run 10	0	1	−1	0.3	18	20	192.70	193.76
Run 11	0	−1	1	0.3	6	30	219.95	218.89
Run 12	0	1	1	0.3	18	30	198.43	204.87
Run 13	0	0	0	0.3	12	25	249.40	256.49
Run 14	0	0	0	0.3	12	25	257.40	256.49
Run 15	0	0	0	0.3	12	25	249.50	256.49
Run 16	0	0	0	0.3	12	25	261.28	256.49
Run 17	0	0	0	0.3	12	25	264.88	256.49

X_1: HCl concentration; X_2: pretreatment time; and X_3: pretreatment temperature.

Biochemical methane potential tests, carried out with fresh, air-dried and ensiled above-ground biomass of Jerusalem Artichoke by Ciccoli et al. (2018), showed that air-dried biomass of Jerusalem Artichoke yielded the highest methane production (133.73 mL g^{-1}VS), which was on the similar level to the raw JAS in this study. In addition, the highest methane production (264.88 mL g^{-1}VS) was achieved after pretreatment at 25°C with 0.3M HCl for 12 h in the current study, which was higher than the same genus Compositae HCl pretreated sunflower stalks (233 mL g^{-1}VS) (Monlau et al. 2012). In comparison with other lignocelluloses biomass, the methane yields of tobacco pretreated with 5% hydrochloric acid for 24 h was 203.30 mL g^{-1}VS (Zhang et al. 2019). The methane yields of switchgrass stalks was 112.6 mL g^{-1}VS with 5%HCl for 12 h at 25°C (Shen et al. 2019). The cumulative methane production of corn straw was 163.4 mL g^{-1}VS for 2%HCl, 25°C, 7 days (Song et al. 2014). For the above lignocellulosic materials, the optimal conditions to reach the highest methane production after hydrochloric acid pretreatment varied, mostly owing to the inherent characteristics of different lignocellulosic materials. Considering the influence of various factors, such as time, temperature and concentration of reagent, the pretreatment conditions applied in this study are more economical and suitable, and can facilitate higher methane production. Hence, we conclude that it is a suitable pretreatment method for JAS.

AD modeling for methane production

The actual methane production of JAS were set as the response values. A three-level factorial arrangement with three factors was used at each of three levels using the Box-Behnken method to investigate the effects of the independent variables HCl concentration (X_1), pretreatment time (X_2), and pretreatment temperature (X_3) on the actual methane production (Table 3). The experimental data were analyzed with RSM using a commercial statistical package and adjusted to a quadratic model. A mathematical regression model was consequently obtained as follows:

$$\text{Methane production (mL g}^{-1}\text{VS)} = 256.49 - 6.04X_1 - 8X_2 + 4.57X_3 + 11.89X_1X_2 + 3.33X_1X_3 + 0.99X_2X_3 - 9.7X_1^2 - 24.35X_2^2 - 24.82X_3^2$$

The statistical significance of the terms in the second-order model was investigated with analysis of variance (ANOVA). The adequacy and significance of the mathematical regression model are shown in Table 3. The model F value of 13.39 indicated that the quadratic model is statistically significantly fit to the methane production data. The lack-of-fit F value of 0.3008 also revealed an insignificant lack of fitting, confirming the good fitness of the model. Moreover, the R^2 of the fitness was 0.9451, indicating that 94.51% of the observed variability in methane production could be explained by the model and that an ideal fit was present between the observed and predicted values (Zhao et al. 2017). An adequate precision value of 10.2904 further indicated that the model could be used to navigate design space.

Statistical significance tests were also conducted for the regression coefficients. Larger F values for the regression parameter coincides with smaller p values, indicating that the parameter had a greater impact on methane production. Among the three operating parameters, pretreatment time (F value = 8.10, $p = 0.0248$) had the largest effect on methane production, followed by HCl concentration (F value = 4.61, $p = 0.0689$), and then temperature (F value = 2.64, $p = 0.1483$) (Table 3).

Optimization of operating parameters for methane production

Experimental results were visualized in three-dimensional response surface plots and corresponding contour plots that showed the simultaneous effects of two independent factors on methane production, with one variable maintained at its central level. The effects of HCl concentration and pretreatment time on JAS methane production at a pretreatment temperature of 25°C are shown in Figure 2(a). JAS methane production significantly increased with increasing HCl concentrations from 0.2 to 0.3 M and with pretreatment time

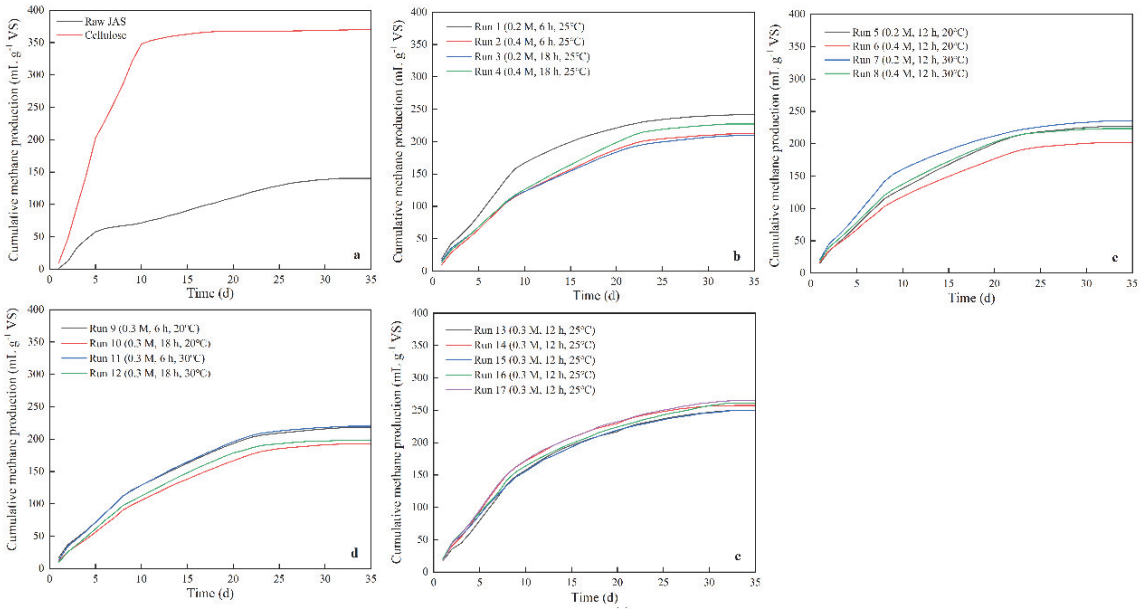

Fig. 1. Comparison of methane productionfrom cellulose (as positive control), raw JAS and pretreated JAS

increasing from 6 to 12 h. JAS methane production then decreased when HCl concentration and pretreatment time increased beyond the aforementioned values. For a fixed duration of pretreatment time, methane production of JAS showed an upward trend with higher HCl concentration and temperature at first, yet gradually decreased (Figure 2 (b)). The higher acid concentration caused a successive decrease in methane production, which could be attributable to the inhibitory effect on the formation of phenolic compounds, furfural, and 5-hydroxymethyl furfural (Nowicka et al. 2021). Meanwhile, Günerhan et al. (2020) investigated the optimization of pretreatment processes of fruit and vegetable harvesting wastes, revealing that a higher HCl concentration would decrease the concentration of soluble sugars that can be converted directly into methane. As seen in Figure 2 (c), for a constant HCl concentration, the methane production of JAS increased at first and then decreased with the elapse of pretreatment time and increasing temperature. This could be attributable to the low pretreatment temperature or the swift duration of pretreatment, which could not effectively

Table 3. Regression model variance analysis of JAS cumulative methane production

Source	Sums of squares	df	Mean square	F value	*p*	
Model	7612.57	9	845.84	13.39	0.0012	Significant
X_1-HCl	291.37	1	291.37	4.61	0.0689	
X_2-Time	511.68	1	511.68	8.10	0.0248	
X_3-Temperature	166.71	1	166.71	2.64	0.1483	
X_1X_2	565.49	1	565.49	8.95	0.0202	
X_1X_3	44.36	1	44.36	0.7021	0.4297	
X_2X_3	3.92	1	3.92	0.0621	0.8104	
X_1^2	396.05	1	396.05	6.27	0.0408	
X_2^2	2,497.23	1	2,497.23	39.53	0.0004	
X_3^2	2,594.55	1	2,594.55	41.07	0.0004	
Residual	442.21	7	63.17			
Lack of Fit	248.91	3	82.97	1.72	0.3008	Not significant
Pure Error	193.29	4	48.32			
Cor total	8,054.78	16				
R^2 = 0.9451; Adeq precision = 10.2904						

X_1: HCl concentration, X_2: pretreatment time, and X_3: pretreatment temperature. $p < 0.05$ indicates model terms are significant.

Table 4. Results obtained from a modified Gompertz model of JAS digestion

Sample	P_{max} (mL g^{-1} VS)	Actual methane production (mL g^{-1} VS)	R_{max} (mL g^{-1} VS d^{-1})	λ(d)	R^2
Raw	158.36	140.27	7.04	5.03	0.992
1	239.58	241.48	16.32	−0.379	0.996
2	216.17	211.90	11.62	−0.399	0.996
3	213.83	209.21	10.78	−1.214	0.996
4	234.91	227.18	11.71	−0.615	0.997
5	232.82	227.21	11.92	−0.945	0.996
6	208.15	202.22	10.27	−1.404	0.996
7	233.25	235.07	14.70	−1.083	0.994
8	227.89	223.39	12.42	−1.164	0.997
9	222.85	218.18	11.48	−1.124	0.996
10	199.32	192.70	9.84	−0.547	0.997
11	224.90	219.95	11.87	−0.820	0.996
12	203.80	198.43	10.96	−0.300	0.996
13	247.32	249.40	15.13	−0.309	0.994
14	258.56	257.40	16.60	−0.519	0.995
15	249.22	249.50	13.84	−1.242	0.995
16	259.74	261.28	13.93	−1.477	0.992
17	267.52	264.88	17.02	−0.494	0.996

decompose the lignocellulose structure of JAS. Conversely, a higher temperature and longer time of pretreatment lead to excessive loss of organic matter. Therefore, an appropriate treatment time and temperature are particularly important to improve the production of methane. Wang et al. (2015) treated rice straw with HCl, and found that increasing temperature and increasing pretreatment time dramatically enhanced glucose conversion. The washing of JAS after pretreatment would lead to the loss of organic matter (such as sugars), hence resulting in the reduction of methane production. In summary, excessive HCl concentration, lengthy pretreatment time, and high temperatures would render the organic matters in JAS excessively destroyed or decomposed, thereby leading to decreased methane output (Kim et al. 2018). These results confirmed that the use of proper pretreatment conditions can maximize the methane output of JAS digestion.

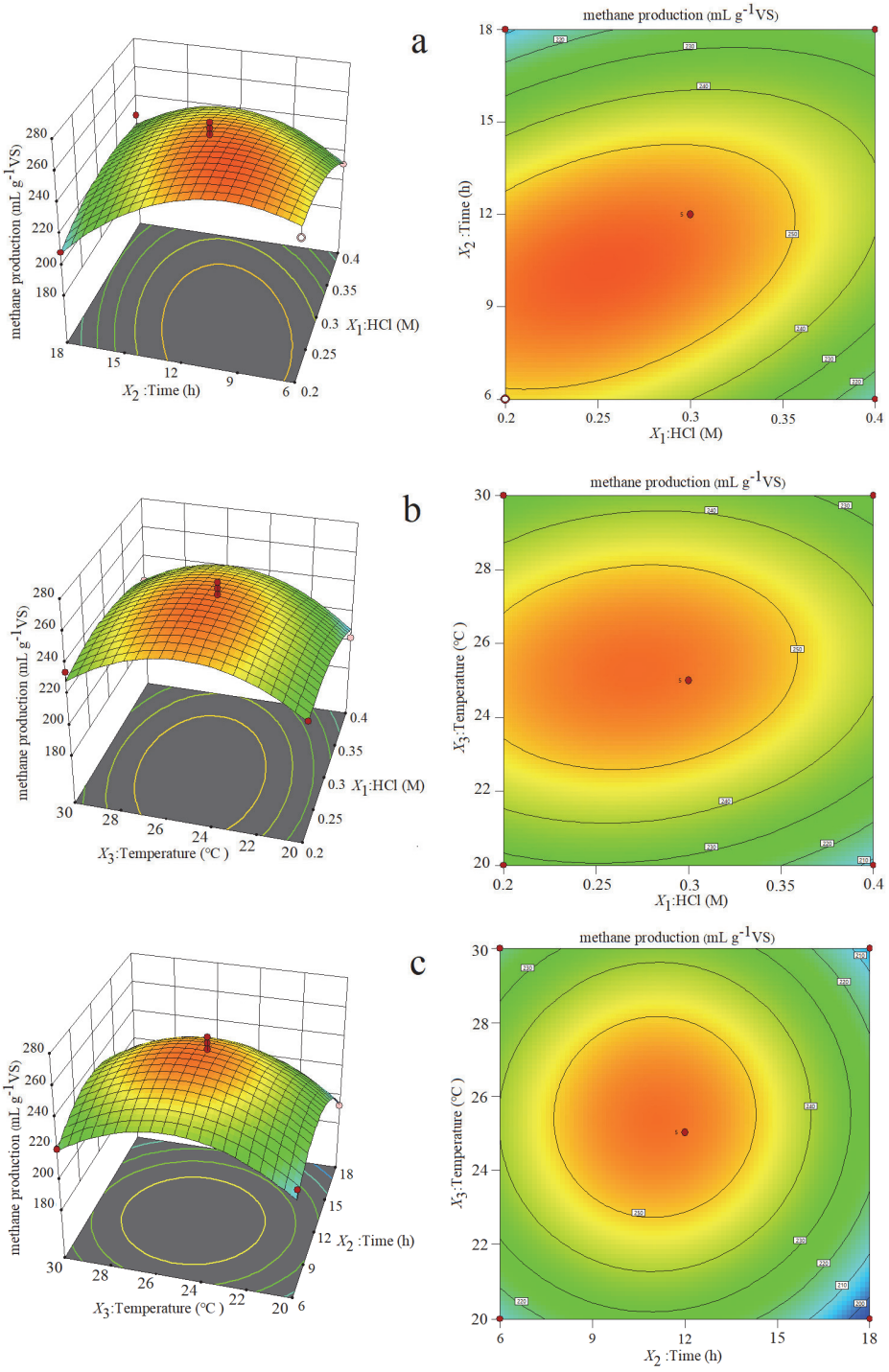

Fig. 2. 3D response surface methodology (RSM) graphs and 2D contour plots reveal the interaction of three factors on methane production: HCl concentration, pretreatment time, and temperature
(a) Interactive effects of HCl concentration and pretreatment time on methane production at a pretreatment temperature of 25°C.
(b) Interactive effects of HCl concentration and pretreatment temperature on methane production for a pretreatment time of 12 h.
(c) Interactive effects of pretreatment time and pretreatment temperature on methane production using an HCl concentration of 0.3 M.

Kinetic analysis

The estimated kinetic parameters using Gompertz modeling are shown in Table 4. The maximum actual methane production of the experimental group was 264.88 mL g^{-1}VS, reaching a 1.89-fold increase over the yields from raw JAS. These results suggest that pretreatment with HCl and employing RSM can greatly enhance the methane production from JAS. The correlation coefficients (R^2) of the model ranged from 0.992 to 0.997, indicating that the model could be appropriately used to describe JAS digestion performance (Cai et al. 2018). The hydrolysis and methanogenesis stages are critical steps during AD. The R_{max} (methanogenesis rate) of runs 1, 13, 14, and 17 were higher than 15 mL g^{-1}VS d^{-1}, implying a high methanogenesis rate. P_{max} (the maximum methane potential) values were substantially higher than those using raw JAS. The lag time (λ) can indirectly reflect digestion efficiency (Kafle et al. 2013). Even though λ is not directly related to digestion performance, a high λ value indicates a longer digestion period, which is unfavorable for large-scale digestion projects. No lag time was observed compared to raw JAS digestion, indicating that pretreatment can shorten the anaerobic digestion cycle and improve efficiency.

Characterization of the effluent and system stability

The final effluent characteristics were measured, including pH, total ammonia nitrogen (TAN), total volatile fatty acids (TVFAs), total alkalinity (TA), and the TVFA/TA ratio, after 35 days of AD (Table 5). AD stability is critical for maintaining sustainable performance of digesters and is reflected by the above parameters. TAN values ranged from 384.91 to 480.60 mg/L, which were well within the range of stable anaerobic digestion system operations (<1,500 mg/L) (Liu et al. 2018). TAN concentrations also affect pH and TA. The pH values observed in the effluent (6.91–7.45, Table 5) fell within the preferred range for methanogenic activity (pH 6.2–8) (Zhao et al. 2017). TVFA/TA ratio is also used to evaluate digestor stability, wherein digestion stably operates at ratios < 0.4 (Zhang et al. 2018a). TVFA/TA values in this study were all lower than 0.4. Thus, the effluent characteristics indicated stable operation for all 17 runs.

Prediction and verification of optimal conditions

The AD parameters were optimized based on the quadratic model using the optimization module of the Design Expert software program. Specifically, response surface optimization was conducted for HCl concentration, pretreatment time, and pretreatment temperatures. Optimal parameter values included 0.25 M HCl, 10.33 h of pretreatment time, and a pretreatment temperature of 25.28°C, which led to the predicted value for cumulative methane production of 259.49 mL g^{-1}VS. Factors were then adjusted based on actual production logistics as follows: HCl concentration of 0.25 M, pretreatment time of 10 h, and pretreatment temperature of 25°C. Three parallel verification digestions were conducted under these optimized conditions, leading to JAS methane production of 256.33 mL g^{-1}VS. The error between the experimental and predicted values was less than 0.5%. Thus, the accuracy and feasibility of the response surface optimization were confirmed.

Fourier transform infrared analyses

The FTIR spectra of JAS before and after HCl pretreatment are demonstrated in Figure 3. The sharp peak at 2,917 cm^{-1} could be attributed to C-H stretching in the CH_2 and CH_3 groups of celluloses, hemicelluloses, and lignin (Gabriel et al. 2020). The absorption peaks of samples after pretreatment weakened compared with raw JAS, indicating that components of carbon chains were destroyed by

Table 5. Final effluent characteristics

Runs	pH	TVFA (mg/L)	TA (mg $CaCO_3$/L)	TAN (mg/L)	TVFA/TA
1	7.45±0.02	591.22±56.05	3,253.25±149.65	384.91±19.25	0.182
2	7.22±0.04	853.93±38.43	3,003.00±78.14	419.10±23.47	0.284
3	7.10±0.06	867.23±39.43	2,752.75±41.66	465.60±26.54	0.315
4	7.27±0.05	1,060.10±47.71	3,003.00±126.62	480.60±27.88	0.353
5	7.07±0.13	1,033.50±76.58	2,752.75±62.33	461.10±27.2	0.375
6	6.92±0.11	966.99±53.52	2,502.50±115.13	465.60±20.02	0.386
7	7.23±0.07	747.52±63.64	3,003.00±41.66	465.60±10.33	0.249
8	7.25±0.03	1073.41±58.30	3,003.00±62.33	461.10±9.82	0.357
9	7.00±0.06	717.59±42.22	2,877.88±20.67	426.00±20.33	0.249
10	6.94±0.15	1,066.76±78.04	2,877.88±41.67	426.30±10.30	0.371
11	6.91±0.09	947.04±62.62	2,877.88±62.33	465.60±18.62	0.329
12	6.95±0.02	1,060.10±47.71	2,877.88±62.33	444.30±18.31	0.368
13	6.97±0.04	887.18±49.93	2,877.88±41.67	461.10±12.78	0.308
14	7.03±0.09	554.64±64.96	2,877.88±20.67	426.30±13.56	0.193
15	7.11±0.02	464.86±22.92	2,877.88±0	452.15±13.83	0.162
16	7.01±0.03	431.60±39.42	2,877.88±20.67	452.15±20.53	0.150
17	6.99±0.05	491.46±42.16	2,877.88±0	452.15±21.30	0.171

TVFA: total volatile fatty acid; TA:total alkalinity; TAN: total ammonia nitrogen

pretreatment. The absorption at 1,700–1,740 cm^{-1} belonged to the non-conjugated carbonyl group which was probably originated from acetyl or uronic-ester of thexylan (Li et al. 2016, Hassan et al. 2020) and its intensity decreased after pretreatment, indicating the acetyl or uronic-ester groups were partly removed during the pretreatment. The absorption of conjugated carbonyl groups at 1,643 cm^{-1} primarily correspond to phenolic acids that possess an unsaturated double bond and a carbonyl group (Li et al. 2016). Decreasing peak intensity at 1,643 cm^{-1} indicated that the chemical bond was broken during treatment. Absorption at 1,510 cm^{-1} corresponds to C=C aromatic skeletal vibration (Hassan et al. 2020) and a corresponding characteristic peak at 1,506 cm^{-1} weakened, indicating that the lignin structure was destroyed and lignin content decreased. A peak at 896 cm^{-1} was associated with the characteristic absorption of β-glycosidic bonds between glucose units (Oh et al. 2005) and its intensity increased after treatment, suggesting increased cellulosic content in the pretreated samples.

Conclusions

This research has conducted the evaluation of anaerobic digestion performance of pretreated JAS. The results revealed that BBD of RSM was an appropriate method for optimizing the pretreatment conditions for the anaerobic digestion of JAS. The results of the analysis of variance showed that pretreatment time exhibited the most predominant effects on methane production, followed by HCl concentration, and pretreatment temperature. Specifically, RSM modeling indicated that an HCl concentration of 0.25 M, a pretreatment time of 10 h, and a pretreatment temperature of 25°C were optimal for obtaining a maximal methane production of 256.33 mL g^{-1}VS. Interestingly, the method led to a shortened fermentation cycle in addition to significant improvements in methane production. Thus, these optimization results could be of interest to commercial biogas plants for shortening residence times without jeopardizing process performance, thereby significantly improving profits.

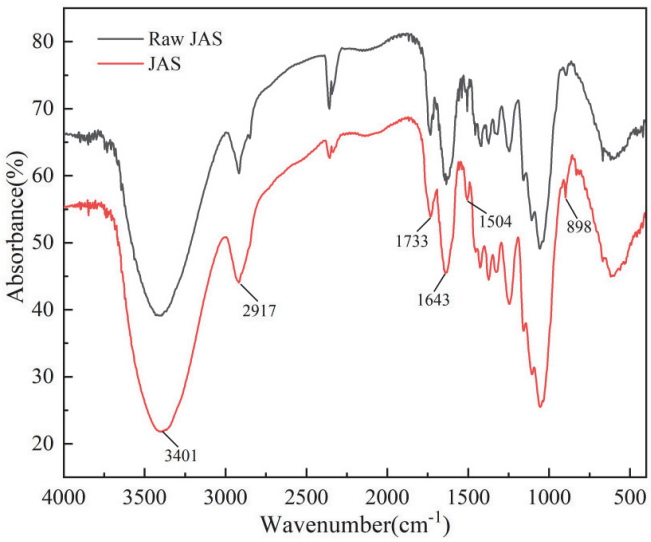

Fig. 3. FTIR spectra of raw JAS and HCL pretreated JAS

Acknowledgments

We gratefully acknowledge financial support from the Natural Science Foundation of Qinghai Province, China (No.2021-ZJ-921).

References

Adeleke, A.O., Latiff, A.A.A., Al-Gheethi, A.A. & Daud, Z. (2017). Optimization of operating parameters of novel composite adsorbent for organic pollutants removal from POME using response surface methodology,*Chemosphere*, 174, pp. 232–242, DOI: 10.1016/j.chemosphere.2017.01.110

APHA. (2005). Standard methods for the examination of water & wastewater, American Public Health (Association. ed.), Washington DC: American Public Health Association.

Cai, Y., Zhao, X., Zhao, Y., Wang, H., Yuan, X., Zhu, W., Cui, Z. & Wang, X. (2018). Optimization of Fe^{2+} supplement in anaerobic digestion accounting for the Fe-bioavailability, *Bioresource Technology*, 250, pp. 163–170, DOI: 10.1016/j.biortech.2017.07.151

Cai, Y., Gallegos, D., Zheng, Z., Stinner, W., Wang, X., Pröter, J. & Schäfer, F. (2021). Exploring the combined effect of total ammonia nitrogen, pH and temperature on anaerobic digestion of chicken manure using response surface methodology and two kinetic models, *Bioresource Technology*, 337, 125328, DOI: 10.1016/j.biortech.2021.125328.

Ciccoli, R., Sperandei, M., Petrazzuolo, F., Broglia, M., Chiarini, L., Correnti, A., Farneti, A., Pignatelli, V. & Tabacchioni, S. (2018). Anaerobic digestion of the above ground biomass of Jerusalem Artichoke in a pilot plant: Impact of the preservation method on the biogas yield and microbial community,*Biomass and Bioenergy*, 108, pp. 190–197, DOI: 10.1016/j.biombioe.2017.11.003

Gabriel, S.A, Funmilayo, D.F. & Evariste, G.K. (2020). Process Optimisation of Enzymatic Saccharification of Soaking Assisted and Thermal Pretreated Cassava Peels Waste for Bioethanol Production, *Waste and Biomass Valorization*, 11, 4, pp. 2409–2420, DOI: 10.1007/s12649-018-00562-0.

Gnansounou, E. & Dauriat, A. (2010). Techno-economic analysis of lignocellulosic ethanol: A review, *Bioresource Technology*, 101, 13, pp. 4980–4991, DOI: 10.1016/j.biortech.2010.02.009

Gunnarsson, I.B., Svensson, S.E., Johansson, E., Karakashev, D. & Angelidaki, I. (2014). Potential of Jerusalem artichoke (Helianthustuberosus L.) as a biorefinery crop, *Industrial Crops & Products*, 56, pp. 231–240, DOI: 10.1016/j.indcrop.2014.03.010

Günerhan, Ü., Us, E., Dumlu, L., Yılmaz, V., Carrère, H. & Perendeci, A.N. (2020). Impacts of Chemical-Assisted Thermal Pretreatments on Methane Production from Fruit and Vegetable Harvesting Wastes: Process Optimization, *Molecules*, 23, 25, 500, DOI: 10.3390/molecules25030500

Hassan, T.M., Hossain, M.S., Kassim, M.H., Ibrahim, M., Mohammad, N.F. & Hussin, M.H. (2020). Optimizing the Acid Hydrolysis Process for the Isolation of Microcrystalline Cellulose from Oil Palm Empty Fruit Bunches Using Response Surface Methods, *Waste and Biomass Valorization*, 11, 6, pp. 2755–2770, DOI: 10.1007/s12649-019-00627-8.

Hossain, M.Z., Suely, A., Yun, J., Zhang, G., Faisal, N.A., Qi, X. & J.N.S. (2019). Recent advances in biological pretreatment of microalgae and lignocellulosic biomass for biofuel production, *Renewable and Sustainable Energy Reviews*, 105, pp. 105–128, DOI: 10.1016/j.rser.2019.01.048

Kafle, Gopi Krishna, Kim & Sang Hun. (2013). Anaerobic treatment of apple waste with swine manure for biogas production: batch and continuous operation, *Applied Energy*, 103, pp. 61–72, DOI: 10.1016/j.apenergy.2012.10.018

Khalid, H., Cai, F., Zhang, J., Zhang, R., Wang, W., Liu, G. & Chen, C. (2019). Optimizing key factors for biomethane production from KOH-pretreated switchgrass by response surface methodology, *Environmental science and pollution research international*, 26, 24, pp. 25084–25091, DOI: 10.1007/s11356-019-05615-y

Kim, M., Kim, B., Nam, K. & Choi, Y. (2018). Effect of pretreatment solutions and conditions on decomposition and anaerobic digestion of lignocellulosic biomass in rice straw, *Biochemical Engineering Journal*, 140, pp. 108–114, DOI: 10.1016/j.bej.2018.09.012.

Kim, S., Park, J.M. & Kim, C.H. (2013). Ethanol production using whole plant biomass of Jerusalem artichoke by Kluyveromycesmarxianus CBS1555, *Applied biochemistry and biotechnology*, 169, 5, pp. 1531–1545, DOI: 10.1007/s12010-013-0094-5

Kozłowski, K., Dach, J., Lewicki, A., Malińska, K., Isaias Emilio Paulino do Carmo. & Czekała, W. (2019). Potential of biogas production from animal manure in Poland, *Archives of Environmental Protection*, 45, 3, pp. 98–108, DOI: 10.24425/aep.2019.128646

Kreuger, E., Sipos, B., Zacchi, G., Svensson, S.E., Bjornsson, L. (2011). Bioconversion of industrial hemp to ethanol and methane: The benefits of steam pretreatment and co-production, *Bioresource Technology*, 102, pp. 3457–3465, DOI: 10.1016/j.biortech.2010.10.126

Li, C., Liu, G., Nges, I.A. & Liu, J. (2016). Enhanced biomethane production from Miscanthuslutarioriparius using steam explosion pretreatment, *Fuel*, 179, pp. 267–273, DOI: 10.1016/j.fuel.2016.03.087

Liu, J., Yang, M., Zhang, J., Zheng, J., Xu, H., Wang, Y. & Wei, Y. (2018). A comprehensive insight into the effects of microwave-H_2O_2 pretreatment on concentrated sewage sludge anaerobic digestion based on semi-continuous operation, *Bioresource Technology*, 256, pp. 118–127, DOI: 10.1016/j.biortech.2018.01.126

Li, W., Zhang, J., Yu, C., Li, Q., Dong, F., Wang, G., Gu, G. & Guo, Z. (2015). Extraction, degree of polymerization determination and prebiotic effect evaluation of inulin from Jerusalem artichoke, *Carbohydrate Polymers*, 121, pp. 315–319, DOI: 10.1016/j.carbpol.2014.12.055

Long, X., Shao, H., Liu, L., Liu, L. & Liu, Z. (2016). Jerusalem artichoke: A sustainable biomass feedstock for biorefinery, *Renewable and Sustainable Energy Reviews*, 54, pp. 1382–1388, DOI: 10.1016/j.rser.2015.10.063

Monlau, F., Sambusiti, C., Barakat, A., Guo, X.M., Latrille, E., Trably, E., Steyer, J.P., Carrere, H. (2012). Predictive models of biohydrogen and biomethane production based on the compositional and structural features of lignocellulosic materials, *Environmental science & technology*, 6, 46, pp. 12217–12225, DOI: 10.1021/es303132t

Nges, A.I., Li, C., Wang, B., Xiao, L., Yi, Z., Liu, J. (2016). Physio-chemical pretreatments for improved methane potential of Miscanthuslutarioriparius, *Fuel*, 166, pp. 29–35, DOI: 10.1016/j.fuel.2015.10.108

Nowicka, A., Zieliński, M., Dębowski, M., Dudek, M. (2021). Progress in the Production of Biogas from Maize Silage after Acid-Heat Pretreatment, *Energies*, 14, 8018, DOI: 10.3390/EN14238018

Oh, S.Y., Yoo, D.I., Shin, Y., Kim, H.C., Kim, H.Y., Chung, Y.S., Park, W.H. & Youk, J.H. (2005). Crystalline structure analysis of cellulose treated with sodium hydroxide and carbon dioxide by means of X-ray diffraction and FTIR spectroscopy, *Carbohydrate Research*, 340, 15, pp. 2376–2391, DOI: 10.1016/j.carres.2005.08.007

Oyekanmi, A.A., Ahmad,A., MohdSetapar, S.H., Alshammari, M.B., Jawaid, M., Hanafiah, M.M., Abdul Khalil, H.P.S. & Vaseashta, A. (2021a). Sustainable Duriozibethinus-Derived Biosorbents for Congo Red Removal from Aqueous Solution: Statistical Optimization, Isotherms and Mechanism Studies, *Sustainability*, 13, 13264, DOI: 10.3390/SU132313264

Oyekanmi, A.A., Alshammari, M.B., Ibrahim, M.N.M., Hanafiah, M.M., Elnaggar, A.Y., Ahmad, A., Oyediran, A.T., Rosli, M.A., Mohd, Setapar, S.H., Nik, Daud, N.N. & Hussein, E.E. (2021b). Highly Effective Cow Bone Based Biocomposite for the Sequestration of Organic Pollutant Parameter from Palm Oil Mill Effluent in a Fixed Bed Column Adsorption System, *Polymers (Basel)*, 27, 14, 86, DOI: 10.3390/polym14010086

Passos, F., Felix, L., Rocha, H., Pereira, Jde, O., de, Aquino, S. (2016). Reuse of microalgae grown in full-scale wastewater treatment ponds: Thermochemical pretreatment and biogas production, *Bioresource Technology*, 209, pp. 305–312, DOI: 10.1016/j.biortech.2016.03.006

Passos, F., Ortega, V. & Donoso-Bravo, A. (2017). Thermochemical pretreatment and anaerobic digestion of dairy cow manure: Experimental and economic evaluation, *Bioresource Technology*, 227, pp. 239–246, DOI: 10.1016/j.biortech.2016.12.034

Paudel, S.R., Banjara, S.P., Choi, O.K., Park, K.Y., Kim, Y.M. & Lee, J.W. (2017). Pretreatment of agricultural biomass for anaerobic digestion: Current state and challenges, *Bioresource Technology*, 245, pp. 1194–1205, DOI: 10.1016/j.biortech.2017.08.182.

Pfariso, M., Eugéne, R., Annie, F.A.C & Johann, F.G. (2021). Maximising the Benefits of Enzyme Synergy in the Simultaneous Saccharification and Fermentation of Jerusalem Artichoke (Helianthus tuberosus) Tuber Residues into Ethanol, *Waste and Biomass Valorization.Waste Biomass Valor*, 13, pp. 535–546, DOI: 10.1007/S12649-021-01488-W

Pokój, T., Gusiatin, M.Z., Bułkowska, K. & Dubis, B. (2014). Production of biogas using maize silage supplemented with residual glycerine from biodiesel manufacturing, *Archives of Environmental Protection*, 40, 4, pp. 17–29, DOI: 10.2478/aep-2014-0035

Shen, J., Zhang, J., Wang, W., Liu, G. & Chen, Ch. (2019). Assessment of pretreatment effects on anaerobic digestion of switchgrass: Economics-energy-environment (3E) analysis, *Industrial Crops & Products*, 145, 111957, DOI: 10.1016/j.indcrop.2019.111957

Song, Z., Yang, G., Liu, X., Yan, Z., Yuan, Y. & Liao, Y. (2014). Comparison of seven chemical pretreatments of corn straw for improving methane yield by anaerobic digestion, *PLoS One*, 2, 9, DOI: 10.1371/journal.pone.0093801

Tian, W., Li, J., Zhu, L., Li, W., He, L., Gu, L., Deng, R., Shi, D., Chai, H. & Gao M. (2021). Insights of enhancing methane production under high-solid anaerobic digestion of wheat straw by calcium peroxide pretreatment and zero valent iron addition, *Renewable Energy*, 177, pp. 21–32, DOI: 10.1016/J.RENENE.2021.06.042

Van Soest P.J., Robertson J.B. & Lewis B.A. (1991). Methods for Dietary Fiber, Neutral Detergent Fiber, and Nonstarch Polysaccharides in Relation to Animal Nutrition, *Journal of Dairy Science*, 74, 10, pp. 3583–3597, DOI: 10.3168/jds.S0022-0302(91)78551-2

Wang, D.L., Ai, P., Yu, L., Tan, Z.X. & Zhang, Y.L. (2015). Comparing the hydrolysis and biogas production performance of alkali and acid pretreatments of rice straw using two-stage anaerobic fermentation, *Biosystems Engineering*, 132, pp. 47–55, DOI: 10.1016/j.biosystemseng.2015.02.007

Wu, Z., Nguyen, D., Lam, T.Y.C., Zhuang, H., Shrestha, S., Raskin, L., Khanal, S.K. & Lee, P.H. (2021). Synergistic association between cytochrome bd-encoded Proteiniphilum and reactive oxygen species (ROS)-scavenging methanogens in microaerobic-anaerobic digestion of lignocellulosic biomass, *WaterResearch*, 15, 190, 116721, DOI: 10.1016/j.watres.2020.116721

Yang, S., Sun, X., Jiang, X., Wang, L., Tian, J., Li, L., Zhao, M. & Zhong, Q. (2019). Characterization of the Tibet plateau Jerusalem

artichoke (*Helianthus tuberosus* L.) transcriptome by de novo assembly to discover genes associated with fructan synthesis and SSR analysis, *Hereditas*, 6, 156, 9, DOI: 10.1186/s41065-019-0086-8

Zhang, H., Khalid, H., Li, W., He, Y., Liu, G. & Chen, C. (2018a). Employing response surface methodology (RSM) to improve methane production from cotton stalk, *Environmental science and pollution research international*, 25,8, pp. 7618–7624, DOI: 10.1007/s11356-017-0682-y

Zhang, H., Ning, Z., Khalid, H., Zhang, R., Liu, G. & Chen, C. (2018b). Enhancement of methane production from Cotton Stalk using different pretreatment techniques, *Scientific reports*, 8, 1, 3463 DOI: 10.1038/s41598-018-21413-x

Zhang, H., Wang, L., Dai, Z., Zhang, R., Chen, C. & Liu, G. (2019). Effect of organic loading, feed-to-inoculum ratio, and pretreatment on the anaerobic digestion of tobacco stalks, *Bioresource Technology*, 298, 122474, DOI: 10.1016/j.biortech.2019.122474

Zhao, C., Cui, X., Liu, Y., Zhang, R., He, Y., Wang, W., Chen, C. & Liu, G. (2017). Maximization of the methane production from durian shell during anaerobic digestion, *Bioresource Technology*, 238, pp. 433–438, DOI: 10.1016/j.biortech.2017.03.184