Waste Management 103 (2020) 61–66

Contents lists available at ScienceDirect

Waste Management

journal homepage: www.elsevier.com/locate/wasman

Continuous hydrogen production from food waste by anaerobic digestion (AD) coupled single-chamber microbial electrolysis cell (MEC) under negative pressure

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ARTICLE INFO

Article history:
Received 19 July 2019
Revised 6 November 2019
Accepted 9 December 2019
Available online 19 December 2019

Keywords:
Food waste
Microbial electrolysis cell
VFAs
Hydrogen recovery
Solid reduction

ABSTRACT

Increased generation of food waste (FW) poses significant risks to the social environment, and therefore it is critical that efficient technology be developed for effective waste valorization. This study used an integrated reactor to combine single-chamber microbial electrolysis cell (MEC) treatment and anaerobic digestion (AD) to achieve efficient hydrogen recovery using FW as substrate. Hydrogen production during continuous AD-MEC operation (511.02 ml H_2 g^{-1} VS) was higher than that achieved by AD (49.39 ml H_2 g^{-1} VS). The hydrogen recovery and electrical energy recovery in AD-MEC were as high as 96% and 238.7 ± 5.8%, respectively. To explore the mechanism of hydrogen production increase, the main components of FW [lipids, volatile fatty acids (VFAs), carbohydrates, and protein] were analyzed to investigate the utilization of organic matter. Compared with AD treatment, the removal rates of carbohydrates and proteins in the soluble phase in AD-MEC were increased by 4 times and 2.3 times, respectively. The removal of VFAs by AD-MEC was increased by 4.7 times, which indicated that the AD reactor coupled with MEC technology improved the utilization of the main organic components and thus increased hydrogen production. This study demonstrates the possibilities of reducing FW quantities along with the production of bio-hydrogen.

1. Introduction

As a major burden on the environment, food waste (FW) is one of the single largest components of the waste stream by weight in China, which disposes of about 1.3 billion tons of food each year (Zhao et al., 2017). The food waste is, for the most part, disposed of in landfill (Hodge et al., 2016). In light of rapidly rising costs associated with energy supply and waste disposal, conversion of food waste to energy is becoming a more economically viable practice. Because of the relatively high moisture content of food waste (Maddi et al., 2017), bioconversion technologies, such as anaerobic digestion (AD) (Al Afif and Amon, 2019), are more suitable than thermochemical conversion technologies, such as combustion (Velasquez et al., 2019) and gasification (Praeger et al., 2019). However, the production of short-chain fatty acids, which are the key intermediate products of anaerobic digestion, is inhibited by the characteristics of high salinity and fat in FW (Pierra et al., 2014), thus limiting the energy recovery of anaerobic digestion in FW. For these reasons, the identification of a new method that can adapt to the high salinity of food waste and accelerate the hydrolysis acidification process is desirable.

Hydrogen is a promising alternative to fossil fuels because it is clean, renewable, and provides a high energy yield (Saidi et al., 2018). Among promising conversion biotechnologies, biological hydrogen production from reproducible resources, especially food waste, has great potential because of its high efficiency, low energy consumption, low pollution, and low cost (Dahiya et al., 2015; Yun et al., 2018; Jarunglumlert et al., 2018). However, biohydrogen that is produced by hydrogen-producing bacteria can be consumed by hydrogen-consuming bacteria such as methanogens to produce biomethane during conventional anaerobic digestion. For example, Han et al. (Han et al., 2004) studied the anaerobic digestion of a mixed food waste collected from university kitchens, hospitals, and markets, and the biogas generated during the 7-day digestion period contained only 19.3% biohydrogen. In addition, the high

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https://doi.org/10.1016/j.wasman.2019.12.015
0956-053X/

salinity of food further inhibits the production of short-chain fatty acids (Zhang et al., 2014), which are important intermediates in hydrogen production. To achieve the goal of efficient hydrogen collection in FW anaerobic digestion, various pretreatment techniques have been applied, including heat shocking (Breunig et al., 2017), acid treatment, or alkaline treatment. However, treatment of FW with heat or with chemicals is costly in industrial applications. Therefore, exploring efficient and environmentally friendly technologies to improve the efficiency of hydrogen energy recovery from food waste with high salt content is necessary.

The microbial electrolysis cell (MEC), developed as a novel anaerobic digestion reactor in recent years, has attracted considerable attention over the past several years as a promising technology for higher hydrogen yields from organic matter (Zhang et al., 2014). It is reported to give a high hydrogen production rate (7 L L^{-1} day^{-1}) and has good salt resistance (Lu and Ren, 2016). Hassanein et al. determined the effect of incorporating a MEC with AD in a single chamber using waste activated sludge as substrate. Cumulative hydrogen production during the AD-MEC batch test for 23-days reached up to 3.39 L hydrogen (Hassanein et al., 2017). However, the hydrogen produced by the cathode could be easily scavenged by methanogens, homoacetogens, and even exoelectrogens (Ruiz et al., 2013) in the single chamber, causing the hydrogen production efficiency of MEC to be seriously inhibited.

Our previous studies have shown that the diffusion and consumption of hydrogen can be effectively controlled by the operation of MEC under negative pressure, which improved the efficiency of hydrogen production (Feng et al., 2018). Based on our previous studies, a further optimized anaerobic digestion reactor combined with MEC technology under negative pressure was constructed to evaluate the hydrogen production performance and hydrogen recovery efficiency using FW as substrate. The roles of MEC are discussed, with a focus on the content of volatile fatty acids (VFAs), protein, and carbohydrate in highly concentrated food waste.

2. Materials and methods

2.1. FW sample preparations

Simulated FW was used as a substrate for the reaction. According to the characteristics of FW in the canteen of Zhejiang Gongshang University (Hangzhou, China), the simulated FW was composed of rice (44%), noodles (16%), vegetables (23%), meat (6%), and tofu (11%). The five components came from the same vendor at Cui Yuan farmers' market (Hangzhou, China). The main characteristics of the FW are listed in Table S1.

2.2. Reactor design and construction

The composite system consisted of an influent unit, a pre-digestion unit, and a combined AD-MEC unit (Fig. S1). As shown in Fig. S1, the negative pressure of the reaction system is controlled by the peristaltic pump, which is stable at about 40.52kpa. For the control group, the system configuration was the same as above except for the absence of the MEC. The influent unit was filled with M9 buffer solution (was composed of 0.1 g/L NH_4Cl, 0.5 g/L NaCl, 4.4 g/L KH_2PO_4, 3.4 g/L K_2HPO_4, 0.1 g/L $MgSO_4$, and 2 g/L $NaHCO_3$) (Huajun et al., 2015). Food waste (wet weight 250 g; 76.6% water) and anaerobic granular sludge (wet weight 137 g; 92.7% water) were added to the 0.5-L pre-digestion unit. The solids content of the mixture was 13.77 ± 0.35% (w/w) (solids content of anaerobic granular sludge was 1.89 ± 0.11%). Before addition to the pre-digestion unit, the anaerobic granular sludge was boiled at 90 for 30 min to inhibit methanogenic activity (WanTaek et al., 2012). The cell anode was a sludge-modified titanium electrode (Guangzhou China, OD 4.2 cm, ID 3.6 cm, height 8 cm), while the cathode was the same as reported previously (Feng et al., 2018). The cathode was a platinum-coated titanium mesh tube (surface area 175 cm^2, outer diameter 7 cm, height 8 cm, mesh thickness 1.2 mm, mesh hole size 3 mm × 6 mm, platinum coating thickness 1 μm). A perspex cap was clamped onto the glass vessel with an O-ring on the cap providing an airtight seal. The five openings on the reactor cap were used for the reference electrode, anode, cathode, gas collection, and gas pressure gauge, respectively. All reported potentials in this study were measured against a Ag/AgCl (3 M KCl) reference electrode. The inoculum for the reaction unit was effluent from an existing acetate-fed MEC running with methanogenic inhibitors (2-bromoethanesulphonate). All tests were conducted in duplicate, and mean values are reported.

2.3. Start up and operation

The MEC experiment was performed at 30 °C under negative pressure (absolute pressure of 40.52 kPa). The reactors were started with a BioLogic VSP potentiostat (SP-50, EC-LAB VMP3, French) by setting the anode potential at −0.2 V (vs. Ag/AgCl) in batch mode. After start up, the reactors were switched to continuous mode with a medium feed rate of 0.347 ml min^{-1}. Using the FW digestive fluid as substrate, the chemical oxygen demand (COD) of the influent water was maintained at 2500–3500 mg L^{-1} by diluting the high-concentration FW digestive fluid for the first 15 days to culture the biofilm and evaluate the utilization situation of organic matter (Li et al., 2014). In the next 20 days, the COD in the influent water was not controlled, and the pre-fermentation liquid was directly used as the influent to the MEC, and two cycles were run for one digestion cycle for 10 days. The solution was continuously mixed using a magnetic stirrer at a speed of 350 rpm throughout the process.

2.4. Analyses

The gas produced from the reactor was collected and its volume was measured using a water displacement column (glass tube, internal diameter 26 mm, 500 ml) which was adjusted to pH 2 with H_2SO_4. The top end of the column was sealed with a rubber stopper so that gas samples could be collected with syringe and needle. The concentrations of hydrogen and methane were determined by gas chromatography (GC7900, Techcomp, Shanghai, China) equipped with a thermal conductivity detector. The concentrations of VFAs (acetic, propionic, and butyric acids) were analyzed using another gas chromatograph (GC7890Ⅱ, Techcomp, Shanghai, China) with flame ionization detection. The three-dimensional excitation and emission fluorescence of protein were measured in a 1-cm cuvette using a fluorescence spectrometer (F-4600, Hitachi, Tokyo, Japan) (specific method is in Supporting Information S-1) (Murphy et al., 2010).

3. Results and discussion

3.1. Performance of current production

Fig. 1 shows the changes in pH and soluble COD for the AD-MEC reactor influent and effluent for days 9–35. The pH in the AD-MEC reactor decreased in each cycle from 7.5 to 6.0 because of hydrolysis and acidification of the organic matter, while the soluble COD reached a maximum of 12,125 mg L^{-1} on days 18 and 28. The maximum current density was 9.87 ± 0.45 A m^{-2}, which was close to that of some high-performance MEC reactors (10.6 A m^{-2}) using sodium acetate as substrate. Previous work has proved that sludge-modified titanium electrode has good biocompatibility and its

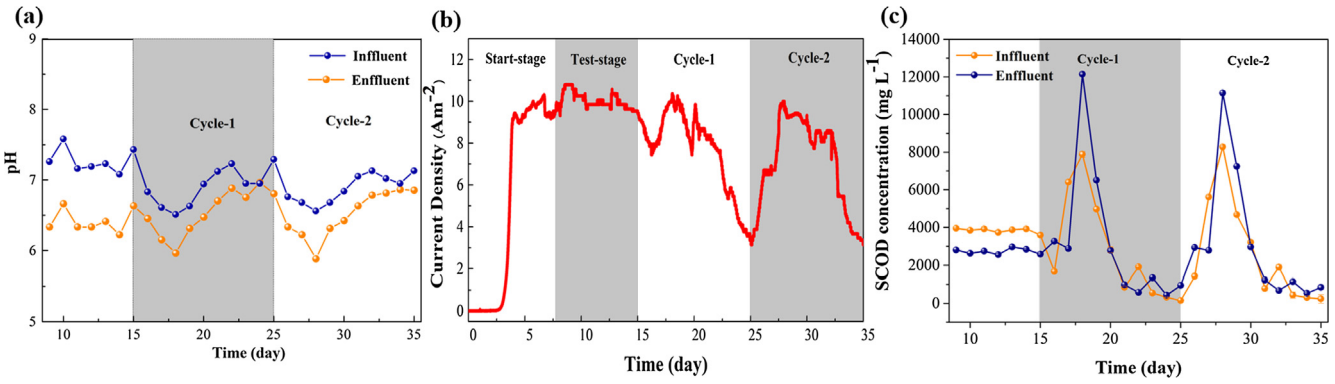

Fig. 1. (a) pH change of AD-MEC reactor in influent and effluent from day 9 to 35. (b) Change of the current production over 35 days. (c) COD change of AD-MEC reactor in influent and effluent from day 9 to 35.

current density is significantly improved (Gu et al., 2017). In this study, the sludge-modified titanium electrode is applied to AD-MEC system, and the multi-fold circular electrode was used to effectively increase the electrode area, which increased the current output density and electron transfer efficiency, and promoted the increase of hydrogen production. Moreover, the high-salt environment in the digestive solution of the food waste increases the rate of electron transfer in the electrochemical system, which helps to increase current density and hydrogen production efficiency. These results suggest that organic matter in food waste with high salinity can be effectively utilized by AD-MEC systems.

3.2. Hydrogen production yield and energy recovery

Fig. 2a shows a uniform trend for changes in hydrogen volumetric yield and current output performance. The hydrogen production in the AD-MEC reactor was 511.02 ml H_2 g^{-1} VS, which was ten times that of the AD system (49.39 ml H_2 g^{-1} VS). Meanwhile, the available organic matter in the solution is gradually reduced in at the end of each operation cycle (Fig. 1(c)), resulting in the decrease of current and hydrogen production. In addition, the corresponding maximum hydrogen volume production of the MEC system (3.48 ± 0.48 L L^{-1} day^{-1}) was also three times that of the AD system (1.55 ± 0.01 L L^{-1} day^{-1}) and FW has significant advantages in hydrogen production compared with other substrates, as shown in Table 1. Borole et al. used the digester effluent as MEC substrate, while the hydrogen production in MECs did not exceed 2 L per L per day and hydrogen recovery rate is less than 20% (Beegle and Borole, 2017). Significant differences in hydrogen production is attributed to different substrates, the digested liquid of food waste has a high organic matter content and salinity, and promoting MEC hydrogen production. The average hydrogen recovery rate of MEC (S-2 Calculations) was as high as 94.6 ± 8.0%, which indicated that AD-MEC efficiently uses FW to produce hydrogen. Methane gas was not detected during operation of the MEC reactor due to the short batch cycle time and the measures taken to inhibit methanogen growth by controlling negative pressure conditions (WanTaek et al., 2012, Feng et al., 2018). Moreover, the mechanism for the increase of hydrogen production is mainly due to the decrease of gas solubility caused by the change of air pressure. According to Henry's law, when the headspace air pressure is nagative, the solubility of hydrogen in the solution is decreased, and it is easy to overflow the water surface (Feng et al., 2018). Therefore, it can effectively reduce the possibility of methane bacteria using hydrogen in liquid phase and increase hydrogen production. by controlling the negative pressure condition of the reactor. In addition, the highest energy recovery of the MEC reactor with an applied voltage of −0.2 V was 175.4 ± 5.8%, higher than the traditional AD reactor, indicating that hydrogen production by MEC treatment is favorable for promoting production capacity (Zhang et al., 2014).

3.3. Changes in organic matter of soluble phase during hydrogen production

To trace the cause of the increase in hydrogen production in AD-MEC, the changes in the main components (proteins, soluble carbohydrates, and VFAs) of COD in the influent and effluent of

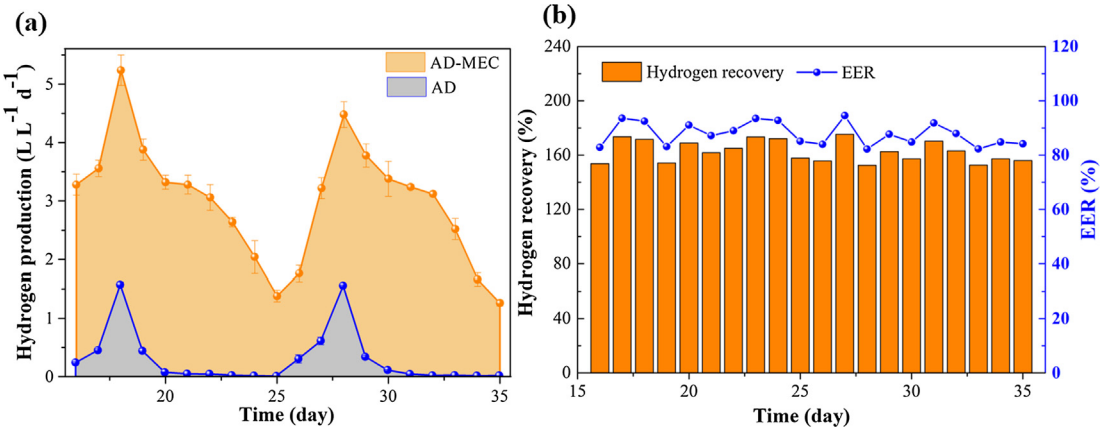

Fig. 2. (a) Comparison of hydrogen production rate by AD-MEC and AD from day 16 to 35. (b) Change of hydrogen recovery and electrical energy recovery for AD-MEC from day 16 to 35.

Table 1
Comparison of current study with others studies dealing with complex substrates in MEC.

Substrate	Reactor configuration	Hydrogen volume yield(L L^{-1} d^{-1})	Electric energy recovery rate(%)	Voltage (V)	References
R-WAS	Double chamber	0.056	176	0.6	Lu et al. (2012a)
A-WAS	Double chamber	0.068	186	0.6	Lu et al. (2012a)
A-WAS	Single chamber	0.91	213	0.6	Lu et al. (2012a)
Glycerin	Single chamber	2.00	139	0.9	Guo et al. (2017)
Crude glycerin	Single chamber	0.41	182	0.6	Guo et al. (2017)
Glycerin	Single chamber	1.30	200	0.6	Feng et al. (2011)
Glycerin	Single chamber	0.60	120	1.0	Feng et al. (2011)
protein	Single chamber	0.42	75	0.8	Lu et al. (2010)
Municipal wastewater	Double chamber	0.015	100	0.9	Escapa et al. (2009)
Corn stalks	Single chamber	3.43	166	0.8	Li et al. (2014)
R-FW	Single chamber	4.86	239	0.8	This study

A-WAS: Alkaline treated waste activated sludge.
R-WAS: Raw waste activated sludge.
R-FW: Raw food waste.

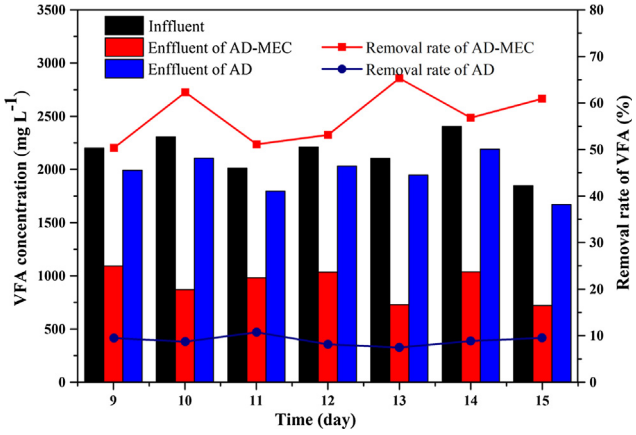

Fig. 3. Efficiency of carbohydrate removal by AD-MEC and AD treatment.

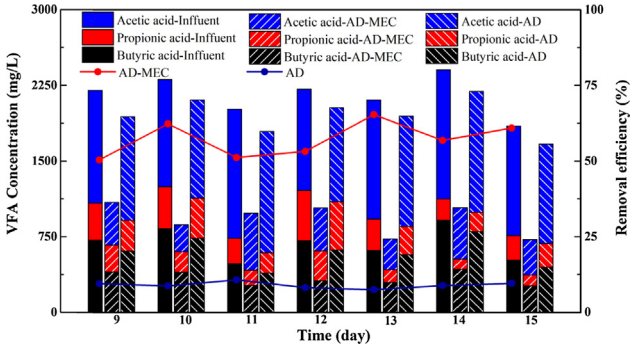

Fig. 4. Concentration and removal efficiency of volatile fatty acids (VFAs) for AD-MEC and AD treatment from day 9 to 16.

the reaction unit were further analyzed. As shown in Fig. 3, the removal rate of soluble carbohydrates by AD-MEC was about 25%, which is about four times higher than the AD reactor (5%). Coupling of the AD reactor with MEC technology promotes the utilization of soluble carbohydrates, which was conducive to increasing hydrogen production.

Pre-digestion results in the dissolution of carbohydrates and conversion to VFAs. The detectable organic acids in the effluent were primarily short-chain VFAs, including acetic acid, propionic acid, and butyric acid. As shown in Fig. 4, the total amount of VFA in the AD-MEC reactor was reduced by 57%, while VFA in the AD reactor was reduced by less than 10%. The efficiency of VFA removal by AD-MEC processing was 4.7 times that of the AD process. The removal of acetic acid, which makes up the highest proportion of VFA, was much higher (49.89%) than that achieved by AD (12.64%), because acetate is the most favorable substrate for exogenous electrons (Lu et al., 2010). The removal rates of propionic acid and butyric acid in AD-MEC were 46.38% and 64.69%, respectively, while the respective removal rates in the AD reactor were 9.78% and 7.51%. The propionic acid and butyric acid were removed less efficiently because they are not ideal substrates for microbial utilization in anaerobic digestion (Lu et al., 2012b). Previous studies have shown that the application of potential in the anaerobic digestion reactor changes the composition of microorganisms and effectively increases the activity of microorganisms, thereby improving the utilization of organic matter (Feng et al., 2015a,b). Taken together, the results show that the MEC reactor increased the anaerobic digestion performance of FW by increasing the utilization efficiency of various organic components in FW.

The changes in protein content in the electrochemical process were fully revealed by three-dimensional excitation–emission matrix (EEM) spectroscopy. The reduction rate of aromatic protein in the AD-MEC reactor was 32.32% (Fig. 5), while the reduction rate of aromatic protein in the AD reactor (Fig. S2) was always less than 10%, suggesting that MEC can also utilize protein to produce hydrogen. Therefore, MEC can more effectively utilize VFAs, carbohydrates, and proteins to increase hydrogen yield.

3.4. Changes in organic matter of solid phase during hydrogen production

AD is commonly used in waste management for FW, with the aim of solids reduction. Previous studies have shown that the reduction of organic solids in FW after AD is generally between 38% and 78% (Brown and Li, 2013; Lim and Wang, 2013; Babel et al., 2004). As shown in Table 2, the solids content (w/w) in the AD system decreased from 13.77 ± 0.35% to 7.12 ± 0.2%, and the solids content (w/w) decreased by 48.3% after 35 days of operation. Furthermore, the MEC system further increased the solids reduction rate to 56.0%. Meanwhile, the degradation rate of total COD in the AD-MEC reactor was 34.9%, which is higher than the degradation rate of 26.1% in AD (Table 2). The results indicated that MEC promoted the hydrolysis of the substrate in FW, thereby achieving a better solids reduction in FW.

Changes in the specific components of total COD (total carbohydrates, total protein, and lipids) were further analyzed. The total carbohydrate and solid phase total protein removal rates were 82.3% and 20.7%, respectively, in the AD-MEC treatment, which were increased over AD treatment by 5.9% (77.7%) a nd 61.7% (12.8%). However, the percentage of lipids in the solid phase after

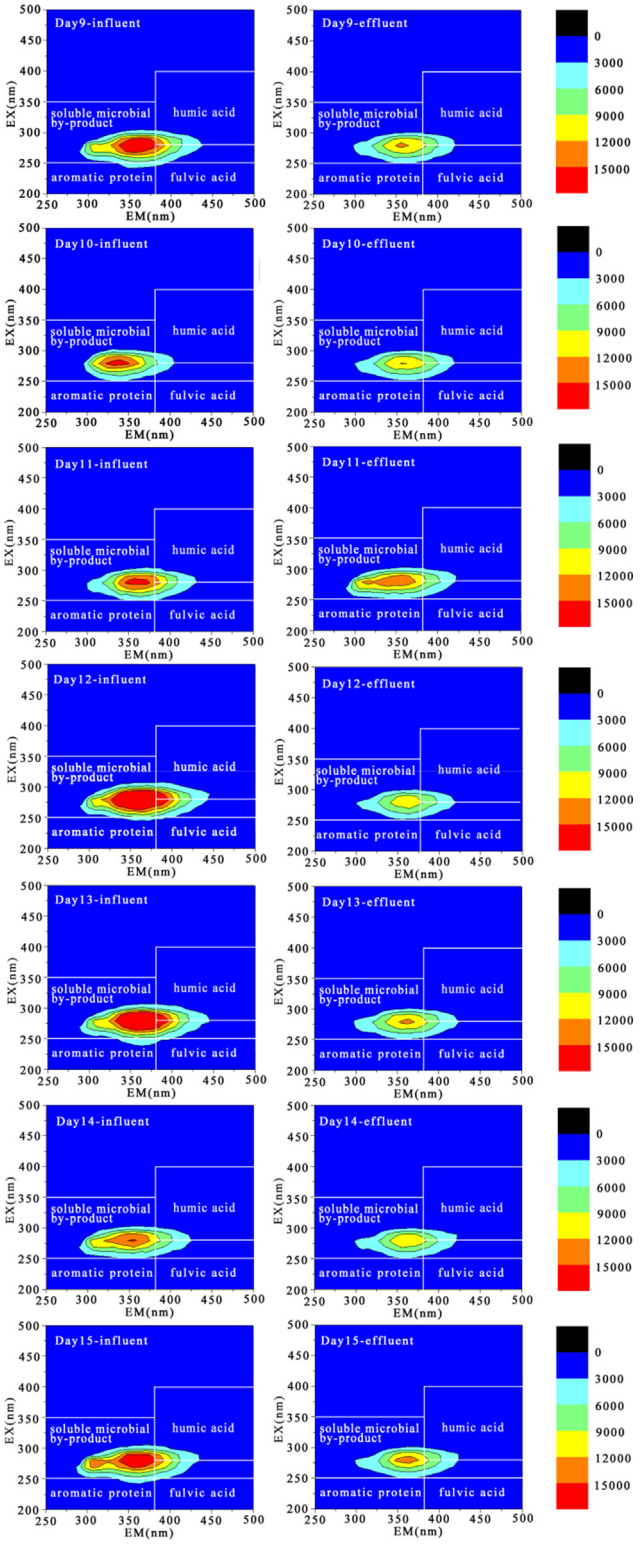

Fig. 5. Three-dimensional excitation–emission matrix fluorescence spectra of aromatic protein, fulvic acid, humic acid, and soluble microbial by-product in the influent and effluent of microbial electrolysis cel.

treatment was higher than the initial value, which was caused by the difficulty of microorganisms utilizing such organic substances, resulting in a gradual increase in lipid content. The results indicate that the efficiency of degradation of organic matter in FW was much higher by MEC treatment than by AD. This was attributed to the increased utilization of carbohydrates and protein by MEC, indicating that MEC can effectively utilize organic matter in FW for hydrogen production.

Table 2
Characteristics of food waste after treatment with different devices.

Parameter	Initial value	After AD-MEC	After AD
Total solid (g kg^{-1})	137.7 ± 3.5	60.6 ± 2.0	71.2 ± 2.0
TCOD (g kg^{-1})	1419.2 ± 66.4	923.5 ± 3.5	1048.5 ± 40.6
TN (g kg^{-1})	57.3 ± 2.8	45.4 ± 3.4	50.0 ± 3.6
Total protein (g kg^{-1})	358.1 ± 17.5	283.8 ± 21.2	312.3 ± 22.8
Total carbohydrate (g kg^{-1})	536.6 ± 9.5	94.9 ± 0.2	119.5 ± 0.5
Lipid(g kg^{-1})	99.3	176.0	268.4
Volatile solids (g kg^{-1})	135.0 ± 3.4	59.4 ± 2.0	69.8 ± 2.0

4. Conclusion

Single-chamber MEC using negative pressure control was found to be a promising treatment technology for increased hydrogen production from food waste. More hydrogen was produced by AD-MEC (511.02 ml H_2 g^{-1} VS) than by AD (49.39 ml H_2 g^{-1} VS), and the energy recovery and hydrogen recovery rates for AD-MEC were as high as 238.7 ± 5.8% and 96%, respectively. Compared with the AD reactor, AD-MEC improved the utilization efficiency of organic matter in soluble phase and soluble phase. Based on the results from this study, MEC technology is considered to be an effective resource utilization method for FW processing and has great engineering application potential.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgments

Funding: This research was supported by the Science and Technology Planning Project of the Science and Technology Department of Zhejiang Province (2018C37057), the National Natural Science Foundation of China (51878611), the Zhejiang Provincial Natural Science Foundation of China (LQ17E080002), and the Xinmiao Talent Project of Zhejiang Province (2018R408076).

Appendix A. Supplementary material

Supplementary data to this article can be found online at https://doi.org/10.1016/j.wasman.2019.12.015.

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