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Enhancement from Anaerobic Digestion of Food Waste by Conductive Materials: Performance and Mechanism

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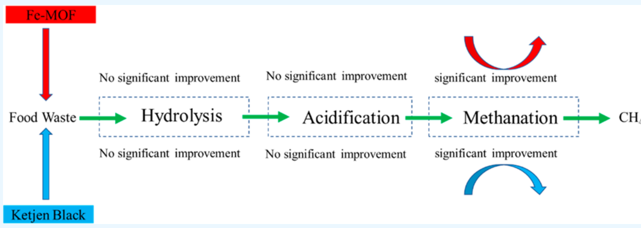

ABSTRACT: Conductive materials (CM) have recently attracted research interest in the anaerobic digestion of food waste to achieve reduction and resource utilization. Fe-metal organic frameworks (Fe-MOF) and Ketjen Black (KB), the conductive materials (CMs), were added for the enhancement of food waste digestion. This study therefore, is intended to fill in this knowledge gap and clarify the underlying mechanism of CM-promoted performance. Batch experiments revealed that the optimal additions of Fe-MOF and KB were 0.5 $g \cdot L^{-1}$ and 0.2 $g \cdot L^{-1}$, respectively. The biogas production increased by 27.50% and 29.45% compared with the blank group, and the removal efficiency of volatile solids (VS), total solids (TS), and chemical oxygen demand (COD) increased by 18.28%, 40.52%, and 15.31%. The lag period was shortened from 3.042 to 2.006 and 1.544 days, respectively. Mechanism studies revealed that Fe-MOF and KB were beneficial to food waste digestion, and the functional groups of Fe-MOF and KB increased the buffer capacity of the system to pH and ammonia nitrogen. The physicochemical properties of Fe-MOF and KB promote the activity of the electron transfer system (ETS); the ETS activity was about 2 times the 11.32 $mg \cdot (g \cdot h)^{-1}$ of the blank group. Zeta potential and electrical conductivity were beneficial to the establishment of intermicrobial direct interspecies electron transfer (DIET).

1. INTRODUCTION

It is about 1/3 of the global food supply for human consumption every year that is lost and wasted in the supply chain of food production, transportation, processing, storage, sales, consumption, etc. How to achieve the sustainable management of food waste has become a global problem.[1] Anaerobic digestion (AD) is a biological process. Special microorganisms break down biodegradable material in the absence of oxygen; simultaneously, a versatile renewable fuel, biogas (methane (CH_4) or hydrogen (H_2)) is recovered.[2] However, due to the complexities of food waste, which are primarily composed of high-biodegradability proteins and carbohydrates,[3] food waste is easily hydrolyzed to monosaccharides and amino acids, which are further converted to volatile fat acids. Acidification of the AD system means that it has a low pH value around 3.5 in food waste (FW) monodigestion, especially when the digesters are overloaded. The lower pH was negative to the activity of methanogenesis and thus reduced methane production.[4]

In the AD system, conductive materials, such as graphene,[5] biochar,[6] magnetite,[7] carbon nanotubes,[8,9] nanoiron tetroxide,[10] and granular activated carbon,[11] were added. It can stimulate direct interspecies electron transfer (DIET) and improve the methanogenesis pathway, thereby promoting methane production and shortening the lag period and enhancing the buffering capacity of the system to ammonia nitrogen and pH. Lin et al.[5] established DIET by adding graphene in anaerobic digestion to increase methane production and methane yield by 25% and 19.5%, respectively, attributed to the high electrical conductivity and specific surface area of graphene. However, the inhibition of adding carbon-based materials, such as activated carbon, carbon nanotubes, and graphene, was a complexity of the preparation process, high economic cost, small effective specific surface area, and low adsorption capacity, which are difficult to further apply in AD. Therefore, it is key to seek one of the additives with high electrical conductivity, large specific surface area, and low economic cost to enhance DIET in AD. Fe-MOFs are a class of coordination polymers constructed by metal ions and organic frameworks through coordination bonds. They have the advantages of high specific surface area, tunable structure, porosity, etc., showing great potential in the field of catalysis.[12] MOF materials have high carbon content, uniform pore

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structure, high porosity, large specific surface area, and abundant active sites, which are conducive to the growth and reproduction of microorganisms and the adsorption of ammonia nitrogen. Owing to the abundant organic ligands and structural diversity in MOFs, MOF-derived porous carbons exhibit uniform pore size, high porosity, and controllable nanoporous structure with high specific surface area and abundant active sites. Compared with traditional carbon materials, MOF-derived porous carbons are simple to prepare, low in cost, and high in adsorption capacity. Most MOFs lack sufficient electrical conductivity to limit their performance. Fe-MOFs are a class of MOFs synthesized with Fe as the metal, due to the advantages of the Fe element, such as low toxicity, low cost, easy preparation, and environmental friendliness.[13] However, the application in anaerobic digestion has not been reported.

In addition, carbon materials have the advantages of being environmentally friendly and inexpensive. KB is similar to carbon nanotubes. Compared with carbon nanotubes, KB has a large specific surface area and high conductivity. It has been widely commercialized for a long time, especially for the high-end batteries. Its performance is enhanced by adding KB. It is speculated that its electrical conductivity and higher specific surface area are beneficial to AD.

In this paper, the effects of Fe-MOF and KB in AD by investigation, organic matter removal efficiency, and biogas production. The mechanisms of Fe-MOF and KB on AD of food waste were studied through the physicochemical properties, ETS, and kinetic analysis.

2. MATERIALS AND METHODS

2.1. Materials. Food waste is used to ensure the uniformity of the digestion substrate and the reliability of the experimental results. The comparison of food waste is as follows: cabbage (20%), pork (10%), chicken (5%), eggs (5%), cooking oil (1%), potatoes (20%), carrots (13.8%), rice (15%), glutinous rice (10%), and salt (0.2%),[14] crushed to a particle size of 1−5 mm, and set aside at −20 °C. The inoculated sludge was taken from the sewage treatment plant in Huaxi District, Guiyang City, Guizhou Province, China. The physicochemical properties of food waste and inoculated sludge are shown in Table 1.

2.2. Conductive Materials. The preparation method of Fe-MOF is as follows: 2.7 g of $FeCl_3·6H_2O$ and 0.824 g of 1,3,5-benzenetricarboxylic acid were dissolved in 70 mL of N,N-dimethylformamide, and the mixture was stirred magnetically for 20 min. It was transferred to a hydrothermal synthesis kettle and heated at 150 °C for 12 h. Fe-MOF was centrifuged and washed several times with absolute ethanol and dried at 60 °C for 6 h to obtain the product, which was ground and placed in a glass drying dish for use. KB was purchased from LION Company in Japan, and its physical and chemical properties are shown in Table 2.

Table 1. Properties of Food Waste and Inoculated Sludge

parameters	food waste	inoculated sludge
pH	5.2	6.8
TS/g·L^{-1}	108.2 ± 4.26	73.5 ± 2.1
VS/g·L^{-1}	87.45 ± 1.6	56.7 ± 1.6
COD/(g·L^{-1})	44.3 ± 15.6	19.5 ± 24.5
ammonia nitrogen/(mg·L^{-1})	235.6 ± 8.4	154.1 ± 4.4
alkalinity /(mg-CaCO$_3$·L^{-1})		1230.3 ± 25.7
C (%TS)	46.3	
H (%TS)	5.4	
O (%TS)	30.1	
N (%TS)	3.6	
theoretical gas production/(mL·g-VS^{-1})	532.8	

Table 2. Physical properties of KB (EC600JD)

volatile (%)	iodine absorption value (mg·g^{-1})	ash (%)	apparent density (kg·m^{-3})	specific surface area (m^2·g^{-1})	specific resistance (Ω·cm)	pH
1(max)	1000−1100	0.1 (max)	100−120	1400	0.3−1	8−10

2.3. Batch AD Experiments. Batch digestions with a working volume of 400 mL were conducted, and each group included three parallel reactors. The top was sealed with rubber to prevent air leakage, and the devices were connected by air pipes. The VS ratio of food waste and inoculated sludge was 1:1. The concentration gradient of Fe-MOF was 0.00, 0.25, 0.50, 0.75, 1.00, and 1.25 g·L^{-1}; the concentration gradient of KB was 0.00, 0.10, 0.20, 0.30, 0.40, 0.50 g·L^{-1}, adjusting the pH value of the substrate within the range of 7.2 to 7.5 before the experiment. Placement occurred in a constant temperature water bath at 36 ± 1 °C, with manual shaking once a day and collection of gas by a drainage method. A set of experiments was set up with only inoculated sludge added to deduct the biogas production from inoculated sludge. All of the batch experiments were performed in duplicate.

2.4. Analytical Methods. *2.4.1. Dynamic Model Analysis.* A modified Gompertz model was used to fit the gas yield of the digestion process.[15]

$$B_{(t)} = f_d \exp\left(-\exp\left[\frac{R_m \cdot e \cdot (\lambda - t)}{f_d} + 1\right]\right) \quad (1)$$

In the formula, $B_{(t)}$ is the cumulative biogas production during the digestion time, mL·g-VS^{-1}; f_d is the theoretical maximum cumulative biogas production, mL·g-VS^{-1}; λ is the lag period, days; R_m is the maximum biogas yield, mL·g-VS^{-1}·d^{-1}; t is digestion time, days; and e is the natural index, 2.7183.

2.4.2. Chemical Analysis. TS and VS were determined by a gravimetric method. The pH value was measured by a portable pH meter (PHB-4, Shanghai Lei Magnetic). COD was determined by a potassium dichromate method. Ammonia nitrogen was determined by Nessler reagent spectrophotometry, according to Wang et al.[16] The method used iodonitrotetrazolium [2-(*p*-iodophenyl)-3-(pnitrophenyl)-5-phenyltetrazolium chloride, INT] to characterize the electron transport system (ETS) activity of the digestive juice; Acid−base titration, zeta potential, and conductivity were measured by a nanoparticle size/zeta potential analyzer (Delsa Nano C, Beckman Coulter, USA). A Fourier infrared spectrometer (Bruker Vertex 70, Bruker, Germany) was used to characterize functional groups of materials. For gas production, using the 3 mol·L^{-1} NaOH solution method to measure, NaOH can absorb CO_2 and H_2S and other gases in the biogas, The methane content was about 95% after the absorption,[17] and the volume of the discharged liquid was recorded every 24 h and expressed as mL·g-VS^{-1}.[18] The C, H, and N compositions of the substrates were determined by using an organic

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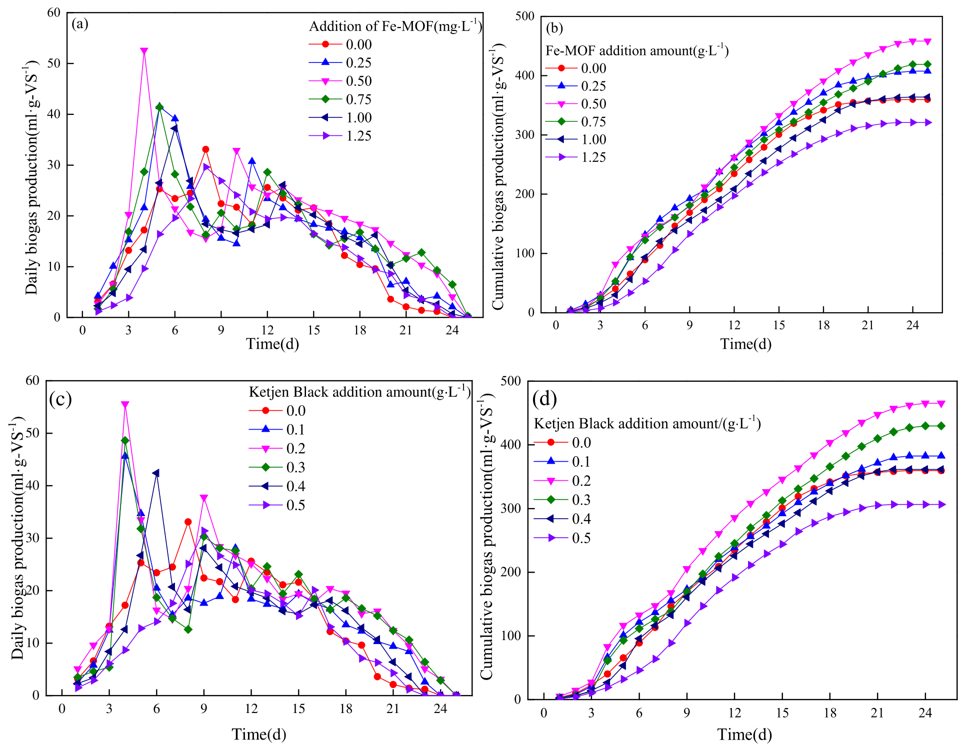

Figure 1. Daily biogas production by adding Fe-MOF (a) and cumulative biogas production by adding Fe-MOF (b). Daily biogas production by adding KB (c) and cumulative biogas production by adding KB (d).

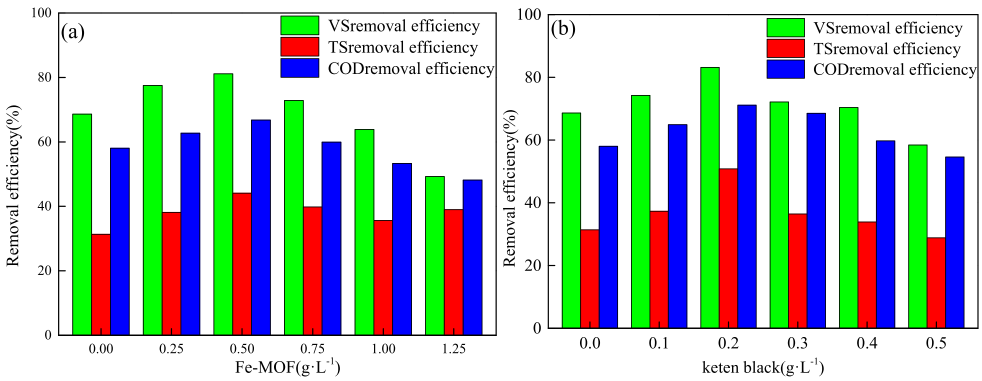

Figure 2. Removal of TS, VS, and COD by adding Fe-MOF (a). The removal of TS, VS, and COD by adding KB (b).

elemental analyzer (Vario EL III, Elementar cube), and the O content of the samples was calculated by the difference method.

3. RESULTS AND DISCUSSION

3.1. Biogas Production. The performance of daily biogas production from AD of food waste is depicted in Figure 1a and c; the daily biogas production has two peaks, which may represent the rapid utilization of easily degradable organic matter by microorganisms and the decomposition of complex structures.[19] The peak of daily biogas production in the blank group appeared on the eighth day, which was 33.1 mL·g-VS^{-1}, and when the Fe-MOF addition amount was 0.50 g·L^{-1}, the peak appeared on the fourth day, which was 52.6 mL·g-VS^{-1}, an increase of 58.91%. When the Fe-MOF addition amount was 1.25 g·L^{-1}, the peak value of the blank group appeared on the eighth day, which was 9.4% lower than that of the blank group. When the addition amount of KB was 0.10, 0.20, and 0.30 g·L^{-1}, the peaks appeared on the fourth day, which were 45.6 mL·g-VS^{-1}, 55.8 mL·g-VS^{-1}, and 48.4 mL·g-VS^{-1}, increased by 37.76%, 68.58%, and 46.22% respectively; when the dose of KB was 0.40 g·L^{-1} and 0.50 g·L^{-1}, the peaks appeared delayed and appeared on the sixth day and day 9. Fe-MOF and KB can increase the daily biogas production, advancing the peak of biogas time. When Fe-MOF and KB are added in excess, the biogas production will be adversely affected. It may be that the microorganism was in direct contact with the surface of the conductive material, which destroys the microbial cell membrane and reduces its activity.[20]

The performance of cumulative biogas production from AD of food waste is depicted in Figure 1b,d. The cumulative biogas production first increased and then decreased with the addition of Fe-MOF, and the cumulative biogas production of KB changed. The trend was the same as that of Fe-MOF. When the Fe-MOF and KB addition amounts were 0.50 g·L^{-1} and 0.20 g·L^{-1}, respectively, the cumulative biogas production reached the maximum, 438.4 mL·g-VS^{-1} and 465.5 mL·g-VS^{-1},

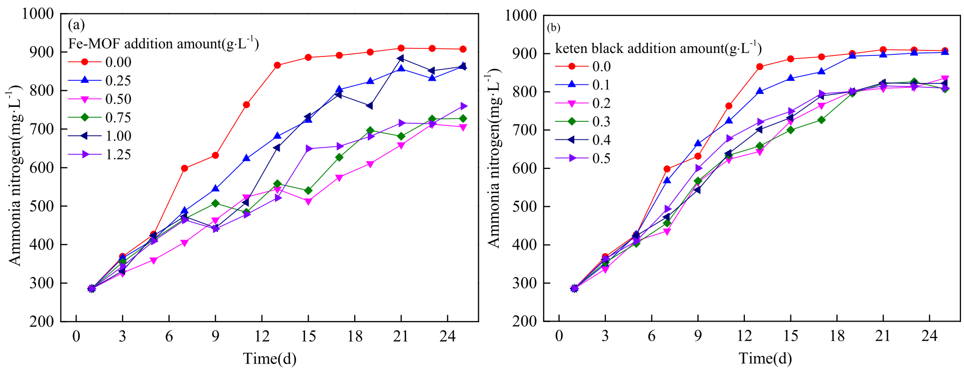

Figure 3. Variations in ammonia nitrogen by adding Fe-MOF(a) The variations in ammonia nitrogen by adding KB (b).

respectively, 21.91% and 29.45% higher than the blank group, respectively; the maximum cumulative biogas production of KB was 6.3% higher than that of Fe-MOF.

3.2. TS, VS. and COD Removal. The removal efficiency of VS, TS, and COD was an important parameter to reflect the reduction of organic matter in the anaerobic digestion system. The higher the removal efficiency, the more obvious the reduction of food waste. It can be seen from Figure 2 that with the addition of Fe-MOF and KB, the removal of VS, TS, and COD first increased and then decreased, and the removal efficiencies of VS, TS, and COD in the blank group were 68.63%, 31.36%, and 58.03%, respectively. When the Fe-MOF addition amount was 0.50 g·L^{-1}, the removal efficiencies of VS, TS, and COD were the highest, which were 81.18%, 44.07%, and 66.81%, respectively, which were increased by 18.28%, 40.52%, and 15.31%, respectively. When the black addition amount was 0.20 g·L^{-1}, the removal efficiencies of VS, TS, and COD were 83.19%, 50.85%, and 71.09%, respectively, which were increased by 21.22%, 62.14%, and 22.51%, respectively. The results show that the addition of conductive materials can improve the removal efficiency of organic matter in the anaerobic digestion process. The higher biogas production, the the higher removal efficiency of organic matte.

3.3. The Variations in Ammonia Nitrogen, pH, and Alkalinity. Ammonia nitrogen represents the content of nutrients in the AD system. Too low of a concentration indicates insufficient nutrients in the system, while too high of a concentration will inhibit the activity of methanogens. The ammonia nitrogen concentration in the digestion system with Fe-MOF and KB was added as shown in Figure 3a,b. The ammonia nitrogen concentration in each group increased with time. The ammonia nitrogen concentration in the blank group tended to be stable after 13 days, and the ammonia nitrogen concentration was 886.1−907.5 mg·L^{-1} and fluctuated. The Fe-MOF addition amount was 0.25, 0.50, 0.75, 1.00, and 1.25 g·L^{-1} The ammonia nitrogen concentration at the end of the experiment was 863.1, 706.1, 727.6, 862.5, and 759.8 mg·L^{-1}, and its ammonia nitrogen concentration was always lower than that of the blank group. The concentration of ammonia nitrogen added with KB was lower than that of the blank group and fluctuated within 800−830 mg·L^{-1}. It can be seen that adding Fe-MOF and KB to the anaerobic digestion system of kitchen waste can reduce the accumulation of ammonia nitrogen, create a favorable growth environment for methanogenic bacteria, and ensure the operation of the anaerobic digestion reaction.

3.4. Mechanism of Conductive Material from AD of Food Waste. *3.4.1. Influence of Physicochemical Properties.* The enhancement of intermicrobial DIET to increase biogas production was related to the zeta potential and conductivity of conductive materials. Weak conductivity requires repeated supply and acceptance of electrons between microorganisms to increase the number of available electrons in the system; strong conductivity can promote DIET between microorganisms.[21,23] The absolute values of the zeta potential of Fe-MOF and KB are 22.34 mV and 22.63 mV, respectively. The larger the absolute value of the zeta potential, the more stable the system, which provides a good environment for the DIET of microorganisms. The conductivities of Fe-MOF and KB are 0.1875 mS·cm^{-1} and 0.2493 mS·cm^{-1}, respectively. The higher the conductivity of the conductive material, the stronger the electron transport ability, and the higher the efficiency of the DIET process mediated by it.[5]

Conductive material can promote DIET between microorganisms and adsorb ammonia nitrogen to improve AD performance through certain electroactive organic functional groups.[18] Figure 4 shows the FT-IR of Fe-MOF and KB, the

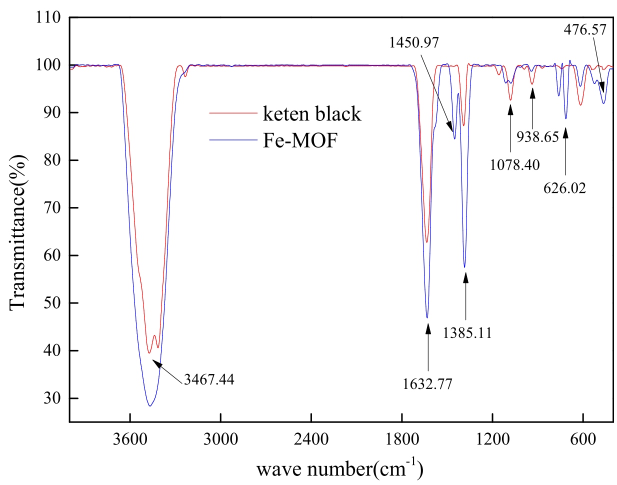

Figure 4. FT-IR spectra of Fe-MOF and KB.

stretching vibration $\upsilon 3$ of the ferrite octahedron near 626.02 cm^{-1}, and the deformation vibration $\delta 1$ of the ferrite octahedron near 476.57 cm^{-1}. These functional groups are related to the literature. The reported synthesized Fe-MOFs are consistent with ref 22, indicating that the Fe-MOFs were successfully prepared. Near 3467.44 cm^{-1} was the characteristic absorption peak of −OH; near 1632.77 cm^{-1} was the antisymmetric stretching vibration of the carboxyl group (υas(COO−)), carbon−carbon double bond (C =C) or

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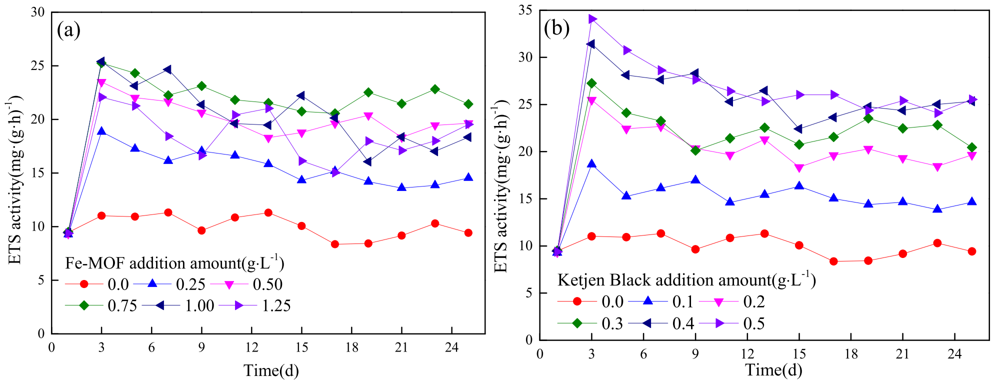

Figure 5. Variations in ETS activity by adding Fe-MOF (a). The variations in ETS activity by addingKB (b).

−NH stretching vibration. Near 1450.97 and 1385.11 cm^{-1} are the symmetrical vibrations of the carboxyl group (υs-(COO−)); near 1078.40 cm^{-1} are the vibrational absorption peaks of C−O−C or the C−O stretching vibration of the alcohol hydroxyl group (υ(C−O)), 1632.77 cm^{-1}. The absorption peak near 1078.40 cm^{-1} was related to amino acids and phospholipids in the cell wall of microorganisms.[22]

The ammonia nitrogen by adding Fe-MOF and KB was lower than that of the blank group (Figure 3a,c), which may be related to the combination of abundant carboxyl groups on the surface of Fe-MOF and KB with NH_3, reducing anaerobic conditions. The concentration of NH_3 in the digestive system reduces the toxicity of NH_3 to microorganisms. The conductive material can enhance the low pH resistance of the system. It may be that when electroactive methanogens reduce CO_2 to produce CH_4, they consume a large amount of H^+ in the cell, which does not affect the activity of intracellular enzymes and the growth of methanogens. Park et al.[24] believed that the biofilm formed on the conductive material may reduce the transfer rate of protons to the cell biofilm, making the cells attached to the biomass carrier resistant to low pH. Some studies have also shown that organic functional groups such as −OH, C═O, C═C, and −NH combine with H^+ to increase the pH and buffering capacity of the system, making the pH value of the system higher, which was beneficial to the growth of methanogens and improve their performance activity and increase biogas production.[25]

3.4.2. Mechanisms. The ETS activity of digested substrates was an indicator to evaluate the electron transfer efficiency of microorganisms or enzymes in anaerobic digestion systems.[26]Figure 5 shows the changes of ETS activity in the anaerobic digestion system with Fe-MOF and KB addition. Compared with the blank group, the addition of conductive material increased the ETS activity in the digestive system, and the ETS activity in the digestive system reached the maximum 3 days before the reaction, which was consistent with the trend of related research.[27]Figure 5a shows that the Fe-MOF addition amounts of 0.25, 0.50, 0.75, 1.00, and 1.25 g·L^{-1} reached the maximum values of 18.85, 23.49, 25.26, 25.41, and 22.08 mg·(g·h)$^{-1}$, respectively. KB (Figure 5b) had the largest gas production when the addition amount was 0.20 g·L^{-1}, and its ETS activity was 25.49 mg·(g·h)$^{-1}$. The ETS activity of the blank group fluctuated in the range of 8 mg·(g·h)$^{-1}$ to 12 mg·(g·h)$^{-1}$. Lizama et al.[28] studied the effect of zerovalent iron nanoparticles on the anaerobic digestion of sludge. The ETS activity increased by 53.2%, and the gas production increased from 63.2% to 77.6%.. When Fe-MOF and KB were added in too high of an amount, the gas production decreased, probably because their toxicity to microorganisms hindered the electron transfer in the respiratory chain of microorganisms. Lin et al.[5] showed that the graphene concentration of 1.00 g·L^{-1} can increase the methane production by 25.0%. After the graphene concentration was further increased to 2.00 g·L^{-1}, the methane production decreased slightly, which may be related to the disturbance of the cell membrane. and synergistic effect of oxidative stress. The above analysis shows that adding an appropriate amount of Fe-MOF and KB can improve the ETS activity of substrates during anaerobic digestion, thereby promoting microbial activity and increasing gas production.

3.4.3. Kinetic Analysis. Process gas production was fitted using a modified Gompertz model. As shown in Table 3, R^2 was greater than 0.99, indicating that the model can accurately reflect the gas production process of anaerobic digestion. The addition of Fe-MOF and KB had a greater effect on the cumulative biogas production and the start-up time of anaerobic digestion. When the Fe-MOF addition amount was 0.50 g·L^{-1}, the predicted biogas production was 518.4 mL·g-VS^{-1}, which was 35.64% higher than that of the blank group, and the lag period was 2.006 days, which was shortened by 34.06%. When the MOF reached 1.25 g·L^{-1}, the predicted biogas production decreased to 337.2 mL·g-VS^{-1}, and the lag period was also prolonged; when the addition amount of KB was 0.20 g·L^{-1}, the cumulative biogas production was 500.5 mL·g-VS^{-1}, 30.96% higher than the blank group, and the lag period was 1.544 days, which was shortened by 49.2% and 29.92% compared with the blank group and Fe-MOF (addition amount of 0.50 g·L^{-1}), respectively. The λ reflects the adaptation time of microorganisms to the environment, and

Table 3. Kinetic model parameters

conductive material	added amount	B_{actual}	f_d	diff (%)	λ (d)	R^2
Fe-MOF	0.00	359.6	382.2	6.28	3.042	0.997
	0.25	364.6	434.7	19.23	2.034	0.995
	0.50	438.4	518.4	13.06	2.006	0.994
	0.75	419.2	454.8	8.49	2.044	0.994
	1.00	363.8	398.3	9.48	2.893	0.994
	1.25	321.1	337.2	5.01	4.116	0.999
KB	0.10	382.6	416.4	8.83	1.824	0.993
	0.20	465.5	500.5	9.67	1.544	0.995
	0.30	429.9	479.8	11.61	2.556	0.995
	0.40	361.4	386.4	6.92	3.038	0.996
	0.50	306.4	322.7	5.32	4.518	0.998

the smaller it was, the faster the microorganisms could adapt to the environment. Wang et al.[29] added 15 $g{\cdot}L^{-1}$ of biochar during the phenol degradation process, the lag period of gas production was shortened from 15.0 days to 1.1−3.2 days, and the maximum gas production rate was accelerated from 4.0 $mL{\cdot}d^{-1}$ to 13.9 $mL{\cdot}d^{-1}$. Wang et al.[30] shortened the lag period from 5.79 to 3.75 d by adding magnetite; the increase in gas production and the shortening of the lag period were attributed to the addition of conductive material in the anaerobic digestion system to establish DIET.

4. CONCLUSIONS

The cumulative biogas production was 438.4 $mL{\cdot}g{\cdot}VS^{-1}$ and 465.5 $mL{\cdot}g{\cdot}VS^{-1}$ by adding Fe-MOF 0.50 $g{\cdot}L^{-1}$ and KB 0.20 $g{\cdot}L^{-1}$, 27.50% and 29.45% higher than the blank group, respectively; the lag period was shortened by 34.06% and 49.2%, respectively. KB was better than Fe-MOF from the AD of food waste.

FT-IR showed that Fe-MOF and KB functional groups are abundant, such as −OH, C═O, and C═C, and −NH functional groups combined with NH_3 or H^+ can increase the buffering capacity of the digestive system to ammonia nitrogen and pH. The zeta potentials of Fe-MOF and KB are 22.34 mV and −22.63 mV, respectively, providing a good environment for the establishment of DIET; the electrical conductivity was 0.1875 $mS{\cdot}cm^{-1}$ and 0.2493 $mS{\cdot}cm^{-1}$, respectively, providing a path for the establishment of DIET.

The physicochemical properties of Fe-MOF and KB promote the activity of the electron transfer system (ETS); the ETS activity was about 2 times the 11.32 $mg{\cdot}(g{\cdot}h)^{-1}$ of the blank group.

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Notes

The authors declare no competing financial interest.

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