

Enhanced startup of full-scale IC reactor treating high-salt fatty acid wastewater

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ABSTRACT

Waste diatomaceous earth from a brewery was used to enhance the startup process of a full-scale internal circulation (IC) reactor treating a high-salt and high-concentration organic wastewater – high-salt fatty acid wastewater, and its treatment efficiency was studied. When the influent chemical oxygen demand (COD) concentration was 15,000 mg/L, the pH value was 3, the sulfate content was 12,000 mg/L, and the effluent COD was about 1,500 mg L, the COD removal rate was above 90%. Experiments have shown that the IC reactor doped with waste diatomaceous earth from the brewery has better performance in forming granular sludge and shortening startup time. The startup was shortened from the original 90–50 d, and the salt-tolerant concentration was increased from 8,000 to 12,000 mg/L. Therefore, dosing waste diatomaceous earth is an economical, applicable, and effective method to strengthen the IC reactor startup.

Keywords: IC reactor; High-salt fatty acid wastewater; Productive startup; Diatomaceous earth; Yeast

1. Introduction

Fatty acid series products are currently the most widely used oleochemical products in the world, and are very important industrial raw materials. The fatty acid series products are produced as follows: The waste materials from vegetable oil and fat processing plants, that is, the saponins, are acidified by sulfuric acid to obtain crude fatty acids, and then the crude fatty acids are subjected to continuous medium pressure hydrolysis and continuous high vacuum distillation to produce high-quality refined fatty acids, stearic acid, and plant pitch. In addition to high-concentration organic substances such as phospholipids and soaps as well as pollutants such as acids and SS, the production wastewater also contains high-concentration salts (mainly sodium sulfate). The concentrations of pollutants in the wastewater are as follows: the concentration of animal oil and vegetable oil is 100 mg/L, the concentration of chemical oxygen

demand (COD) is 30,000–60,000 mg/L, and the concentration of SS is 1,200–3,000 mg/L. The pH value is about 3.0, and the salt content is 3%–5% (mainly sodium sulfate salt).

This type of wastewater is a typical kind of high-salt and high-concentration organic wastewater, which is difficult to treat. Usually, there are two treatment methods for this type of wastewater: the first method is evaporation. However, there is no ideal way to remove the concentrated liquid after evaporation. Since there is a high treatment cost for hazardous waste disposal and high energy consumption, this method is barely considered by companies. The second method is biochemical treatment. Because the B/C ratio of this wastewater is 0.4–0.45, the biochemical treatment is relatively easier. Thus, the conventional treatment method is physicochemical + biochemical treatment. The anaerobic process can be used to treat high-concentration organic wastewater and recover biogas. However, due to the

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high salinity of this wastewater, sulfate ions in the wastewater are very unfavorable for biochemical treatment in the conventional physicochemical + biochemical treatment methods. During anaerobic digestion process, sulfate-reducing bacteria (SRB) compete with methanogens (MPB) for substrates and produce inhibitory effects on MPB. At the same time, sulfides produced by SRB reducing sulfate also produce toxic effect on MPB and other anaerobic bacteria, thus affecting the treatment effect of anaerobic reactor [1–4]. With the deepening of SRB research and the development of anaerobic reactors, some new high-efficiency anaerobic reactors such as upflow anaerobic sludge blanket (UASB) have been gradually applied to the treatment of high-sulfate wastewater [5-13]. The anaerobic reactor with internal circulation (IC) is the third-generation of high-efficiency anaerobic reactor. It has the characteristics of high-volume load, small footprint, high impact resistance, and the ability to treat wastewater containing toxic substances [14]. Since it was developed and used in the production by the company PAQUES in the Netherlands in 1985, IC reactor has been widely used in the practice of treating high-concentration organic wastewater in papermaking and other industries [15,16]. However, it has been rarely used in treating high-salt and high-concentration organic wastewater.

Other studies have shown that salt-tolerant yeasts can be used to treat high-organic and high-salt wastewater and have better performance than ordinary aerobic or anaerobic bacteria [17]. Since the 1970s, some people in Japan have studied the treatment of high-concentration organic wastewater such as fermentation wastewater using yeast. In the late 1980s, a Japanese company solved the practical problem of yeast treatment technology [17-19]. In high-salt conditions, yeast has higher substrate utilization rate, higher maximum specific sludge growth rate, larger half-rate constant, and higher nutrient removal capacity [20]. Preliminary degradation with yeast and then treatment with bacteria is a feasible and effective way to treat wastewater with high organic matter and high salt content [20]. At present, there are few engineering examples of using salt-tolerant yeast to treat high-salinity and high-concentration organic wastewater. Thus, further studies on yeast treatment are needed.

Brewery diatomaceous earth is used as a filtration aid in the beer production process to filter fermentation broth. As brewery diatomaceous earth traps and adsorbs the proteins, yeast, bacteria, and other organic matter, it appears dark brown. The waste diatomaceous earth obtained from the brewery contains a large amount of yeast, and the diatomaceous earth used in the brewery is a high-quality product with a very high internal porosity. Since the rapid and stable startup of the IC reactor is the key to the smooth operation of the entire wastewater treatment project, this study used a high-salt fatty acid production wastewater from an enterprise in Jiangxi Province, China as the treatment research object. During the startup of the full-scale IC reactor, we attempted to add brewery waste diatomaceous earth and control the operating conditions. The method and start-up characteristics of an enhanced IC reactor treating high-salt fatty acid wastewater were investigated, which provided a reference and basis for the efficient startup and application of IC reactor in the treatment of high-salt and high-concentration organic wastewater.

2. Materials and methods

2.1. Experimental setup

A custom-designed and custom-installed full-scale IC reactor was used in this test. Fig. 1 shows the on-site physical device diagram of the IC reactor. The reactor contains a total of seven cylindrical steel structures. The volume of the treatment water was designed to be 70 T/h. Two of the steel structures were selected in the study, which were labeled as 1# reactor and 2# reactor. The height of the reactor is 19.4 m, the effective height is 16 m, the diameter is 8 m, and the volume of a monomer reactor is 804 m³. The reactor is composed of two reaction chambers stacked on top of each other, where the first chamber is located at the height of 1-10 m and the second chamber is located at the height of 11.2-17.2 m. A circular gas-liquid separator with a height of 2.5 m and a diameter of 2 m is built on the upper part of the main body. The bottom of the reactor is filled with water via an annular water inlet, the internal epoxy resin is anti-corrosive, and the silicate board is used for heat insulation. The sampling ports are located at 3, 6, and 12 m from bottom. The platinum thermal resistance was installed at 1.5 m from the bottom of the reactor



Fig. 1. On-site physical device diagram of IC reactor.

and the temperature was displayed digitally on the screen. The temperature in the reactor was controlled at $35^{\circ}C \pm 5^{\circ}C$.

The internal structure of the IC reactor is shown in Fig. 2. The inlet water enters the first reaction chamber from the bottom of the reactor and is mixed with anaerobic sludge. Most of the organic matter is degraded and converted into biogas in the first chamber. The produced biogas is collected by the collecting hood in the three-phase separator of the first reaction chamber, and rises along the ascending tube. While the biogas rises, the mixed liquid in the first reaction chamber is lifted to the gas-liquid separator at the top of the reactor. The biogas is discharged from the pipe at the top of the gas-liquid separator. The separated mud-water mixture is returned to the bottom of the first reaction chamber along the return pipe, and is fully mixed with the granular sludge and the inlet water at the bottom, which realizes the internal circulation of the mixture. After being treated in the first reaction chamber, the wastewater automatically enters the second reaction chamber and continues to be treated. Wastewater is further purified. The generated biogas is collected by the gas collecting hood of the three-phase separator in the upper part of the second reaction chamber, enters the gas-liquid separator through the ascending tube, and is discharged by the gas collecting device through the top ducts. The supernatant from the treatment of the mixed liquid in the second reaction chamber is drained by the outlet pipe, and the precipitated sludge is automatically returned to the second reaction chamber.

2.2. Experimental water

A fatty acid producer in Jiangxi Province, China, the wastewater was used for this experiment after neutralization, air flotation and oil removal, and hydrolysis and



Fig. 2. Internal structure of IC reactor.

acidification. The quality of the inlet water for IC reactor is shown in Table 1.

2.3. Inoculated sludge

The inoculated sludge used in this experiment was anaerobic biological pond granular sludge from the sewage treatment plant of Jinshawan Industrial Park, Hukou, Jiujiang, Jiangxi, China. The color of the inoculated sludge was dark brown. The volatile suspended solids (VSS) concentration of the sludge was 46.7 g/L, and the total suspended solids (TSS) concentration was 59.4 g/L.

The sludge was screened before inoculation. The sludge was fed into the IC reaction tower and domesticated with glucose as a carbon source for artificial water distribution (COD of about 6,000 mg/L). The inoculation volume of the sludge in the reactor was ½ of the total of the reaction volume.

2.4. Analytical method

COD_{Cr} was measured by potassium dichromate method (GB11914–89); volatile fatty acid (VFA) was measured by titration method; alkalinity (ALK) was measured by titration method; SS sludge concentration was measured by gravimetric method; salt content was measured by gravimetric method; and PH is measured by acidimeter.

2.5. Experimental methods

During the startup operation, the inlet water volume was constant, while COD concentration and salinity of the inlet water were gradually increased. The volume of the inlet water volume was 10 t/h.

The COD concentration of fatty acid wastewater was very high. Even after the neutralization reaction, air floatation, and hydrolytic acidification treatment, the COD concentration was still around 25,000 mg/L. High-concentration water was not conducive to the rapid startup of the reactor. Therefore, before entering the IC reactor, the highconcentration wastewater was diluted by low-concentration wastewater (anaerobic effluent from the IC reactor), and freshwater was also added to control the COD concentration of the influent in the range of 5,000-6,000 mg/L. In addition, the low pH of the fatty acid wastewater and the large concentration of SO_4^{2-} in the wastewater were unfavorable for the startup of the reactor. Therefore, the hydrated lime was added at the initial stage of the startup to neutralize the acid and alkali, adjust the pH to 8.5~9.0, and the addition amount of lime was about 5,000 g/m³.

Table 1 Inlet water quality of IC reactor

pН	8.5
COD (mg/L)	25,000
BOD (mg/L)	15,000
SS (mg/L)	100
NH ₃ –N (mg/L)	100
Animal and vegetable fat (mg/L)	30
Salt (mg/L)	15,000

At the same time, a waste diatomite from a nearby brewery was prepared and sludge was added to the IC reaction tower to reach a certain concentration of 20,000~40,000 mg/L. After inoculation, the sludge was domesticated and water was artificially dispensed with dextrose with the addition of a small amount of fatty acid wastewater, approximately 20 m³/d. The COD concentration was controlled at about 5,500 mg/L and the domestication time was 7–10 d to adapt to the wastewater quality as soon as possible. After domestication, the initial startup of the 1# and 2# reactors began according to the debugging plan. The brewery diatomite earth was not added to 1# reactor and only added to 2# reactor.

The initial startup of the reactors took 61 d. The waste diatomite earth from the brewery was added to the salted wastewater in the 2# reactor to cultivate and domesticate the sludge by wastewater as follows: the ratio of activated sludge to diatomite was 150-180:1. The cultivation and domestication were conducted according to the wastewater containing 3,000-4,000 mg/L salt and 6,000-7,000 mg/L COD concentration. The ratio of COD and salt was controlled to 1:0.5-0.6. When the salt concentration reached 7,000-8,000 mg/L, the diatomaceous earth of the brewery was re-added at the dosage of 40%-50% of the initial addition. At the same time, the effluent of the IC reaction tower was partially refluxed to the hydrolysis and acidification tank, and the inlet water was adjusted adaptively. When the salt content in the inlet water reached 12,000 mg/L, the salt concentration of the influent should stop increasing, the COD of the influent was controlled at 14,000-15,000 mg/L, and the COD load of the IC reaction tower was controlled at 4-4.5 kg/m³ d. The IC reaction was operated stably, and the return flow rate of the outlet water was simultaneously controlled between 100% and 120%.

The initial COD concentration of the reactor inlet water was 6,000 mg/L, and the COD of the outlet water was about

500 mg/L. Under the condition that the COD removal rate of the effluent was stabilized at 90%, the organic load of the influent was increased by about 20% every week, and finally, the COD concentration of influent was stabilized at 15,000 mg/L.

During the startup of the reactor, the COD, salt content (SO_4^{2-}) , temperature of the reactor inlet, and the COD, PH, VFA, and ALK of the reactor outlet water were continuously measured every day. The shape and particle size of granular sludge after were analyzed after the startup of the reactor.

3. Results and discussions

3.1. Effect of influent water quality on COD removal

During the startup process, the COD, and salt concentration of the reactor inlet water were gradually increased. The relationships between the COD and salt concentration of the influent and the removal rate of COD and COD in the effluent for both reactors are shown in Fig. 3. The COD concentration of the influent increased from 6,000 to 14,500 mg/L, and the salt concentration of the influent increased from 3,000 to 12,500 mg/L. As can be seen from Fig. 3a, for 1# reactor, the COD removal rate is maintained over 90% in the first 40 d and the highest removal rate, 92.5%, is obtained on the 3rd day. The COD concentration of the effluent is below 1,000 mg/L in the first 40 d. However, after the 41st day, the influent COD concentration exceeds 11,000 mg/L, and the salt content exceeds 9,000 mg/L, the effluent COD concentration gradually increases, and the COD removal rate gradually decreases. On the 61st day, the COD concentration in the effluent has increased to 3,500 mg/L, and the COD removal rate has dropped to 77.5%. For the 2# reactor, the COD concentration in the effluent basically remains stable below 1,500 mg/L within the first 61 d, and the COD removal rate is above 89.5% with



Fig. 3. Relationship between influent COD and salinity and effluent COD and COD removal rate: (a) 1# reactor and (b) 2# reactor.

the peak value of 92.5%. In the later stage after 41 d, the COD removal rate of the 2# reactor is stabilized between 90% and 91.5%, which is significantly different from that of the 1# reactor. This result indicated that the reactor with added waste diatomaceous earth was resistant to high load during startup.

Organic loading rate (OLR) is an important operation control parameter of anaerobic biological treatment system. Its change will have a significant impact on the microbial community structure and operation efficiency of the system [21,22], and the organic load will affect the COD removal rate [23]. The relationship between the COD of the effluent and the organic load for the 1# and 2# reactors was compared, as shown in Fig. 4. With the increase of the inflow, the organic load OLR increased from the initial 1.75 to 4.5 kg/m³ d. After the 1# reactor was started up, the COD concentration was first reduced to as low as 500 mg/L, and then sharply increased from 1,200 mg/L on the 41st day when the organic load reached 3.65 kg/m³ d. The COD concentration in the effluent of the 2# reactor was basically stable between 500 and 1,500 mg/L, which indicated that the 2# reactor had better load resistance than 1# reactor.

3.2. Influence of COD/SO_4^2 on COD removal

Anaerobic biological treatment is the main technical means for the treatment of sulfate organic wastewater. COD/SO_4^{2-} is an important factor affecting the anaerobic digestion process [24–27] and plays a decisive role in the substrate competition between SRB and MPA in the system. The relationship between the COD/SO_4^{2-} in the influent and the COD and COD removal rate in the effluent of the reactor is shown in Fig. 5. When the COD/SO_4^{2-} is reduced from 1.8 to 1.2, the COD concentration in the effluent of the 1# reactor begins to increase and reaches 3,250 mg/L on the 61st day (Fig. 5a), while the COD removal rate



Fig. 4. Comparison of the relationship between COD and organic load in effluents of 1# and 2# reactors.



Fig. 5. Comparison of the relationship between COD and COD removal rate of effluent and COD/SO_4^2 for 1# and 2# reactors. Relationship between (a) effluent COD and influent COD/SO_4^2 and (b) COD removal rate and influent COD/SO_4^2 .

decreases significantly to 77.5% (Fig. 5b). The results indicated that when the salt ratio increased, the 1# reactor's anaerobic digestion capacity changed weakly, and the salt tolerance became poor. However, in the 2# reactor, as COD/ SO_4^{2-} reduced in the same way, the COD concentration of the effluent only slightly increased with the peak value of 1,500 mg/L (Fig. 5a), and the COD removal rate was stabilized above 90% (Fig. 5b), indicating that the 2# reactor had a certain salt resistance. The results demonstrated that the adding waste diatomaceous earth can increase the salt resistance of the reactor during the startup.

3.3. Changes in effluent pH and VFA of IC reactor

PH and VFA are important control parameters of anaerobic reactor, which can reflect the actual operation conditions of the reactor. The changes in pH and VFA during the startup process were monitored. VFA indicates the content of volatile organic acids (an important intermediate product in the anaerobic digestion process) in the anaerobic treatment system. It is an important indicator reflecting the effect of the anaerobic bioreactor [28]. Methane bacteria mainly use VFA to form methane, and only a small part of methane is generated from CO₂ and H₂. The accumulation of VFA in the anaerobic reactor indicates the inactive state of the methane bacteria or the deterioration of the operating conditions of the reactor. A higher VFA (e.g., acetic acid) concentration has an inhibitory effect on the methane bacteria. Therefore, in the operation of the reactor, the VFA in the effluent is used as an important control index. When the VFA is too low, the materials that can be used by methane are reduced, and the decomposition degree of organic matter in the anaerobic reactor is low. When the VFA exceeds the amount that can be used by methane bacteria, it will cause excessive accumulation of VFA, which will lead to the decreased of the pH in the reactor and affect the normal function of methanogens. At the same time, when methanogen is damaged by various reasons, the utilization rate of VFA is reduced, which in turn causes the accumulation of VFA and forms a vicious circle.

The relationship between the pH value of the effluent and the VFA during the startup process of the 1# and 2# reactors is shown in Fig. 6. The pH value of the effluent of the 1# reactor is initially 7.5~8.5 and decreased to 6.5 on the 41st day (Fig. 6a), while the PH value of the 2# reactor effluent is initially 7.5~8.5 and basically remains stable in the range of 7–7.5 (Fig. 6b). During the startup process, the VFA value of the effluent of 1# reactor has been increasing from 120 to 700 mg/L (Fig. 6a), while the VFA value of the effluent of 2# reactor has only increased to 620 mg/L. This result indicated that the 2# reactor has more active methanogen than the 1# reactor and has a better operating environment.

3.4. Changes in effluent PH value, influent water quality, and effluent VFA/ALK

VFA/ALK can reflect the accumulation degree of intermediate metabolites in the anaerobic treatment system [29-31]. The VFA/ALK and PH of the reactor effluent were further observed, as shown in Fig. 7. The VFA/ALK of the 1# reactor effluent is in the range of 0.15–0.22 on day 5-23. On day 23-41, VFA/ALK gradually increases to 0.3. After day 49, it sharply increases to exceed 0.4, and then increases to the maximum value of 0.5. Meanwhile, the PH value also decreases to 6.2, indicating abnormalities have occurred in the internal system of the 1# reactor, the intermediate metabolites cannot be decomposed and used by methanogens in time, and measures need to be taken to solve the problems. The VFA/ALK effluent of the 2# reactor is basically stable in the range of 0.15–0.3, the pH value is stable at 7-8, and the system operates normally. The results indicated that the methanogen can normally digest in the 2# reactor with the added diatomite earth during the startup. In contrary, the 1# reactor without the addition of diatomite earth collapsed under the influence of high salinity and high concentration of organic wastewater.

Based on the comparison on the relationship between COD, salt content in the influent water in the influent water, and VFA/LAK in Fig. 8, it can be seen that the VFA/ALK of the 2# reactor effluent can be kept below 0.3 under the



Fig. 6. Changes in effluent pH and VFA: (a) 1# reactor and (b) 2# reactor.

condition that the COD and salt concentration in the influent water gradually increase. In contrary, the VFA/ALK of the 1# reactor effluent increases sharply after the 47th day and eventually reaches 0.5, indicating system anomalies. Therefore, it is further demonstrated that the 2# reactor has higher salt resistance and higher organic load resistance than 1# reactor.

3.5. Comparison of size of sludge particles

After the startup process was complete, the sludge was collected at the sampling port of the 2# reactor, and it was observed that the sludge was obviously granulated (Fig. 9b). In contrary, no particles were formed in the 1# reactor, it is flocculent sludge (Fig. 9a). The particle size of the sludge from the 2# reactor was analyzed to be in the range of 2–5 mm, as shown in Fig. 10.

3.6. Role of waste diatomaceous earth

Due to the high porosity of the diatomaceous earth in the brewery, it has a unique porous structure and is a good carrier for the growth of sludge. The yeast in the diatomaceous earth is also a catalyst for the growth of anaerobic bacteria. The sludge in the IC reaction tower grows rapidly in short time and achieves the treatment effect of the IC reaction tower at the fastest speed. In addition, due to the weight of the diatomite earth and the mutual aggregation effect, the anaerobic sludge produces particles. The formation of sludge particles is related to EPS [32], and the nutrients adsorbed on the surface of the diatomaceous earth promote the formation of sludge particles, thereby improving the removal effect of COD.

The porous structure of diatomite earth allows microorganisms to accumulate. When the diatomite earth is impacted by high-salt wastewater containing sulfate ions, the protective film outside the diatomite can play an anti-impact effect. The carrier is conducive to protecting the basement membrane and prolonging the residence time of sludge, which is also reflected in reference [33,34]. In addition, due to the large porosity, the diatomite earth has a larger surface area at the same volume, so that the treatment load can be greatly improved. As the operating time



Fig. 7. Comparison of relationship between PH value of effluent and VFA/ALK.



Fig. 8. Comparison of the relationship between influent water COD, salinity, and VFA/LAK.



Fig. 9. On-site photos of granular sludge: sludge in (a) 1# reactor and (b) 2# reactor.



Fig. 10. Particle size of sludge in 2# reactor (minimum scale:mm).

of the IC reactor increases, the microorganisms inside the diatomaceous earth produce some salt-loving bacteria, thus ensuring the subsequent stable operation of the IC reactor.

4. Conclusions and recommendations

• The IC reactor with the addition of waste diatomaceous earth from the brewery started up after 50 d. Under the condition of high salt-containing fatty acid influent COD concentration of 15,000 mg/L and salt content of 12,000 mg/L, the effluent COD concentration was around 1,500 mg/L, and the COD removal rate reached 90%. However, the COD removal rate of the IC reactor without the addition of diatomaceous earth from the brewery was reduced to 77%, and the best performance was obtained under the conditions of influent COD concentration of 10,000–11,000 mg/L and the salt content of 8,000–9,000 mg/L. The results demonstrated that the IC reactor with the addition of brewery waste diatomaceous



earth was more resistant to high salt and high load than the IC reactor without the addition of the diatomaceous earth.

- The IC reactor with the addition of waste diatomaceous earth from the brewery had better performance in forming particulate sludge than the IC reactor without the addition of waste diatomaceous earth.
- When the brewery diatomaceous earth was added into the IC reaction tower, the porous structure of the brewery's diatomaceous earth can be used as a "sequestration bed" for sludge growth and reproduction. Meanwhile, the active enzymes on the diatomaceous earth had "catalysis" effect for sludge reproduction, which shortened the startup process of the IC reaction tower to half (from the original 90–50 d), which can indirectly improve the production efficiency of the enterprise. At the same time, the waste is recovered and comprehensively utilized, which is suitable for the startup of the treatment of high-salinity and high-concentration organic wastewater.

• Due to the high salt content in the influent water, although the waste diatomite earth from the brewery was added to enhance the startup of the IC reactor, the COD treatment load was still only 4.5 kg COD/m³ d. The COD treatment load was far lower than other waster loads treated by the IC reactor, and the advantage of the IC reactor was not exerted. Therefore, further research is needed to increase its load.

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186