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Enhancement of anaerobic digestive efficiency by the use of exchange resin to remove cations in sewage sludge

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ABSTRACT

Hydrolysis is considered as the rate-limiting stage of anaerobic digestion which is one of the most applied stabilization processes in the disposition of sludge. It is urgent to accelerate the hydrolysis of the sludge particles and improve its biodegradability. This study utilized cation-exchange resin (CER) to adsorb divalent cations in the supernatant of activated sludge with the purpose of making the sludge floc disintegrated. The results showed that the biopolymers incorporated in the tightly bound extracellular polymeric substances can be released to the bulk using CER to remove cations. However, the lack of essential elements led to a much lower methane yield of treated sludge than that of activated sludge. The treated sludge got a higher methane production rate constants after added Fe^{2+}. It is necessary to add Fe^{2+} or regenerated liquid of resin-containing essential elements in order to maintain the activities of microbial life.

Keywords: Cation-exchange resin; Anaerobic digestion; Disintegration; Methane production rate

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1. Introduction

The treatment of municipal wastewater results in a great quantity of sewage sludge to be disposed of reduction, hygienization, and stabilization. Anaerobic digestion is an attractive method to stabilize sewage sludge for its ability to achieve both pollution control and energy recovery [1]. Meanwhile, after aerobic treatment in a wastewater treatment plant (WWTP), most of the residual biodegradable material is either enclosed inside the microbial cell wall [2] or enmeshed in an extracellular polymeric matrix [3], which reduces its availability to anaerobic micro-organisms and further contributes to limit the volatile solids (VS) reduction of these sludges from 35 to 45% [4]. When degrading this kind of complex organic solid waste, the biological hydrolysis is so difficult that it is usually considered as the rate-limiting stage. A size reduction of the particles and the resulting enlargement of the available specific surface can support the biological process by reducing the technical digestion time [5]. Hence, further studies are needed to accelerate the hydrolysis phase and improve the anaerobic digestive efficiency.

In order to reduce the impact of the rate-limiting stage and improve the digestive efficiency, an

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extensive work has been done on pretreatments for sludge by solubilizing and disintegrating the decomposable organic substances in the sludge. Various pretreatment methods, such as mechanical [6], ultrasonic [7,8], microwave [2], chemical [9], and thermal [9], have been proposed to accelerate the hydrolysis of the sludge particles with the purpose of improving its biodegradability under anaerobic conditions. Nevertheless, the methods mentioned above are all not perfect and have some disadvantages to overcome.

Previously, it has been reported that extracellular polymeric substances (EPS) is a major sludge floc component holding the floc together in a three-dimensional matrix [10] formed by extracellular polymers and bivalent cations [11,12]. Higgins and Novak [13] have shown that excess bivalent cations can cause an improvement in floc structure and settling properties. Some researchers found that qualitatively different polymeric materials could be extracted through reducing the salt level of the sludge by filling and drawing elution with distilled water [14]. Hence, it is necessary to find out an approach to remove most of the bivalent cations in the sludge floc which could be called deflocculation; then the cell components and other organic matter can be released from internal microbial floc to enhance the anaerobic digestive efficiency.

Resins, which combine both chemical and physical mode of cation extraction, were first used by Rudd et al. [10] to extract EPS. Frølund et al. demonstrated that using a cation-exchange resin (CER) can extract more EPS from waste-activated sludge (WAS) as compared with mechanical and/or chemical methods [15]. So, the CER is a good choice to achieve the removal of the bivalent cations in the sludge floc. However, existing researches used the CER as a method of EPS extraction from WAS rather than a pretreatment method of sludge in anaerobic digestion.

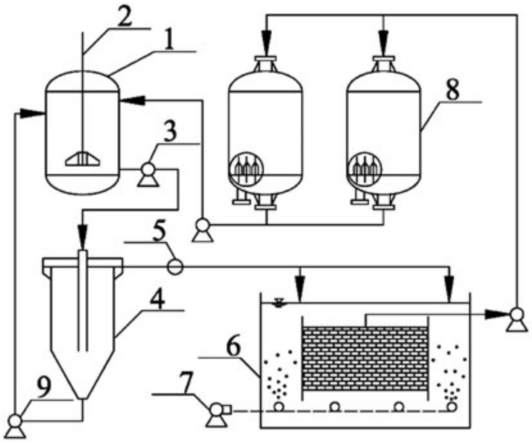


Fig. 1. Experimental device: 1—contact reactor; 2—stirring system; 3—peristaltic pump; 4—settling tank; 5—metering pump; 6—membrane system; 7—aerator; 8—resin softener; and 9—peristaltic pump.

In this investigation, CER is used to remove the bivalent and trivalent cations in the supernatant of the sewage sludge, which can promote the diffusion of the cations enmeshed in the extracellular polymeric matrix or even enclosed inside the microbial cell wall by the concentration gradient between the supernatant and sludge floc. Then, a new device using CER is designed in order to increase the soluble organic matter content and enhance the anaerobic digestive efficiency. Based on this device, the removal of cations in different floc fractions was researched; the shift of organic matters in different floc fractions was studied; and the enhancement of the anaerobic digestive efficiency was investigated.

2. Materials and methods

2.1. Reactors and operation

This study was carried out in a lab-scale experimental device (Fig. 1). The device consists of a contact reactor followed by a settling tank, a membrane system and a resin softener. Contact reaction happened in a 6.0l cylindrical vessel which was continuously stirred by a paddle, and the stirring intensity of the stirring system is 100 rpm. The hydraulic retention time (HRT) of the contact reactor was 1 h. The settling tank was cylindrical with a radius of 0.1 m and an effective volume of 7.0l, respectively. The membrane system held 0.1 m^2 flat-sheet membranes (SINAP, porous size of 0.1 μm). The CER was a chelating ion-exchange resin in the sodium form (IRC748, Rohm and Haas, German). There were three resin columns, and the bed volume and height of each resin column were 300 cm^3 and 500 mm, respectively.

First, the feedstock (raw sludge) was put into the contact reactor. Then, a peristaltic pump (BT100-2 J, Baoding Longer, China) was used to convey the sludge to the settling tank. After sedimentation, the sediments were conveyed back to the contact reactor by another identical peristaltic pump, while the supernatant was filtrated through the membrane system to the resin softener. During this run, the membrane module was cleaned through aeration. The bivalent and trivalent cations in the supernatant were removed by CER in the resin softener; after that the filtrate returned to the contact reactor. The device was operated with HRT of 1.0 h during the sampling campaign. A control group was setup in another cylindrical vessel, the same as the contact reactor, and stirred in the same stirring intensity. The sludge in the control group was not treated by CER system and only influenced by the shear force. So, the influence of shear force can be deducted from the experimental group.

To start the device, 6.0l activated sludge was added into the contact reactor. Meantime, the settling tank and membrane system were filled with the supernatant of the activated sludge. During the operation time, the sludge in the settling tank was returned back to the contact reactor by the peristaltic pump continuously.

2.2. Feedstock

Raw sludge for the treatment was collected from Quyang WWTP (Shanghai, China). The plant treats mainly domestic wastewater, and the treatment process is PASF (remove phosphorous by activated sludge and biofilm technology). The sludge was collected from the aeration tank. Characteristics of the feedstock are listed in Table 1.

2.3. Biochemical methane potential test

2.3.1. Seed inoculum

The mesophilic seed sludge for biochemical methane potential (BMP) test was the samples from a single-stage completely mixed semi-continuous anaerobic digester with a liquid working volume of 6.0l. The digester has been operated to treat dewatered sludge of feeding TS 20% under mesophilic conditions with SRT of 20 d for 3 months. Characteristics of the inoculums are listed in Table 1.

2.3.2. BMP assay

The BMP test was conducted using the method of Owen et al. [16] with a few modifications. Biodegradability assays were realized in batch mode in 1L conical flasks. Twenty grams of anaerobic sludge was put in each bottle as inoculum. Biomass and inoculum quantity were prepared according to a calculated ratio 2:1 (biomass to inoculum) based on their VS content. All media preparations and transfers were performed in an atmosphere of nitrogen and carbon dioxide (70:30, v/v). Sealed bottles were maintained at 35 ± 1℃ under shaking (220 rpm). Each assay was performed in triplicate and accompanied with blank controls containing only inoculated medium. Biogas production was measured in each test bottle. Enhancement of biodegradability was evaluated by comparing the biogas volumes produced by treated (experimental groups) and untreated (control groups) samples.

When the BMP test is deficient in nutrients, necessary nutrients, micronutrients, and vitamins are needed for the optimal function of anaerobic microorganisms. The chemicals given below were concentrations in g/l in assay vessel of BMP test: $FeCl_2 \cdot 4H_2O$, 2; $MnCl_2 \cdot 4H_2O$, 0.05; $CoCl_2 \cdot 6H_2O$, 0.05; $NiCl_2 \cdot 6H_2O$, 0.092; $CaCl_2 \cdot 2H_2O$, 5; $MgCl_2 \cdot 6H_2O$, 10; $AlCl_3$, 0.05; $ZnCl_2$, 0.05; $CuCl_2 \cdot 2H_2O$, 0.038 [17].

2.3.3. B_0 and k

The degradation of each sample was assumed to follow a first-order rate of decay [18]. Thus, the production of methane was assumed to follow the equation

$$B = B_0\left(1 - e^{-kt}\right) \qquad (1)$$

where B is the cumulative methane yield at time t. B_0 was assumed to equal the final B (after 100 d of fermentation). k is the methane production rate constants and estimated by taking the reciprocal of the time from the start of the BMP assay until B equals to 0.632 B_0 [19]. k can be calculated using a fitting software called CurveExpert (version 1.4) with cumulative methane yields of all periods imported.

2.4. EPS extraction

A heat extraction method [20,21] was modified to extract the EPS extractions from the activated sludge. In brief, activated sludge samples (50 ml) were collected from the reactor and first centrifuged at 6,700 rpm and 4℃ for 15 min; then the bulk solution which composed of soluble organic matters was

Table 1
Characteristics of the feedstock and the inoculums

Parameters	pH	TS (%, w/w)	VS/TS (%)	MLSS (mg/l)
Feedstock	7.8 ± 0.1	1.08 ± 0.05	63.2 ± 0.5	10,473 ± 100
Inoculums	7.8 ± 0.1	13.5 ± 0.2	40.1 ± 0.5	–

All measurements were made in triplicate for each sample.

collected. Subsequently, the collected bottom sediments were re-suspended to their original volume using the NaCl buffer solution (0.5% w/w) that had a similar salinity to the solution in the activated sludge reactors which was preheated to 70℃, mixed at 500 rpm for 10 min, and next the suspensions were centrifuged again at 6,700 rpm and 4℃ for 15 min with the bulk solution, and the solid phase was collected separately. The organic matter in the bulk solution was readily extractable EPS and was regarded as the LB-EPS of the biomass. The collected sediments were re-suspended to its original volume of 50 ml again by the aforementioned NaCl solution at room temperature and heated in a water bath at 60℃ for 30 min. Then, it was mixed at 500 rpm for 10 min, and next the suspensions were centrifuged again at 6,700 rpm and 4℃ for 15 min with the bulk solution, and the solid phase was collected separately. The organic matter in the bulk solution was the tightly bound EPS (TB-EPS), while the residues (solid phase) that re-suspended again with the aforementioned buffer solution to the original volumes were the pellets. The pellets fraction is mainly formed by the cells in the residue after EPS extraction. After all the EPS fractions were extracted, membranes of 0.45 μm polytetrafluoroethylene were used to filter out the particulates present in the soluble, LB-EPS, and TB-EPS solutions.

2.5. Analytical methods

The analysis of metal cations concentration (including 16 cations) in different EPS fractions was carried out using ICP method (Agilent 720ES, USA).

Chemical oxygen demand (COD) was measured using HACH methods.

TS and VS concentrations were measured according to the procedures in standard methods [22].

All analyses were carried out in triplicate. The equation used to calculate the standard deviation (SD) is as follows:

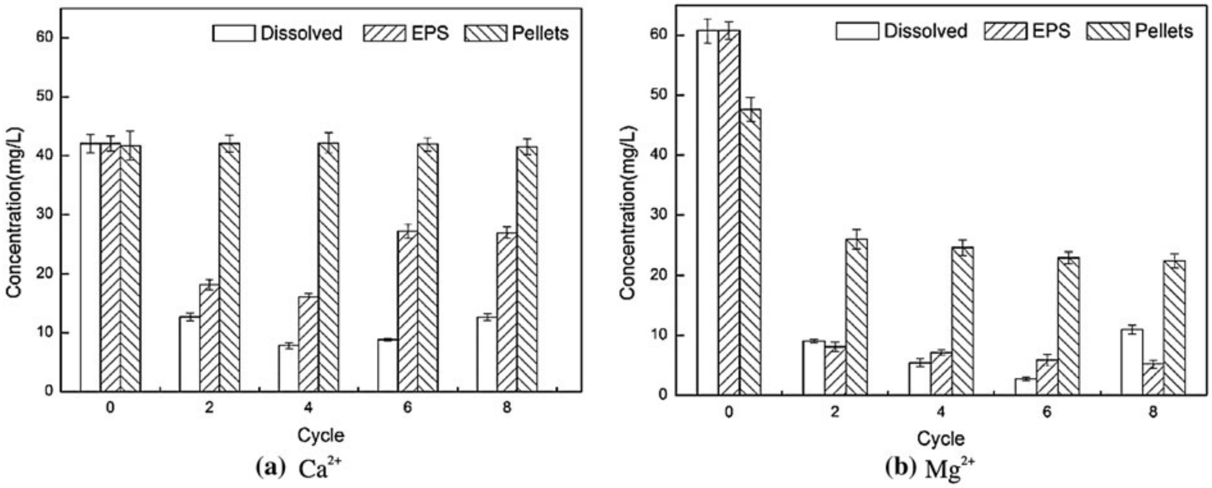
$$s = \sqrt{\frac{\sum (x_i - x)^2}{n - 1}} \tag{2}$$

where s is the SD, x_i is the value of measurement, x is the sample mean, n is the number of measurements. Sample mean was calculated as the rate of the sum of all measurements and the amount of measurements carried out.

3. Results and discussion

3.1. Device utilization in cation adsorption and organic matters release

Cations especially bivalent cations are an integral part of the floc structure, linking separate biopolymer components together into floc structure [11,12]. To confirm the effect of CER, the distribution of cations in different EPS fractions before and after treated was measured. The results showed that the main cations in the sludge were Ca^{2+} and Mg^{2+}. The distribution of Ca and Mg in different EPS fractions is shown in Fig. 2. And the distribution of all nine cations (Ca, Mg, Al, Fe, Zn, Mn, Cu, Co, and Ni) in activated sludge is shown in Table S1. The distribution of Ca,



(a) Ca^{2+} **(b)** Mg^{2+}

Fig. 2. The distribution of Ca and Mg in different EPS fractions. (a) Ca and (b) Mg. (Error bars: one SD. All measurements were made in triplicate for each sample.)

Mg, Al, and Fe in different EPS fractions of experimental groups after 8 cycles is shown in Table S2.

As shown in Fig. 2(a), Ca^{2+} was basically uniformly distributed in dissolved EPS and pellets of activated sludge. During the CER treatment, Ca^{2+} in dissolved form and EPS form was removed as 81.3 and 61.8%, respectively, after 4 cycles (maximally), while in the pellets, it was slightly removed. When compared with Ca^{2+}, the CER treatment could achieve a higher removal proportion of Mg^{2+}, and above 50% of Mg^{2+} can be removed even in the pellets. This result is consistent with the report of Frølund [15]. This result indicates that the method in this paper can remove most of the bivalent cations, such as Ca^{2+} and Mg^{2+}, in dissolved form and that enmeshed in an extracellular polymeric matrix. For Mg^{2+}, the method can even remove half of them enclosed inside the microbial cell wall.

The content of organic matters in different EPS fractions of activated sludge, control group, and experimental group is shown in Fig. 3. As shown in Fig. 3, organic matter (COD) of activated sludge was mainly (80.5%, respectively) in the TB-EPS fraction. The organic matter enmeshed in the TB-EPS fraction is hard to be hydrolyzed, and it is necessary to transfer the organic matters in the TB-EPS fraction to the LB-EPS and the soluble fractions. In the experimental group, dissolved organic matter increased fourfold (from 4.5 to 22.7%) than the original activated sludge after the treatment of the device described in 2.1. Meantime, organic matters in the LB-EPS also increased, and organic matters in the TB-EPS were decreased. While in the control group, a small portion of organic matters in the TB-EPS was transferred to the LB-EPS, and the soluble fractions only increased a little (from 4.5 to 8.6%) after being stirred. This suggested that the method in this paper could promote the disintegration of sludge floc and release the biopolymers incorporated in the TB-EPS to the supernatant where the most organic matter is soluble.

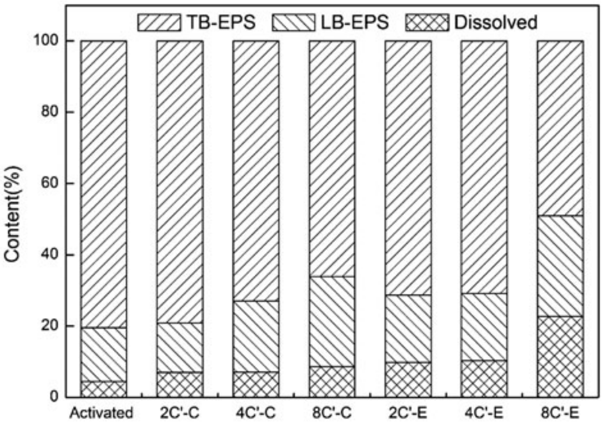


Fig. 3. The proportion of COD in different EPS fractions: Activated—activated sludge; C′—cycle; C—control group; and E—experimental group.

3.2. *Anaerobic digestive efficiency of treated sludge*

A temporal plot of the cumulative methane yields, corrected for their inoculum methane yields, of the activated sludge, control groups, and experimental groups is shown in Fig. 4. The control groups had a higher methane potential and biodegradability than activated sludge and could produce methane continuously in 160 h as well as activated sludge. However, experimental groups produced methane only in the first 25 h, no matter it was after 4, 6, or 8 cycles (Fig. 4).

According to the results mentioned above, it may imply that the CER not only removed the calcium and magnesium, but also some trace elements which are necessary for micro-organism such as iron, aluminum, and so on. The availability of certain trace elements has been reported to impact the biogas production strongly. Some trace elements are crucial for the activity of enzymes in methanogenic systems. A suitable concentration of trace metals could enhance the anaerobic digestion and stimulate the methanogenic activity. Adding moderate metal ions to anaerobic digestion system can accelerate the degradation of organic matter and enhance the methane production [23]. So, it is necessary to add trace elements into the system, and find out the cation that plays a key role in anaerobic digestion.

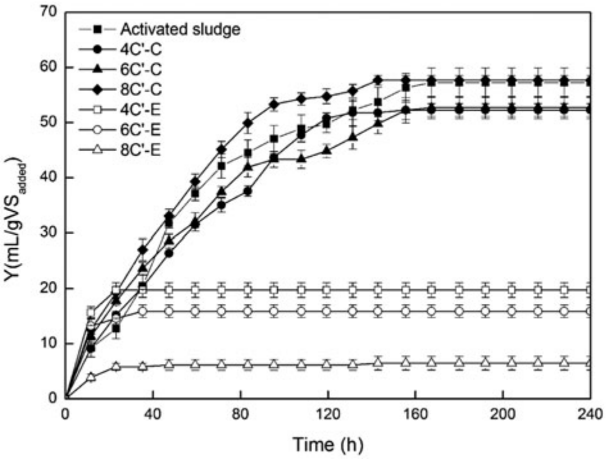


Fig. 4. BMP cumulative methane production of activated sludge, control groups, and experimental groups. (Error bars: one SD. Each assay was performed in triplicate for each sample.)

3.3. Improvement of anaerobic digestion efficiency by cation addition

Several researches indicated that the anaerobic digestion might be limited or inhibited if metals such as Cu, Co, Fe, Mn, Mo, Ni, and Zn were not properly added or overdosed [24–26]. So Fe, Mn, Co, Ni, Ca, Mg, Al, Zn, and Cu were added individually to experimental groups after 8 cycles. The concentrations of the nine cations are listed in material and methods (2.3.2).

The temporal plots of the cumulative methane yields of the activated sludge, experimental groups, and experimental groups which added Fe, Mn, Co, Ni, Ca, Mg, Al, Zn, and Cu and all nine cations are shown in Fig. 5. The results of experimental group which added Fe and all nine cations, including methane yields data and methane production rate constant of activated sludge, are summarized in Table 2. As shown in Fig. 5, the methane potential and biodegradability of experimental groups which added Co, Mn, Al, Zn, and Cu were just a little higher than the experimental groups which added no elements, while experimental groups which added Ni were less. Meanwhile, experimental groups which added Ca and Mg were higher than the experimental groups which added no elements but still lower than activated sludge. And the methane potential and biodegradability of experimental group which added Fe is the highest in the all 13 groups. The methane production rate constant of experimental groups which added Fe and all nine cations was much higher than the untreated sludge in all the 190 h (Table 2). Though the experimental group which added all nine cations had nearly the same ultimate methane yield as activated sludge, it needed 110 h only to reach the ultimate methane yield while 190 h for activated sludge. In addition, the experimental group which added Fe had the highest methane production rate constant (Table 2). The relatively lower ultimate methane yield of experimental group which added all nine cations than that added only Fe may be attributed to the excess to threshold values of another eight cations, resulting in the inhibition of biogas production [25].

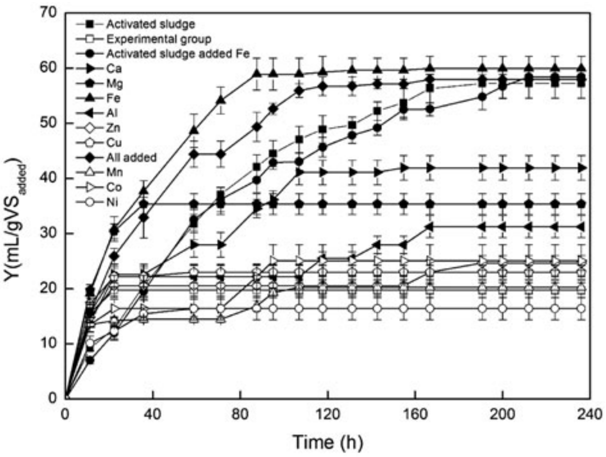


Fig. 5. BMP cumulative methane production of activated sludge, experimental groups after 8 cycles, and experimental groups after 8 cycles which added Fe, Mn, Co, Ni, Ca, Mg, Al, Zn, and Cu and all nine cations. (Error bars: one SD. Each assay was performed in triplicate for each sample.)

Table 2
Methane production rate constant of activated sludge and experimental group which added Fe and all nine cations (Mean ± SD)

Sample	Ultimate methane yield (ml/gVS$_{added}$)	Methane production rate constant (d^{-1})
Activated sludge	57.23 ± 2.65	0.277 ± 0.019
Experimental group added Fe	59.96 ± 2.20	0.725 ± 0.047
Experimental group added all nine cations	57.97 ± 0.86	0.560 ± 0.023

The results indicate that the CER removed some trace elements which are necessary for micro-organism such as iron, and then due to the lack of trace elements, the experimental groups could not reach the similar ultimate methane yield as activated sludge. When added Fe, the experimental groups could reach the similar ultimate methane yield as activated sludge and a higher methane production rate constant thanks to the increase of dissolved organic matters.

4. Conclusions

The device in this paper using CER to remove cations can promote the hydrolysis and solubilization of the particulate organic substances, which increased the methane production rate of the sludge. However, due to the lack of elements such as Fe, the experimental groups could not reach the similar ultimate methane yield as untreated sludge. Hence, it is needed to add Fe or eluate of resin-containing essential elements into the treated sludge in order to maintain the activities of microbial life.

Supplementary material

The supplementary material for this paper is available online at http://dx.doi.10.1080/19443994.2015.1007171.

Acknowledgments

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