



# New insight into fouling behavior and foulants accumulation property of cake sludge in a full-scale membrane bioreactor



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## ARTICLE INFO

### Article history:

Received 30 November 2015

Received in revised form

24 February 2016

Accepted 25 February 2016

Available online 2 March 2016

### Keywords:

Full-scale membrane bioreactor

Cake sludge

Fouling behavior

Extracellular polymeric substances

Microbial community structure

## ABSTRACT

Cake sludge attached on membrane surfaces was collected and characterized in a full scale membrane bioreactor (MBR) compared with bulk sludge. The morphological, chemical and microbial properties were examined through microscopic observations, particle size distribution (PSD) analysis, chemical analysis, Fourier transform infrared (FTIR) spectroscopy, scanning electron microscopy (SEM) and energy-dispersive X-ray (EDX) analysis, specific oxygen utilization rate (SOUR) measurements and Biolog assay. The results showed that fiber-like substances might have served as the skeleton of larger size aggregates in cake sludge. Moreover, much more polysaccharides and inorganic elements such as multivalent cations were accumulated in cake sludge than proteins and humics. Cake sludge showed lower microbial activity for aerobic degradation than bulk sludge, but higher metabolic activity for the degradation of refractory substances (aromatic proteins and humics) other than polysaccharides. Based on batch filtration experiments, it was found that cake sludge had much higher cake layer fouling potential but lower membrane pore blocking resistance, probably due to the heterogeneous structure of cake sludge resulting from accumulation and interaction of various inorganic and organic foulants. This investigation could assist in obtaining a better understanding of the fouling behavior and foulants accumulation properties of cake sludge in the full-scale MBRs.

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## 1. Introduction

Recently, significant progress has been achieved in both research and application of membrane bioreactors (MBRs), which exhibit several advantages over conventional activated sludge processes during wastewater treatment and reclamation by adopting microfiltration/ultrafiltration for solid/liquid separation. However, membrane fouling remains not well resolved, which contributes considerably to the operating and maintenance costs and limits the more widespread applications of MBRs [1,2]. Membrane fouling is caused by the complex interaction between the membrane and mixed liquor component, including suspended solids, colloids, biopolymers, and solutes, which is introduced from raw wastewater or produced during biomass growth and decay [3].

The contributions of different sludge fractions to membrane fouling have been extensively studied [4–8]. Among the complex foulants, biopolymers, including soluble microbial products (SMP)

and extracellular polymeric substances (EPS), are widely accepted as primary fouling-causing substances [4]. It was found that due to the membrane rejection effect, SMP was more easily accumulated in MBRs, resulting in the poor filterability of sludge suspension [5]. Additionally, in a lab-scale submerged anaerobic MBR, it was observed that fine particles and a higher level of EPS preferentially accumulated in the cake sludge compared with bulk sludge [6]. Moreover, inorganic elements, especially metal cations (Mg, Al, Fe and Ca), were found to accumulate in cake sludge and enhance cake layer formation [7,8]. The above analysis indicated the important effects of various foulants on cake layer formation.

Nevertheless, cake layer formation on the membrane surface was inevitable, and cake layer fouling accounted for over 80% of the total filtration resistance in most MBR studies [9–11]. During long-term filtration, there is a continuous increase in filtration resistance caused by the accumulation and compression of the cake layer. Thus, recent efforts have been devoted to characterizing the cake layer, including the physical morphology and structure [9,12], chemical composition [8] and microbial properties [13], theoretical analysis and modeling [12]. One study using a series of analytical methods showed that structures, EPS concentrations

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and microbial communities changed significantly along the cake depth, and the continuous accumulation and interaction of foulants on the membrane surface decreased cake layer porosity, which was closely related to the TMP increase [10]. It was also reported that cake sludge had a specific filtration resistance nearly three orders of magnitude higher than that of bulk sludge [11], which was mainly due to the accumulation of the biopolymer clusters (BPC) within the cake sludge pores. Thus, the cake layer can be defined as a porous and inhomogeneous media formed on membrane surfaces due to the adsorption, deposition and accumulation of various foulants [14]. The above lab-scale/pilot-scale studies indicated that cake sludge has quite different fouling behavior from that of bulk sludge due to foulant accumulation, especially in MBRs during long-term operation; thus, it is highly appropriate to comprehensively characterize cake sludge to facilitate cake layer fouling control in MBR applications.

Most of the studies involving cake sludge characterization were conducted in lab-scale/pilot-scale MBR systems mainly fed with synthetic wastewater. Therefore, the results obtained may be inappropriate to be extended directly to full-scale MBR applications because of the difference in fouling behavior between lab-scale/pilot-scale and full-scale MBRs, as confirmed by several researchers [4,15], and also limited work has been done in full-scale MBR until present. Thus, the objective of this study is to investigate the characteristics of cake sludge in terms of fouling behavior, foulant accumulation and microbial properties in a full-scale MBR treating domestic wastewater for approximately four years by various analytical methods. The results obtained in this study are expected to provide new insights into cake sludge properties and membrane fouling control in full-scale MBR applications.

## 2. Materials and methods

### 2.1. The full-scale MBR system and operation

The MBR system with a treating capability of 2000 m<sup>3</sup>/d for domestic wastewater treatment and reclamation is located in the university campus of Xi'an, China. The biological treatment unit was divided into four sequential zones, namely, the anaerobic tank (150 m<sup>3</sup>), the anoxic tank (420 m<sup>3</sup>), the oxic tank (480 m<sup>3</sup>) and the MBR tank (120 m<sup>3</sup>), in series (Fig. S1 of Supporting information). 216 submerged PVDF hollow fiber membrane modules (MUNC-620AII type, Asahi Kasei Chemicals Corp., Ltd., Japan) with a nominal pore size of 0.1 µm and total membrane area of 5400 m<sup>2</sup> were installed in the MBR tank. The effluent was extracted by suction pumps at a fixed flow rate (16 L/m<sup>2</sup> h) under an intermittent operation mode (9 min on/1 min off). Air was monitored by flow rate meters and continuously supplied through the air diffusers to the oxic tank (2000 m<sup>3</sup>/h) and MBR tank (2000 m<sup>3</sup>/h) to provide oxygen demanded by microorganisms and scour the membrane surface, respectively. Accordingly, the concentration of dissolved oxygen in the oxic tank and MBR tank was in the range of 2–5 mg/L and 4–7 mg/L, respectively. After pretreatment by screening (coarse screen and fine screen with bar clearances of 5 mm and 1 mm, respectively) and regulation, domestic wastewater collected from the university campus was supplied into the AAO-MBR system. The characteristics of the raw wastewater can be found elsewhere [16]. The system was equipped with a programmable logic controller (PLC) for automatic control of the operation, including equipment involved in the influent, aeration, sludge recirculation and effluent. TMP was monitored by an on-line pressure gauge. The hydraulic retention time (HRT) was approximately 12.5 h, and the sludge retention time (SRT) was set at 20–40 d.

### 2.2. Analytical methods

#### 2.2.1. Cake sludge collection and fractionation

Cake sludge formed on the membrane surface was scraped off by using a plastic sheet as the membrane module was taken out for physical cleaning, which was operated in accordance with the reported methods [7,10]. The exact time period for sampling was between June and July 2015. All of the collectable cake sludge from 12 membrane modules was collected to address the observed problem of an uneven distribution of cake layers on the membrane surface when the modules were lifted for physical cleaning (once per year). Additionally, within the sampling period, cake sludge was sampled more than three times from different membrane modules. The collected sample was diluted with deionized water to achieve the same MLSS level as that of bulk sludge, and then was placed on a magnetic blender and gently mixed. However, it was noted that after a long period (several hours to days) of mixing, a certain amount of large particles remained in the mixed cake sludge, which disrupted the accurate analysis of cake sludge properties, due to the difficulty in sampling. Therefore, further fractionation of cake sludge was proposed and conducted by using a series of stainless steel sieves, with sieve pore sizes of 2 mm, 0.5 mm and 0.2 mm, respectively. In addition, the effect of the proposed fractionation method on cake sludge properties is discussed in a subsequent section.

#### 2.2.2. EPS extraction and analysis

EPS extraction from bulk sludge and cake sludge samples was performed using the thermal treatment method [16]. The content of the extracted EPS samples was analyzed in terms of proteins and polysaccharides. Proteins were quantified using the modified Lowry method, in which bovine serum albumin was used as the standard [17]. The polysaccharide concentration in EPS was determined according to the anthrone method, in which glucose was used as the standard [18].

#### 2.2.3. PSD analysis

PSD of the cake sludge and bulk sludge was analyzed using a laser granularity distribution analyzer (LS 230/SVM+, Beckman Coulter Corporation, USA) with a detection range of 0.4–2000 µm. The detailed pretreatment for cake sludge before the PSD analysis is described in Section 2.2.1. Three measurements of each sample were taken and typical PSD curves were reported.

#### 2.2.4. Batch filtration experiment

The filtration properties of cake sludge and bulk sludge were evaluated by batch filtration experiments using a stirred dead-end cell (MSC300, Mosu corp., Shanghai, China) [19,20]. To avoid the effect of MLSS on filterability of different sludge samples, the MLSS concentration of raw cake sludge was diluted to the same level as that of bulk sludge (MLSS=4 g/L). The membranes employed for filtration were hydrophilic polyvinylidene fluoride (PVDF) flat sheet membranes with a nominal pore size of 0.1 µm and an effective membrane area of 28 cm<sup>2</sup> (VVLP9050, Millipore Corp., USA), which were the same material and pore size as the membrane used in the full-scale MBR process. Nitrogen gas was adopted to allow for a constant pressure for filtration. The permeate flux data were continuously logged using a top-loading electronic balance (Sartorius, BSA2202S, Germany) connected to a personal computer. Prior to each filtration, each of the new membranes was soaked in ultrapure water for 24 h and further cleaned through filtering ultrapure water for 30 min at a pressure of 25 kPa to remove any impurities and to stabilize the permeate flux. The filtration pressure was maintained constant at 10 kPa and the stirring speed in the cell was set at 200 rpm throughout the experiments. All of the experiments were conducted at room

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