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# Dynamic membrane bioreactor for wastewater treatment: Operation, critical flux, and dynamic membrane structure

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## ABSTRACT

This research investigated the characteristics of the dynamic membrane bioreactor (DMBR) for wastewater treatment, including operation performance, critical flux of dynamic membrane (DM), and structure of the cake layer. Various analytical methods were used. The DMBR exhibited excellent pollutant-removal efficiencies, and the critical flux of DM was in the range of 70–75 L/m<sup>2</sup>h. The DM operated at sub-critical flux exhibited longer filtration time and smaller flux decline in the filtration process. The DM formed by activated sludge exhibited a hierarchical structure, and the order of mean particle size of three cake layers was as follows: top cake layer < middle cake layer < bottom cake layer. Protein (PN) was the major part of extracellular polymeric substances (EPS) in the three layers and its concentration did not show obvious decline from the top cake layer to the bottom cake layer, whereas the quantity of polysaccharides (PS) exhibited an increased tendency, which was also proved by confocal laser scanning microscopy (CLSM) image analysis. Total EPS (T-EPS) content did not show obvious variations among the three cake layers. The middle cake layer contained the most high-molecular weight (MW) substances with MW larger than 160 kDa and the bottom cake layer contained the lowest high-MW substances. The tight bound EPS (TB-EPS) was the major content of T-EPS in the residual substances on the backwashed stainless steel mesh. The quantity of PN attached to the mesh was much greater than that of PS. More residual substances were attached on the outer support mesh. Residual substances of the inner support mesh were mainly attached on the intersection of the two stainless steel wires, whose interaction was enhanced by the inorganic ions.

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## 1. Introduction

The DM formed on large-pore support mesh is a new type of separation method, which exhibited the advantages of high filtration flux, low cost of membrane module, and easy backwash [1,2]. Compared with conventional membrane bioreactors, the DMBR could also be operated successfully combined with water treatment processes, such as the anoxic/aerobic process [3,4]. In the above-mentioned reports, DMs were not operated based on the theory of critical flux/sub-critical flux. The critical flux hypothesis for microfiltration is that on start-up there exists a flux below which a decline of flux with time does not occur; above it, fouling is observed [5]. The critical flux of microfiltration/ultrafiltration membranes is commonly determined by continuous flux or trans-membrane pressure (TMP) step methods, where membrane fouling is indicated and evaluated by the increase in filtration resistance at each step [6–8]. The critical flux of a self-forming

DM on the polyester nonwoven fabrics by activated sludge was investigated, which focused on the effect of membrane compressibility on critical flux [6]. Because the DM formation materials and the support meshes vary from case to case, the critical fluxes of different DMs should be investigated specifically.

The cake layer on the support mesh is the indispensable part in the DM separation process. The DM formed on the relatively large-pore mesh increases the intrinsic membrane retention capacity, and the fouling of DM has a different meaning. DM fouling was interpreted as follows: the initial deposited materials on the underlying support mesh are actually DM-forming components (cake layer), which are necessary and desired; the subsequent deposited materials are foulants, which induce additional increase of filtration resistance of DM [1]. Although cake layer formation is a key factor of the DM process, the studies of cake layer to date are insufficient [9]. In the previous studies, we reported that the bi-diatomite membrane exhibited dynamic semi-compressibility [10], and the biologically enhanced powder activated carbon diatomite DM performed a two-layer structure [11]. These conclusions were mainly drawn from scanning electron microscopy analyses, and specific and further researches of the DM structure are needed.

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The aim of this study was to investigate characteristics of DMBR for wastewater treatment, including operation performance, critical flux of activated sludge DM formed on the stainless steel mesh with an equivalent aperture of 38  $\mu\text{m}$ , and structure of the cake layer. Analytical methods, such as particle size distribution (PSD) analysis, Fourier transform infrared (FTIR) spectroscopy, scanning electron microscopy (SEM)-energy-dispersive X-ray analyzer (EDX), gel filtration chromatography (GFC) analysis, CLSM analysis, three-dimensional excitation-emission matrix (3D-EEM) fluorescence spectra, and extraction and chemical analysis of EPS, were adopted. This study would provide a new insight with regard to the critical flux and specific structure of DM formed on large-pore stainless steel mesh, which is necessary for the development of DM.

## 2. Materials and methods

### 2.1. Experimental methods

The DMBR with a total effective volume of 20 L consisted of three parts: anaerobic zone (5 L), anoxic zone (5 L), and aerobic zone (10 L) in sequence, as shown in Fig. S1. The anaerobic environment was created by a water seal on the top of the anaerobic zone. The flat-sheet DM module used a stainless steel mesh with an equivalent aperture of 38  $\mu\text{m}$  as the support layer, and its double effective filtration area was 0.042  $\text{m}^2$ . A synthetic wastewater recipe simulating real domestic sewage was adopted (see Supporting Information). It took 20 days for the inoculation process in the DMBR, and the MLSS in the anaerobic zone, anoxic zone, and aerobic zone were 2460–2576 mg/L, 3545–3754 mg/L, and 5558–5940 mg/L, respectively.

The DMBR was operated continuously for 157 days. Mixed liquors of the aerobic zone and the anoxic zone were recycled to the anoxic zone (recirculation R) and the anaerobic zone (recirculation r), and the recycle rates were controlled at 2 times and 1 times of the influent flow rates, respectively. The sludge retention time of the DMBR was maintained at 20–40 d by discharging the mixed liquor from the aerobic zone daily. The dissolved oxygen concentration of the anaerobic zone, anoxic zone, and aerobic zone was in the range of <0.2 mg/L, 0.3 mg/L, and 1–2 mg/L, respectively.

The operation process of DM could be divided into three stages: pre-coating, filtration, and backwash [3,12]. In the pre-coating stage, the DM was formed by activated sludge and effluent of the DM was recycled into the aerobic zone, which lasted about 20 min. In the filtration stage, filtration fluxes of 60–120  $\text{L}/\text{m}^2\text{h}$  (9 min filtration/1 min relaxation) were applied. A high-precision vacuum pressure gauge was connected to the effluent pipe to measure the TMP of DM. The filtration was stopped once the operating pressure reached 40 kPa, and air backwash was started and performed through the bottom outlet of the membrane module using an air pump (Maple Mini Air Compressor, China).

### 2.2. Analytical methods

#### 2.2.1. Analysis of constant flux

Critical flux value of DM was tested by the standard flux-step method, and a step height of 5  $\text{L}/\text{m}^2\text{h}$  and 15 min duration were chosen [8].

#### 2.2.2. Cake layer collection

At the end of the filtration stage, the DM was sampled and divided into three cake layers; top cake layer, middle cake layer, and bottom cake layer, which were obtained by gently scraping off cake layers from the membrane surface using a plastic sheet [1,9]. The top cake layer of the cake was loose and not obviously

compressed, and the middle cake layer was moderately compressed, which can be felt when using a blade to divide them [9]. The bottom cake layer was near the stainless steel mesh and constituted approximately 10% of the total cake layer thickness [1]. The three divided cake layers of the dynamic membrane were cut into horizontal slices using a blade, and the distance from the cake layer surface down to the bottom was controlled by a series of proof sticks. The residual substance (named gel layer) attached to the stainless steel mesh was brushed off with distilled water. Samples of different cake layers were mixed with de-ionized water, followed by mixing on a magnetic stirrer to form uniform liquor, and subjected to the following measurements.

#### 2.2.3. EPS extraction

EPS extraction of samples was carried out according to the reported thermal treatment method [13]. The total EPS was expressed as the sum of polysaccharides and proteins. The backwashed stainless steel support mesh was cut from the DM module and then subjected to ultrasound at 40 kHz and by 300 W for 30 min to extract the PS and PN completely. PS in EPS was measured by the anthrone method with glucose as the standard. Protein content was determined by the Folin-Phenol method with bovine serum albumin (BSA) as the standard.

#### 2.2.4. GFC analysis

EPS from the three divided cake layers of DM were filtered with a 0.45  $\mu\text{m}$  membrane, and the filtrates were fractionated by a GFC analyzer. The GFC system consisted of a TSK G4000SW-type gel column (TOSOH Corporation, Japan) and a liquid chromatography spectrometer (LC-10ATVP, SHIMADZU, Japan). Polyethylene glycols with MWs of 1215 kDa, 274.4 kDa, 128 kDa, 11.84 kDa, 6 kDa, and 194 Da (Merck Corporation, Germany) were used as standards for calibration. The elution at different time intervals was collected by an automatic fraction collector and automatically analyzed using a UV spectroscopy analyzer to obtain an MW distribution curve.

#### 2.2.5. PSD analysis

The sampled three cake layers combined with de-ionized water were well mixed on a magnetic stirrer to form uniform liquor and get particle sizes approaching those in the cake layer best. PSDs of three cake layers were tested by a focused beam reflectance measurement (EyeTech particle size and shape analyzer, Ankersmid, Holland). Mixed liquor in the aerobic zone of DMBR was also measured to verify the differences between them.

#### 2.2.6. SEM and EDX analysis

After dehydrating by natural evaporation for 48 h and coating with gold using a sputter coater, the samples were examined by SEM (XL-30ESEM, Philips, Holland). The EDX analyzer (INCA, UK) was also employed to determine the inorganic components of residual substances on the stainless steel support mesh.

#### 2.2.7. CLSM analysis

The three different cake layers were observed using a CLSM (FV1000, Olympus, Japan). For PS and PN staining, two probes were collectively applied: Sypro Orange (Invitrogen) to target all the proteins and Concanavalin A, Alexa Flour 633 conjugate (5 mg/L, Invitrogen) to target the PS ( $\alpha$ -Man,  $\alpha$ -Glu (polysaccharide)) in the EPS matrix [9]. After staining, the samples were washed gently with a phosphate buffer to remove unbound probes. After that, the treated samples were immediately observed under CLSM.

#### 2.2.8. FTIR analysis

Samples extracted from the three different cake layers were dried in a vacuum freeze-drying machine (FD-1C-50, Boyikang

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