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Investigation of the role of biopolymer clusters in MBR membrane fouling using flash freezing and environmental scanning electron microscopy

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ABSTRACT

The technique that employs flash freezing and environmental scanning electron microscopy (ESEM) was utilised for detailed investigation of the fouling materials in a membrane bioreactor (MBR). The method involves the flash freezing of a wet sample in liquid nitrogen for 10 s to preserve its structure for direct ESEM observation with a high image resolution. ESEM images show that the sludge cake formed by simple filtration of the MBR bulk sludge has a highly porous, sponge-like structure with a fairly low resistance. However, the fouling layer attached to the membrane surface contains a thin gel layer under the main body of the sponge-like sludge cake, which is similar to that formed by filtration of a dispersion of biopolymer clusters (BPCs). It is apparent that BPCs tend to accumulate on the membrane surface, and the gel layer is largely responsible for the high filtration resistance of the cake layer on the fouled membranes.

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1. Introduction

Membrane bioreactors (MBRs) are increasingly applied to biological wastewater treatment owing to their ensured solids–water separation and excellent effluent quality for reuse purposes (Judd, 2006; Yang et al., 2006). However, membrane fouling, which is caused primarily by foulant deposition on the membrane surface, remains far and away the major limitation to the cost-effectiveness of MBRs for large-scale applications (Asatekin et al., 2007). Numerous efforts have been devoted to obtaining a fundamental understanding of the membrane fouling mechanisms (Le-Clech et al., 2006) that is essential for the development of effective fouling control technologies. It is generally believed that the deposition of a fouling (cake or gel) layer on the membrane surface is the major form of membrane fouling during MBR operation (Chu and Li, 2005; Wang et al., 2007). A number of foulants have been identified that would be responsible for the fouling layer formation, including biomass sludge (Defrance et al., 2000), the extracellular polymeric substances (EPS) in sludge (Nagaoka et al., 1996; Drews et al., 2006), soluble microbial products (SMP) and other forms of organic matter in the liquid phase (Rosenberger et al., 2006; Liang et al., 2007). Therefore, the roles played by different foulants, and their interactions in membrane fouling during MBR operation, however, still require investigation.

The supernatant of the MBR sludge mixture has been found to have a consistently higher organic concentration than the effluent from the MBR (Shin and Kang, 2003; Holakoo et al., 2006). It is therefore believed that the organic materials in the sludge suspension contribute significantly to the development of membrane fouling (Judd, 2006; Ng et al., 2006; Rosenberger et al., 2006; Liang et al., 2007). Studies have further indicated that biopolymer clusters (BPCs) are one of the primary foulants in the MBR system (Wang et al., 2007; Sun et al., 2008; Wang and Li, 2008). BPCs are formed by the clustering of SMP and loose EPS in the sludge cake. BPCs are much larger in size than SMP, and they differ from bacterial flocs in that they are composed of few microorganisms. It has become clear that the difference in organic concentration between the supernatant of the MBR sludge and its permeate effluent is due to the retention of BPCs by membrane filtration. Meanwhile, BPC formation and accumulation in turn would cause serious membrane fouling during MBR operation (Sun et al., 2011b). However, the role played by BPCs in fouling layer formation and its effect on membrane permeability remain to be determined.

Detailed examination of the fouling layer structure on the membrane surface is greatly needed for better understanding of the MBR fouling mechanisms and the interactions of different foulants during the fouling process. Such examination is also extremely important to the development of more effective membrane fouling alleviation strategies. For example, a further increase in shear intensity may not be effective for membrane fouling reduction if the top layers of the sludge cake contribute little to its filtration

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resistance. Similarly, the commonly applied back-flushing technique (Wu et al., 2008) may have a low degree of effectiveness if BPCs accumulate mainly at the bottom of the sludge cake and cover the membrane surface. Chemical cleaning from the permeate side may be more effective in this case (Chang et al., 2002). The advanced microscopic techniques used to date to examine foulants and fouling layers, including scanning electron microscopy (SEM), confocal laser scanning microscopy (CLSM) and atomic force microscopy (AFM), are unsatisfactory. Conventional SEM examination requires samples to undergo dehydration followed by sputter coating (Miura et al., 2007), whereas samples for CLSM must be stained using specific fluorescent dyes before observation (Chu and Li, 2005; Hwang et al., 2008). As the foulants are highly hydrated, porous and soft, the SEM sample pretreatment steps can cause significant deformation, or even collapse, of the structure and morphology of the foulants and fouling layers (Fig. 1a and b). AFM scan requests little sample treatment and the images can have a fairly high resolution. This, however, is the case only for rather hard surfaces. The AFM images of the fouling layers on membrane are usually blurry owing to the soft nature of the foultants (Huisman et al., 2000; Martinez et al., 2000; Song et al., 2004). Moreover, AFM as a surface scanning technique is apparently not suitable for examination of thick sludge cake layers, as is also the case for CLSM. In the latter, the free dyes may remain in the cake, and the fouling layers may produce false images that are difficult to discern.

Environmental SEM (ESEM, or, more generally, variable-pressure SEM) is another technique employed for the direct observation of highly hydrated samples including fouling layers (Le-Clech et al., 2007), but requires no dehydration and sputter coating steps. Omission of the dehydration step allows preservation of the sample contents and structure. However, the maximum magnification possible for ESEM observations at room temperature could be restricted, being determined by the limitation of the useful specimen distance, which may lead to a loss of specimen details. Thus, most ESEM images of the fouling layers on the membrane surface look rather blurry (Le-Clech et al., 2007). The other problem for ESEM is the specimen dehydration resulted from water evaporation at room temperature in the low-pressure (one to several hundred Pa) specimen chamber, which often leads to significant sample shrinkage and structure deformation. This problem is more severe for highly hydrated specimens, as is the case for the gel and/or cake layers responsible for membrane fouling (Fig. 1c and d). However, both the magnification and resolution can be significantly improved and the specimen dehydration can be greatly minimised if the specimen is cryogenically fixed and maintained frozen on the cold stage during ESEM examinations (Santiwong et al., 2008; Wang and Waite, 2009).

In this study, the flash freezing technique with liquid nitrogen coupled with ESEM examination was adopted for the first time to examine the shape and structure of the MBR foulants and fouling layers. In view of the known role of BPCs in membrane fouling, focuses were placed on the characterisation of the fouling properties of BPCs and determination of the spatial distribution of BPCs in the sludge cake layer. The findings would provide important insight into the mechanisms of membrane fouling in MBRs.

2. Materials and methods

2.1. Sludge and BPC samples

The sludge and BPC samples were obtained from a submerged MBR that had been in stable operation for more than 4 yr (Sun et al., 2011b). A 0.2 m^2 polyethylene hollow-fibre membrane module was immersed in the cuboid plexiglass reactor, which had a working volume of 5 L. The feed to the reactor was a mixture of synthetic wastewater and actual domestic sewage. The synthetic wastewater was prepared according to the basic recipe of AEESP (2001) to supply about 90% of the organic load in the influent, and the actual sewage was collected from a local wastewater treatment plant (Stanley Sewage Treatment Works, Hong Kong). The influent had a total organic carbon (TOC) concentration of around 220 mg L^{-1}, and the concentration of the mixed liquor suspended

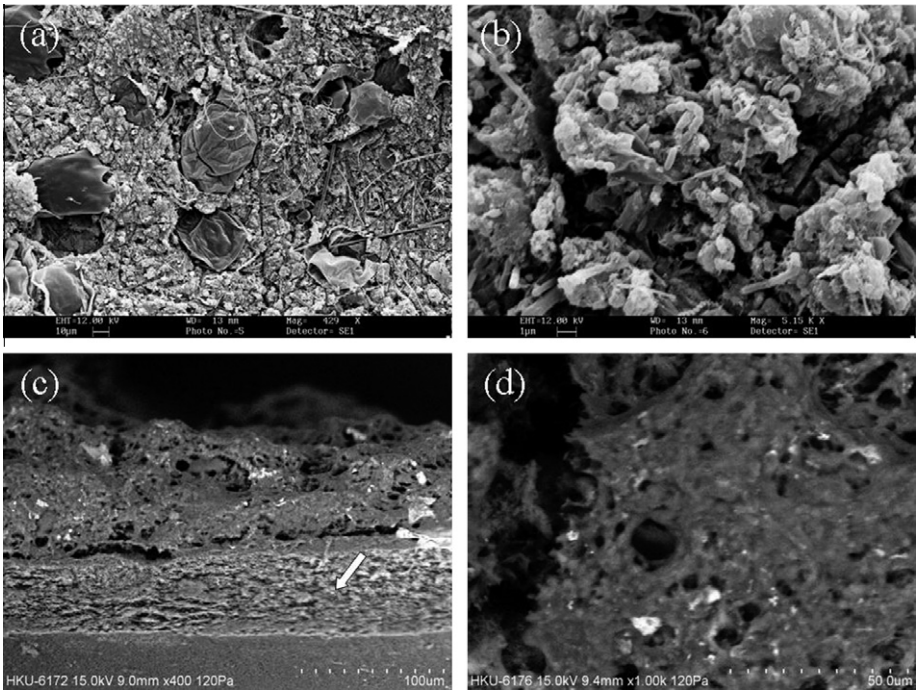

Fig. 1. Micrographs of the activated sludge cake layer obtained (a and b) with a conventional SEM after sample pretreatment involving dehydration and sputter coating and (c and d) with an ESEM at room temperature without prior flash freezing. The arrow points to the membrane filter.

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