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Evaluating the feasibility of pyrophyllite-based ceramic membranes for treating domestic wastewater in anaerobic ceramic membrane bioreactors

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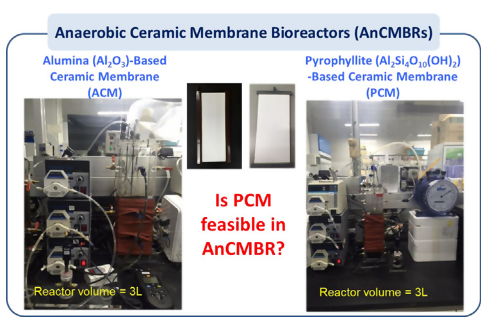
HIGHLIGHTS

- Pyrophyllite-based ceramic membranes applied to anaerobic membrane bioreactors.
- This novel approach achieved higher organic removal and methane yield at long HRTs.
- Short HRT operations resulted in substantial levels of sludge washout.

GRAPHICAL ABSTRACT



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ABSTRACT

This study laid great emphasis on anaerobic ceramic membrane bioreactor (AnCMBR) treatment of domestic wastewater for facile and enhanced energy recovery. To this end, the performance of the natural-based ceramic (*i.e.*, pyrophyllite-based) membranes was mainly explored in this study by evaluating filtration and treatment performances. 92.9 ± 5.5% chemical oxygen demand (COD) removal and stable methane production were successfully achieved in a bench-scale AnCMBR while maintaining a slightly long hydraulic retention time (HRT). Comparative filtration experiments with commercialized ceramic membranes suggested that the pyrophyllite-based membrane separation in AnCMBR treatment of wastewater at long HRT is feasible. However, short HRT operations resulted in substantial levels of sludge washout. Future improvements of AnCMBR technology in cost-effective ceramic membrane development, increased flux, and harsh environmental conditions would make AnCMBR competitive with anaerobic membrane bioreactor (AnMBR) technology.

1. Introduction

Anaerobic treatment provides a great venue for recovering energy (*i.e.*, biogas: CH_4) from diverse organic substrates, such as municipal wastewater, food wastewater, sewage sludge, and livestock manure [1–3]. Integrating membrane filtration with

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anaerobic treatment has been widely applied to achieve high quality effluents by solid-liquid separation and a relatively small footprint that would be suitable for domestic and industrial wastewater treatment [4–6]. Recently, anaerobic membrane bioreactor (AnMBR) systems have become an attractive technical approach for treating domestic wastewater because it allows to long biomass retention for methanogenesis regardless of short hydraulic retention or poor biomass settling properties [2,5,7,8]. Despite the numerous technical merits, one of the demerits of AnMBR treatment has been pointed as severe fouling problem, which leads to high operating cost and impeded its practical employment [9–12].

In this respect, a great deal of researches has been conducted intensively to develop effective fouling control strategy. Most previous studies focused on the optimization of operating conditions, such as gas scouring intensity, hydraulic retention time (HRT), sludge retention time (SRT), and temperature, *etc.* [2]. Modifying broth properties (*i.e.*, adding additives, such as coagulants or fluidized media) has been also tried to minimize the membrane fouling in AnMBR systems [10,13,14]. Indeed, these fouling control strategies generally enhanced the process performance, but they also had diverse negative impacts on membrane fouling along with operating conditions or types of additives [2,15]. In addition, some researchers have tried to modify membrane surface using plasma treatment or surface grafting, which significantly enhanced membrane permeability [2,16,17]. However, the technical limitations (*i.e.*, membrane production or module design) have been a bottleneck for widespread practical use [18].

Recently, employing a ceramic membrane has drawn great interests due to its technical advantages. Ceramic membrane allows to achieve high water flux and to alleviate fouling problem compared with conventional polymeric membrane [19–21]; for this reason, using ceramic membrane can improve the process efficiency of AnMBR system [18,22,23]. Given that ceramic membrane is resistant to harsh environmental conditions due to its higher chemical stability, the aggressive chemical cleaning strategy could be used for fouling control of anaerobic ceramic MBR (AnCMBR) [15,23,24]. Furthermore, in engineering aspects, it is difficult to exchange the membranes submerged in AnMBR systems because anaerobic conditions should be maintained without being exposed to the atmosphere and the systems require a long start-up period. Therefore, AnCMBR with relatively less fouling potential can be a suitable option to treat organic substrates, such as domestic wastewater. Nevertheless, its practical implementation in AnCMBR has been limited because raw materials used for most commercialized ceramic membranes (i.e., alumina, silicon carbide, and titania) are quite expensive. For this reason, several researchers have developed cost-effective ceramic membranes using natural-mineral based materials, such as pyrophyllite, dolomite, kaolin, and Moroccan clay [25,26]. Pyrophyllite ($Al_2Si_4O_{10}(OH)_2$), one of the abundant natural-minerals in South Korea, can be easily obtained from mine waste. Considering that this reusable ceramic material is cheaper than purified alumina, pyrophyllite-based ceramic membrane can be an effective way to reduce manufacturing cost [26]. For instance, the sintering temperature of pyrophyllite (*i.e.*, 1,300 °C) is substantially lower than that of alumina (*i.e.*, 1,600 °C) [27]. In this context, AnCMBR using pyrophyllite-based ceramic membrane can be an attractive means for treating domestic wastewater.

However, so far, natural-mineral based ceramic membrane has not been applied to AnMBR systems. Therefore, this study evaluated the feasibility of AnMBR system using the pyrophyllite-based ceramic membrane, which was developed in our recent previous study [18]. To the best of our knowledge, this study can be the first work suggesting that application of pyrophyllite-based ceramic membrane for the anaerobic treatment of domestic wastewater. For this, two AnCMBRs using pyrophyllite- (*i.e.*, target experiment) or alumina-based ceramic membrane (*i.e.*, control experiment) were operated and their performance was evaluated with filtration efficiency, organic removal, methane content, and biomass properties.

2. Materials and methods

2.1. AnCMBRs and experimental setup

Two acrylic AnCMBRs with a working volume of 3 L were operated in parallel to compare the biogas production performance and filtration efficiency along with the types of membrane materials (Fig. 1). In each anaerobic MBR system, a flat-sheet pyrophyllite-based ceramic membrane (*i.e.*, AnCMBR-PCM) or a flat-sheet alumina-based ceramic membrane (*i.e.*, AnCMBR-ACM) was mounted (Table 1). The AnCMBR systems were operated in the inside-out mode and were equipped with pH, OPR, and level sensors. The fouling control was conducted through biogas scouring with a flow rate of 2 L min^{-1}. The produced biogas was recirculated to score the membrane surfaces using a gas diaphragm pump (Boxer Pumps 10K, London, U.K.). The volume of biogas was measured by a drum-type gas meter (TG 05, RITTER, Germany).

2.2. Operating parameters for AnCMBRs

Two AnCMBRs (*i.e.*, AnCMBR-ACM and AnCMBR-PCM) were inoculated with the anaerobic sludge collected from a full-scale domestic wastewater treatment plant in South Korea. The initial dose of mixed liquor suspended solid (MLSS) in both AnCMBRs was set as 10,000 mg L^{-1}. The synthetic wastewater containing glucose as a sole carbon source (*i.e.*, 878.6 mg L^{-1} based on chemical oxygen demand (COD)) was fed into the reactors (Table 2). The influent COD concentration adopted in this study was relatively high level, but similar AnMBR studies have been selected higher strength wastewater containing a COD of 600–800 mg/L [7,9]. A newly developed pyrophyllite-based ceramic membrane was tested to determine whether it is capable of being applied to AnMBR treatment, thus the high-strength domestic wastewater would be beneficial in improving biogas production, particularly helpful in removing organic matters in anaerobic treatment.

The filtration cycle was performed as follows; 4 min on (*i.e.*, suction) and 1 min off (*i.e.*, stop). A digital pressure gauge (KELLER PR-21Y, Switzerland) was connected between the membrane and a peristaltic permeate pump (Masterflex L/S, Cole-Parmer, IL) to measure trans-membrane pressures (TMPs) in each AnCMBR system. The sludge of both AnCMBRs was withdrawn only as a sample for analysis; therefore, the solids retention time (SRT) of the bioreactors could be considered as infinite. Both AnCMBRs were operated under two different HRT conditions; therefore, the operation periods were classified into two phases (*i.e.*, phase I and phase II). The detailed information for operating conditions in this study is summarized in Table 3.

2.3. Analytical methods

Liquor samples were periodically collected from each AnCMBR at three different positions; i) influent, ii) mixed liquor, and iii) permeate. The concentrations of MLSS and mixed liquor volatile suspended solid (MLVSS) were measured according to the Standard Methods 2540 D/E [28]. COD was analyzed using a HACH DR/3900 spectrophotometer following the user guidance. The dissolved organic carbon was measured using a TOC analyzer (TOC-L CPH, Shimadzu Corp., Japan). The biogas sample was collected from