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Removal of trace organic contaminants by a membrane bioreactor–granular activated carbon (MBR–GAC) system

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ABSTRACT

The removal of trace organics by a membrane bioreactor–granular activated carbon (MBR–GAC) integrated system were investigated. The results confirmed that MBR treatment can be effective for the removal of hydrophobic ($\log D > 3.2$) and readily biodegradable trace organics. The data also highlighted the limitation of MBR in removing hydrophilic and persistent compounds (e.g. carbamazepine, diclofenac, and fenoprop) and that GAC could complement MBR very well as a post-treatment process. The MBR–GAC system showed high removal of all selected trace organics including those that are hydrophilic and persistent to biological degradation at up to 406 bed volumes (BV). However, over an extended period, breakthrough of diclofenac was observed after 7320 BV. This suggests that strict monitoring should be applied over the lifetime of the GAC column to detect the breakthrough of hydrophilic and persistent compounds which have low removal by MBR treatment.

1. Introduction

The presence of trace organics including pharmaceutically active compounds (PhACs), endocrine disrupting chemicals (EDCs), and various industrial compounds in the aquatic environment is of great concern due to their potential adverse effects on human health and the ecosystem (Cirja et al., 2008; Clara et al., 2005). These trace organics can be found in municipal wastewater in the range of up to several μg/L. Therefore, it is essential to effectively remove these contaminants to protect the environment and drinking water resources. However, conventional water and wastewater treatment processes (e.g. coagulation, sand filtration, conventional activated sludge treatment) may not provide effective barriers against trace organic contaminants (Cirja et al., 2008; Ternes et al., 2002).

The superiority of the membrane bioreactor (MBR) systems over conventional activated sludge process (CAS) in terms of basic effluent quality has been widely reported (Melin et al., 2006). Longer retention time due to adsorption, potentially followed by biodegradation, has been reported to be a key removal mechanism for trace organics by MBR (Cirja et al., 2008). Although better and/or more stable removal of trace organics possessing moderate to high biodegradability by MBR treatment in comparison to that of CAS has been reported, significant variation in MBR removal performance has also been noted in several recent studies, particularly for biologically persistent hydrophilic compounds (Clara et al., 2005; Joss et al., 2005; Tadkaew et al., 2011). Therefore, post-treatment of MBR permeate or application of hybrid MBR processes appears to be a logical means to prevent trace organics dispersion in the environment via incompletely treated wastewater.

The potential of activated carbon in the removal of pesticides in drinking water treatment has been demonstrated (Snyder et al., 2007; Yu et al., 2008). Several studies have also evaluated the adsorption of other emerging trace organics including a range of PhACs and ECDs on activated carbon in both laboratory systems and full scale drinking water treatment plants (Kim et al., 2010; Ternes et al., 2002). Elimination of various trace organics by adsorption onto GAC has been generally found to be satisfactory. In comparison to investigations involving drinking water treatment, only a few studies have investigated GAC adsorption as an option for tertiary treatment of conventional biologically treated wastewater (Dickenson and Drewes, 2010; Grover et al., 2011; Hernández-Leal et al., 2011). It has been noted in those studies that the adsorption of trace organics on activated carbon decreased due to competition with bulk organic matter for adsorptive sites. In fact, competition with bulk organic matter for adsorptive sites has important implications to the lifetime and serviceability of GAC columns. For efficient adsorption of trace organics, it is essential that the feed to GAC column has a low bulk organic content. Because MBR can produce suspended solids-free permeate with low total organic carbon content (Melin et al., 2006), GAC can be an excellent post-treatment option for MBR permeate. In such a system, GAC can specifically target the residual trace organics in MBR permeate without any significant interference from the bulk organics.

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Evidence from the literature indicates that neither MBR nor GAC on its own can adequately remove all trace organics of concern. Only a few studies concerning trace organics removal have investigated and reported encouraging results from hybrid PAC–MBR systems wherein PAC was directly added into the bioreactor (Li et al., 2011; Zhang et al., 2008). However, there has been no extensive study on the efficiency of the sequential combination of these two processes for the removal of trace organics. In this study, trace organics removal via sequential application of GAC adsorption following MBR treatment was systematically investigated. The extent of overall removal of a set of selected compounds possessing varieties of chemical structures was assessed. Diclofenac, which has been suggested as a possible anthropogenic marker for trace organics in the aquatic environment, was selected for extended monitoring to study the breakthrough of hydrophilic and biologically persistent compounds during GAC operation.

2. Methods

2.1. Model trace organics and synthetic wastewater

A set of 22 compounds was selected to represent four major groups of trace organics, namely PhACs, pesticides, hormones and industrial chemicals. The selection of these model compounds was also based on their widespread occurrence in domestic sewage and environmental water as well as their diverse physicochemical properties (Supplementary Data Table S1). A combined stock solution was prepared in pure methanol, kept in at −18 °C and used within a month. Once stable operation had been achieved (Section 2.2), trace organics were continuously introduced to the feed solution to achieve a constant concentration of approximately 5 μg/L of each selected compound. This initial feed concentration was selected to avoid any ambiguity from the effluent concentrations being too close to the analytical limit while it was still representative of the environmental concentration. The chemical analysis of the influent samples confirmed the accuracy and consistency of this dosing process throughout the duration of the experiment.

A synthetic wastewater comprising of glucose (400 mg/L), peptone (100 mg/L), urea (35 mg/L), KH_2PO_4 (17.5 mg/L), $MgSO_4$ (17.5 mg/L), $FeSO_4$ (10 mg/L) and sodium acetate (225 mg/L) was utilized. The chemical oxygen demand (COD), total organic carbon (TOC) and total nitrogen (TN) of the wastewater was 600, 180 and 25 mg/L, respectively.

2.2. Laboratory-scale MBR–GAC set up and operation protocol

A 5.5 L laboratory scale MBR system (Supplementary Data Fig. S2) was used in this study. The MBR was seeded with activated sludge from another lab-scale MBR system which had been in continuous operation for over 3 yr (Tadkaew et al., 2011). The hydraulic retention time was set at 24 h. Apart from the samples for mixed liquor suspended solid (MLSS) and mixed liquor volatile suspended solid (MLVSS), no sludge was withdrawn from the MBR at any stage of this study, theoretically meaning an infinite sludge retention time (SRT). The temperature and dissolved oxygen concentration in the bioreactor were maintained at 20.0 ± 0.1 °C and 3 ± 1 mg/L, respectively. The pH of the mixed liquor was monitored daily and remained stable in the range of 7.2–7.5. After an initial start up period of 6 weeks, once stable operation of the MBR in terms of TOC and TN removal had been achieved, the selected trace organics were added to the feed and the MBR was operated for further 6 weeks under these conditions.

A borosilicate glass (Omnifit, Danbury, CT, USA) column filled with 7.5 g of GAC was used as a post-treatment unit for the MBR permeate. The column had an internal diameter of 1 cm and an active length of 22 cm, resulting in a bed volume (BV) of 17 mL. GAC-1200 (Activated Carbon Technologies Pty Ltd., Victoria, Australia), was used as the adsorbent. The physical and chemical characteristics of the GAC used are outlined in Supplementary Data Table S3. The GAC was washed with Milli-Q water to remove fine particles, and then dried at 105 °C for 24 h and stored until use. The MBR permeate was pumped through the GAC column in an up-flow mode at a flow rate of 2.4 mL/min (equivalent to 8.5 BV/h), resulting in an empty bed contact time (EBCT) of 7 min. The GAC post-treatment column was attached to the MBR setup 2 weeks after the start of spiking the synthetic wastewater with trace organics, and it was operated for the remaining 4 weeks (equivalent to 7320 BV).

2.3. Analysis of trace organics and other basic parameters

The trace organic compounds in feed and permeate samples were extracted using Oasis HLB cartridges (Waters, Milford, MA, USA). The trace organics were then eluted from the cartridges and the eluents were evaporated to dryness. Finally, the dry residues in the vials were derivatized, cooled to room temperature and subjected to GC–MS analysis using a Shimadzu GC–MS (QP5000) system. The GC–MS system was equipped with a Shimadzu AOC 20i autosampler and a Phenomenex Zebron ZB-5 (5% diphenyl–95% dimethylpolysiloxane) capillary column (30 m × 0.25 mm ID, d_f = 0.25 μm). Details of sample preparation and GC–MS operation are available elsewhere (Hai et al., 2011b). Removal efficiency was calculated as, $R = 100 \times \left(1 - \frac{C_{Eff}}{C_{Inf}}\right)$ where C_{Inf} and C_{Eff} are influent and effluent (permeate) concentrations of the trace organics, respectively. Complete degradation of an organic compound may follow different pathways and undergo several steps; therefore the term removal here does not necessarily indicate complete degradation of the trace organics, but rather a loss of the specific trace chemical molecule, either by a chemical change or sorption to solid surfaces.

Basic parameters such as total organic carbon (TOC), total nitrogen (TN), chemical oxygen demand (COD), MLSS and MLVSS contents were measured according to the methods utilized in our previous studies (Hai et al., 2011a,b; Tadkaew et al., 2011).

3. Results and discussion

3.1. Basic performance of the MBR system

In this study, a synthetic wastewater was used to ensure a consistent influent composition. The MBR showed stable and good performance with respect to all key basic water quality and operating parameters (see Supplementary Data Table S4).

Turbidity of MBR permeate was consistently below 0.2 NTU during the entire period of operation. In addition, a stable TOC removal (97–99%) was achieved and TOC concentration of the permeate was typically less than 5 mg/L. The MBR system was operated under aerobic conditions and, therefore, is not expected to have high nitrogen removal via denitrification. Accordingly, the TN removal in our study ranged from 31% to 68% (Supplementary Data Table S4). Notably, nitrogen in the synthetic feed solution was supplied mostly in organic-bound form (from peptone and urea). The ratio of influent COD, total nitrogen and total phosphorous (COD_{in}:TN:TP) in the synthetic feed solution was 150:6.5:1, and residual ammonia at a concentration of 6 mg/L was detected in the MBR permeate. This suggests that partial nitrification occurred following the hydrolysis of the organic-bound nitrogen to ammonia. Since no sludge withdrawal was conducted, the MLSS in the reactor increased from 4.9 to 7.4 g/L over the operation period of 6 weeks. Nevertheless, such increase in MLSS concentration

did not lead to any significant variation in TOC and TN removal (Supplementary Data Table S4). TMP across the membrane module was also stable throughout the operation period (data not shown), probably because the membrane was operated at a relatively low flux. Therefore, it can be stated that we were able to observe trace organics removal by MBR treatment under stable conditions.

3.2. Removal of trace organics by MBR system

Given the diverse physicochemical properties of the 22 compounds selected in this study, it is not surprising that their removal efficiency by MBR varied quite significantly. Little or no removal was observed for carbamazepine, diclofenac and fenoprop, while 80–99% removal of all five steroid hormones and four alkyl phenolic trace organics could be observed (Fig. 1). It has been suggested that the removal of the significantly hydrophobic ($\log D > 3.2$) compounds such as the hormones and alkyl phenolic compounds utilized in this study is probably dominated by sorption to the activated sludge facilitating enhanced biological degradation in some cases (Tadkaew et al., 2011; Wells, 2006). On the other hand Tadkaew et al. (2011), proposed that functional groups play an important role in determining the extent of biodegradation of compounds possessing lower hydrophobicity ($\log D < 3.2$). They systematically demonstrated that compounds with strong electron withdrawing groups (EWG) are more resistant to MBR treatment, while the removal of compounds possessing both electron donating group (EDG) and EWG can substantially vary depending on the number and type of the functional groups. The low to moderate removal of six significantly hydrophilic compounds (i.e. carbamazepine, diclofenac, fenoprop, naproxen, ketoprofen and metronidazole) in this study, therefore, can be attributed to the presence of one or more strong EWG (such as chlorine atom, amide group and nitro group) or absence of strong EDG in their structures (Supplementary Data Table S1). Our results regarding the removal efficiency of these biologically persistent compounds are in line with previous reports (Clara et al., 2005; Joss et al., 2005; Radjenovic et al., 2007; Tadkaew et al., 2011). One anomalous result obtained was the high removal of primidone, despite containing a strong EWG (amide) (Tadkaew et al., 2011). A possible explanation may be that the presence of methyl groups (weak EDG) led to conversion of the methyl group to alcohol (Shaw and Harayama, 1992), bypassing the problematic amide conversion. On the other hand, in good agreement with the literature reports (Visvanathan et al., 2005), among the less hydrophobic compounds ($\log D < 3.2$) those containing the strong EDG hydroxyl group (i.e., acetaminophen, salicyclic acid, pentachlorphenol) were consistently removed to a high degree in our study. It is noteworthy that in line with the observations reported by Hai et al. (2011a), the removal of the halogenated organics correlated better with the ratio of halogen content to $\log D$ rather than $\log D$ only. This substantiates that the former is a better indicator for the prediction of halogenated trace organics removal by MBR treatment.

In addition to adsorption and biodegradation, volatilization may also contribute toward the removal of highly volatile trace organics from an aqueous solution. The removal of a trace organic compound due to aeration during wastewater treatment depends on its vapour pressure (Henry's constant) and hydrophobicity (Cirja et al., 2008). However, given the very low Henry's constant (H) and low $H/\log D$ ratio of all compounds selected in this study (Supplementary Data Table S1), their removal by volatilization is expected to be negligible. Except for MLSS sampling, no sludge was withdrawn from MBR in this study. The removal via sludge wastage, therefore, can also be considered to be insignificant.

3.3. Complementary effect of GAC post-treatment

Several recent studies have pointed out that MBR treatment may not be effective for the removal of hydrophilic and biologically persistent trace organic compounds (Joss et al., 2005; Tadkaew et al., 2011). In good agreement with the literature, results presented in Section 3.2 confirm low removal efficiency of several hydrophilic compounds (i.e. fenoprop, ketoprofen, naproxen, diclofenac and carbamazepine). As a result, it is necessary to further polish the MBR permeate. High removal efficiency of all selected trace organic compounds following GAC post-treatment was observed (Fig. 2) in this study. These results demonstrate that GAC post-treatment could significantly improve the removal of the compounds which were poorly removed by MBR treatment. For example, all five compounds, which were removed by MBR treatment with efficiencies below 40% (i.e. fenoprop, ketoprofen, naproxen, diclofenac and carbamazepine), achieved overall removal efficiencies of 98% or above following GAC treatment (Fig. 2). Our results are consistent with several previous studies where GAC filtration has been found to be a viable tool for the elimination of trace organics from surface water or biologically treated wastewater (Dickenson and Drewes, 2010; Grover et al., 2011; Hernández-Leal et al., 2011; Kim et al., 2010; Ternes et al., 2002).

Adsorption onto a given adsorbent through hydrophobic interactions increases with an increasing log$D$ value of the adsorbates (Snyder et al., 2007; Yu et al., 2008). Therefore significantly

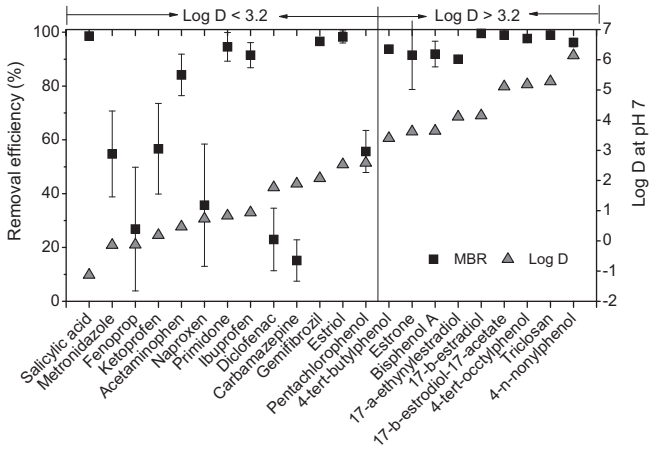

Fig. 1. Removal efficiency of the trace organic contaminants by MBR. Duplicate samples were taken each week. Error bars represent standard deviation of 12 measurements over 6 weeks.

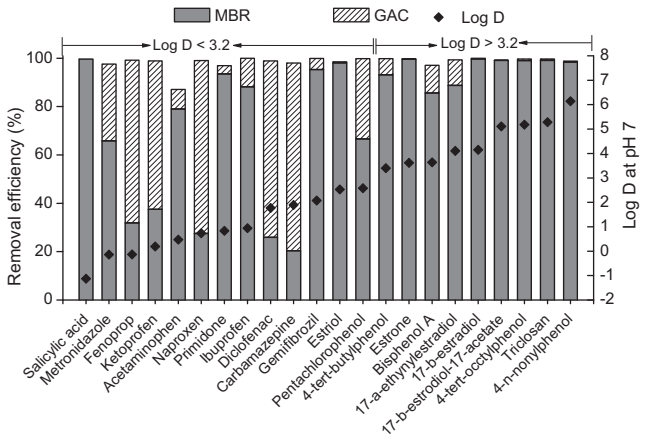

Fig. 2. Overall removal efficiency of the trace organic contaminants by the MBR–GAC integrated system at 406 BV.

hydrophobic compounds ($\log D > 3.2$) are likely to attain high removal through GAC adsorption. However, in this study all hydrophobic compounds had been already significantly removed by MBR treatment. Therefore, under a reduced competition for the adsorptive sites, the GAC post-treatment helped to enhance the removal of less hydrophobic compounds from MBR permeate (Fig. 2). High removal of the hydrophilic compounds can be explained by the fact that hydrophobicity-independent mechanisms such as ion exchange, surface complexation and hydrogen bonding also play significant roles in sorption of trace organics onto GAC (Dickenson and Drewes, 2010; Yu et al., 2008). In fact, results contrary to the trend expected from hydrophobicity considerations can also be found in the literature (Li et al., 2011; Yu et al., 2008). For instance, Yu et al. (2008) reported higher adsorption of carbamazepine as compared to nonylphenol although the latter possesses a significantly higher value of $\log D$ and thus is significantly more hydrophobic. Dickenson and Drewes (2010) reported that $\log D$ (at pH 7) did not correlate well with the Freundlich adsorption capacity of ibuprofen, naproxen, and diclofenac, suggesting that specific polar interactions governed the sorption processes. In this study, in line with our initial expectation, high adsorption onto GAC of the compounds resistant to MBR treatment ensured that the combined process resulted in an overall near-complete removal of the compounds.

3.4. Breakthrough of biologically persistent hydrophilic compound from GAC column

Although adsorption on GAC may lead to high removal of trace organics initially, over time, the adsorption capacity of the GAC column will eventually become exhausted (Hernández-Leal et al., 2011). Therefore, short term observation of removal performance is not adequate. Breakthrough profiles provide important information for the design of a specific GAC system and for subsequent regeneration of the spent carbon. For a full scale installation, monitoring the breakthrough of a large set of compounds may not be always feasible. Instead, the concentration of a hydrophilic, persistent marker in the GAC effluent can be monitored to detect the point when GAC would have to be regenerated. In this study the two lowest removal efficiencies by MBR treatment were achieved for diclofenac and carbamazepine, respectively. Given the lower $\log D$ value of diclofenac (1.77) compared to carbamazepine (1.89) (Supplementary Data Table S1), diclofenac was selected for extended monitoring of its concentration in the GAC effluent. A significant increase in diclofenac concentration in GAC effluent as a function of BV was observed in this study (Supplementary Data Fig. S5). While the initial concentration of diclofenac in GAC effluent was only 55 ng/L, within 7320 BV of operation its concentration exceeded 5000 ng/L. Hernández-Leal et al. (2011) investigated the removal of 16 trace organics in the concentration range of 0.1–10 μg/L using GAC adsorption and reported that at a flow rate of 0.5 BV/h the removal efficiency of these trace organics spiked into an aerobically treated greywater was higher than 72%. They also reported that continuous operation of up to 1728 BV did not lead to the breakthrough of TOC or any of the trace organics selected. In the present study, there was a gradual increase in diclofenac concentration in the GAC effluent (Supplementary Data Fig. S5). Breakthrough profiles are influenced by the characteristics of the target trace organics, the activated carbon properties, and the influent water quality along with other operational conditions. Given the significantly higher flow rate and the difference in the type of GAC used in our investigation, the difference in the time scale for breakthrough compared that of Hernández-Leal et al. (2011) study is not surprising. Although a fresh GAC column may achieve high removal of all trace organic contaminants, our results reaffirm that strict monitoring should be in place over the lifetime of the GAC column to detect the breakthrough point of hydrophilic and hardly biodegradable compounds which have low removal by MBR treatment.

4. Conclusion

Our results confirm that MBR treatment can effectively remove hydrophobic ($\log D > 3.2$) and readily biodegradable trace organics but is less effective for the removal of hydrophilic and persistent compounds. GAC post-treatment was observed to significantly complement MBR treatment to obtain high overall removal of less hydrophobic and biologically persistent trace organics. However, breakthrough of diclofenac, whose concentration in the GAC effluent was monitored for extended period, indicated that strict monitoring should be applied over the lifetime of the GAC column to detect the breakthrough point of hydrophilic and persistent compounds which have low removal by MBR treatment.

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.biortech.2011.10.051.

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