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Comparison of microporous and nonporous membrane bioreactor systems for the treatment of BTEX in vapor streams

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Increased regulatory constraints on industrial releases of atmospheric volatile organic compounds (VOCs) have resulted in an interest in using biofilters, bioscrubbers and air/liquid membranes for treatment of vapor phase waste streams. In this report, we describe the comparison of the use of two fundamentally different types of membrane module systems that allow the rapid diffusion of vapor phase aromatics and oxygen to an active biofilm for subsequent biodegradation. One system used a commercial membrane module containing microporous polypropylene fibers while the other used a nonporous silicone tubing membrane module for the delivery of substrate (a mixture of benzene, ethylbenzene, toluene, and xylenes [BTEX]) and electron acceptor (O₂). Tests of the systems under similar conditions with BTEX in the vapor feed stream showed significant performance advantages for the silicone membrane system. The average surface-area-based BTEX removal rate for the microporous membrane system over 500 h of operation was 7.88 μ g h⁻¹ cm⁻² while the rate for the silicone membrane system was 23.87 μ g h⁻¹ cm⁻². The percentages of BTEX removal were also consistently better in the silicone membrane system versus the microporous system. Part of the performance problem associated with the microporous membrane system appeared to be internal water condensation and possible plugging of the pores with biomass over time that could not be resolved with vapor phase backflushing.

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Introduction

Volatile organic compounds (VOCs) represent a large and increasingly regulated group of hazardous wastes in the world today. With increasing air and vapor pollution standards being enacted the use of alternatives to traditional industrial processes are being developed and implemented. Over the past several decades, the biological treatment of degradable VOCs has become an accepted industrial approach for handling hazardous vapor streams [8,13,16,21]. The use of biological systems when compared to traditional physicochemical methods such as scrubbing, adsorption and condensation may include the advantages of lower costs and complete degradation of VOCs to nontoxic byproducts such as CO₂ and water [3,13,23]. Biological processes most often used in industrial vapor treatment include biofiltration, bioscrubbers and trickling bed filters [2,3,7,8,16,23]. These approaches have limitations such as overgrowth of biomass within the filtration unit and moisture control due to evaporation at high vapor flow rates [2,13,16]. Overgrowth of biomass can lead to backpressure fluctuations, channeling of the vapor stream, and acidification of the filter bed over time. Moisture loss can lead to deterioration of the system's biomass and a reduction in its degradative capacity.

Membrane based systems have been evaluated as a method of better controlling and optimizing the biofilm environment for reactor systems [5,6,10,25]. Both VOCs [9,10,19-21] and oxygen

[4,18] are transferred to the actively metabolizing biofilms using microporous membranes. These systems have demonstrated favorable results for low organic loading situations but under high loading conditions the micropores tend to plug with biomass and require continual backflushing [13,15,16,19]. In addition, microporous systems can be costly to purchase. Nonporous membrane systems have been heavily investigated in terms of both liquid oxygenation and substrate delivery [5,6,12,17,25]. In particular, extractive membrane bioreactors (EMBs) in which a liquid waste stream is passed along one side of a nonporous permeable membrane and an oxygenated liquid biomedium is passed along the other side have shown excellent promise [5,6,14,17,24-26]. These systems, which are usually composed of silicone rubber tubing or plates, have also been investigated for the treatment of contaminated vapor streams with promising results [1,11].

Using a multisubstrate blend of benzene, toluene, ethylbenzene, m-xylene, p-xylene and o-xylene (BTEX) as vapor contaminant, a comparison was made of the biodegradative performance of a microporous membrane system against the performance associated with using a nonporous silicone membrane system previously reported [1].

Materials and methods

Medium

The basal mineral salts medium used in all of the reactor studies contained the following (per liter of distilled water): K_2HPO_4 , 5.0 g; NaH₂PO₄, 2.5 g; (NH₄)₂SO₄, 1 g; disodium nitrilotriacetate, 15 mg; MgSO₄·7H₂O, 30 mg; CaCl₂, 10 mg; MnSO₄·H₂O, 5 mg; FeSO₄·7H₂O, 1 mg; CoCl₂, 1 mg; ZnSO₄·7H₂O, 1 mg; CuSO₄·

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246

5H₂O, 0.1 mg; H₃BO₃, 0.1 mg; Na₂MoO₄·H₂O, 0.1 mg. The pH was adjusted to 7.0 with 1 N NaOH.

Bacterial inoculants

For the bioreactor studies a dual culture of *Pseudomonas putida* BTE1, which grows on benzene, toluene and ethylbenzene, and *P. putida* TX1, which grows on toluene, m-xylene and p-xylene, were used [1]. Both *Pseudomonas* isolates exhibited co-oxidative activity against o-xylene.

Analytical procedures

Benzene, ethylbenzene, toluene, *m*-xylene, *p*-xylene and *o*-xylene were analyzed using a Hewlett-Packard 5890A gas chromatograph (GC) equipped with a 30 m Hewlett-Packard HP-624 column and FID detector (Hewlett-Packard, Wilmington, DE). Aromatic hydrocarbon standards were made by adding known amounts of neat BTEX components to serum bottles that had been volumetrically measured and were capped with Teflon[®]-lined rubber septa. The aromatic compounds were allowed to volatilize for 1 h at 25°C prior to sampling the vapor phase with a 50- μ l gas-tight syringe. Concentrations of the six compounds were combined and listed as one BTEX value.

Membrane bioreactor designs

The microporous and nonporous membrane systems tested utilized a common bioreactor mainframe design as the introductory source of the bacterial cultures as well as the control and monitoring point for the various environmental parameters of the system. It was composed of a gas-tight 4.5-1 fermentor with automatic controls and monitoring systems for pH, temperature, and dissolved oxygen (DO). To it was attached either a microporous or nonporous membrane module through which contaminated vapor could travel while basal salts medium from the fermentor recycled over the exterior of the membrane tubing. The mainframe design is described in more detail elsewhere [1].

The two module systems, which were tested at separate times, allowed the passage of BTEX-containing air through the dry interior of the membrane tubing or fibers. This in turn provided the diffusion of BTEX out to the active aqueous biofilm as seen in Figure 1. The membrane contactor systems provided both BTEX



Biofilm - Membrane Systems

Figure 1 Representation of the two biofilm-membrane systems.

and the electron acceptor (O_2) concurrently to the biofilm. The systems also allowed for diffusion of the metabolic byproduct, CO_2 , back into the airstream for removal. Both delivery systems utilized a commercial dry air stream in order to minimize differences in moisture delivered to each membrane contactor. Thus, any moisture observed in the vapor-carrying portion of the membrane system results from diffusion from the culture medium. Figure 2 shows a schematic of the entire mainframe and module systems. Besides the membrane modules, the only other difference between the two operational designs was the BTEX delivery system. Liquid BTEX was volatilized into the airstream of the microporous membrane module via a syringe infusion pump (Cole-Parmer #74900 series, Vernon Hills, IL) while the nonporous membrane module utilized saturated BTEX vapors from a sealed flask that had been diluted with clean air to provide the proper BTEX concentration [1].

Microporous module system

The module for this system contained a 2.5X8 Liqui-Cel[®] membrane contactor obtained from Hoechst Celanese (Charlotte, NC). The Liqui-Cel^(m) contactor was composed of numerous polypropylene microfibers and had the specifications listed in Table 1. The fermentor portion of the system was inoculated with 100 ml of a mixture of P. putida BTE1 and P. putida TX1 that had been grown overnight on BTEX. The inoculant was added to the sealed fermentor vessel containing 4.5 l of sterile basal salts buffer along with 50 μ l of equimolar BTEX and grown with agitation at 25°C. Active growth of the culture rapidly reduced the DO in the fermentor so periodic air sparging was required to provide oxygen for continued growth. Since sparging also stripped some of the BTEX from the reactor vessel, periodic readditions of BTEX were made. Following growth on BTEX, the cell mass reached an OD₆₆₀ of 0.178 and liquid recycle through the microporous module was initiated at 1 l min⁻¹. Airflow through the module was started at 250 ml min⁻¹ and increased periodically during the run. To backflush the membrane, the BTEX syringe pump was turned off and the vapor exit valve on the membrane module was closed. The airflow to the module was increased to $2 \, 1 \, \text{min}^{-1}$ forcing air through the micropores out into the recycling culture liquid. The lengths of the intermittent backflushings ranged from 15 to 30 min. Afterwards, the vapor exit valve was reopened and the airflow rate was returned to 250 ml min⁻¹. The BTEX pump feed was then reinitiated.

Nonporous module system

The module for this system was constructed with two 15.2-m sections of silicone tubing and had the specifications shown in Table 1. The bioreactor system was inoculated with *P. putida* BTE1 and TX1 in similar fashion as the microporous system and initiated as previously described [1]. Airflow through the module was maintained at approximately 250 ml min⁻¹ during the run.

The two systems tested had vapor sampling ports on both the inlet and outlet of the membrane modules as well as the top of the 4.5-1 fermentor. All aromatic hydrocarbon analysis was carried out via direct injection of vapor sample into a gas chromatograph. The rapid recycle $(1 \ 1 \ min^{-1})$ of culture medium between the modules and the fermentor also allowed for representative monitoring of environmental parameters for the membrane module in the fermentor vessel. It was not possible to monitor biomass growth directly on either membrane system, as that would have required dismantling the modules. Consequently, culture liquid recycling through the system was monitored (OD) and quantitated for cell



Figure 2 Schematic diagram of the membrane module systems and the bioreactor mainframe. Vapor flow is represented by dashed lines while liquid flow is represented by solid lines. For the microporous membrane test liquid BTEX was volatilized into the air inlet line with a syringe pump. For the nonporous membrane test saturated BTEX vapor was diluted into the air inlet line under flowmeter control.

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	Table 1	Specifications of the membrane modules

Characteristic	Microporous membrane module	Nonporous membrane module
Membrane source	Hoechst Celanese membranes	Dow Corning Silastic membranes
Module housing	7.7×25.8-cm polypropylene	7.7×25.8-cm polypropylene
Membrane length	6.35×20.3 - cm polypropylene fibers cartridge with \sim 7430	30.4 m of silicone tubing
Membrane diameter	300 μm OD, 240 μm ID	2.16 mm OD, 1.02 mm ID
Surface area	$14,000 \text{ cm}^2$	2068 cm ²
Pore size	0.05 μm	_
Porosity	40%	-

growth. The temperature in both systems was maintained at 25° C. Sterile basal salts medium was introduced into the fermentor vessel and waste liquor was removed at a total system dilution rate of 0.14 day⁻¹. The vessel liquid was mixed at an agitation rate of 300 rpm. All system components making liquid and vapor contact including the membrane modules were sterilized by autoclaving on dry cycle prior to use except for several pressure gauges, which were sterilized with ethanol.

Results

Microporous module system

Within 48 h after the start of liquid recycle through the system the module was removing about 90% of the BTEX feed stream as seen in Figure 3. The BTEX loading was subsequently increased up to approximately 25 μ g h⁻¹ cm⁻². By 110 h clear liquid droplets were observed exiting the vapor line indicating that condensation within the microfibers was occurring. Under microscopic examination, the liquid droplets did not appear to contain bacteria, indicating that a breach in the membrane had not occurred. The BTEX-removal performance for the system began declining as can be seen by the increasing differential between the loading and degradation values (Figure 3). This was probably due to condensation on the interior of the membrane limiting mass transfer to the exterior of the membrane through the micropores. Cell biomass forming on the exterior module fibers was also observed through the semitransparent polypropylene at this time and may have contributed to limitation of vapor mass transfer as a result of blockage within the micropores. To attempt to alleviate these problems the system was backflushed periodically at the times indicated by the arrows in Figure 3A. There was limited, short-term, improvement with the initial backflushing attempts, but the system's performance never returned to the >90% BTEX removal observed within the first 100 h of operation (Figure 3B). Over the course of the run a thick biomass formed within the module's fiber matrix even with the rapid liquid recycle designed to keep the membrane system cleared. The cell density within the recycling culture liquid increased up until around 170 h, at which point it began to decline slowly (Figure 3C). The two OD₆₆₀ spikes at 170 and 318 h represent cell biomass dislodged from the membrane module during 30-min backflushes. The overall drop in OD₆₆₀ from 170 h probably did not denote an actual decrease in the total cell biomass within the system but represented attachment of the free-floating biomass to the membrane over time. This speculation is based on the observation of a slight increase in liquid

recycle backpressure through the module over time suggesting the increased fixed biomass was restricting liquid flow. Furthermore, upon termination of the run when the module was disassembled and a thick biofilm was observed throughout the membranes further arguing that the contactor might have acted as a filter to trap recycling cell mass. DO was never a constraint, either because its mass transfer was not significantly impeded even with the condensation/plugging problem or there was simply not enough BTEX transferred to the microbial community to reduce the DO level significantly.

Nonporous module system: The silicone tube module was inoculated, and within 72 h of the start of liquid recycle was removing almost 100% of the BTEX feed stream (Figure 4B). BTEX loading was increased until a change in performance was observed. At no time during this run was condensate or culture



Figure 3 Mineralization of BTEX by a biofilm established on a microporous membrane system. Symbols: (\bigcirc) BTEX loaded (μ g h⁻¹ cm⁻²); (\bigcirc) BTEX degraded (μ g h⁻¹ cm⁻²); (\blacksquare) BTEX removed (%); (\square) dissolved oxygen (%); (\blacktriangle) culture OD at 660 nm. Arrows represent points at which the BTEX feed was terminated, the membrane module outlet was closed, and clean air was forced through the membrane at approximately 2 l min⁻¹ for either 15 or 30 min.

liquid observed in the vapor outlet line. When the BTEX loading was increased above $25 \,\mu g \,h^{-1} \,cm^{-2}$ (approximately between 300 and 400 h), the system exhibited a drop in removal efficiency to less than 90%. This was not due to oxygen limitations (Figure 4A and B) but rather the result of higher BTEX loading, which exceeded the biofilm's ability to maintain complete transfer and elimination through the membrane. Once the BTEX loading was reduced after 400 h the system returned to its previous performance level of 95% or greater. The recycle liquid cell density increased up until approximately 300 h at which point a consistent decline was apparent (Figure 4C). As with the microporous membrane test, the reduction in reactor cell population was likely due to the attachment or entrapment of the free cells to the membrane over time. However, backpressure development was not observed using this module. This was likely due to the greater open area available in this module. In support of this argument, a large amount of fixed biomass was observed through the semitransparent housing and when this module was opened, upon termination of the reactor run,



Figure 4 Mineralization of BTEX by a biofilm established on a nonporous membrane system membrane. Symbols: (\bigcirc) BTEX loaded (μ g h⁻¹ cm⁻²); (\bigcirc) BTEX degraded (μ g h⁻¹ cm⁻²); (\blacksquare) BTEX removed (%); (\square) dissolved oxygen (%); (\blacktriangle) culture OD at 660 nm.

Table 2 The performance of the two module systems tested

Characteristic	Microporous membrane module	Nonporous membrane module
Vapor retention times (s)	8-16	6-7.5
Average surface area loading (μ g h ⁻¹ cm ⁻²)	12.97	24.97
Average surface area removal (μ g h ⁻¹ cm ⁻²)	7.88	23.87
Average degraded (%)	61	95
Maximum surface area removal (μ g h ⁻¹ cm ⁻²)	16.84	31.4
Average volume removal $(g m^{-3} min^{-1})$	27.5	29.8
Maximum volume removal (g m ^{-3} min ^{-1})	57.6	43.4

a large viscous biomatrix was present on and between the silicone tubing. The data described in Figure 4 represent an operational period of 480 h in order to compare it to the microporous membrane data described in Figure 3. In fact, the nonporous system operated for 1250 h and continued to behave consistently during that time with no deterioration in performance [1].

In either of the two test systems there was no preferential removal of the separate BTEX components. Each of the compounds appeared to be degraded at essentially the same rate except for o-xylene which, being co-oxidized, was the slowest to be removed. Both bacterial isolates, BTE1 and TX1, were present in consistent concentrations throughout both module tests and contaminants were not observed in the time frames evaluated.

Discussion

Previous investigators discussed the performances of various membrane-based bioreactor systems for treatment of VOCs [9,10,17,21,24,25]. Reij *et al* [21] and Sirkar [22] showed that the mass transfer coefficient (K) for the aromatic components from the vapor phase to the biofilm is defined by a collective series of transfer resistance coefficients shown in Eqs. (1) and (2):

Nonporous membrane
$$\frac{1}{K} = \frac{1}{k_{\text{g}}m} + \frac{1}{k_{\text{mp}}m} + \frac{1}{k_{\text{we}}}$$
 (1)

Microporous membrane
$$\frac{1}{K} = \frac{1}{k_{gm}} + \frac{1}{k_{mnp}m} + \frac{1}{k_{we}}$$
 (2)

where k_g =gas phase mass transfer coefficient, *m*=Henry's partition coefficient, k_{mnp} =nonporous membrane mass transfer coefficient, k_{we} =exterior water phase mass transfer coefficient and k_{mp} =microporous membrane mass transfer coefficient.

Though it is not possible to quantify the individual resistances indicated in Eqs. (1) and (2) without data involving variations in flow rates, these equations provide a framework for understanding the performance of the microporous and nonporous membranes. The difference between the two equations is the difference between the two membrane mass transfer coefficients:

$$k_{\rm mnp} = \frac{S_{\rm m} D_m}{\delta} \qquad k_{\rm mp} = \frac{D \epsilon}{\delta \tau}$$

where D_m =membrane diffusion coefficient, S_m =solubility in membrane and δ =membrane thickness, D=diffusion coefficient, ϵ =membrane porosity and τ =membrane tortuosity.

Reij et al [21] further pointed out that if the biofilm forms in direct contact with either the nonporous silicone membrane or the open pores of the microporous membrane and is actively removing aromatic substrates, then the water phase mass transfer resistance (k_{we}) would probably be slight. If on the other hand, water forms on the interior of either of the membrane systems, this transfer resistance can be significant. Also, since the microporous membrane is impermeable to aromatic substrates except at open pores, the total porosity of the membrane (ϵ) is key to the overall transfer to the active biofilm. If the porosity is reduced through blockage with biofilm debris and inorganic matrix material, then the microporous membrane transfer resistance (k_{mp}) will increase, and the transfer rate will decrease. Since water clearly condenses within the microporous membrane system within 100 h of normal use, another interior water resistance $(1/k_{wi})$ needs to be added to Eq. (2). This is assuming that a uniform layer of water was formed inside the tubes. In reality, there may have been a blend of complete water occlusion of some microfibers and insignificant water in others.

The microporous system tested was operated with the culture liquid flowing around the outside (shell side) of the microfiber and the gas phase flowing through the interior (lumen side). An explanation of the mediocre BTEX removal and degradation performance over time to water formation in the microfibers is supported by Pressman et al [19] who observed similar condensation problems with their initial tests of TCE degradation. They resolved their problem by running the culture liquid through the lumen side and passing the gas through the shell side, which minimized water obstruction of the gas flow. However, it is likely that long-term use of a system in this manner would still have significant potential for micropore blockage and thus reduced performance over time, requiring some type of backflushing regimen. Ergas et al [9] by contrast did not report a decline in their microporous bioreactor system treating toluene vapors. They obtained maximum volume-based toluene removal rates of 42 g m⁻³ min⁻¹ compared to our maximum volume-based BTEX removal rate of 58 g m⁻³ min⁻¹. That maximum rate was achieved prior to the observation of condensation in the system evaluated here and could not be attained again after the system's performance deteriorated.

For the biotreatment of vapor streams containing aromatic contaminants, the nonporous silicone membrane system delivered superior performance over the microporous membrane system in terms of surface-area-based removal rates, long-term operational stability and maintenance. Substrate removal rates in excess of 25 μ g h⁻¹ cm⁻² were possible with the nonporous system (Table 2). Although the vapor-retention times observed and substrate loadings presented in this study were different from those used in other investigations, the aromatic hydrocarbon removal efficiencies and rates of removal with the nonporous membrane system were comparable or higher than those reported using other systems [3,9,13,15,23].

The nonporous system showed no deterioration in performance over 500 h of operation while the microporous system exhibited a consistent decline after 100 h of operation. The microporous systems exhibited significant liquid condensation in the interior of the membrane tubes after approximately 100 h of operation under the conditions tested, which probably caused mass transfer performance problems. Biomass plugging of the micropores also may have contributed to performance deterioration. This condensation/biomass plugging problem was ongoing despite attempts to backflush the system periodically. The construction of silicone tubing systems is simple and relatively inexpensive compared to microporous membrane systems and requires no ongoing backflushing regimen to remove condensation or pore blockage. With vapor retention rates of less than 8 s, the nonporous system shows promise for vapor treatment of biodegradable VOCs such as BTEX which are readily diffusible through the membrane.

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250

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