Desalination and Water Treatment
www.deswater.com
doi: 10.1080/19443994.2016.1154897

57 (2016) 29439–29447
December

Taylor & Francis
Taylor & Francis Group

Arsenate removal using a hybrid system of adsorbents and a microfiltration membrane

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Received 14 December 2015; Accepted 8 February 2016

ABSTRACT

Arsenate in drinking water is a serious concern to millions of people around the world. Although various technologies have been developed in recent years, one of the most sustainable technologies for removing arsenate from water in developing countries is the use of absorption in the treatment of drinking water. This study determined the surface characteristics and arsenic removal efficiency of a new adsorbent Fe–Mn–Si (FM-α) and compared the performance of a hybrid system using FM-α and a microfiltration membrane with a hybrid system using a traditional adsorbent (zero-valent iron, ZVI) and a microfiltration membrane. Surface area analyses employing electron microscopy and energy-dispersive X-ray spectroscopy obtained a specific surface area of FM-α (17.2 m^2/g) that is 14 times that of ZVI (1.2 m^2/g). The arsenate removal efficiency of FM-α was 51 and 14% at pH 4 and 7, respectively, in the presence of phosphate, and 89 and 94% at pH 4 and 7, respectively, in the presence of humic acid; all these values were higher than those of ZVI under the same treatment conditions. Hybrid systems using FM-α had a higher bed volume than hybrid systems using ZVI. For example, the hybrid system using FM-α fed with only As(V) had a bed volume of 7,600, which is more than 10 times that of the hybrid system using ZVI. In terms of flux and pressure, hybrid systems using FM-α had more stable flux and pressure during operation over 47 d than the hybrid systems using ZVI. Additionally, hybrid systems using FM-α tended to rapidly and almost completely return to initial flux levels after backwashing, compared with recovery of only ~25% of the initial flux in hybrid systems using ZVI. The hybrid systems using FM-α accumulated more arsenate than the hybrid systems using ZVI. Overall, hybrid systems using FM-α had a higher bed volume, more stable flux and pressure, and better arsenate removal efficiency even in the presence of competing substances. Thus, these systems are a promising sustainable solution for the removal of arsenate and organic matter from water in developing countries.

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Presented at the 8th International Conference on Challenges in Environmental Science & Engineering (CESE-2015) 28 September–2 October 2015, Sydney, Australia

Keywords: Arsenate; Organic matter; Drinking water; Adsorbent; Microfiltration membrane

1. Introduction

Arsenic is a metalloid substance that has a high level of toxicity and is generated by natural processes and human activities [1,2]. It is well known that arsenate in drinking water causes cancer in the liver, kidney, lung, and other tissues [3]. As a result, the World Health Organization has set the standard for arsenate in drinking water at 10 μg/L, while the United States Environmental Protection Agency recently lowered its drinking water quality standard for arsenate from 50 to 10 μg/L. In case of Cambodia, which is located southeast Asia where contains serious global arsenic pollution problem, it is inappropriate to use the groundwater as drinking water since organic matters are included in groundwater as well as arsenic pollution [4,5]. In water, arsenic exists as inorganic arsenic either inorganic arsenite or arsenate. Since arsenite has higher mobility and toxicity than arsenate [6,7], the most common method of removing arsenate from water is to convert arsenite into arsenate using an oxidant [8–10]. Numerous methods have been developed to remove arsenate from drinking water, e.g. absorption through ion exchange [11,12], adsorption on activated carbon [13,14] or activated alumina [15], coagulation and filtration using iron chloride [16], and hybrid system incorporating pipe reactor and microfiltration [17]. As a general arsenate removal method, in relation to absorbent, Fe–Mn binary oxide (FMBO) [18] and nanozero-valent iron (nZVI) are widely developed and applied related to absorption [19]. Among the various methods, adsorbents such as zero-valent iron (ZVI) are widely used because they are cheap, easily available, and effective in removing arsenate from water [20]. However, the removal of arsenate from water by adsorbents including ZVI is affected by the presence of phosphate, chloride, nitrate, bicarbonate, sulfate, humic acid, and other species [21–27]. Recently, we developed a new adsorbent, FM-α. Our preliminary experiments showed that the arsenate removal capacity of FM-α is as good as that of ZVI. The purpose of this study was to determine the surface characteristics of FM-α and to determine the arsenate removal efficiency of FM-α under different pH conditions and in the presence of competing substances. In addition, we combined FM-α with a microfiltration (MF) membrane to produce a hybrid system that removes both arsenate and organic matter together, and we then compared the performance of the hybrid system with the performances of other hybrid systems using ZVI and an MF membrane.

2. Materials and methods

2.1. Chemicals and their analytic methods

A stock solution of arsenate (1,000 mg/L) was made by dissolving 4,284 mg of $Na_2HAsO_4 \cdot 7H_2O$ (Wako Pure Chemical Industries, Osaka, Japan) in distilled water. A working solution of arsenate (1 mg/L) was made by diluting the stock solution with distilled water. The concentration of arsenate in samples was measured by inductively coupled plasma mass spectrometry (Perkin-Elmer, DRC-e, Massachusetts, USA) after filtering with a 0.45 μm GF/C filter and oxidizing with 98% nitric acid. A stock solution (1,000 mg/L) of phosphate was made by dissolving 4,393 mg of KH_2PO_4 (Sigma-Aldrich, St Louis, USA) in distilled water. A working solution of phosphate (5 mg/L) was made by diluting the stock solution with distilled water. The concentration of phosphate was measured by a DR 5000 spectrophotometer (Hach Co. Ltd), and the amount of HA was determined using a total organic carbon analyzer (Shimadzu Corp. TOC-L Series). A stock solution of HA was made by dissolving 0.15 mg of HA (Sigma-Aldrich, St. Louis, USA) in 1,000 ml of distilled water at pH 12. A working solution of HA (5 mg/L) was made by diluting the stock solution with distilled water.

2.2. Adsorbent preparation

FM-α was prepared employing the coprecipitation method. Briefly, 7.3 g of $FeCl_3$ was dissolved in 200 mL of distilled water and then mixed with 12.5 g of $FeSO_4 \cdot 7H_2O$ and 18 g of SiO_2. The mixture was then vigorously mixed with 2.4 g of $KMnO_4$ in 200 mL of distilled water while the pH value was adjusted to 4–5. After mixing for 1 h, the solution was allowed to precipitate for 4 h at room temperature, the supernatant was removed, and the precipitate was dried in a dry oven for 12 h at 90°C. After cooling, FM-α was stored in a desiccator. Meanwhile, the traditional adsorbent ZVI was purchased from Samchun Chemicals (Pyeongtaek, South Korea).

2.3. Schematic of hybrid systems

Four types of hybrid systems were used in this study as shown in Fig. 1. Two were filled with FM-α and two with ZVI. Each hybrid system consists of two

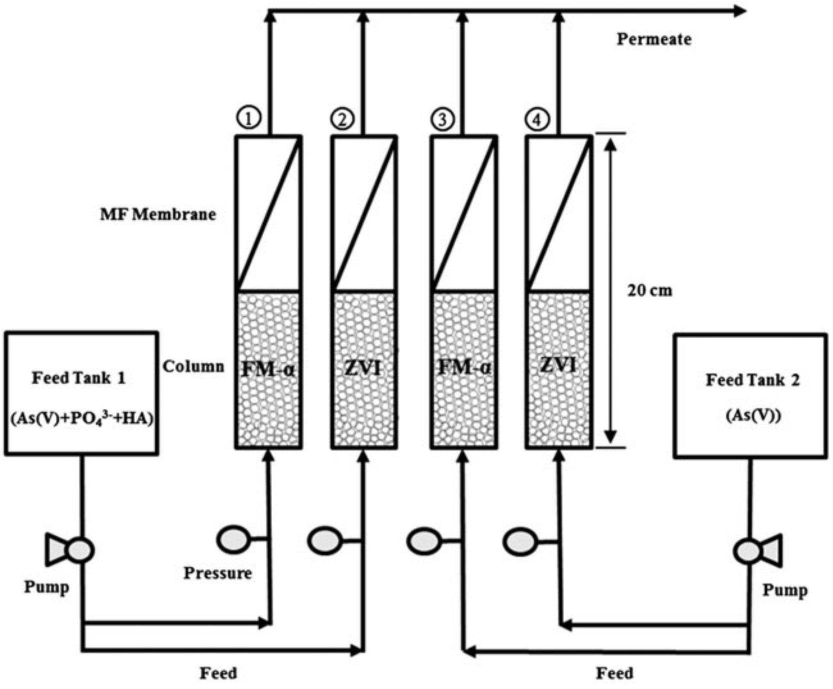


Fig. 1. Schematic diagram of the hybrid systems; column combined with membrane.

parts. One part is an MF membrane, and the other part is a column filled with either FM-α or ZVI adsorbent. Hybrid systems 1 and 3 were filled with 35 g of FM-α and hybrid systems 2 and 4 with 35 g of ZVI. For each system, the combined height of the membrane and column was 20 cm and the diameter was 4 cm. Two types of synthetic water were fed into the hybrid systems. One type of synthetic water contained arsenate only and was fed into hybrid systems 3 and 4. The other contained arsenate and competing substances (phosphate and HA) and was fed into hybrid systems 1 and 2. The configurations of the hybrid systems are given in Table 1, and the membrane properties are given in Table 2. Synthetic water was fed into the four hybrid systems with a peristaltic pump at an upward flow rate of 13.9 mL/min. The hybrid systems were operated for 47 d at room temperature with pH 7 ± 0.5.

Table 2
Properties of the membrane

Item	Specification
Type	Hollow fiber (MF)
Material	Polysulfone
Effective area	0.015 m^2
Pore size	0.1 μm
pH range	2–11
Module type	Pressure driven

2.4. Surface characterization of the adsorbents

The surface characteristics and chemical compositions of adsorbents were determined using an energy-dispersive X-ray spectrometer (Quantax-10, Bruker) and scanning electron microscope (JSM-6510, JEPL Ltd, Billerica, USA). The particle size distribution and specific surface area of the adsorbents were analyzed

Table 1
Configurations of the hybrid systems

Hybrid system	Arsenate + PO_4^{3-} + HA	Arsenate	FM-α	ZVI
1	O		O	
2	O			O
3		O	O	
4		O		O

employing a surface area analyzer (QD-SI/MP, Quantachrome, FL, USA) and the Brunauer–Emmett–Teller method.

2.5. *Effect of pH and competing substances on arsenate removal efficiency*

The removal efficiency of arsenate is highly affected by the presence of competing substances such as SiO_3^{2-}, PO_4^{3-}, CO_3^{2-}, and SO_4^{2-}. In this study, we examined the effects of phosphate and HA on the adsorption capacity of the two adsorbents under different pH conditions with/without competing substances, i.e., a mixture of 1 mg/L arsenate, 5 mg/L phosphate, and 5 mg/L HA was fed into hybrid systems 1 and 2, while only arsenate was fed into hybrid systems 3 and 4.

2.6. *Comparison of hybrid systems*

The flux and pressure of each hybrid system were monitored during the 47 d of operation. Additionally, after the operation, the flux of each component of the hybrid system was measured to determine the effect of adsorbents on the overall flux of the hybrid systems. After 47 d of operation, the membranes in the four hybrid systems were backwashed using physical and chemical methods. Distilled water was used for physical backwashing, and 0.1 M HNO_3 and 0.1 M NaOH were used for chemical backwashing. The flux of backwashed membranes was then measured and compared with the initial and final fluxes of the system.

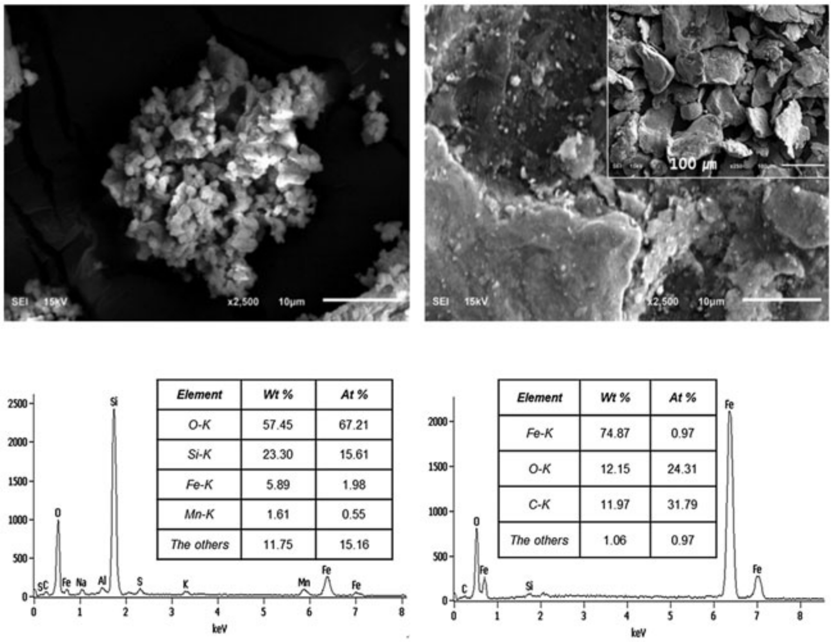
2.7. *Analysis of resistance and solid on the membrane*

After 47 d of operation, the membrane resistance was analyzed by measuring R_m, R_c, and R_f, where R_m is intrinsic resistance of the membrane, R_c is the cake resistance, and R_f is the fouling resistance. The amount and composition of accumulated solute on each membrane were analyzed after backwashing. Not only the total solid (TS) that caused fouling in membranes but also the solutes from volatile solid (VS) and fixed solid (FS) were analyzed to determine the proportion of organic and inorganic matter of foulants. Finally, the accumulation of arsenate on the membranes in the four hybrid systems was measured.

3. Results and discussion

3.1. *Surface characterization of absorbents*

Fig. 2 shows scanning electron microscopy (SEM) (2,500×) and energy-dispersive X-ray spectroscopy (EDS) surface analyses of FM-α and ZVI. In the figure, FM-α appears to have a rough and porous surface with many microparticles. The surface of ZVI does not appear to be as rough as that of FM-α and has larger particles. In terms of composition, FM-α contained



Element	Wt %	At %
O-K	57.45	67.21
Si-K	23.30	15.61
Fe-K	5.89	1.98
Mn-K	1.61	0.55
The others	11.75	15.16

Element	Wt %	At %
Fe-K	74.87	0.97
O-K	12.15	24.31
C-K	11.97	31.79
The others	1.06	0.97

Fig. 2. SEM micrograph (2,500×) and EDS surface analysis of FM-α (left) and ZVI (right).

5.89% Fe, 1.61% Mn, and 23.30% Si, whereas ZVI was primarily Fe (74.87%). According to surface area analysis, the specific surface area of FM-α was 17.2 m^2/g, which is 14 times that of ZVI (1.2 m^2/g). Owing to its larger surface area, it is likely that FM-α has better absorption efficiency than ZVI.

3.2. *Effect of pH and competing substances on the arsenate removal efficiency of absorbents*

Figs. 3 and 4 show the arsenate removal by the two adsorbents at different pH values (4 and 7) in the presence of competing substances (phosphate and HA). In the presence of phosphate, 51 and 14% of arsenate were removed at pH 4 and 7, respectively, by FM-α, compared with 46 and 32%, respectively, by ZVI. In the presence of HA, 89 and 94% of arsenate were removed at pH 4 and 7, respectively, by FM-α, compared with 20 and 80%, respectively, by ZVI. It appears that the removal of arsenate by FM-α was affected by the presence of phosphate but not by HA, which is consistent with the results obtained for other adsorbents in a previous study [18].

3.3. *Bed volume of the hybrid systems*

Fig. 5 shows breakthrough curves of the four hybrid systems. In the figure, hybrid systems using FM-α (hybrid systems 1 and 3) had higher bed volumes of 766 and 7,600, respectively, than hybrid systems using ZVI (hybrid systems 2 and 4), which had bed volumes of 379 and 619, respectively. As expected, the hybrid systems fed with competing substances (hybrid systems 1 and 2) had small bed volumes of 766 and 379, respectively, relative to the hybrid systems fed with only arsenate (7,600 and 619, respectively). It is interesting that the bed volume of hybrid system 3 is 10 times that of hybrid system 4, which is probably because ZVI absorbed arsenate during the process of oxidation from 0 to 3 [28].

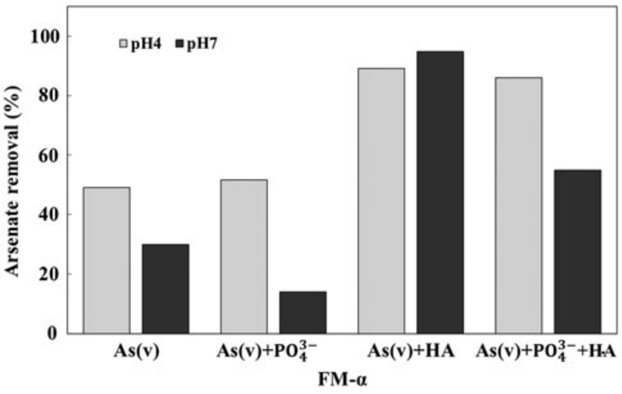


Fig. 3. Arsenate removal (%) by FM-α at different pH values and in the presence of competing substances.

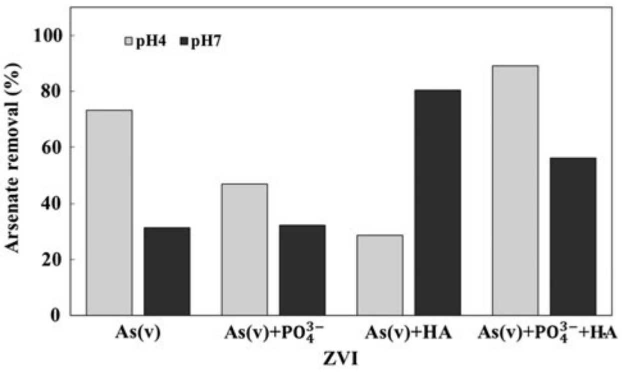


Fig. 4. Arsenate removal (%) by ZVI at different pH values and in the presence of competing substances.

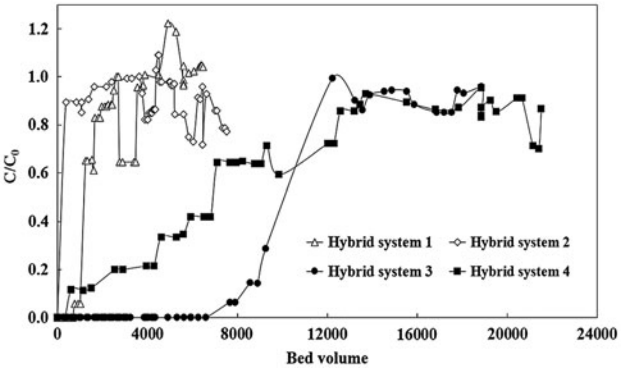


Fig. 5. Arsenate breakthrough curves of the four hybrid systems.

3.4. *Comparison of flux and pressure on the hybrid system*

Fig. 6 shows the changes in flux and pressure for the four hybrid systems during the 47-d operation. In the figure, the flux decline was greater for the hybrid systems using ZVI (hybrid systems 2 and 4). For example, hybrid system 2 had a 90% decline from initial flux after 16 d and hybrid system 4 had an 80% decline after 43 d. Meanwhile, hybrid system 3 using FM-α had more stable flux and pressure throughout the operation period. Fig. 7 shows the distribution of the rate of flow between the membrane and column in the four hybrid systems. In the hybrid systems using FM-α (hybrid systems 1 and 3), the flow rate of columns contributed 40 and 35% of the total flux, respectively, while the contributions were somewhat lower, 30 and 20% respectively, in the hybrid systems using

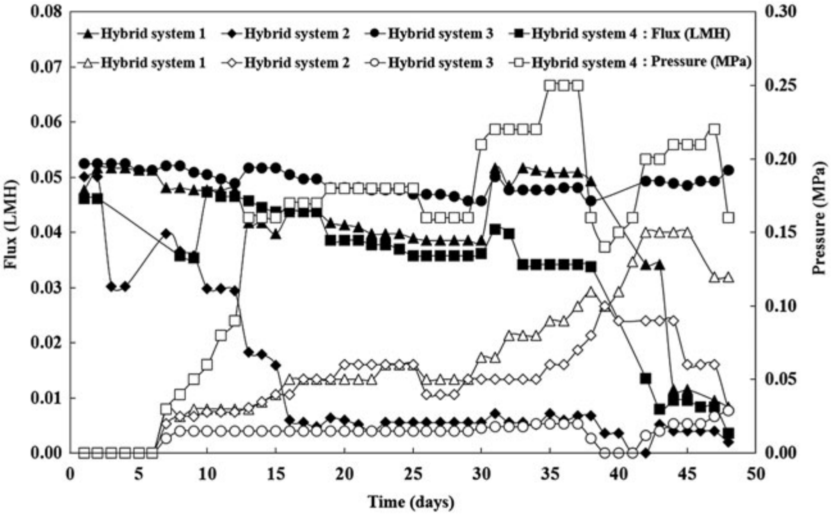


Fig. 6. Comparison of the four hybrid systems in terms of flux and pressure.

ZVI (hybrid systems 2 and 4). FM-α had a greater contribution to the overall flux, probably owing to its higher absorption efficiency.

3.5. Comparison of flux recovery after backwashing

Fig. 8 and Table 3 present the flux recovery at each membrane after physical and chemical backwashing. As seen in Table 3, hybrid systems 1 and 3 had relatively small flux declines during operation. After backwashing, the fluxes of the two hybrid systems rapidly returned to initial levels. Meanwhile, hybrid systems 2 and 4 had larger flux declines during operation, and the flux recovery was slow and only reached 25.5 and 20%, respectively, of the initial flux after backwashing. It is possible that the fouling of the surface of the membrane by the oxidation of ZVI is difficult to remove by either physical or chemical backwashing.

3.6. Comparison of membrane resistance

Fig. 9 shows the proportion of resistance in different parts of each hybrid system. In the figure, R_c accounted for more than 50% of the membrane resistance in all hybrid systems, which indicates that the fouling formed by the cake layer appreciably affected the surface of the membrane. Meanwhile, the R_f values of the membranes in hybrid systems 1 and 2 were more than 10% higher than those of the membranes in hybrid systems 3 and 4, which is probably due to membrane fouling by competing substances in feed tank 1.

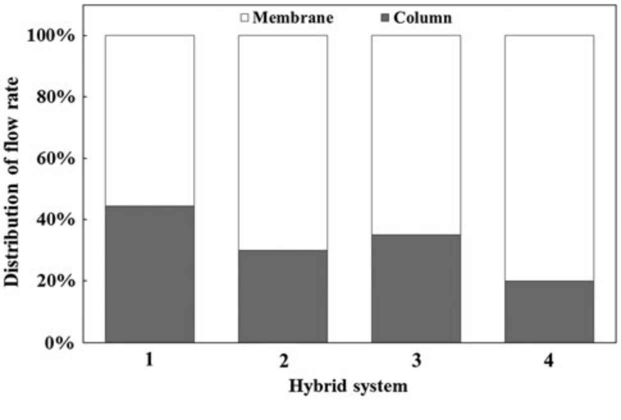


Fig. 7. Distribution of the flow rate in the four hybrid systems.

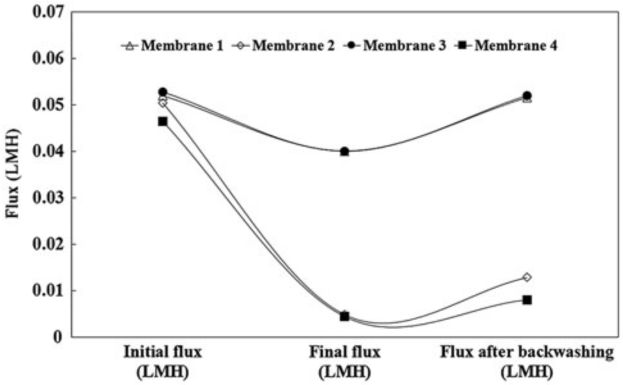


Fig. 8. Flux recovery of the membrane after backwashing.

Table 3
Initial flux, final flux, and flux after backwashing

Membrane	Initial flux (ml/min)	Final flux (ml/min)	Flux after backwashing (ml/min)
1	13.0	10.0	12.9
2	12.6	1.2	3.2
3	13.2	10.0	13.0
4	11.6	1.1	2.0

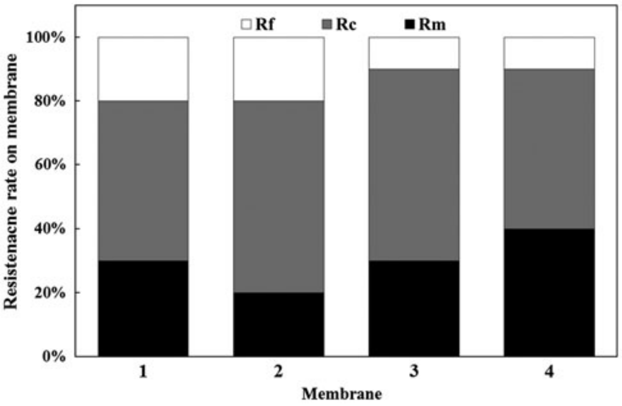


Fig. 9. Proportion of resistance in different parts of each hybrid system.

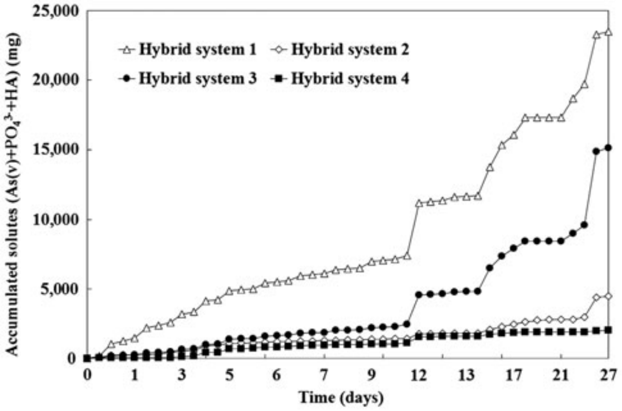


Fig. 10. Accumulation of solutes in the four hybrid systems.

3.7. TS and accumulated solutes

Table 4 shows the amounts of TS, VS, and FS obtained from the membranes of the four hybrid systems after backwashing. In the table, hybrid system 1 using FM-α had 3.49 g of VS and 3.27 g of FS, which are larger amounts than those of hybrid system 2 using ZVI (2.92 g of VS and 2.56 mg of FS). Meanwhile, there was no appreciable difference in the amount of FS when there were no competing substances (hybrid systems 3 and 4). Fig. 10 shows the accumulation of solutes (arsenate, phosphate, and HA) in the four hybrid systems. In the figure, hybrid systems 1 and 3 had greater accumulations of solutes than hybrid systems 2 and 4, which indicates that FM-α was more efficient in removing solutes than ZVI. Fig. 11 shows the accumulation of arsenate in the four hybrid systems. In the figure, hybrid systems 3 and 4, which were fed arsenate only, had more accumulated arsenate than hybrid systems 1 and 2, because there were no competing substances in hybrid systems 3 and 4. Hybrid system 3 using FM-α had more accumulation of arsenate than hybrid system 4 using ZVI, possibly because of the larger adsorption capacity.

Table 4
Amounts of solids obtained from membrane after backwashing

Membrane	TS (g)	VS (g)	FS (g)
1	6.76	3.49	3.27
2	5.48	2.92	2.56
3	2.28	0	2.28
4	2.29	0	2.29

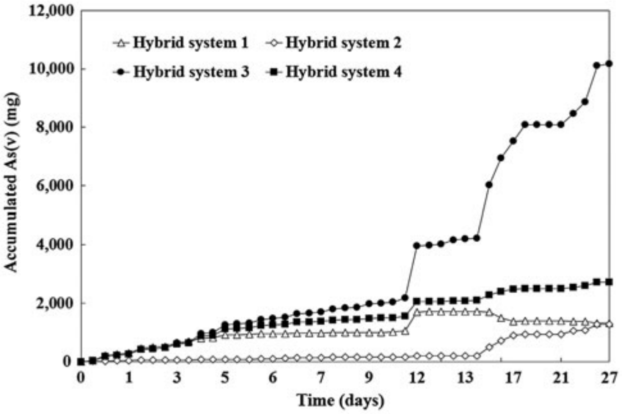


Fig. 11. Accumulated arsenate in hybrid systems.

4. Conclusions

In this study, we determined the surface characteristics of FM-α and the arsenate removal efficiency of FM-α under different pH conditions and in the presence/absence of competing substances. In addition, we combined FM-α with an MF membrane to make a hybrid system and then compared the performance of the hybrid system with other hybrid systems using ZVI and an MF membrane. According to surface area analysis using SEM and EDS, FM-α had a specific surface area of 17.2 m^2/g, which was 14 times that of ZVI (1.2 m^2/g). The arsenate removal efficiency of FM-α was 51 and 14% at pH 4 and 7, respectively, in the presence of phosphate, and 89 and 94% at pH 4 and 7, respectively, in the presence of HA; all these values were higher than those of ZVI under the same treatment conditions. Hybrid systems using FM-α had higher bed volumes than hybrid systems using ZVI. In terms of flux and pressure, hybrid systems using FM-α had more stable flux and pressure during the operation than hybrid systems using ZVI. Additionally, hybrid systems using FM-α tended to rapidly return to the initial flux after backwashing, but hybrid systems using ZVI recovered only 25.5% of their initial flux after backwashing. In terms of the accumulation of solutes, hybrid systems using FM-α accumulated more arsenate than hybrid systems using ZVI. Overall, hybrid systems using FM-α had a higher bed volume, more stable flux and pressure, and better arsenate removal efficiency even in the presence of competing substances. Thus, a hybrid system using FM-α is a promising sustainable solution for the removal of arsenate and organic matter from water in developing countries.

Acknowledgment

This research was supported by the National Research Foundation of Korea (NRF-2013K1A3A9A04043230) and partly funded by Korea Ministry of Environment (MOE) as "Advanced Technology Program for Environmental Industry" (E315-00015-0412-1).

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