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Removal of fluoride and total dissolved solids from coalbed methane produced water with a movable ultra-low pressure reverse osmosis system

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ABSTRACT

The aim of this project was to establish an economical and environmentally benign technology for removing fluoride (F^-) and total dissolved solids (TDS) from coalbed methane (CBM) produced water. The proposal involved a movable wastewater treatment system comprised of a flocculation sedimentation pretreatment unit and an ultra-low pressure reverse osmosis unit in Liulin County, Shanxi Province, China, where concentration of F^- ranged from 1.3 to 18.2 mg/L and concentration of TDS ranged from 2800 to 6,600 mg/L. When the hydraulic load was 2–4 m^3/day with the running mode in 10 h cycles at 10–15 days interval, the removal efficiency was 94.7% for F^- and 98.1% for TDS with 55% water recovery from June to November 2011. Concentrations of F^- and TDS in effluent were lower than the permissible discharge standard values of pollutants for irrigation and livestock. The flexible deployment and small footprint affordable was appropriate for removing pollutants from CBM mining areas with low water production and discommodiously decentralized treatment.

Keywords: Coalbed methane produced water; Ultra-low pressure reverse osmosis; Total dissolved solids; Fluoride

1. Introduction

Exploration of natural gas field results in coalbed methane (CBM) produced water, which is generated in gas production by dewatering the trapped gas bubbles from groundwater. CBM produced water management has become one of the key factors in the feasibility of gas field development. Produced waters vary widely in composition because they originate from separate geological formations with dissimilar gas hydrocarbon compositions, and differ by well development and maintenance [1]. This significantly affects the technical and economic feasibility of employing treatment technologies for beneficial utilization of produced water. CBM produced water is mostly characterized with high suspended solids and total dissolved solids (TDS), which is typically high in sodium (Na^+), bicarbonate (HCO_3^-), and chloride (Cl^-) [2]. It might also contain other large waste sources, e.g. iron, manganese, fluoride (F^-), boron, and other trace elements [3].

Large volumes of CBM produced water were reported to cause many adverse environmental effects on animal and plant growth, soil quality, and ground-

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water and surface water systems [4,5]. The discharge of CBM produced water has the potential to increase the concentration of TDS in soils, which could interfere with nutrient uptake by plants, possibly resulting in a decline in the growth of agricultural crops and native plants [6]. The continuous use of water with high amount of F^- has been shown to be toxic to humans and animals [7]. Regions where CBM exploration is underway often lack sufficient water for irrigation and livestock water consumption; therefore, the beneficial use of CBM produced water has become a possible means of water management by providing additional and reliable water supplies and reducing the disposal cost of produced water [1]. In China, surface discharge and evaporation are widely adopted for disposing CBM produced water [8]. Therefore, alternative methods for cost-effective and feasible water management due to limited land for evaporation pond and increased regulatory restrictions are being sought.

Various measures have been proposed for treating CBM produced water, e.g. ion exchange [5], nanofiltration [9], reverse osmosis (RO) [9], capacitive desalinization [10], and free-thaw evaporation [11]. However, these technologies focused on costly centralized wastewater treatment, collecting and treating decentralized CBM produced water with conventional wastewater treatment facilities. Small-scale and flexible shuttling between CBM wells for collecting low water production and removal of pollutants might be a reliable option, as it is uneconomical and inconvenient to use a centralized collection system to treat wastewater from CBM mining areas which are located in mountainous areas, especially with low volume water production.

RO has been found to be a dominating method for desalting water [12]. However, there remain several concerns regarding RO treatment. Application of a conventional RO membrane is limited due to high operation and maintenance costs as RO requires high pressure in the system and needs extensive pretreatment [13]. The ultra-low pressure reverse osmosis (ULPRO) membrane with a high desalting degree might offer a viable option for produced water treatment, because it has been shown to be efficient in rejecting organic and inorganic species as compared with the conventional RO membranes while requiring considerably less feed pressure, thereby resulting in lower operation costs [1,14]. The feed water to an ULPRO membrane needs to attain a turbidity goal of 1 NTU (turbidity unit) for normal RO membrane operation [15], thus ULPRO membranes are inadequate for collecting and directly treating pollutants from CBM produced water. A pretreatment unit for reducing turbidity merits consideration. Ultrafiltration was used as a means of pretreatment for treating surface water with ULPRO and preventing a RO membrane from fouling [16]. By the application of polyaluminum chloride, coagulation, sedimentation, and filtration or its usage in combination results in a high removal of turbidity in wastewater as a pretreatment for RO application [17–19]. Groundwater turbidity produced from pretreatment, consisted of sand filter, activated carbon, and cartridge filter, could undergo the strongest reduction (87%) [20].

This work is focused on CBM produced water from the Liulin County mining area, Shanxi Province, China. It also evaluated an integrated model and useful technology based on flocculation sedimentation (FS) combined with an ULPRO membrane unit, which was mounted on a movable equipment for treating CBM produced water from site to site as water production shifted from each well for removing F^- and TDS from CBM produced water in order to render it usable.

2. Materials and methods

2.1. Study area

The study site was located in Yangjiayu village, Zhuangshang town, Liulin County, western Shanxi Province, China, which is a semi-arid region with an average annual precipitation ranging from 400 to 500 mm and average annual mean temperature of 10.5℃. The precipitation is unevenly distributed throughout the year with the majority occurring from June to August [21]. The aquifer layer is controlled by a westward-inclining nose structure, topography, and annual precipitation and is mostly dominated by sodium bicarbonate and sodium chloride solutions [22]. The types of soil in this region are Typic Hapli-Ustic Argosols [23] and the average soil moisture content is 11.7% in maize cropland [24].

2.2. Investigation of the chemical characteristics of CBM produced water in the study area

A total of five wells were selected to investigate the chemical characteristics of CBM produced water in the study area. A water sample was monthly collected at each well from August to October 2010. Before sampling, the water pH, turbidity, and TDS were measured *in situ* at the discharge outlets separately using a portable Hanna Instrument (HI98185A), turbidimeter (US61-1900C), and conductivity meter (TDS890001).

Acid-washed polyethylene sample bottles were rinsed with wellhead discharge water, and then a 500 ml water sample was collected at the wellhead of each discharge outlet. Water samples were acidified with nitric acid to a $pH < 2$ and placed in a cooler box ($t < 4$℃) for transportation to an analytical laboratory for chemical analysis. F^- was measured with ion selective electrode (FOO1508, VanLondon-Phoenix) and chemical oxygen demand (COD) of the water samples was measured utilizing the potassium dichromate method [25]. For the remaining ions analysis, part of water samples was filtered through a 0.45 μm Millipore filter. The Cl^-, SO_4^{2-}, and HCO_3^- ions were measured with ion chromatography (Dionex IonPac AS14). The cations were measured with ICP-OES (OES Opima 5300 DV, America PE, USA).

The sodium adsorption ratio (SAR) is used to assess the suitability of water for agricultural irrigation, as determined by the relative concentration of Na^+ to Ca^{2+} and Mg^{2+} in water [5]. It is defined as:

$$\mathrm{SAR}\,(\mathrm{mmol}^{1/2}/\mathrm{L}^{1/2}) = [\mathrm{Na}^+]/[\mathrm{Ca}^{2+} + \mathrm{Mg}^{2+}]^{1/2}$$

where Na^+, Ca^{2+}, and Mg^{2+} are the concentrations of the respective ions (mmol/L) [5]. The SAR for CBM produced water was calculated based on the method described above.

2.3. Description of the movable CBM produced water treatment system

The movable CBM produced water treatment system was situated in a container (7 × 2.5 × 2.5 m) with four wheels, including the pretreatment unit for reducing turbidity and the ULPRO unit for removing F^- and TDS from CBM produced water. All components were installed inside of the container, which was movable

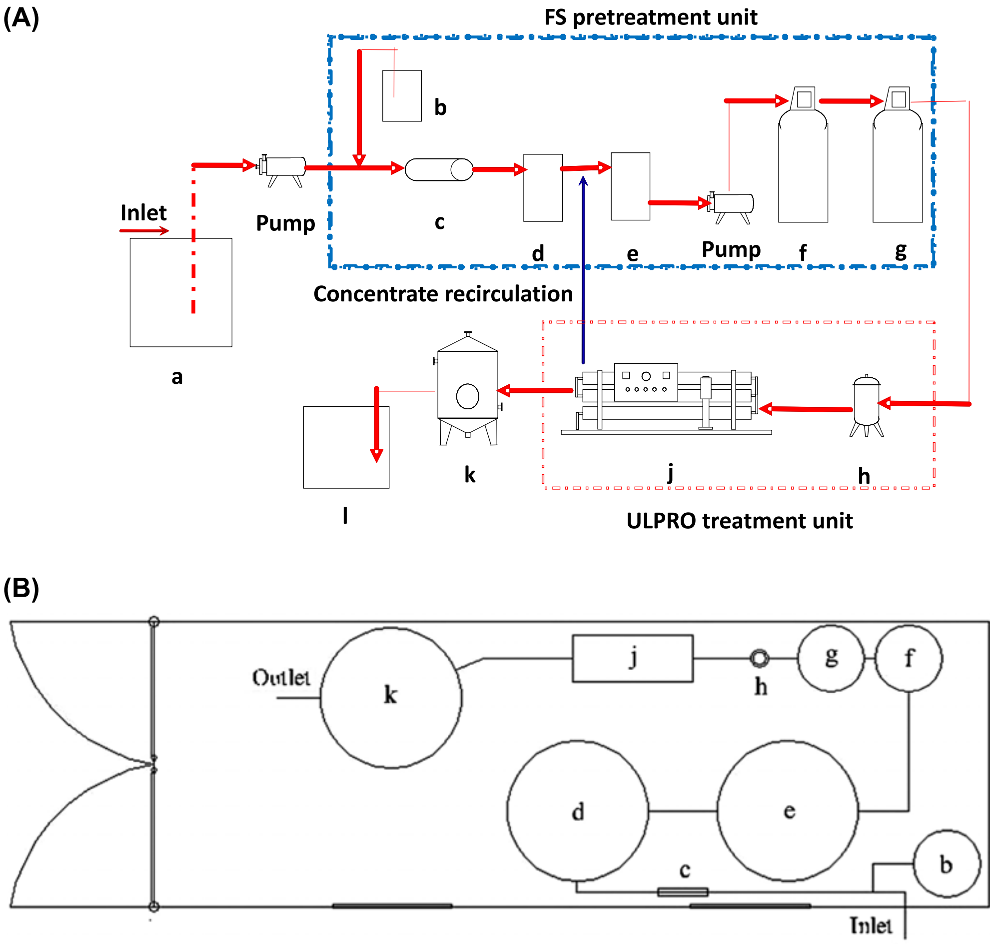

Fig. 1. (A) A movable treatment system for CBM produced water in Liulin County. (B) Layout scheme of CBM produced water treatment facilities in a movable container. (a): wastewater retention pond; (b): dosing device (polyaluminum chlorides); (c): static mixer tank; (d): flocculation and sedimentation tank; (e): regulating tank; (f): quartz sand filter; (g): activated carbon filter; (h): precision filter; (j): ULPRO device; (k): pure water storage tank; and (i): pure water retention pond. The pretreatment unit consisted of a dosing device, static mixture, a flocculation and sedimentation tank, a regulating tank, a quartz sand filter, and an activated carbon filter. The ULPRO treatment unit consisted of a precision filter and four ULPRO membranes.

from site to site on an automated trailer. The flow diagram of entire water treatment system is shown in Fig. 1. CBM produced water was added into constructed wastewater retention pond (a), and then pumped (the flow rate was $2\,m^3/h$) to a static mixing tank ($V = 1.5\,m^3$) (c). Polyaluminum chloride as a coagulant at a dose of 2 mg/L (the influent rate was 1 L/h and the reagent concentration was 4 g/L) was added into the static mixing tank through a dosing device (b). The water flowed into a flocculation and sedimentation tank (d), and then flowed into the regulating tank ($V = 1.5\,m^3$) (e). The effluent was pumped ($v = 2\,m^3/h$) through a quartz sand (4–6 mesh) filter ($\varphi 0.40 \times 1.65\,m$) (f) and an activated carbon (10–24 mesh) filter ($\varphi 0.40 \times 1.65\,m$) (g). Then water passed through the precision filter (5–10 μm) (h). The filtered water was fed into an ULPRO unit (j), subsequently water flowed into the water storage tank ($V = 1.5\,m^3$) (k), and then was discharged into a water retention pond (i). The ULPRO unit consisted of four ultra-low pressure aromatic polyamide membranes (ESPA2−4040). The ULPRO membrane was operated at a pressure of 1.05 MPa with a feed flow rate of $1.2\,m^3/h$. The remaining concentrated water completely flowed back into the regulating tank (e).

2.4. Experimental methodology

The movable CBM produced water treatment system as described in Fig. 1 was employed for experimental studies. Assessment of F^- and TDS removal efficiency (RE) from CBM produced water was at number 8 well in the study area. The air temperature ranged from 10 to 33 ℃ during the experimental period. Approximately $2–4\,m^3/d$ of produced water was discharged from the number 8 well during the study period. Water sampling sites were located at the wastewater retention pond (defined as influent) (a), at the outlet of the activated carbon filters (g), and at the outlet of the ULPRO device (defined as effluent) (j). The movable CBM produced water treatment system was periodically processed at an interval of about 10–15 days with a running mode of 10 h cycles per day, from June to November 2011.

3. Results and discussion

3.1. Characteristics of CBM water in the experimental area

The properties of CBM produced water in the experimental area were investigated from August to October 2010, and are summarized in Table 1. Water turbidity ranged from 45 to 193 NTU with an average of 110 NTU. The TDS values varied widely, ranging from 2,800 to 6,600 mg/L with an average of 4,300 mg/L. It was evident that CBM produced water was characterized by both high suspended solids and high TDS. The high TDS in the produced water was attributable to sodium, bicarbonate, and chloride ions and was predominantly sodium bicarbonate or sodium-bicarbonate-chloride. Being high in sodium and low in calcium/magnesium meant very high values for the SAR, ranging from 67.2 to 96.4 $mmol^{1/2}/L^{1/2}$. The F^- in the CBM produced water ranged from 1.3 to 18.2 mg/L with an average of 5.8 mg/L. Concentrations for most elements in CBM produced water were notably low or below the detection limit.

CBM produced water in the experimental area was predominately by sodium bicarbonate or sodium bicarbonate chloride, which was similar to what was investigated in the Ferron, San Juan, Black Warrior, and Powder River Basins in the USA [2,26–27]. CBM produced water was characterized by high suspended solids and high TDS, as have been previously shown in research from other CBM areas [2,28]. However, the calcium and magnesium concentrations in Liulin County are rather low, which could be associated with the large concentrations of bicarbonate that are a factor in the carbonate equilibrium controlling the

Table 1
The chemical analysis data of CBM produced water from the Liulin County area from August to October 2010 ($n = 15$)

Component	Concentration	Component	Concentration
TDS (mg/L)	2,800–6,600	Cl (mg/L)	562–1,985
Turbidity (NTU)	45–193	SO_4 (mg/L)	28.40–168.40
SAR ($mmol^{1/2}/L^{1/2}$)	67.2–96.4	F (mg/L)	1.30–18.20
pH	7.49–8.60	Fe (mg/L)	0.054–0.564
COD (mg/L)	2.10–2.91	Mn (mg/L)	0.031–0.673
Na (mg/L)	1,360–1,852	Cu (mg/L)	<0.001
Ca (mg/L)	8.10–23.34	Pb (mg/L)	<0.001
Mg (mg/L)	8.20–31.09	As (mg/L)	<0.001
HCO_3 (mg/L)	1,454–2,150	Cd (mg/L)	<0.001

concentrations of calcium and magnesium under reduction conditions [2].

High sodium levels and low calcium/magnesium levels indicate very high values for the SAR, which are comparable to those investigated in other CBM mining areas [28]. The pH of CBM produced water ranged from 7.49 to 8.60, and it is prone to be slightly alkaline for containing high HCO_3^-. Therefore, the direct discharge of CBM produced water tends to result in high risk of soil salinization, which leads to adverse impacts on sensitive plant growth and soil quality due to salinity and Na^+ build up [29,30]. Additionally, CBM produced water was also polluted by F^- (Table 1). The 77% of water samples exceeded the standards for irrigation water quality [31]. Soil F^- content in Liulin County ranged from 451 to 469 mg/kg, which exceeded the average soil F^- in China (440 mg/kg), as well as internationally (220 mg/kg) [32–33]. Soil F^- is therefore a threat to local soil and water systems while discharging CBM produced water with a high F^-. Therefore, CBM produced water in Liulin County requires treatment before surface discharge in order to minimize the risk of CBM water induced pollution.

3.2. Removal of pollutants from CBM produced water

The turbidity in CBM produced water was typically high as shown in Table 1. The average removal efficiencies for turbidity in the FS pretreatment unit during the experimental period ranged from 99.1 to 99.4% (Fig. 2). The monthly average turbidity reduced from 85.9–175.7 NTU (av. 105.5 NTU for 6 months) in influent to 0.3–1.0 NTU (av. 0.7 NTU for 6 months) in effluent for the FS pretreatment unit (Fig. 2). The FS pretreatment unit was effective for removing the turbidity of CBM produced water, which caused the feed water of the ULPRO treatment unit to meet the turbidity goal of 1 NTU for normal RO membrane operation.

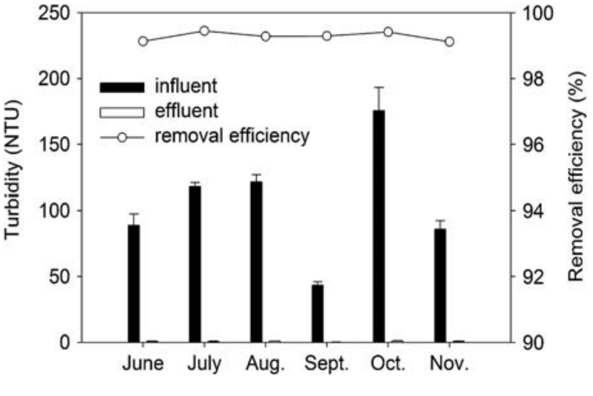

Fig. 2. RE of a FS pretreatment unit for turbidity in CBM produced water from June to November 2011.

The TDS concentration in CBM produced water was usually fairly high as shown in Table 1. The average TDS removal efficiencies in the entire movable treatment system during the experimental period ranged from 96.9 to 98.9% with 55% water recovery (av. 98.1%) (Fig. 3(f)). The ULPRO treatment unit was a major factor in TDS removal because the average removal efficiencies ranged from 96.3 to 98.9% (av. 97.8%) (Fig. 3(d)). The removal of TDS in produced water by the ULPRO treatment unit accounted for 87% (Fig. 4). The monthly average TDS reduced from 2,743–6,227 mg/L (av. 4,049 mg/L for 6 months) in influent to 33–120 mg/L (av. 75 mg/L for 6 months) in effluent through use of the free-movable treatment system (Fig. 3(f)). The average SAR reduced from 81.1 to 7.2 $mmol^{1/2}/L^{1/2}$ after treatment of the entire movable system (Table 2). The removal of TDS and SAR in CBM produced water by the movable treatment system is due to efficient removal of Na^+, HCO_3^-, and Cl^-, which were the predominate dissolved ions in the produced water (Tables 1 and 2).

The average F^- removal efficiencies in the entire movable treatment system during the experimental period ranged from 92.1 to 98.3% (av. 94.7%) with 55% water recovery (Fig. 3(e)). The ULPRO treatment unit was a major factor in F^- removal because the average removal efficiencies for F^- in ULPRO treatment unit during the experimental period ranged from 89.9 to 98.2% (av. 93.7%) (Fig. 3(c)). The ULPRO treatment unit reduced F^- in CBM produced water by 89.0% (Fig. 4). The monthly average F^- reduced from 4.4–17.8 mg/L (av. 8.4 mg/L for 6 months) in influent to 0.1–0.9 mg/L (av. 0.45 mg/L for 6 months) in effluent for the entire movable treatment system (Fig. 3(e)).

3.3. Rationality for an FS pretreatment unit combined with an ULPRO treatment unit

CBM produced water in the experimental area is characterized by high TDS, high turbidity, high SAR, and a predominance of sodium, bicarbonate, and chloride ions, which contain a high F^- level (Tables 1 and 2). Therefore, benign technologies may be effective for removing F^- and TDS from CBM produced water before surface discharge. RO [34] and ULPRO [35] were reported to have high RE for TDS. RO with zeolite membranes was utilized to treat CBM water with TDS of 18,600 mg/L, which was able to reduce at an overall TDS rejection rate of 83.5% due to the ionic sieving effects of zeolite through RO operation at a pressure of 4.1 MPa [34]. However, ULPRO exhibited a high permeate flux while displaying a competitive

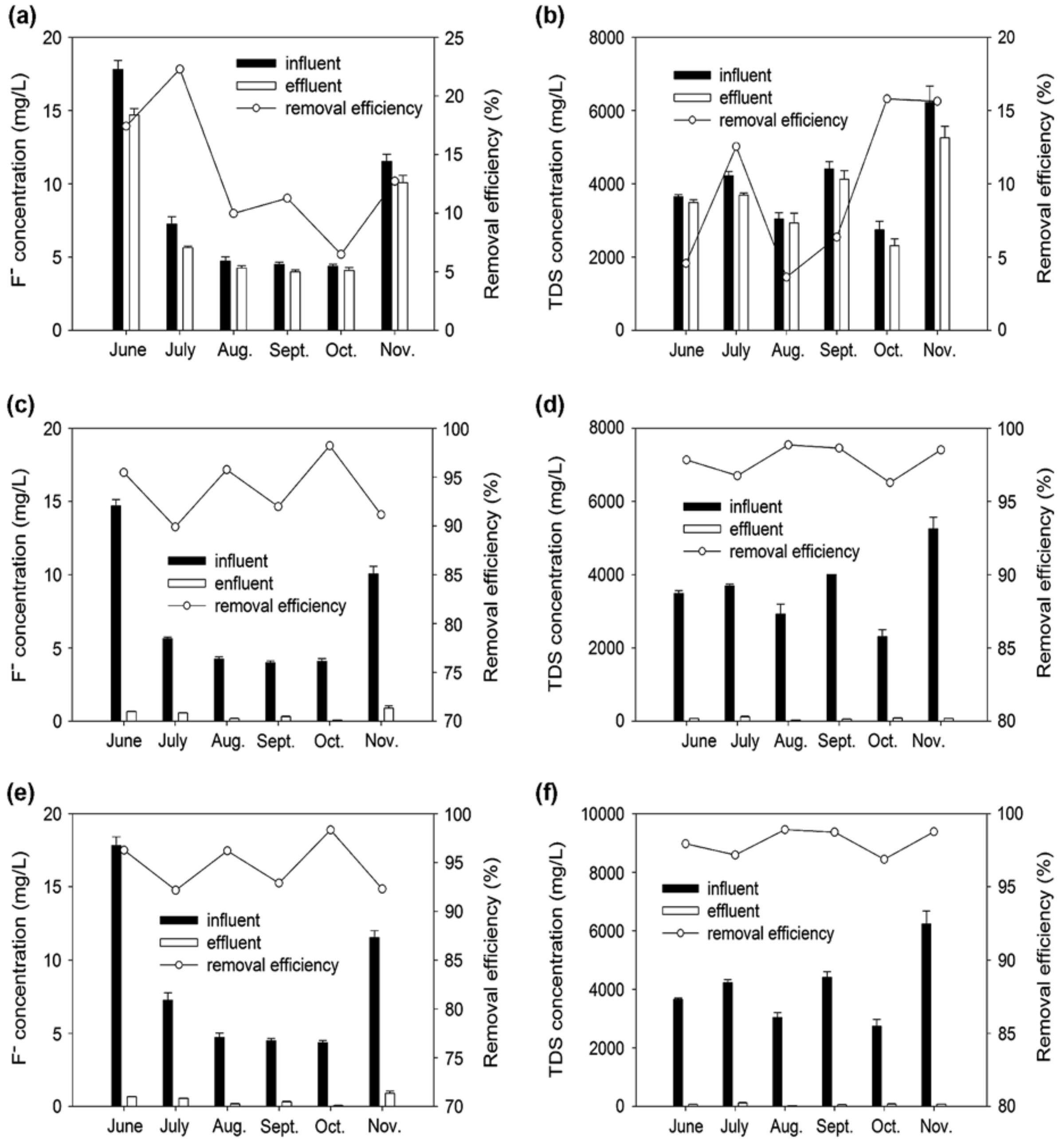

Fig. 3. RE of FS pretreatment units (a, b), an ULPRO treatment unit (c, d), and a entire movable treatment system (e, f) for F⁻ and TDS in CBM produced water from June to November 2011.

rejection in comparison to a conventional RO membrane [35]. The ULPRO was reported to treat produced water more economically than conventional RO membrane [35]. Particles cause colloidal/particle fouling of RO membranes in which a cake of particles form, thereby reducing the permeate flux [36]. One pretreatment criterion for the water used with RO membranes is the turbidity of the water following pretreatment. A turbidity goal of 1 NTU for the present experimental RO system was the target, while other membrane developers accept a lower turbidity value (e.g. 0.5 NTU) [37].

The average turbidity fluctuated between 0.3 and 1.0 NTU in effluent for the FS pretreatment unit though the monthly average turbidity ranged from 85.9 to 175.7 NTU (Fig. 2). It is obvious that the FS pretreatment unit adopted in this project efficiently reduced the turbidity of CBM produced water to meet turbidity goal of 1 NTU for reliable operation of RO membranes. Polyaluminum chlorides as

Fig. 4. The average removal percentage of a FS pretreatment unit and an ULPRO unit for F^- (left) and TDS (right) in CBM produced water from June to November 2011.

Table 2
RE of an entire movable CBM produced water treatment system for SAR, pH, Cl^-, Na^+, and HCO_3^- from June to November 2011

Parameters	pH	HCO_3^- (mg/L)	Cl^- (mg/L)	Na^+ (mg/L)	SAR ($mmol^{1/2}/L^{1/2}$)
Influent	7.87	1970.0 ± 206.1	775.0 ± 33.7	1547.1 ± 92.0	81.1 ± 12.0
Effluent	6.93	55.8 ± 29.8	33.7 ± 13.7	37.8 ± 19.6	7.2 ± 1.9
RE (%)		97.2	95.6	97.5	91.1

flocculent are able to form corresponding hydroxides, which neutralize the surface charges of colloidal particles [38]. This precipitation process produces new particles that incorporate raw water particles into flocs removing floatable particles, resulting in significant turbidity reduction [38]. The F^- was removed along with the flocculated material by the adsorption of F^- into the diffuse layer of the flocculent or through the combination with precipitated alum [39]. The turbidity RE of the FS pretreatment unit is high, with a TDS removal rate of 13% by utilization of the FS pretreatment unit (Fig. 4). Therefore, ULPRO is the key unit for the removal of three main ions (HCO_3^-, Cl^-, and Na^+) (Fig. 3(d)). Likewise, ULPRO is the key unit for the removal of F^-, which removed 89% of F^- in the produced water (Fig. 4). The present ULPRO membranes were reliably operated at an ultra-low pressure of 1.05 MPa. In the standards for irrigation water quality [31] and in the livestock and poultry drinking water standard [40] of China, F^- and TDS are, respectively, set at a maximum allowable limit of 2 and 1,000 mg/L and 2 and 2,000 mg/L.

Therefore, the employment of entire removal system consisting of FS pretreatment unit combined with an ULPRO treatment unit simultaneously efficiently removes F^- and TDS in CBM produced water. The final effluent of the RO process meets the standards imposed for water reuse in irrigation and livestock water consumption. These potential beneficial uses are relevant in such semi-arid areas of China, as the Liulin County, where water is an essential resource.

3.4. *Applicability of movable equipment for CBM produced water treatment*

The development process for CBM is generally divided into three phases: exploration, test production, and mining [41]. Differences of water production were observed with relevance to the development phases of CBM and of well sites. It is estimated that they produce approximately 8–80 L of water per minute, which varies according to the aquifer system pumped [2,42]. However, a single well had water production of 4–10 m^3/d in the early stage and reached a 20 m^3/d for normal extraction in the Liulin County [41], and the number 8 well of the experimental study produced 2–4 m^3/d of water. A centralized system would be capable of treating a relatively large quantity of CBM produced water at a single well and/or multi-well concentrated distribution with relatively small amounts of CBM produced water. However, it is inadequate for collecting and treating decentralized CBM produced water. Wastewater collection by pipeline from various wells with a small amount of CBM water, especially in mountainous areas, is a relatively costly process.

Therefore, small-scale and flexible shuttling among a CBM wells system for collecting and treating low water production may be worthwhile. Small-scale mobile units were developed to inexpensively treat relatively small amounts of oilfield brines by nanofiltration and RO [43]. Several case studies further suggested that mobile electrodialysis treatment units are able to treat produced water with TDS ranging from 11,400 to 27,000 mg/L [44], recovering 80–90% of brackish water. However, electrodialysis process is challenged by complex operation, high cost, and poor removal of both organics and microbiological organisms [45]. The movable ULPRO treatment system with compact treatment units has a smaller footprint, and is easily deployed. The operation of the system was stable and it simultaneously reduced the F^-, TDS, and SAR of produced water with 55% water recovery, making it suitable for irrigation and livestock water consumption. Additionally, the system may be rapidly deployed to each site to treat CBM water from unlined ponds. For changing capacity demands, this equipment is more widely applicable to a range of CBM wells with low water production and is discommodious for a decentralized system.

4. Conclusions

The pilot experiments showed that the movable treatment system is capable of removing F^- at a rate of 98.6% and TDS at a rate of 96.4% with 55% water recovery from June to November 2011. After appropriate FS pretreatment, the ULPRO membrane exhibited a high efficiency in removing dissolved ions and the average SAR values were reduced from 81.1 to $7.2\,\text{mmol}^{1/2}/\text{L}^{1/2}$. It is feasible to treat CBM produced water for being used in irrigation and livestock, especially in those CBM mining areas lacking water. The flexible deployment, small footprint, and environmentally benign nature of the treatment system is a promising measure for removing wastewater pollutants from CBM mining areas with low water production and is discommodious for a decentralized system.

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