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Nutrient recovery of source-separated urine via forward osmosis and a pilot-scale resource-oriented sanitation system

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ABSTRACT

Urine contains large amounts of nutrients and can be used as fertilizer. However, these nutrients are difficult to eliminate when they infiltrate sewage, and thus become pollutants. A new toilet system using forward osmosis (FO) as the key element was designed to recover resources from human excreta. In this system, FO was used to harvest the nutrients in urine and also could be regarded as a pretreatment for reverse osmosis. In this study, the influence of membrane material, draw solution concentration, flow rate, etc. was investigated for application of FO in urine enrichment. The differences between synthetic urine and natural urine before and after enrichment were compared and analyzed. With pH adjustment, the rejection of N, P, K, and other nutrients in urine was >80%. The results of the pilot-scale toilet system showed high potential for the adoption of FO to recover the nutrients in urine. By integrating this system with other treatment techniques, toilet wastewater will no longer be a burden but rather a resource.

Keywords: Forward osmosis; Source separation; Resource recovery; Sanitation system; Urine

1. Introduction

Toilet wastewater is one of the main sources of pollutants in municipal sewage. The commonly used end-of-pipe systems consume large amounts of land, energy, and water, resulting in high operation and maintenance costs [1]. On the other hand, human excreta contain large amounts of nutrients such as N, P, and K. These nutrients are crucial for plant growth and are costly to eliminate from wastewater [2]. Alternative toilet systems have been developed, such as ecosanitation systems [3], that use different methods and techniques, e.g., struvite crystallization [4,5], nitrification and distillation [6], and biochar adsorption [7,8], to achieve closed-loop cycles of water and nutrients. However, these techniques barely recover many of the nutrients in urine, and the water remaining after nutrient recovery via these methods needs further treatment.

Forward osmosis (FO) is a green membrane technology using the osmotic pressure differential between the feed solution and the draw solution as the driving force for enriching the feed solution. In addition, this technology has drawn growing attention in recent years because of the following characteristics [9–11]:

- No need for external high hydraulic pressure compared with other membrane processes;
- Low fouling intensities and easy recovery after fouling;
- Lower strength requirements for the membranes; and
- Energy savings if draw solution recovery is not considered.

Because of the characteristics of FO, it can be used to enrich solutions with high concentrations and large amounts of pollutants. Source-separated urine contains high concentrations of nutrients and some organics, and these nutrients are always difficult to eliminate when they are in municipal

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wastewater. Therefore, some researchers have studied the possibility of adopting FO for urine treatment. Zhang et al. [2] used synthetic urine to study the performance of a cellulose triacetate (CTA) membrane on urine enrichment and found that the rejection of organic nitrogen was rather low, only about 20%–50%; rejection of ammonia was higher 50%–80%; and rejection of both potassium and phosphorus was >90%. Xue et al. [11,12] found that the rejection of phosphorus was high (>90%) and almost constant at different pH levels, but rejection of nitrogen was sensitive to pH and varied greatly, from <20% to >90%. Using treated municipal wastewater, they also found that the initial water flux (about 4.5 LHM) could be maintained at its initial level for 30 d, and even longer with predisinfection. Ansari et al. [13] observed a 92% recovery of phosphate and a water flux decline of 30% from their initial values as digested sludge was concentrated threefold. However, drawbacks of applying FO in urine enrichment still exist: low water flux owing to the high concentration of urine, low rejection of nitrogen, draw solution contamination, etc. [2,14]. Moreover, most of the studies used synthetic urine with small-scale reactors in the lab, which could lead to different results than those for practical situations.

In this study, methods to improve the efficiency of urine enrichment using FO were studied and results from using synthetic urine or natural urine were compared. The optimization of operation conditions can help to promote water flux and rejection of nutrients, leading to decreased contamination of the draw solution. Furthermore, a pilot-scale sanitation system using FO as the key element was designed and built to verify the effectiveness of the FO process.

A urine-diversion vacuum toilet was adopted in the pilot-scale toilet. The source-separated urine and corresponding flushing water were enriched by FO, and the diluted draw solution from the FO process was further recovered by reverse osmosis (RO). After enrichment, the volume of urine was dramatically reduced, becoming convenient for transportation. The enriched urine with high concentrations of nutrients could be utilized as liquid fertilizer. The feces were digested to eliminate pathogens under mesophilic anaerobic conditions. A schematic diagram of the pilot-scale toilet system is shown in Fig. 1.

2. Materials and methods

2.1. Experimental setup

A diagram of the FO experiment is shown in Fig. 2(a). The experiment platform consisted of a flat sheet FO membrane module, peristaltic pump, flow meter, containers of feed solution and draw solution, hoses, electronic scales (Napco, JA31002 USA), conductivity meter (WTW, multi 3420, German), and data acquisition PC. The liquids and their velocity was driven and controlled by the pump. The water flux could be calculated from the weight increment of the draw solution, which was measured with a scale and recorded by the PC. A conductivity meter was used to record the conductivity of the liquids. Fig. 2(b) displays the module design of the FO chamber in the lab; the effective contact area was 0.023 m^2 (10 cm × 23 cm). Experiments were conducted intermittently, running for 4 h each at room temperature (25°C ± 2°C) in FO mode (feed facing the active layer).

The bench-scale experiments were planned in order to study the influencing factors of the FO process and to determine the relative parameters for designing the pilot-scale FO module.

2.2. Materials

2.2.1. Membrane

Five types of membranes were tested in the bench-scale FO process, namely CTA-NW and thin film composite (TFC) membranes (Hydration Technology Innovations, LLC, USA),

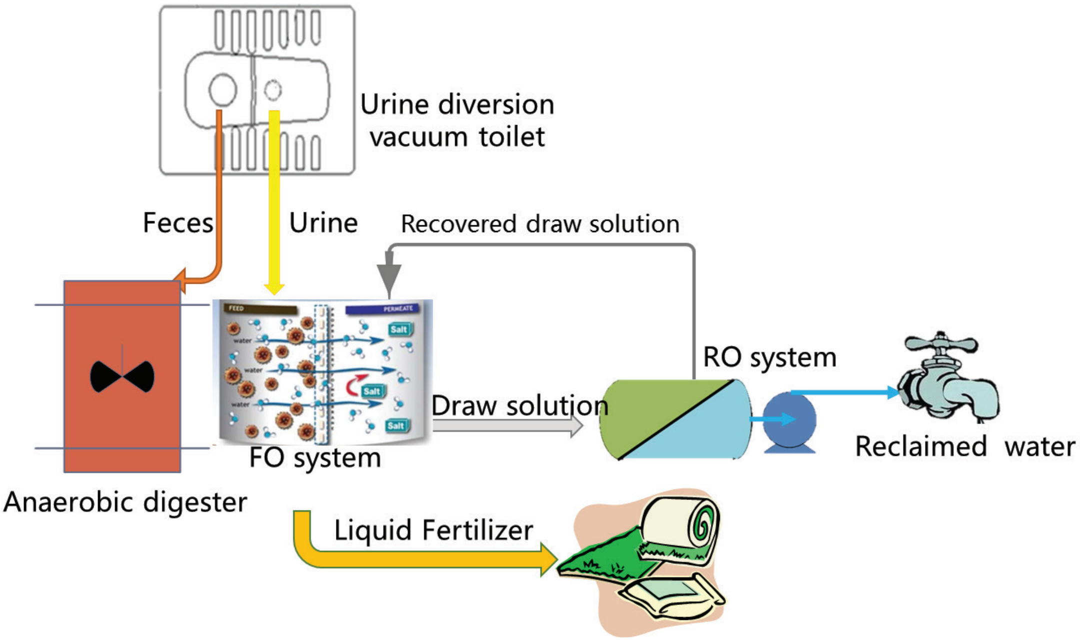

Fig. 1. Schematic diagram of the pilot-scale toilet system.

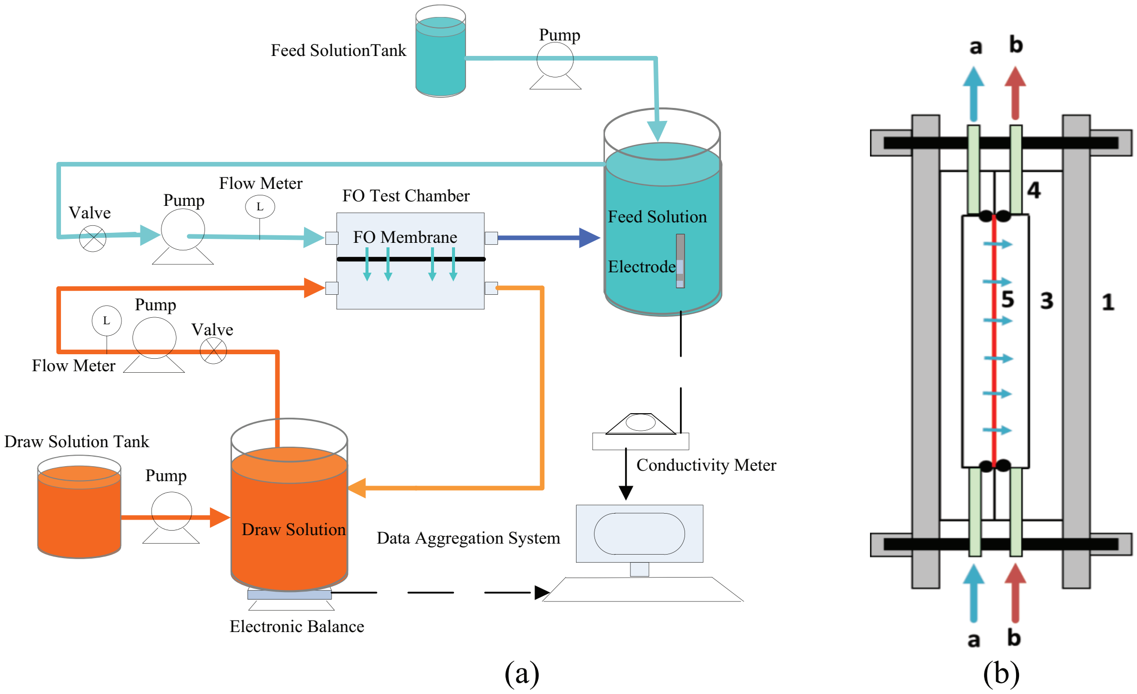

Fig. 2. Experimental setup for the (a) FO system and (b) FO module. 1: Fixed splint; 2: strap bolt; 3: flat-water channel; 4: draft tube; 5: FO membrane; a: feed solution; and b: draw solution.

AQuaPorin (AQP) membranes (FO AIM4010, Aquaporin, Denmark), and RO membranes (FELMETIC SW30, DOW, USA; directly used as an FO membrane) either unmodified or modified through elimination of the supporting layer. All membranes were soaked in ethanol for 2 h before the experiments to maintain their hydrophilicity and then washed with deionized water.

2.2.2. Feed solution

The feed solutions included synthetic urine and natural urine taken from the pilot-scale onsite toilet we designed and installed in Tsinghua Primary School in Beijing, China. The composition of synthetic urine was made according to Zhang's recipe for SU [2].

2.2.3. Draw solution

For the bench-scale process, the draw solution concentration was 0.5–4.0 M NaCl (AR, Sinopharm Chemical Reagent Co., Ltd, China).

Allowing for recovery via the RO process, the draw solution concentration for the pilot-scale toilet was 2.0 M NaCl.

2.3. Data collection and analysis

The weight loss or volume change of the feed was measured to calculate the water permeation of the membrane. Feed samples were taken before and after FO enrichment, and their ion and nutrient contents were analyzed. The ion concentrations were measured with an ion chromatograph (Dionex, ICS 2000, USA), and TN, TP, and NH_3–N were measured using alkaline potassium persulfate digestion UV spectrophotometric, ammonium molybdate spectrophotometric, and Nessler's reagents spectrophotometer, respectively. Scanning electron microscopy (SEM) was employed to observe the surface of the FO membrane.

The water flux (J_w, LHM) was calculated using Eq. (1):

$$J_w = \Delta w/(\rho s t) \tag{1}$$

where Δw is the weight incremental of draw solution (kg); ρ is the density of liquid (kg/L), s is the effective area of membrane (m^2), and t is the time interval (h).

The rejection of nutrients in the feed was calculated with Eq. (2):

$$R_f = 1 - \frac{C_p}{C_f} \tag{2}$$

where C_f is the concentration of the feed solution (mol/L), and C_p is the concentration of the permeate (mol/L). In this study, the average ± standard deviation and error bars were based on triplicate measurements.

2.4. Pilot-scale toilet system

After bench-scale experiments, the main parameters of the onsite system were determined. The pilot-scale toilet with resource recovery system was installed at the northwest corner of the playground of Tsinghua University Primary School. The system consisted of a toilet with six squatting positions and a treatment system, and its capacity was 100 users per day. A schematic diagram of the pilot-scale toilet system is shown in Fig. 1. The main design parameters of the treatment system are listed in Table 1.

Table 1
Main design parameters of the treatment system

Units	Indicator	Value
Toilet	Flush water	Urine <0.1 L/flush Feces <1.0 L/flush
FO module	Module type	Flat sheet
	Membrane area	20 cm × 60 cm × 100 pieces Effective area = 7.5 m^2
	Operation pressure	0.2 MPa
	Flow rate	3,500 L/h
RO module	Module type	Spiral wound
	Membrane area	Effective area = 8.7 m^2
	Operation pressure	6.8 MPa
	Flow rate	1,000 L/h
Anaerobic digester	Volume	1.5 m^3
	Retention time	25 d
	Temperature	35°C

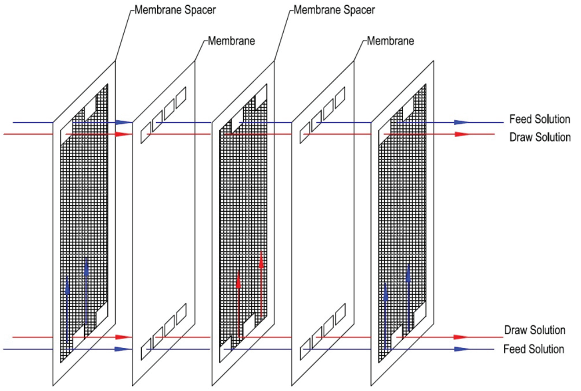

Fig. 3. Structure of the FO module.

To increase the packing density of the FO membrane, a flat-sheet module with 100 pieces of membrane was designed, referring to the module design of electro-osmosis. The structure and the flow within the module are shown in Fig. 3. The mesh spacer was 60 cm × 20 cm, and the pore size of the mesh spacer was 2 mm × 2 mm.

3. Results and discussion

3.1. Results of the bench-scale FO process

3.1.1. Separation performance comparison of different membranes

The most commonly used commercial membranes were selected to test their basic performance under a “standard” condition (1.0 M NaCl as draw solution and deionized water as feed solution) in the FO mode (active layer facing the feed). These basic tests were aimed to contrast these membranes under comparable conditions as those in other studies and screen out the unreliable membranes.

The comparison of water flux and rejection of different membranes can be seen in Fig. 4. The results showed that the CTA and TFC membranes achieved the best performance with water fluxes of 23.6 and 20.5 LHM, respectively, and rejection of 99.8% and 99.6%. The RO membranes had slightly higher rejection, but the water flux was only 1/3 for the CTA/TFC membranes. The modification of the RO membrane contributed to an increase in water flux mainly due to the decrease of concentration polarization [15]. This improvement in water flux made it possible to adopt the modified RO membrane in the FO process, to overcome the unavailability and expensive price of FO membranes [16]. However, the strength of the RO membrane was reduced after modification of eliminating the supporting layer. The newly commercialized AQP membrane had a rather unsatisfying performance, with water flux of only 3.12 LHM and rejection of 85.6%. The AQP membrane incorporates the functional water channel protein aquaporin to improve water productivity [17], however, the test results showed that this commercialized AQP membrane was insufficient in application in the FO process yet.

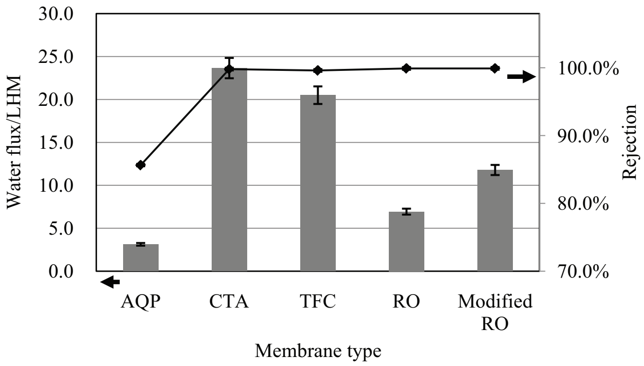

Fig. 4. Water flux and rejection of NaCl for the five membrane types studied.

3.1.2. Influence of draw solution concentration on water flux

A range of different concentrations of NaCl was applied to explore the most suitable concentration for practical application. The influence of draw solution concentration on water flux is revealed in Fig. 5.

As is shown in Fig. 5, the water flux was supposed to increase linearly with the draw solution concentration, however, the increasing rate of the curve declined with higher draw solution concentration. This was mainly due to the loss in ionic activity and the increase in dilutive internal concentration polarization and concentrative external concentration polarization [18].

In the aspect of draw solution recovery, the increment in concentration of draw solution would lead to more energy consumption [19]. As the diluted draw solution was designed to be recovered by the RO process in the toilet, the cost/benefit needed to be considered as well as the limitation of the pump pressure of the RO process. Therefore, 2.0 M NaCl solution was chosen as the draw solution [20]. Under this concentration of draw solution, a high-pressure pump with a pressure of 6.80 MPa was needed in the RO system for the recovery of draw solution.

3.1.3. Influence of cross-flow velocity on water flux

The influence of the cross-flow velocity on water flux is demonstrated in Fig. 6. Cross-flow velocity mainly affects the external concentration polarization next to membranes. Higher flow rate contributes to the turbulence next to membranes and mass transfer across membranes [21]. Moreover, higher velocity has a positive effect on the decrease of membrane fouling because of the turbulence [22]. However, these effects became limited when the cross-flow velocity increased to a certain level, around 1,200 mL/min. After that, the promotion in flow velocity helped little in increasing efficiency of the process yet consumed more energy. In the module design and operation, approximately 1,100 mL/min (equivalent to 11.3 cm/s) of cross-flow velocity suitably balanced the efficiency and energy consumption [23]. With this parameter, the FO module in the onsite test used circulation pumps with pumping capacity of 3,500 L/h (equivalent to 11.8 cm/s).

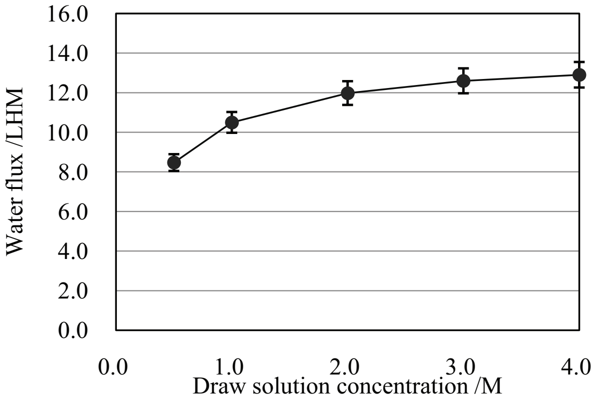

Fig. 5. Influence of draw solution concentration on water flux.

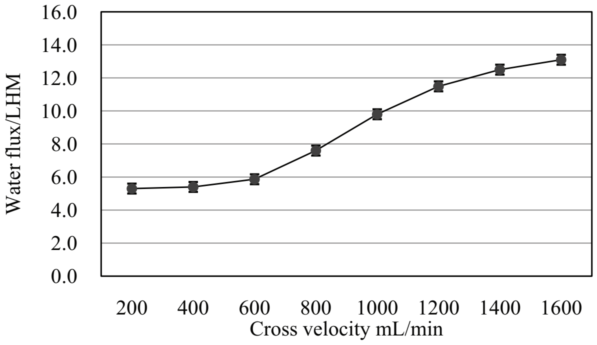

Fig. 6. Influence of water flow rate on water flux.

3.1.4. Concentration effect using synthetic and natural urine

The enrichment results for synthetic and natural urine via the FO process are listed in Table 2. The CTA membrane was used to conduct the urine enrichment experiment, and the draw solution concentration was 2.0 M NaCl. The urine was concentrated twice (volume reduced to 1/2 after concentration). The synthetic urine was made according to Zhang's recipe for SU [2], and the natural urine was taken from the pilot-scale onsite toilet we installed at Tsinghua Primary School.

The results showed that the natural urine had lower nutrient concentrations in comparison with the synthetic urine, especially for nitrogen. It was estimated that the difference came from the excessive flushing and cleaning water. In addition, the volume and composition of kids' urine (the natural urine was collected from the pilot-scale toilet, whose users were mostly primary students) could be different from those of adults (Zhang's recipe for SU [2] was based on the average composition of urine from Euro-American adults). Moreover, huge differences exist in the dietary habits of Chinese residents and residents in other countries. Chinese people usually have lower protein intake, resulting in lower nitrogen contents in their excreta. A minor reason for the low concentration of nitrogen in the natural urine was the ammonia loss during urine storage.

Another obvious difference between synthetic urine and natural urine was that most of the nitrogen (NH_3–N/TN = 98%) in the synthetic urine was NH_3–N but this ratio in natural urine was only 66%. The reason for this difference is that in Zhang's recipe for SU [2], the nitrogen was provided almost entirely by ammonium carbonate ($(NH_4)_2CO_3$), whereas natural urine contained other forms of nitrogen such as organic nitrogen and nitrites and nitrates.

For the FO enriching results, the rejections of TP and K were approximately 97% and 85%, respectively; however, the rejection of nitrogen was not ideal. Because the most important principles for the rejection of particles are physical screening effect and electrostatic interaction [2], the low rejection of nitrogen primarily occurred because of its small particle size and low charged state.

3.1.5. Influence of pH on nutrient rejection

The pH of the feed was adjusted to promote the rejection of nutrients, especially nitrogen. Rejection of single nutrients under different pH levels was studied using KCl, NH_4Cl, and NaH_2PO_4, with the same nutrient concentrations according to Zhang's recipe for SU [2]. The pH of these solutions was

Table 2
Nutrient concentrations of urine before and after the FO process

Urine type		TP (g/L)	TN (g/L)	NH_3–N (g/L)	COD_{Cr} (g/L)	K (g/L)
Synthetic urine	Before	3.21	12.5	12.3	4.55	0.84
	After	6.24	16.9	16.3	7.63	1.48
	Rejection	97.2%	73.4%	65.8%	83.9%	88.1%
Natural urine	Before	2.11	2.46	1.63	4.03	0.58
	After	4.14	3.16	1.78	6.96	0.97
	Rejection	97.9%	64.5%	54.7%	86.3%	84.1%

adjusted with 1 M NaOH or HCl. The results illustrated that the rejection of K and P remained almost constant under different pH levels, however, the rejection of ammonia changed dramatically. The rejection of each nutrient is shown in Fig. 7.

The reason for the differences in rejection change is that K remains as K^+ under different pH levels and P particles are large enough to be rejected by the membrane, thus they do not vary. However, nitrogen was basically in the form NH_4^+ when pH < 7, and the membrane had higher rejection of these charged ions than it did of the uncharged molecules (NH_3). When pH increased, the NH_4^+ converted into NH_3. Though the concentrations of nutrients were much lower than those in urine, a similar phenomenon was observed in municipal wastewater by Xue et al. [12].

As ammonia rejection was most sensitive to pH variation, the rejection of total nitrogen was further compared in the synthetic and natural urines. The results are presented in Fig. 8. If the synthetic urine was sufficient for simulation of natural urine, the rejection under different pH levels should be similar. However, the results showed obvious differences in rejection. The highest rejection was achieved at pH = 7 for natural urine whereas for synthetic urine it was at pH = 5.

The nitrogen rejection of synthetic urine was similar to the rejection curve of ammonia (Fig. 7), because in Zhang's recipe for SU [2], the only nitrogen form was NH_4^+–N, provided by $(NH_4)_2CO_3$.

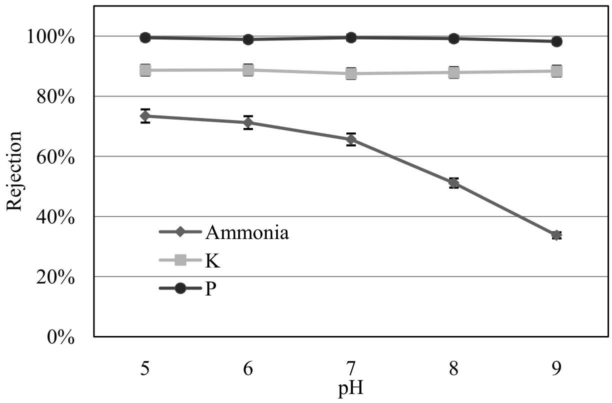

Fig. 7. Influence of pH on the rejection of each nutrient.

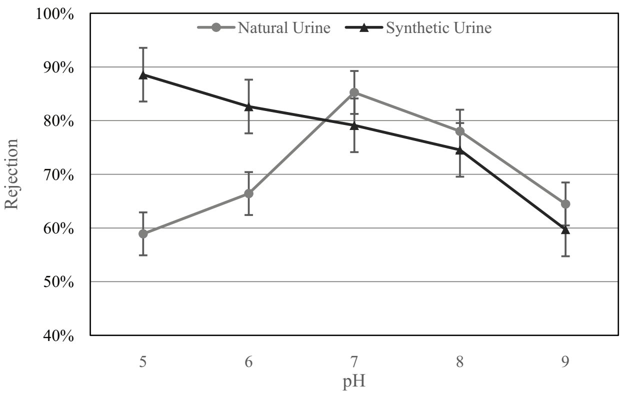

Fig. 8. Influence of pH on nitrogen rejection in natural urine and synthetic urine.

However, the natural urine was collected from the pilot-scale toilet. In this practical situation, the urine diversion toilet is unable to separate urine from feces completely, and toilet paper could also get into the urine. The composition of natural urine could be much more complex than synthetic urine under these conditions. Thus, the nitrogen in natural urine could exist in different forms, such as NH_4^+–N, NO_2^-, and NO_3^-. When pH = 7, ammonia mainly existed as NH_4^+, and nitrate had relatively higher rejection than at lower pH. When the pH increased, the rejection of nitrite was higher, but the rejection of ammonia decreased. Taken together, the highest rejection of total nitrogen was achieved when pH = 7 [24,25]. This result corresponded to the research of Xue et al. [12]. This result indicated that although the synthetic urine was used as a simulation of natural urine, some features were not suitable enough because practical situations can be complicated. Though nutrient concentrations in the urine were much higher than in municipal wastewater, the constituents of nitrogen were similar in certain ways.

3.2. Operational results of the pilot-scale system

3.2.1. Overall appearance of the system

The layout of the toilet wastewater treatment system is shown in Fig. 9(a), and the urine diversion toilet is shown in Fig. 9(b). The human excreta were collected separately owing to the special design of the toilet stool, which was divided into two parts, a collecting bowl for urine and one for feces.

3.2.2. Performance of the whole toilet system

The collected urine was diluted about 1.3–1.5 times by the flushing water (0.1 L of flushing water and 0.2–0.3 L urine). After the FO process, the urine could be enriched 2.5-fold, the volume reduced enormously, and the concentration of nutrients increased. The rejection of nutrients in the pilot-scale FO module was similar to the results in the bench-scale experiments. Thus, the processed urine could be used as a liquid fertilizer.

The reclaimed water was produced from urine, after pretreatment by FO, with the RO process producing a very high-quality reclaimed water for reuse of toilet flushing and irrigation with slightly high total dissolved solids (TDS) (generally came from the unrejected NaCl in the draw solution). Moreover, the clean water reclaimed from the system could not satisfy the need for flushing completely if there were not enough users (<50 users per day) of the system.

The main onsite operation results of the pilot-scale sanitation system are listed in Table 3.

3.2.3. Performance of the FO module

The FO module ran intermittently, depending on the production of urine. In the FO process, the initial water flux was approximately 2.0–2.5 LHM, but the water flux decreased sharply within 30 min, to <1 LHM. After each process, the module was backwashed with clean water, and the water flux recovered to almost 100% of its initial value. It was estimated that the decrease in water flux mainly came from clogging

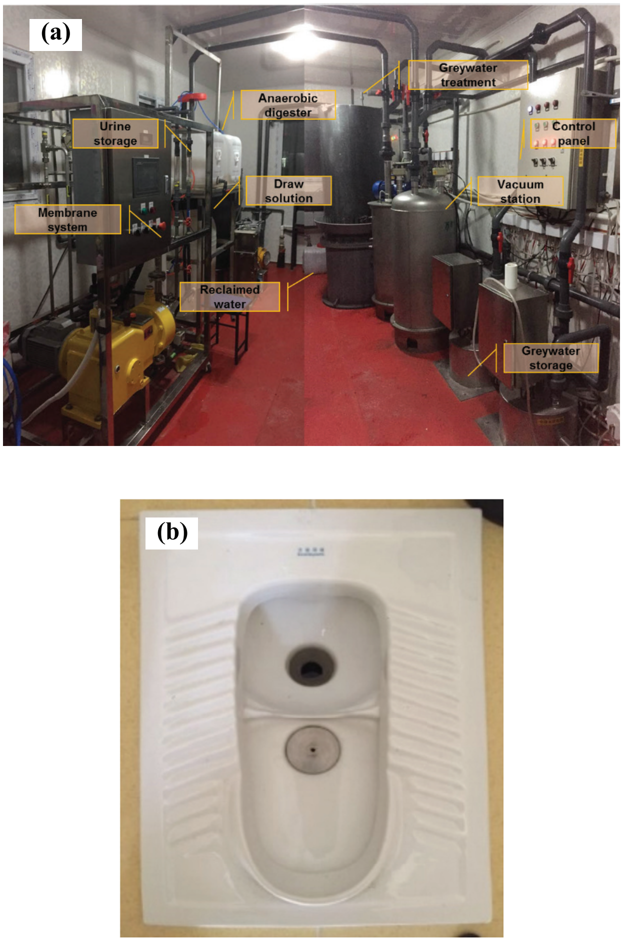

Fig. 9. The pilot-scale toilet wastewater treatment system (a); and the urine diversion toilet (b).

Table 3
Main onsite operation results of the pilot-scale sanitation system

Stream type	Indicator	Value
Urine concentration	Volume	Before enrichment: ~15 L/d After enrichment: ~6 L/d
	pH	9.1–9.3
	N + P + K	Before enrichment: ~5 g/L After enrichment: ~10 g/L
Reclaimed water	Volume	~9 L/d
	Quality	Completely safe for toilet flushing with slightly high TDS ~900 mg/L
Feces	Volume	<1 L/d
	Sterilization	Egg removal >99%; Fecal *E. coli* $<10^{-3}$
	Retention time	>40 d

and fouling of the FO module by particles in the urine. In the end, the average water flux for the FO module was only 0.5–0.7 LHM. For a better understanding of the decrease in water permeability, SEM of the membrane was performed to see the fouling condition after operation for 1 month. Comparing with an unused membrane, some pollutants could be seen on the membrane surface, clustered as irregular crystals, but the inner pores of the membrane remained clean.

This phenomenon demonstrated that the pollutants were merely adhering to the surface; thus, they could be easily removed by simple physical backwash. Moreover, it indicated that the FO process behaved well regarding membrane fouling and flux recovery [26,27] under the application situation.

The performance of the FO module showed high potential for the adoption of the FO process to enrich urine, but because of the imperfections of the separated collection of urine and membrane material, the system is not ideal for now. For further study of the application of the FO process in urine enrichment, promotion of the urine diversion toilet, integration of pretreatment methods for urine, and improvement in membrane materials and FO module design are necessary.

4. Conclusions

In this study, the influence of membrane material, draw solution concentration, flow rate, etc. was studied for application of the FO process in urine concentration. The most suitable operation condition was achieved with a CTA membrane, 2 M NaCl as the draw solution, and 11.3 cm/s crossflow velocity. The most suitable pH for the enrichment of source-separated urine was 7.0. Under these circumstances, the rejection of N, P, and K reached 80%, 98%, and 85%, respectively. Nutrient concentration increased along with a reduction in urine volume, thus indicating the potential for use as liquid fertilizer. The results showed a promising method of urine utilization via enrichment using the FO process to recover nutrients. Integrated with other techniques, toilet wastewater will no longer be a burden but rather a resource.

Acknowledgments

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