

Polyethylene Glycol-Coated Magnetic Nanoparticles-Based Draw Solution for Forward Osmosis

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Abstract

Forward osmosis (FO) is a promising technology for urine volume reduction to ease the recovery of nutrients. Its efficiency is highly dependent on the draw solution. Hence, functionalization of novel draw solutions for forward osmosis (FO) has become a subject of intense investigation. Coated magnetic nanoparticles (MNPs) based solutions offer great potentials for their ability to generate osmotic pressure as well as their easy recovery. Since concentrated urine features high osmotic pressure, we aim to synthesize a high osmotic pressure generating draw solution. Polyethylene glycol (PEG), is an attractive coating agent as aqueous Polyethylene Glycol solutions are known to generate high osmotic Pressure. Moreover, PEG are hydrophilic and expected to have physical aggregation suppression. In this study, we adopted coprecipitation method to synthesize PEG 4000 coated MNPs as draw solution, and studied the influence of the initial coating agent amount and sonication effect on the coating ratio and the osmotic pressure generation of solutions made of the synthesized particles. We found that initial PEG to MNP ratio affects the coating ratio. Indeed, higher coating ratio is obtained with higher initial PEG to MNP ratio. A PEG to MNP ratio of 1 to 4 led to 31% coating ratio. This coating ratio can be slightly increased if the synthesized draw solution is treated with ultrasound for 30 minutes. Water flux data collected from forward osmosis experiment revealed that this novel draw solution generates osmotic pressure. The osmotic pressure obtained from a draw solution containing a given amount of PEG 4000, is larger than the osmotic pressure if the same amount of PEG 4000 is used alone. Fate of the osmotic pressure of the novel draw solution following recycling of the synthesized MNPs is also evaluated. It was found that the drop in osmotic pressure of the regenerated draw solution is insignificant, proving possibility to reuse this draw solution for many cycles.

Keywords: urine, nutrients, forward osmosis, draw solution, MNPs, PEG 4000

Introduction

Urine diverting toilets were proposed to provide safe and affordable sanitation and to facilitate the separation of human waste products for easy resource recovery (Rieck et al. 2012). The urine fraction is a nutrient-rich solution that can be used as a liquid fertilizer (Sene et al. 2012; 2013). However, its large volumes make its transportation to farmland uneconomical (Pahore et al. 2010), making urine volume reduction necessary. However, this process faces not only the challenge of treatment costs but most available volume reduction technologies are energy demanding as well. Hence, forward osmosis (FO)—a naturally occurring phenomenon—has emerged as a promising

low-energy technology for urine volume reduction and resource recovery (Nikiema et al. 2017). The driving force behind FO is the osmotic pressure difference between the feed and draw solutions of different concentrations that are separated by a semi-permeable membrane. Although the operation of FO requires low energy overall, recovery and regeneration of the draw solution requires a high-energy input. Therefore, the choice of draw solution is of the utmost importance. Investigations on novel draw solutions have interested many researchers as exemplified by the works of Guizani et al. (2019), Ge et al. (2011), Yang et al. (2013), Campos et al. (2015), and Ling et al. (2010) among others. Special interest has been given to the functionalization of coated magnetic nanoparticles (MNPs) due to their ease of recovery with a magnet (Ling et al. 2010). The ultimate objective of our research was to synthesize an easy-to-recycle and high osmotic pressure-generating draw solution. It is worth mentioning that urine that has been concentrated 5 folds is characterized by an osmotic pressure exceeding 10 MPa (Nikiema et al. 2017). However, due to their large size, in comparison to the common electrolytes, MNPs do not exhibit high osmotic pressure properties when present in solutions on their own (Guizani et al. 2018). To overcome this limitation, MNPs are coated with osmotic pressure-generating polymeric agents. Researchers have evaluated the usage of different coating materials, such as dextran, oleic acid, polyacrylic acid, and tri-sodium citrate (Zhang 2006; Hong et al. 2009). While many coating agents were tested here, it was possible to suppress aggregation by covering the particle surface with a hydrophilic coating agent to increase the coating ratio and particle concentration. However, the osmotic pressure achieved was low and impractical, and thus, insufficient for concentration of solutions, such as urine or wastewater. Polyethylene glycol (PEG) is an attractive coating agent, owing to the capability of aqueous polyethylene glycol to generate high osmotic pressure (Money 1989). In this study, PEG 4000 (with an average molecular weight of 4000), which is a hydrophilic compound expected to suppress physical aggregation, and thus, generate high osmotic pressure, was used as a coating agent.

Micro-emulsion, thermal decomposition, and chemical coprecipitation are the major common methods used to synthesize iron oxide MNPs (Majidi et al. 2016). Amongst these, thermal decomposition is the most common method for the purpose. It is reported that PEG can synthesize uniform particles of about 10-nm diameter/radius via the thermal decomposition method and generate an osmotic pressure of about 2.8 MPa (Ge et al. 2011). However, this route was recently proven to induce defects and negatively affect the properties of the MNPs. In this study, we adopted the co-precipitation method to synthesize PEG 4000-coated MNPs as a draw solution due to its convenience, simplicity, and low cost; we also studied the influence of the initial amount of coating agent on the coating ratio and the osmotic pressure of a draw solution prepared from the synthesized particles. The fate of the osmotic pressure of the novel draw solution, following recycling of the synthesized MNPs, was also evaluated.

1. Material and Methods

1.1. Draw solution synthesis

Magnetic nanoparticles were prepared using the co-precipitation technique (Figure 1). Two-hundred millilitres solutions of analytical grade iron chloride (III) · hexahydrate (8.59 g) and iron chloride (II) · tetra-hydrate (23.35 g) were mixed thoroughly. Afterwards, 100 mL of alkaline sodium hydroxide solution (5 M) was added to the blend under conditions of nitrogen gas purging and was kept for an hour in a warm bath (80°C). Upon addition of the alkaline solution, a black precipitate was formed. It has been confirmed in an earlier study (Guizani et al. 2018) that this black precipitate is magnetite (Fe_3O_4), whose formation takes places through reactions described in equations (1), (2), and (3). As per these chemical equations, a yield of 10 g Fe_3O_4 was expected from the aforementioned recipe, which was confirmed experimentally. Following the addition of the aqueous solution of sodium hydroxide and an hour of continuous mixing, 100 mL each of solutions containing 2.5, 5, or 40 g PEG 4000 was added; the

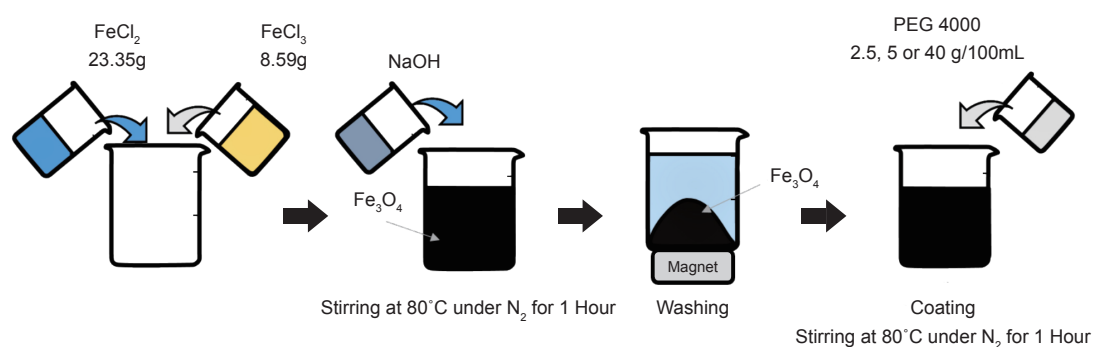


Figure 1. Synthesis of PEG-coated magnetic nanoparticles by the co-precipitation method.

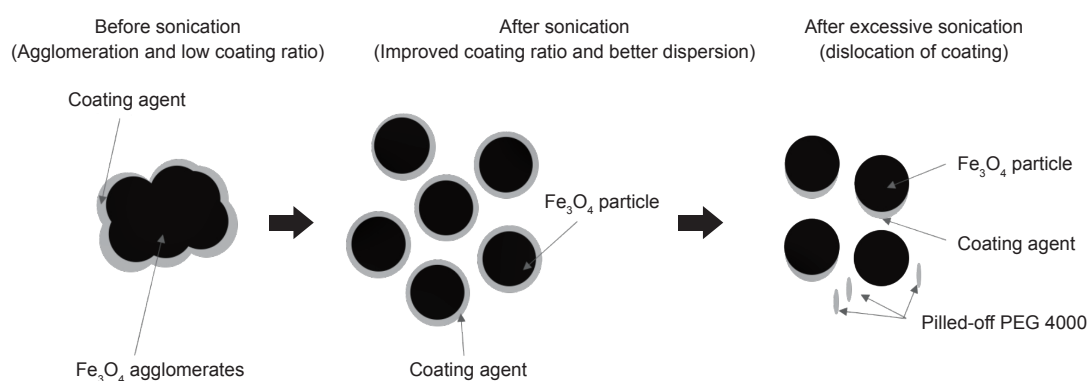
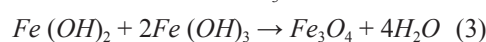


Figure 2. The mechanism of working of the ultrasonic homogenizer (UH 50) used in this study. (Adopted from Guizani et al. 2019)

mixtures thus prepared were stirred at 800 rpm for 1 h at 80°C. These amounts corresponded to the initial MNP to PEG ratios of 1:0.25, 1:0.5, and 1:4. The synthesized particles were washed several times until the pH was approximately 7. A neodymium magnet was used to recover magnetite particles at each washing step.



1.2. Assessment of the coating ratio

Coating ratio was determined using the gravimetric method. After the coated MNPs were dried at 105°C; they were then burned at 800°C. The amount of mass reduction with respect to the initial mass of MNPs was taken as the coating ratio.

1.3. Ultrasonic treatment

To improve coating ratio, ultrasonication was applied. In an earlier study by Guizani et al. (2019), it was confirmed that the average particle diameter of magnetite decreases by application of ultrasonic treatment. Moreover, the agglomeration of particles during particle recovery using a magnet was confirmed. Therefore, it was expected here that the coating ratio could be increased as a result of increase in surface area. The ultrasonic treatment was carried

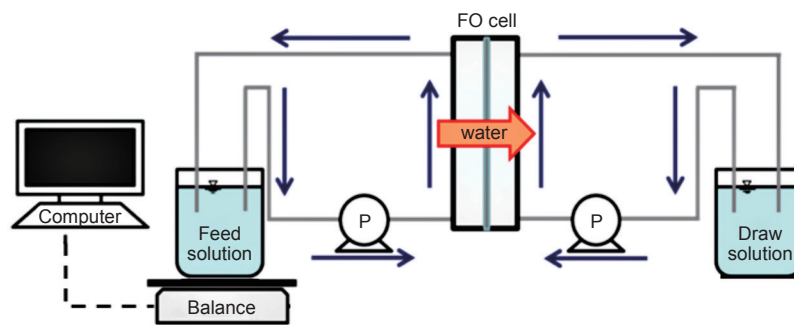


Figure 3. Forward osmosis experiment (draw solution + magnetic nanoparticles; feed solution deionized water).

Table 1. Forward osmosis experimental conditions.

| | |
|-------------------------|----------------------------|
| Flow rate | 0.20 m/s (co-current) |
| Operation time | 1 h |
| Membrane | Cellulose triacetate (CTA) |
| Effective membrane area | 98 cm ² |

out using a 20-kHz, 50-watt probe ultrasonic homogenizer, UH 50 (SMT Corporation, Japan). Figure 2 illustrates the working mechanism of sonication and its expected effect on dispersion and coating ratio. The mixture of MNPs and PEG 4000 was sonicated for 30 min and then stirred for another 60 min.

1.4. Synthesis of the draw solution

An FO device with an effective membrane area of 98 cm² was used with a cellulose triacetate (CTA) membrane sandwiched between symmetrical flow paths with a width of 1 cm and a height of 0.2 cm (Figure 3). The CTA membrane was purchased from Fluid Technology Solutions (FTS, Inc., Albany, USA). The feed (deionized water) and driving (PEG-coated MNPs) solutions were circulated for an hour in co-current mode at a flow rate of 0.2 m/s using two peristaltic pumps. The objective of this experiment was to assess whether this synthesized solution could be used as a draw solution. In other words, we aimed to know whether this solution would generate an osmotic pressure that would allow water to move across the semi-permeable membrane from the feed solution toward itself. For this purpose, the mass change on the feed side was measured using an electronic balance and the water flux was calculated. Equal volumes of 200 mL each of the draw and feed solutions were used in the experiment. Three different coated MNP-based solutions were used as draw solutions with particle concentrations of 2.9, 4.9, and 9.6 wt.%. The feed solution consisted of deionized water. Details of the operational conditions are presented in Table 1.

1.5. Estimation of differential osmotic pressure

Osmosis is the movement of water from an area of low concentration toward that of high concentration. The higher the osmotic pressure difference is, the higher is the water flux. In our experimental setup, the movement

of water depended on the strength of the osmotic pressure difference between the feed and draw solutions. The osmotic pressure can be calculated from the molar concentration of each solution as given in equation (4).

$$\Delta\pi = iMRT \quad (4)$$

Where M is the molar concentration of dissolved species (mol L^{-1}), R is the ideal gas constant ($0.08206 \text{ L atm mol}^{-1} \text{ K}^{-1}$), T is the temperature (K), and i is the van't Hoff factor for the solute.

It has been suggested that water flux is linearly proportional to the osmotic pressure difference as given in equation (5).

$$J_w = k\Delta\pi \quad (5)$$

Where J_w is the water flux ($\text{L m}^{-2} \text{ h}^{-1}$), k is the water permeability coefficient ($\text{L m}^{-2} \text{ h}^{-1} \text{ bar}^{-1}$), and $\Delta\pi$ is the osmotic pressure difference (bar).

It is worth mentioning that for each new membrane lot used, the membrane permeability coefficient was determined. An FO experiment was conducted using deionized water as the feed solution and NaCl as the draw solution. Pure water flux was measured during the first 10 min of the operation of FO; its values were plotted against the osmotic pressure applied. The membrane permeability coefficient (k) was obtained from the slope of equation (5). Once the permeability coefficient of the membrane was known, the unknown osmotic pressure of a newly tested draw solution could be obtained by dividing the water flux obtained from the FO experiment by the permeability coefficient. However, Wang et al. (2016) have reported that the water fluxes of CTA membranes deviated from the theoretical fluxes obtained from this linear equation. Indeed, concentration polarization makes the flux drop below the theoretical values. At higher osmotic pressure, the concentration polarization is larger, leading to a larger difference from theoretical values. A similar phenomenon was observed by Matsuda (2017) and Nikiema et al. (2017).

For the PEG-coated MNPs, the generation of osmotic pressure in the coated ferromagnetic particles is still not understood well. Therefore, no theoretical equation is available in the literature to mathematically represent the phenomenon. For the sake of simplicity, the differential osmotic pressure has been estimated from the water flux data using the aforementioned linear equation.

2. Results and Discussion

2.1. Coating ratio

As illustrated in Figure 4, the coating ratio increased as a result of an increase in the amount of the coating agent. The coating ratio increased from 3% to 6% and then to 31%, respectively, for 2.5, 5, and 40 g of PEG 4000. In other words, for 10 g of magnetite, only 0.3, 0.6, and 3 g of PEG 4000 effectively coated the magnetite, respectively.

The work of Guizani et al. (2018) revealed that beyond a certain initial MNP:coating agent ratio, the coating ratio will not be improved, rather it will only decrease. This is attributed to the fact that excessive amounts of coating agent will cause it to peel off from the MNPs leading to a drop in the coating ratio. The results obtained in this paper do not reveal the optimum coating ratio.

As shown in Figure 5, the use of ultrasonic treatment improved the coating ratio. Indeed, an increase of coating ratio from 31% to 40% was achieved. This increase was most likely due to the dispersion of aggregated particles by sonication, which led to a larger surface area being exposed for coating. A detailed study on the effect of the

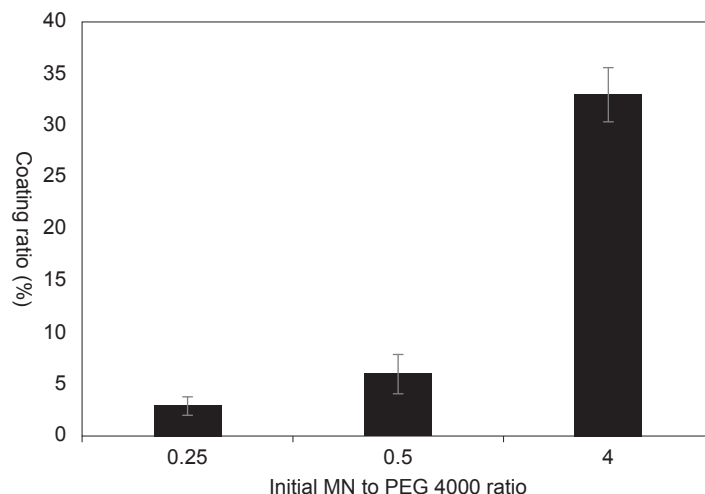


Figure 4. Effect of initial magnetic nanoparticles to polyethylene glycol 4000 ratio on the coating ratio. (Error bars show standard deviation (SD))

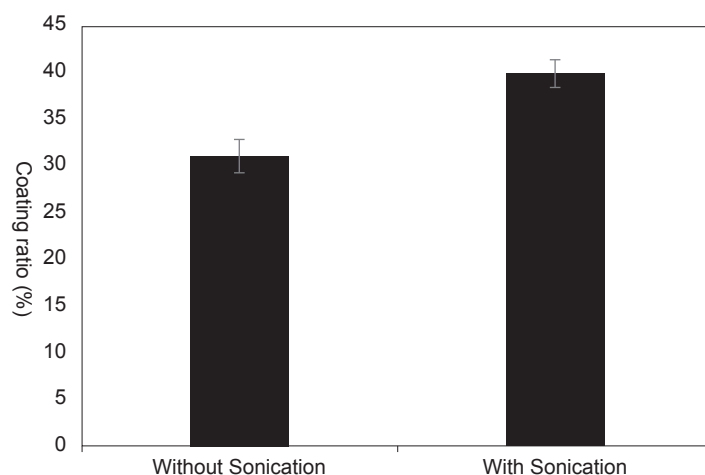


Figure 5. Effect of sonication on the coating ratio. (Error bars show standard deviation (SD))

ultrasonic treatment can be found in the work by Guizani et al 2019.

Optimized conditions for synthesis and coating were adopted for the rest of the study. In other words, further experiments were conducted using an initial MNPs to PEG 4000 ratio of 1 to 4. After sonication, the coating ratio was 40%. Solutions containing wt.% of 2.9, 4.9, and 9.6% were prepared for measurement of water flux and estimation of osmotic pressure. The amount of PEG 4000 in the solution was calculated using the 40% coating ratio.

2.2. Water flux measurements and estimation of osmotic pressure

Figure 6 illustrates the time course of weight change in the feed tank (feed solution weight). The change reflects the movement of water from the feed to the draw side as the amount of water decreases in the former. The experiment was conducted for an hour, followed by calculation of the water flux. As an example in Figure 6, the water drops from 200 g to 191 g over a one-hour period, providing a flux of one litre per square meter per hour (LMH). Using the permeability constant, differential osmotic pressure was obtained. Matsuda et al. (2017)

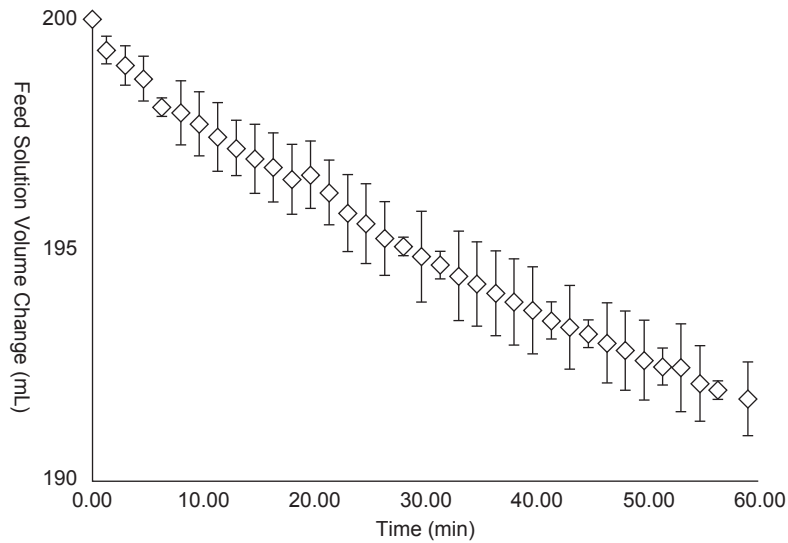


Figure 6. Time course of weight change in feed solution reservoir: Draw solution composed of coated magnetic nanoparticle solution with particle concentration of 9.6 wt.%. (Error bars show standard deviation (SD))

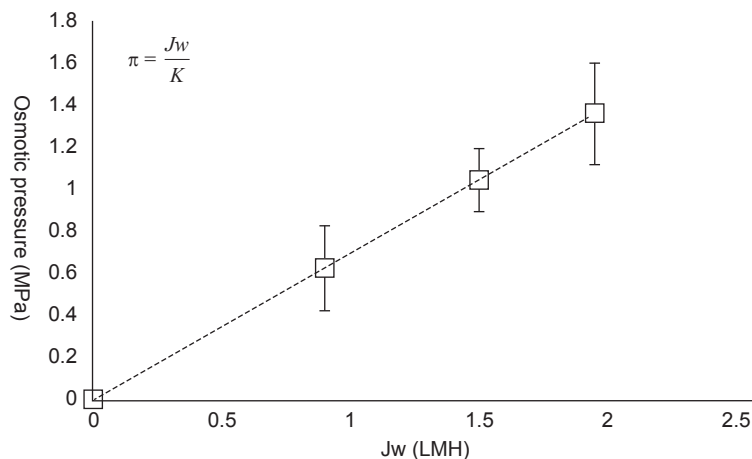


Figure 7. Estimated osmotic pressure versus water fluxes. (Error bars show standard deviation (SD))

measured the permeability of the membranes used in this study with the same method described in Section 2.5. The authors reported an average value of $1.44 \text{ L m}^{-2} \text{ h}^{-1}$ per MPa. Using this permeability value, osmotic pressure ranges were estimated to be 0.6, 1.04, and 1.37 MPa, respectively for 2.9, 4.9, and 9.6% wt.%, respectively (Figure 7). However, as reported by Wang (2015) and Matsuda (2017) for CTA membranes, water flux does not obey the linear relationship due to the polarization of internal concentration. In our study, for a given osmotic pressure, the observed flux was recorded to be much lower than the ones estimated using the linear relationship, i.e. the osmotic pressure estimated using permeability coefficient was underestimated.

2.3. Osmotic pressure versus wt.%

In case of an ideal solute, the Van't Hoff's relationship is linear between osmotic pressure and molarity; this

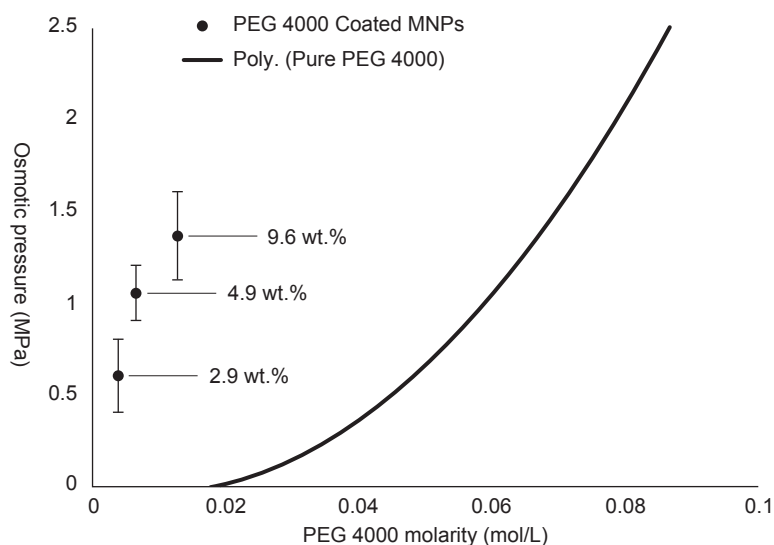


Figure 8. Relationship between concentration (wt.%) and osmotic pressure for PEG 4000-coated MNPs.
(Error bars show standard deviation (SD))

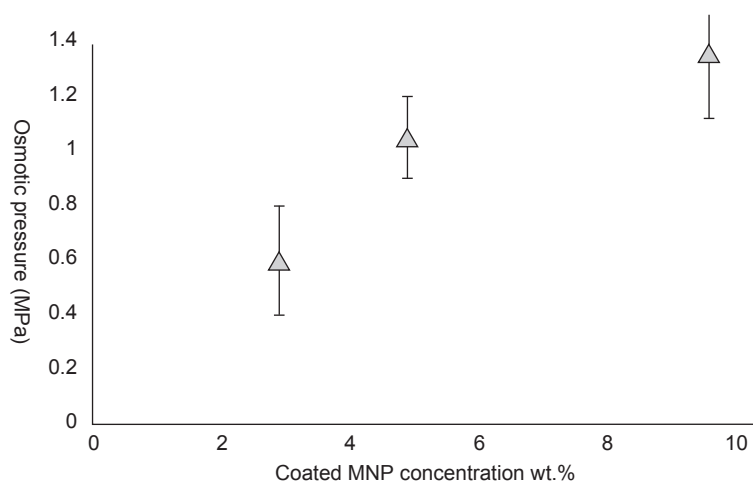


Figure 9. Relationship between concentration (wt.%) and osmotic pressure for PEG 4000 coated MNPs.
(Error bars show standard deviation (SD))

applies to solutions of inorganic salts, such as sodium chloride and potassium chloride. However, polyethylene glycol is reported to not behave as an ideal solute in an aqueous solution. Money (1989) reported that the relationship between concentration and osmotic pressure is not linear, instead it is a second-order polynomial relationship (Figure 8). Steuter et al. (1981) explained this using the fact that, in the case of high-molecular weight solutes, large polymers break into subunits; the higher total number of subunits may then lead to a higher osmotic pressure.

Figure 8 illustrates the osmotic pressure of PEG-coated MNPs and an aqueous solution with PEG 4000 alone. It reveals that PEG 4000-coated MNPs could generate an osmotic pressure of 0.6, 1.05, and 1.37 MPa, respectively, at

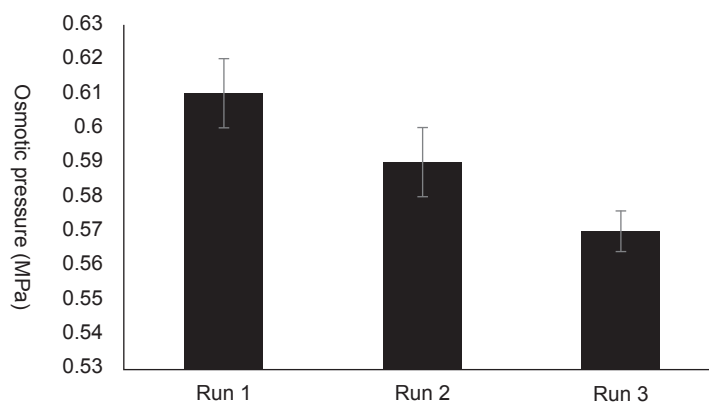


Figure 10. The osmotic pressure of the regenerated draw solution.
(Error bars show standard deviation (SD))

weight concentrations of 2.9, 4.9, and 9.6 wt.%, respectively. It is worth mentioning that at the same concentration levels of PEG, higher osmotic pressure was generated by PEG-coated MNPs, in comparison to the aqueous solution containing PEG only. This could be attributed to the fact that contained electrolytes produce stronger osmotic pressure than free uncontained electrolytes, such as polyelectrolytes contained in ion-exchange resin or in MNPs nano-particles. It is worth mentioning that similar observations were reported by Dey and Izake (2015). These observations led us to conclude that osmotic pressure can be generated under non-colligative conditions too. However, the osmotic pressure generated is not high enough to concentrate urine and recover nutrients. Urine that has been concentrated 5 folds requires a minimum osmotic pressure of 11.62 MPa (Nikiema et al. 2017). It is worth mentioning that osmolality of more than 2.5 osmol/kg has been reported using a 0.087 mol/L poly-acrylic acid solution (Johnson 2017). In addition, in this study, we observed that with the initial ratio of 1:4 of an MNP-coating agent, coating ratio saturation could not be confirmed, which suggested that a higher osmotic pressure could be achieved by increasing the initial amount of coating agent. Furthermore, it is already established that higher molecular weight PEG generates higher osmotic pressure. Therefore, high-molecular weight coating agent, such as PEG 10000, is expected to improve the osmotic pressure of the draw solution. With respect to the agglomeration of particles, performing coprecipitation in the presence of a dispersant is likely to reduce surface tension of the synthesized particles and lead to the formation of smaller particles, and thus, a higher coating ratio, which eventually leads to a higher osmotic pressure.

The relationship between concentration (wt.%) and osmotic pressure for PEG 4000-coated MNPs is illustrated in Figure 9. At higher wt.%, higher osmotic pressure was recorded. Further investigation is required to understand the relationship between osmotic pressure and the concentration of PEG-coated MNP solution.

2.4. Repetitive use of PEG 4000 MNP-based draw solution

Three FO experimental runs were performed, where the draw solution of PEG-coated MNPs was recovered and reused as a regenerated draw solution, assuming no loss of particles had occurred. Figure 10 illustrates the osmotic pressure of the draw solution as calculated from flux at each run. Results show that the osmotic pressure dropped from 0.61 MPa to 0.59 MPa in the second run and to 0.57 MPa in the third run. The loss of osmotic pressure was not significant. Assuming that the drop proceeds linearly, multiple reuses may be possible. We conclude based on our observations that, although there was only a slight drop in the osmotic pressure, repetitive use of the novel draw solution is possible. However, an evaluation of its use for several times is required.

Conclusion and Recommendations

This study aimed to synthesize a novel draw solution for concentration of urine via the FO process. In this study, we successfully synthesized a novel draw solution made of PEG 4000-coated magnetic nanoparticles. The highest osmotic pressure generated by this novel draw solution was 1.37 MPa. At the same concentration level, PEG 4000 alone generates a lower osmotic pressure. Repetitive use of this novel draw solution revealed that its osmotic pressure drops slightly after each use. Our study helps to solve an important challenge hindering the common use of the FO system. An easy-to-recover draw solution was prepared that was also free of reverse diffusion. However, higher osmotic pressure is needed to concentrate urine. Therefore, although the draw solution cannot be used to concentrate urine in its current form, this study offered a better understanding of its synthesis. Further investigations are needed to obtain a higher osmotic pressure.

Acknowledgements

Our research activities were supported by “The Sanitation Value Chain: Designing Sanitation Systems as Eco-Community Value System” Project funded by the Research Institute for Humanity and Nature (RIHN, Project No. 14200107). Acknowledgement is also due to the Global Station for Food, Land, and Water Resources (GSF), a project of Global Institution for Collaborative Research and Education at the Hokkaido University, for providing support and partial funding to conduct this research.

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