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Enhanced water flux using uncoated magnetic nanoparticles as a draw solution in forward osmosis desalination

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ABSTRACT

Pure uncoated magnetic nanoparticles (MNPs) were synthesized and studied as a potential draw solution (DS) in forward osmosis (FO) desalination process. Saturation magnetization was found to be 3.79 emu/g and the formation of Fe_3O_4 MNPs was confirmed by Fourier transform infrared and X-ray diffraction spectra. The osmotic potential of Fe_3O_4 MNPs was evaluated using a bench-scale crossflow FO unit. The feed solution (FS) of deionized water and NaCl solutions of different salinities (0.5, 1, and 2 g% NaCl) were evaluated. The water flux was found to decrease with the increase in water salinity of the FS by 62%, 78%, and 87% for the 2.5 g% MNPs, respectively. Furthermore, the Fe_3O_4 MNPs concentration (2.5 and 5 g%) was found to have a substantial impact on water flux with a three-fold increase in the pure water flux for the higher concentration. Results confirmed that the concentration of both FS (NaCl) and DS (MNPs) have a considerable effect on the produced pure water flux. This research demonstrated that the proposed uncoated Fe_3O_4 MNPs can be used as a potential draw solution for water desalination.

Keywords: Desalination; Draw solution; Forward osmosis; Magnetic nanoparticles

1. Introduction

Freshwater accessibility represents one of the most challenging environmental problems of the 21st century. Shortage of fresh water supply is attributed to the rapid increase in agricultural, industrial, and household water uses (Shannon et al. [1]; Montgomery and Elimelech [2]). Water desalination is one of the key solutions to this problem since global water demand is expected to increase to 6.9 trillion m^3 by 2030. However, water and energy are interrelated, since most water treatment techniques are currently energy-intensive [3]. Among various water desalination techniques, distillation, and membrane technologies are the most common. Currently, forward osmosis (FO) is being recognized as a promising alternative to other membrane processes for water desalination, such as reverse osmosis (RO) [4]. FO is an "osmotically driven membrane" process which makes use of the difference in osmotic pressure across a semipermeable membrane between two solutions of different salt concentrations [5]. FO is more energy-efficient, compared to RO which needs a higher applied hydraulic pressure. In addition, FO shows less fouling tendency thereby decreasing the capital and operating cost of the desalination process [6,7]. FO was studied for desalination

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[8], agricultural fertilization [6,9], food processing [10], and wastewater treatment [11]. On the other hand, commercial applications of FO for water treatment are still limited due to the poor recovery of fresh water. This problem is mainly associated with the choice of the proper draw solute (DS).

An ideal draw solution should generate a high osmotic pressure by reducing the water chemical potential to draw purified water from the feed solution. In addition, the draw solutes are expected to be easily recovered to produce purified water and regenerate the draw solution. Furthermore, ideal draw solution should be: environmentally-friendly, cost-effective, inert, large enough to prevent back diffusion, and stable chemically and thermally. Various draw solutes have been used in FO processes such as volatile compounds [12], inorganic salts [13], fertilizer drawn processes [6], ionic liquids [14], switchable polarity solvents [15], polyelectrolyte [16], hydrogels [17], and nanoparticles [18]. Recovery of the DS can be achieved by using heat [19], pressure [20], magnetic separation [21], electrolysis [22], and chemical precipitation [23]. However, the limited use of FO, compared to RO, is attributed to the proper choice of the DS that produces high osmotic pressure and can easily be reused after the desalination process [4]. Organic based DS were extensively studied such as carbohydrates [24], polyelectrolytes [16], ionic liquids [25], switchable polarity solvents [15], organic ionic salts [26], and polymer-based hydrogels [27].

More recently, magnetic nanoparticles (MNPs) were evaluated as superior DS due to their advantageous properties. MNPs provides elevated ratio of the surface area/volume and the tunability of the surface functional groups to affect the osmotic pressure of the MNPs as DS. In addition, MNPs are easily recovered and recycled using simple magnetic field as a result of their superparamagnetic properties. Furthermore, MNPs are generally non-toxic with low reverse solute flux due to relatively larger particle size than the pores of the membrane. These properties rely mostly on the nature and structure of the organic coating that is generally hydrophilic in nature to improve the osmotic pressure of the DS. Different organic coatings were evaluated in FO driven desalination process such as polyethylene glycol [28], polyacrylic acid (PAA) [29], dextran [30], citrate [31], pectin [7,32] to render the surface of the MNPs used as DS more hydrophilic to increase the osmotic pressure in the DS side. However, choosing the proper organic coating is crucial as it can affect the generated osmotic pressure, superparamagnetic properties, and consequently the recovery of the DS [8,29,33]. Coating of the MNPs with the sodium salt of PAA was achieved successfully by Dey and Izake [34]. The water flux was ≈5 LMH. The recovery of the DS was achieved by applying external magnetic field for 5 min. Co-precipitation was used by Na et al. [31] to prepare citrate coated MNPs. The as-prepared NPs were less than 10 nm in diameter and produced 17 LMH water flux. However, the interaction between the negative citrate coating and the cellulose triacetate FO membrane decreased the water flux sharply. Pectin coated MNPs prepared by Attallah et al. [7] were used as a DS and produced 0.82 LMH water flux and a salt rejection of more than 97%. Our previous study on pectin coated MNPs showed a 25% decrease in the pure water flux after pectin coating. The decrease in the produced water flux with the increase in pectin concentration was attributed to the increase in particle size of the MNPs after coating [32]. The appropriate tailoring of the fabrication technique to prepare uncoated MNPs that produces a significant osmotic pressure as DS in FO desalination and easily recovered using magnetic separation, is still needed.

In this research, the preparation of MNPs with superior properties as DS in FO desalination processes was evaluated. The prepared nanoparticles exhibit acceptable water dispersibility to generate a high osmotic pressure for desalination applications with higher water flux compared to most reported organic-coated MNPs in both pure and saline water. This is combined with the easy recovery of the DS using magnetic separation for reuse, rendering this investigation convenient for sustainable low-energy water desalination using FO.

2. Materials and method

2.1. Materials

Anhydrous ferric chloride ($FeCl_3$) and ferrous sulfate heptahydrate ($FeSO_4·7H_2O$) were obtained from Sigma-Aldrich (USA). Analytical grade ammonia solution (33 wt.%) was used as received.

2.2. Synthesis of MNPs

MNPs were prepared using a co-precipitation method as reported elsewhere [32,35]. Briefly, a 2:1 molar ratio of anhydrous ferric chloride ($FeCl_3$) and ferrous sulfate heptahydrate ($FeSO_4·7H_2O$) in deionized water was allowed to stir for 20 min. Dark colored precipitate was observed after the dropwise addition of ammonia under vigorous stirring. The addition of ammonia was resumed till the formation of a completely black precipitate of magnetite. The solution was allowed to stir for extra 30 min to confirm the complete formation of magnetite. The particles were cooled to room temperature, washed with water, and dried in the oven at 65°C. The chemical reaction can be indicated as the following equation [36]:

$$Fe^{2+} + 2Fe^{3+} + 8OH^{-} \rightarrow Fe_3O_4 + 4H_2O \tag{1}$$

The resulting sample was carefully grinded and sieved using a USA standard mesh of 106 µm sieve (Newark Wire Cloth Co., USA) in order to decrease the average particle size and the particle size distribution.

2.3. Characterization

The particle size and particle size distribution of the prepared MNPs was measured using DLS (dynamic light scattering, Malvern Instruments Ltd., UK). The crystal phase structure of MNPs was examined using XRD (X-ray diffraction, D8 Bruker, USA). The surface morphology of the formed MNPs was examined by FESEM (field emission scanning electron microscope, JEOL JSM-7401F, USA). The structural characteristics of the MNPs were investigated using FTIR (Fourier transform infrared spectroscopy, Thermo-scientific Nicolet 380, USA). The magnetic properties of MNPs were evaluated using VSM (vibrating sample magnetometer, LakeShore 7300, Germany) (3.2 Tesla), the applied magnetic field used was in the range of −20,000 and 20,000 Oe. Thermal analysis of the MNPs was investigated using TGA (thermogravimetric analyzer, TA Instruments, New Castle, DE). The readings were recorded in controlled atmosphere under N_2 from 25°C to 650°C at 10°C/min heating rate.

2.4. Forward osmosis process

The FO experiment using MNPs as the DS was conducted using a bench-scale FO filtration system in a crossflow mode as represented in Fig. 1. The membrane was polyamide in composition (PA, Porifera Inc., USA) and was used in the all FO experiments. The feed solution tested were saline water of different salinities (5, 10, and 20 g/L NaCl) and deionized water as our control experiment. The diameter of the FO cell is 40 mm and the active membrane area of 1.257×10^{-3} m^2 as provided from the supplier.

The FO experiments were conducted at a crossflow velocity of 12.9 cm/s, which is equivalent to a crossflow rate of 0.22 L/min. The feed and draw solutions were operated in countercurrent directions using a double-headed peristaltic pump (Stenner, model 170DMP5, USA). The temperature of both FS and DS were kept constant at 25°C ± 1°C using a water bath temperature controller (Polyscience, model 9106A12E).

The changes in the volume of both DS and FS were recorded simultaneously by connecting the DS and FS to a computer software for inline data entry every 150 s. The FO experiments were carried out for 2 h each and ultrasonication of the DS for 10 s was employed with the aim to decrease the aggregation of MNPs and hence maintain their particle size distribution. The starting volume of both the DS and FS was 200 mL each to decrease the effect of concentration change and each solution was allowed to return to its tank after passing through the membrane (Fig. 1). Both DS and FS tanks were tightly covered during the FO experiment to avoid water losses due to evaporation. The MNP DS were constantly stirred during the FO experiment using a mechanical stirrer, Hei-TORQUE Precision 400, to ensure consistent dispersion of the MNP in the DS.

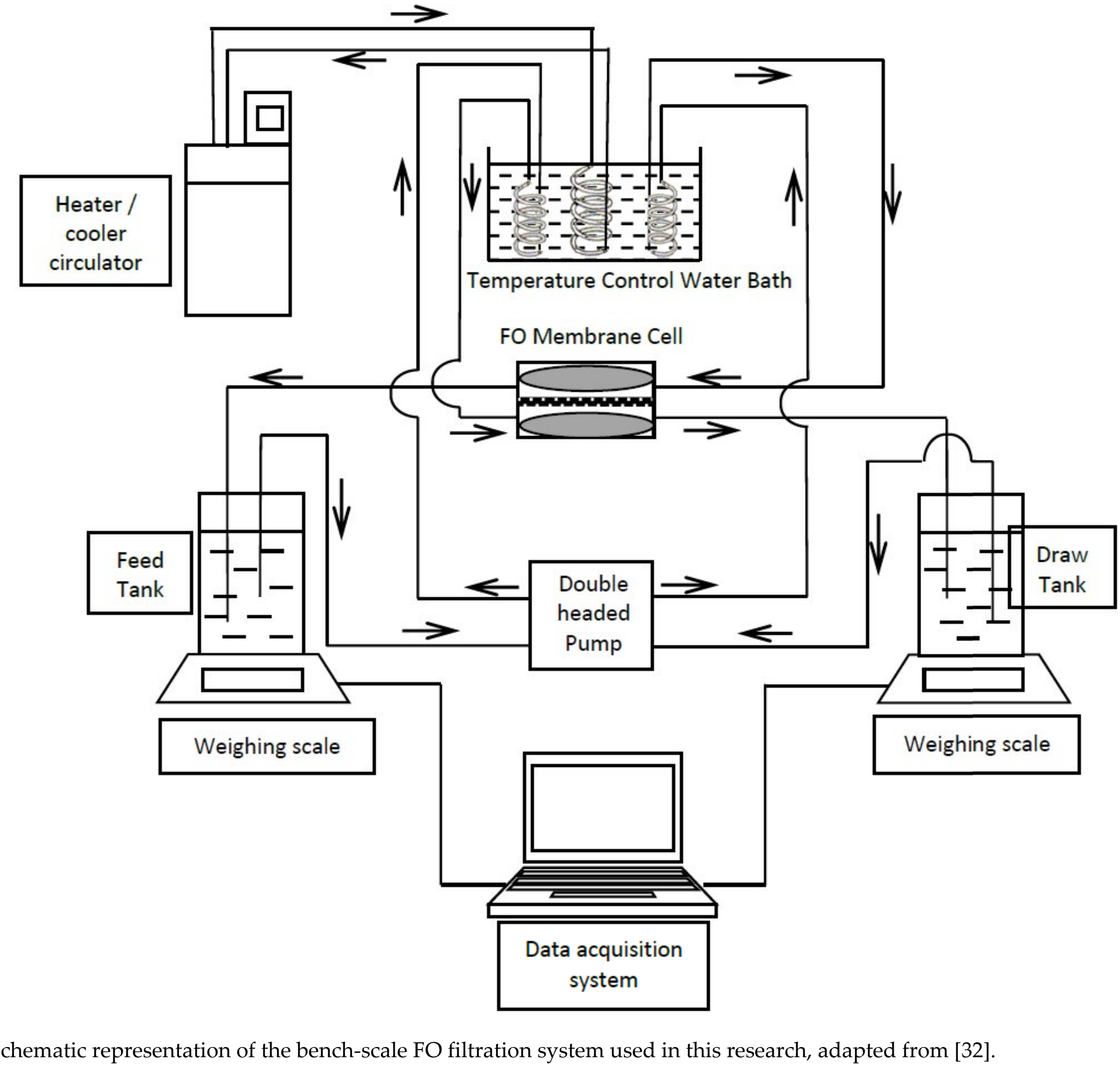

Fig. 1. Schematic representation of the bench-scale FO filtration system used in this research, adapted from [32].

The pure water flux, J_w (LMH, Lm^{-2} h^{-1}), is calculated using the following equation:

$$J_w = A\,\sigma\,(\pi_{DS} - \pi_{FS}) \tag{2}$$

where J_w is the obtained pure water flux, A is the permeability coefficient of the membrane, σ is the reflection coefficient, which is usually assumed unity indicating complete rejection of the solute, π_{DS}, π_{FS} are the bulk osmotic pressure of the draw solution and the feed solution, respectively.

3. Results and discussion

3.1. Characterization of the prepared MNPs

3.1.1. Determination of crystal phase

The magnetite (Fe_3O_4) structure formation was verified using XRD (X-ray diffractometer), as indicated in Fig. 2. The illustrated diffraction peaks at 220, 311, 400, 422, 511, and 440 indicate the presence of the magnetite cubic spinel phase of Fe_3O_4 as reported in the literature. [37].

3.1.2. Particle size and morphology

The particle size of Fe_3O_4 MNPs was measured using DLS. The addition rate of the base, ammonia, during the preparation was found to greatly affect the particle size and particle size distribution of the prepared samples. The particle size of the prepared Fe_3O_4 MNPs was 127 ± 5 nm with a low surface charge of 2.25 ± 0.15 mV. This relatively small MNPs diameters will increase the number of dissolved MNPs per unit volume and hence increase the osmolality of the DS. This can be ascribed to the fact that osmolality is a colligative property that is affected by the number of dissolved solutes [29,38]. The hydrophilic small particle size, low surface charge, and the continuous stirring during the FO process contribute to an overall decrease in the viscosity with a consequent increase in the osmotic pressure [39,40]. Particle size and surface charge were found to have noticeable effect on the osmotic pressure of the DS and thus affect the pure water flux in the FO desalination process. Cannon et al. [41] reported that proper tailoring of the particle size and surface charge can enhance the produced osmotic forces of the draw solution. Furthermore, sieving using a USA standard sieve was employed to reduce the particle size distribution to obtain better mono-disperse nanoparticles, as evidenced elsewhere [42].

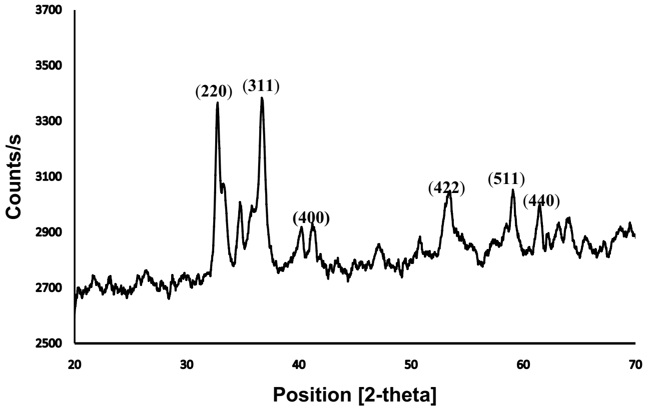

Fig. 2. XRD pattern of the prepared Fe_3O_4 MNPs.

The shape and morphology of Fe_3O_4 MNPs were examined using scanning electron microscope. Fig. 3 shows that the prepared Fe_3O_4 MNPs having a spherical shape as confirmed using scanning electron microscopy.

3.1.3. Fourier transform infrared

Fig. 4 shows the FTIR spectrum of the prepared Fe_3O_4 MNPs. The absorption bands at 455 and 1,400 cm^{-1} are attributed to the Fe–O stretching mode. Other broad absorption band at ≈3,200 cm^{-1} corresponds to O–H stretching mode and this suggest that the surface of the MNPs had a large number of hydroxyl groups with an overall positive effect on the pure water flux during the FO process [43] Thus, such findings confirm the formation of Fe_3O_4 MNPs [44].

3.1.4. Thermogravimetric analyzer

The weight losses of the Fe_3O_4 MNPs was measured and shown in Fig. 5. The weight loss of Fe_3O_4 MNPs (17%) occurred higher than 100°C was ascribed to the loss of the physically and chemically adsorbed moisture water molecules, as reported previously [37,45].

3.1.5. Measurement of magnetic properties

The magnetic properties of Fe_3O_4 MNPs were examined using VSM. Fig. 6 shows the prepared Fe_3O_4 MNPs have zero remanence magnetization. Thus, Fe_3O_4 MNPs exhibit the superparamagnetic characteristics [28]. Moreover, the saturation magnetizations of Fe_3O_4 MNPs was 3.79 emu/g. A substantial decrease in the saturation magnetizations of MNPs coated with organic materials, such as pectin-coated MNPs, in comparison to the uncoated MNPs was reported in the literature. This decrease was attributed to the formation

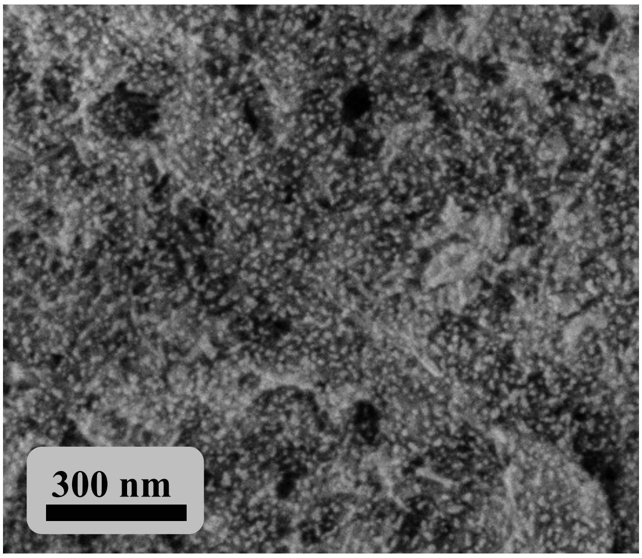

Fig. 3. SEM image of the prepared Fe_3O_4 MNPs.

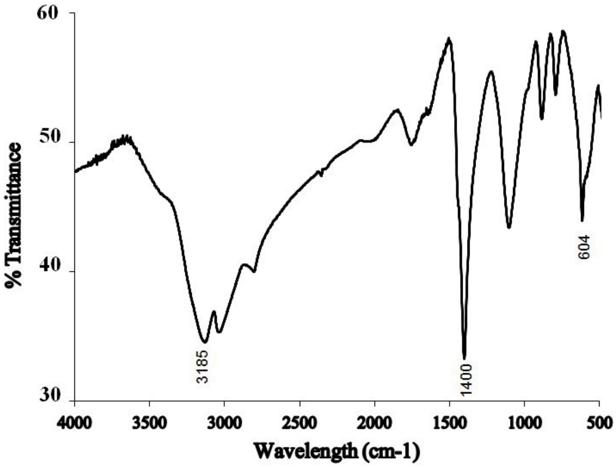

Fig. 4. FTIR spectrum of the prepared Fe_3O_4 MNPs.

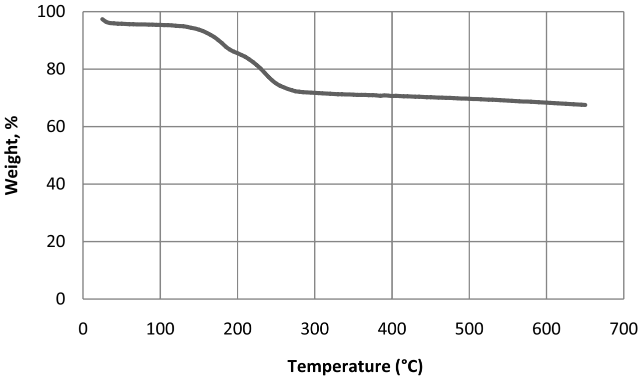

Fig. 5. TGA analysis of the prepared Fe_3O_4 MNPs.

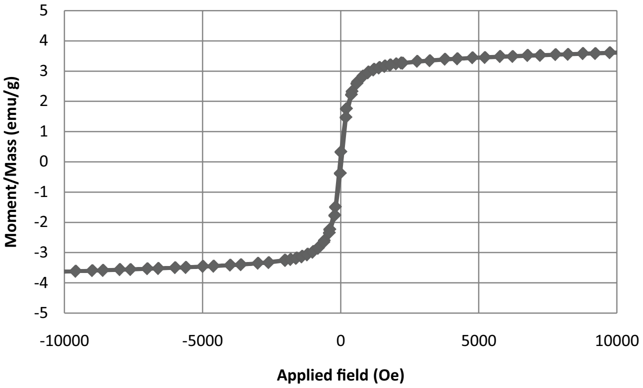

Fig. 6. Hysteresis loop of the prepared Fe_3O_4 MNPs.

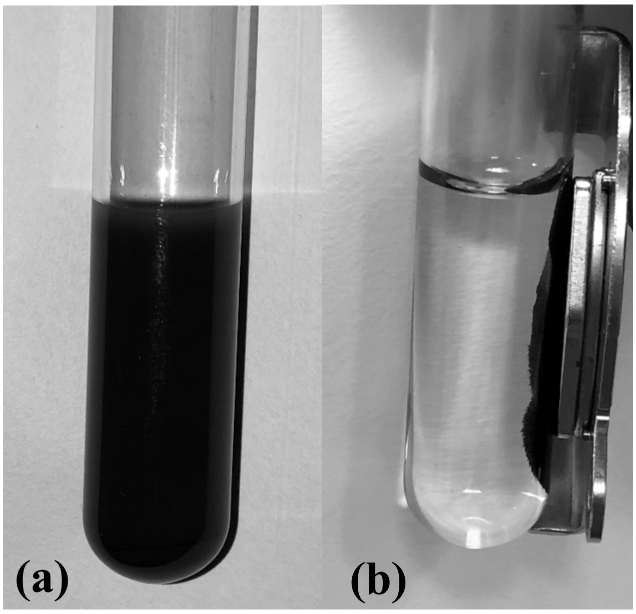

Fig. 7. Recovery of the prepared Fe_3O_4 MNPs (a) without external magnet and (b) in presence of simple magnet for 7 min.

of a non-magnetic masking coating layer on the MNPs surface, and a decrease in the overall magnetic properties of the coated MNPs [7]. These findings confirm the need to prepare "uncoated" MNPs with superior properties to be used as DS in FO desalination [32].

The regeneration of the prepared MNPs was attained with a simple magnet as shown in Fig. 7. It is interesting to note that the solution takes a short time of 7 min to become clear, indicating the potential use of MNPs as sustainable, easy-recovered, and energy-efficient DS for FO desalination processes in the future.

3.2. FO process performance

Two different concentrations of the Fe_3O_4 MNPs (2.5 and 5 g%) were studied as the DS in a bench-scale FO filtration system in a crossflow mode for water desalination applications. The change in the weight was measured simultaneously between the weight decrease in the FS side and increase in the DS side and used to measure the pure water flux according to the aforementioned equations. Due to large particle size, compared to membrane pores, pure water was only allowed to pass through the membrane and the MNPs were retained in the DS tank. This means that the increase in the weight of the DS tank is mainly due to the pure water flux from the FS tank. Experiments were run in duplicates for statistical analysis of the results.

Fig. 8 demonstrates the obtained pure water flux when using deionized water and saline water of different salinities (0.5, 1, and 2 g% NaCl), where 1 g% equals 10,000 ppm, as the FS, and Fe_3O_4 MNPs with an initial concentration of 2.5 g% as the DS. The gradual decrease in the pure water flux with time is expected due to the concentration decrease in the DS tank by the permeated pure water with subsequent reduction in the osmotic pressure at the DS tank. However, this concentration decrease in the DS has a limited effect on the pure water flux over 2 h during the FO process. Pure water flux, in FO mode, was calculated as 11.91 LMH when deionized water was employed as the FS, this flux is superior to most values in the literature for pure water flux using coated-MNPs. In addition, the pure water flux of 4.45, 2.55, and 1.45 LMH were obtained when using 0.5, 1, and 2 g% NaCl (W/V) as the FS, respectively. The reverse relationship between the obtained pure water flux and NaCl concentration

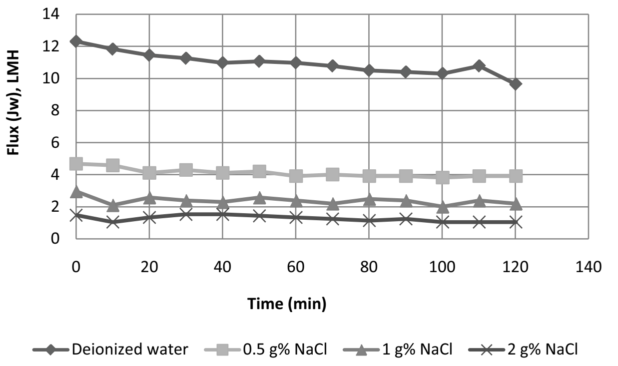

Fig. 8. Pure water flux values in FO experiment using DS of MNPs 2.5 g% W/V against different FS.

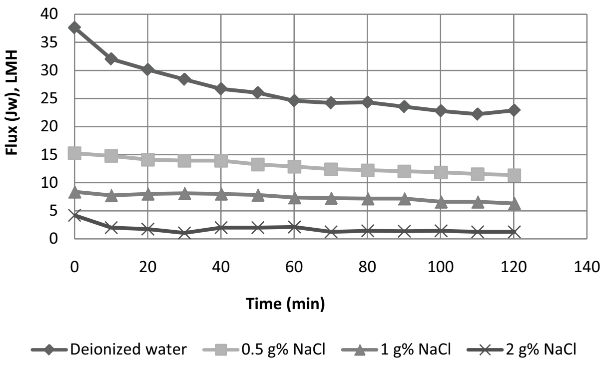

Fig. 9. Pure water flux values in FO experiment using DS of MNPs 5 g% W/V against different FS.

shows that the difference in the osmotic pressure across the membrane between the FS and DS is the key driving force in this experiment. Hence, lower NaCl weight % produces larger osmotic pressure difference across membrane and improves the pure water flux from FS tank to DS tank.

Furthermore, to investigate the effect of MNPs concentration in the DS tank on the obtained pure water flux during the FO desalination, another concentration of Fe_3O_4 MNPs (5 g%, W/V) was utilized as the DS and the obtained pure water flux when using deionized water and saline water of different salinities (0.5, 1, and 2 g% NaCl) as FS were calculated. As shown in Fig. 9, the obtained pure water flux was calculated as 35.69 LMH when deionized water was employed as the FS, and the pure water flux of 14.97, 8.37, and 2.55 LMH were obtained when using 0.5, 1, and 2 g% NaCl (W/V) as the FS, respectively.

Fig. 10 shows that the measured pure water flux increases significantly with the increase in the MNPs concentration from 2.5 to 5 g%, as the DS against deionized water and saline water of different NaCl concentration as the FS. These findings demonstrate that Fe_3O_4 MNPs with the proper particle size distribution, zeta potential and concentration as the DS in FO desalination can produce osmotic pressure that is high enough to generate an acceptable pure water flux, as illustrated in this experiment. It is noteworthy that our reported pure water flux results using "uncoated" MNPs as the DS are much higher than water fluxes reported by other coated-MNPs, as shown in Table 1. This indicates that the fabrication technique and proper tailoring of the fabrication conditions of the prepared MNPs used as DS have substantial effect on the generated osmotic pressure across the membrane and the obtained pure water flux in FO desalination. The use of a relatively higher MNPs concentration (5 g%) had a major effect in increasing the pure water flux however, the effective MNPs concentration depends on the amount of aggregated MNPs which will increase the size of the MNPs and lead to an overall decrease in the effective osmotic pressure. The decrease in the MNPs aggregation during the FO process was achieved through continuous stirring during the FO process to decrease the aggregation and settling of the MNPs combined to the sonication prior to the process. The high concentration of the MNPs combined with the vigorous stirring during the FO experiment using a mechanical stirrer have substantial impact on the obtained pure water flux using the fabricated MNPs as the DS.

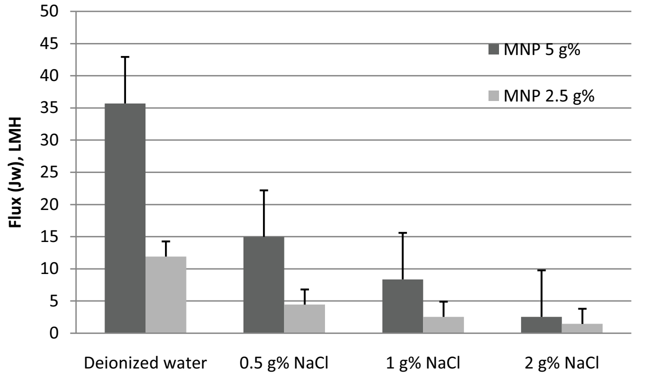

Fig. 10. Pure water flux of different concentrations of Fe_3O_4 MNPs (2.5 and 5 g%) as DS in FO desalination.

Statistical analysis of the results using analysis of variance (ANOVA), Design Expert® software, confirmed that the concentration of both FS (NaCl) and DS (MNPs) have a substantial impact on the produced pure water flux in the FO desalination process, the *p*-value of both factors is less than 0.05 and the model *F*-value is 13.56, as shown in Table 2. In addition, the effect of the FS concentration is more significant on the resultant water flux than the DS concentration with negligible significance of the interaction between the two variables.

4. Conclusion

A forward osmosis desalination system utilizing uncoated MNPs as the draw solution was proposed. Our proposed system utilizes the difference in the osmotic pressure exerted by the MNPs in the DS tank to stimulate the pure water flux across the semipermeable membrane from the FS to DS. The membrane helps also to reject NaCl throughout the FO desalination process with a significant decrease in the required energy compared to other membrane desalination techniques. Two different MNPs concentrations (2.5 and 5 g% W/V) were studied as DS. FS used in our experiments were deionized water and NaCl solution with different concentrations (0.5, 1, and 2 g% W/V).

Table 1
Water flux of some coated-MNPs as DS in FO desalination as reported in the literature

DS	Initial concentration	FS	Water flux (LMH)	References
Pectin-coated MNPs	2 wt.%	DI	0.82	[7]
Chitosan-coated MNPs	0.06 g/L	DI	5.5	[46]
Citrate-coated MNPs	20 mg/L	DI	6.2	[31]
Dextran-coated MNPs	2.0 M	DI	8	[37]
Oxalic acid-coated MNPs	0.8 g/mL	DI	10	[8]
This work (MNPs)	2.5 g%	DI	11.91	–

Table 2
ANOVA of MNPs as DS for FO desalination in the present study

Source	Sum of squares	df	Mean square	*F*-value	*p*-value Prob > *F*	
Model	1,194.45	4	298.61	13.56	0.0003	Significant
A-DS	144.84	1	144.84	6.58	0.0263	
B-FS	1,049.6	3	349.87	15.88	0.0003	
Residual	242.32	11	22.03			
Lack of fit	84.86	3	28.29	1.44	0.3023	Not significant
Pure error	157.45	8	19.68			
Correlation total	1,436.76	15				

Experimental and statistical results, using ANOVA, demonstrated that the preparation method was successful, and the careful control of the preparation parameters have substantial impact on the obtained pure water flux during the FO experiment using MNPs as DS with pure water flux up to 35 LMH. Further investigation on the economic analysis of the proposed setup and the possibility of large-scale FO application are ongoing.

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