Exploring the limitations of forward osmosis for direct hydroponic fertigation: Impact of ion transfer and fertilizer composition on effective dilution

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A B S T R A C T

There is a need for water reuse technologies and applications to minimize the imminent water crisis, caused by the world population growth, the reduction of freshwater resources and the increasing water pollution. Fertilizer-drawn forward osmosis (FDFO) is a promising process capable of simultaneously extracting fresh water from low-quality sources as feed water (e.g., wastewater or greywater), while diluting fertilizer solutions for direct fertigation, avoiding the demand for freshwater for irrigation. Achieving an adequate level of dilution for direct fertigation is a key element to be evaluated for the implementation of FDFO. This study assessed the performance of the forward osmosis process to dilute fertilizer solutions to be applied directly in hydroponic systems. Experiments were carried out under conditions close to osmotic equilibrium to evaluate the process performance up to the maximum dilution point. Tests were carried out with individual and blended fertilizers (*i.e.*, $(NH_4)_2HPO_4$ or DAP, and KNO_3) used as draw solution (DS) and with deionized water or individual salts (NaCl, $MgCl_2$, Na_2SO_4, $MgSO_4$) in the feed solution (FS). Water fluxes and reverse salt fluxes indicated that both fertilizer DS composition and concentrations play a fundamental role in the process. Suitable nutrient concentrations to be directly applied without further dilution for N, P and K (119, 40, 264 mg.L^{-1} respectively) were obtained with deionized water as FS and blended DAP (0.025 M) and KNO_3 (0.15 M) as DS. However, important fertilizer losses from DS to FS were observed, being the highest for NO_3^- (33–70% losses from DS to FS). The presence of salts in FS decreased the water fluxes and the DS dilution due to the osmotic equilibrium caused by a greater loss of nutrients from DS to FS (up to 100%), compared with tests using just deionized water as FS. This study points out the potential limitations of the FDFO process, due to the high solute fluxes and low water fluxes in conditions close to osmotic equilibrium.

1. Introduction

United Nations has estimated that by 2050 nearly 6 billion people will suffer from clean water scarcity (Boretti and Rosa, 2019). Thus, future scenarios drive the need to improve water management practices and strategies to ensure water supply. Within this context, water reuse is a promising option to alleviate water stress, while moving towards the Circular Economy principles. Nonetheless, water reuse remains a limited practice due to barriers ranging from technical and economic feasibility to legislative restrictions and social acceptance. Therefore, to solve the imminent water crisis, it is necessary to develop efficient technologies that will make water reuse a sustainable and affordable practice to be

widely implemented. With irrigation being the world largest water consumer, the application of reused water for irrigation purposes is a crucial strategy capable of significantly decreasing the demand for freshwater and therefore reducing water stress. Among agricultural techniques, hydroponics is a promising approach that can be implemented worldwide. In this soilless cultivation technique, plants grow in direct contact with water that contains the required nutrients for their development. The typical concentrations of nitrogen, phosphorous and potassium (NPK, main nutrients for plants) of common hydroponic solutions are diverse (Table 1), as the nutritional requirements for the plants depend on many factors, such as plant type, stage of plant growth, seasonal differences or weather conditions (Resh, 2013).

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Table 1
NPK concentrations of standard nutrient solutions for hydroponics according to previous studies. Adapted from Trejo-Téllez and Gómez-Merino (2012).

Reference	Nutrient concentration, mg.L^{-1}			Nutrient concentration, mmol.L^{-1}		
	N	P	K	N	P	K
Hoagland and Arnon (1938)	210	31	234	15.0	1.0	6.0
Hewitt (1966)	168	41	156	12.0	1.3	4.0
Cooper (1979)	200–260	60	300	14.3–18.6	1.9	7.7
Steiner (1984)	168	31	273	12.0	1.0	7.0

Given that the hydroponic technique requires large amounts of freshwater (Chekli et al., 2017a), it is of great interest to study the potential of technologies capable of treating alternative water sources for subsequent reuse in hydroponics. Among the variety of available technologies, forward osmosis (FO) emerged as a promising solution for water treatment and reuse, as it can recover fresh water from low quality water sources such as seawater or wastewater (Coday et al., 2014; Zhou et al., 2014). In FO, a highly concentrated solution (draw solution: DS) extracts water from a low concentration solution (feed solution: FS), and the water is transported through a dense membrane (Phuntsho et al., 2013). FO membranes exhibit high pollutant rejection and have low fouling propensity, and the process does not require hydraulic pressure as it is driven by the difference in osmotic pressures between FS and DS (Van Der Bruggen and Luis, 2015). One of the main drawbacks of FO is the reverse salt flux (J_s) (Holloway et al., 2015); *i.e.*, solute losses from draw to feed per membrane area and time (Jamil et al., 2016). J_s plays a fundamental role in the design of osmotically driven processes (Phillip et al., 2010), since it decreases the osmotic driving force (Phuntsho et al., 2011), represents economic losses (fertilizer losses in FDFO) and causes difficulties with feed concentrate management (Phuntsho et al., 2013), hence jeopardizing the benefits of the FO process (Chekli et al., 2012). As pointed out by Zou et al. (2019) in a review of approaches to reduce reverse solute fluxes in FO, it is crucial for FO operations to control and reduce J_s, and they also highlighted the lack of J_s data in FO studies. Therefore, detailed studies of solute fluxes in FO are of great interest to assess their impact on FO performance.

One of the practical applications of FO is the osmotic dilution of soluble fertilizers for irrigation purposes (Sahebi et al., 2015), as most of them are capable of generating a high osmotic potential (Phuntsho et al., 2013). In fertilizer-drawn forward osmosis (FDFO), the osmotic dilution of the fertilizer DS occurs, with the aim of later being used for direct fertigation (application of fertilizer nutrients for irrigation purposes) since it contains the essential nutrients for plant growth. FDFO concept was mainly developed in the last decade and has shown promising results. FDFO is particularly interesting when applied to low quality sources as feed water, such as brackish water or greywater, avoiding the demand for freshwater for irrigation.

Most FDFO studies have focused on the performance of different fertilizer salts as DS (Chekli et al., 2017b; Lotfi et al., 2015; Majeed et al., 2015; Phuntsho et al., 2011, 2012) and their interactions with different membranes (Corzo et al., 2017; Phuntsho et al., 2013). Some recent works have even used commercial fertilizers as DS (Chekli et al., 2017a; J.E. Kim et al., 2019; Xie et al., 2015; Zou and He, 2016). Besides, it should be noted that in most of the previous FDFO studies, authors highlight the need for further dilution because final concentration of nutrients in DS were above the threshold tolerated by the plants. In previous cases, proper DS dilution for direct fertigation was only achieved after coupling FO with other technologies (Jamil et al., 2015; Luo et al., 2015) or by applying additional pressure (Chekli et al., 2017a; Kim et al., 2017; Sahebi et al., 2015). Overall, most of FDFO studies were devoted to demonstrating proof of concept for the use of fertilizers as draw solutions and did not focus on the impact of FS salinity, nor on achieving an optimal dilution to the required level of nutrients for plants, especially when approaching osmotic equilibrium. Final DS

concentrations suitable for direct fertigation - without further dilution of the final draw solution - are therefore essential for the success of FDFO and more studies are required on the practical application of the process (Phuntsho et al., 2012). Finally, even if some studies have focused on bidirectional diffusion of the various ions present in both FS and DS (Hancock et al., 2011; Hancock and Cath, 2009), all were carried out on a very small experimental scale and under conditions far from osmotic equilibrium.

For FDFO to be applicable on a full scale, relatively low DS concentrations are required, and it is of interest to achieve the desired concentrations in a single step. Given the current limitations of FDFO concerning the dilution factor of the fertilizer for direct fertigation, it is crucial to conduct more experiments close to osmotic equilibrium, as it will have a great impact on the achievable dilution rate, filtration kinetics and is expected to depend on FS initial salinity and reverse salt diffusion. Within this framework, this study aimed to evaluate the suitability of the FDFO process to achieve an effective DS dilution to generate a suitable nutrient solution for direct application in hydroponic systems (as means of nutrients: N, P and K content in solution). The performance of the FO process at conditions close to osmotic equilibrium, as well as ion fluxes through the membrane were also experimentally evaluated.

2. Materials and methods

2.1. Materials

The tests at lab scale were performed with commercial FO hollow fiber modules (Aquaporin Inside HFFO2, Aquaporin A/S, Denmark). The HFFO2 module, made with inner-selective biomimetic active layers, contains 13,800 membrane fibers 270 mm long, an inner diameter of 195 μm and total effective area of 2.3 m^2 (Nikbakht Fini et al., 2020; Sanahuja-Embuena et al., 2019).

Deionized (DI) water was used as feed solution in most of the experiments; and some tests were then conducted using single salt solutions (MgCl$_2$, MgSO$_4$, Na$_2$SO$_4$ or NaCl) as FS (Table 2). Feed salts MgCl$_2$.6H$_2$O and MgSO$_4$.7H$_2$O (99%) were purchased from Scharlab and Na$_2$SO$_4$ was purchased from Merck. NaCl (Sea salt, >99.4% NaCl) was purchased from Vicens i Batllori S.L. (Banyoles, Spain). The FS salts were chosen because they are commonly found in waste-, brackish-, and

Table 2
List of tests (6.5 mM for the salts in FS, when applicable).

	FEED	DRAW		
	Content	Content	DAP (M)	KNO$_3$ (M)
Baseline tests	DI water	DAP	0.05	
		KNO$_3$		0.05
		MIX 1	0.05	0.05
Effect of draw solute concentration	DI water	DAP	0.50	
		KNO$_3$		0.50
		MIX 0.5	0.50	0.50
Optimal nutrient solution	DI water	MIX 2	0.050	0.10
		MIX 3	0.030	0.08
		MIX 4	0.050	0.20
		MIX 5	0.025	0.15
Effect of salts in the feed solution	NaCl	DAP	0.05	
		KNO$_3$		0.05
		MIX 1	0.05	0.05
	MgCl$_2$	DAP	0.05	
		KNO$_3$		0.05
		MIX 1	0.05	0.05
	Na$_2$SO$_4$	DAP	0.05	
		KNO$_3$		0.05
		MIX 1	0.05	0.05
	MgSO$_4$	DAP	0.05	
		KNO$_3$		0.05
		MIX 1	0.05	0.05

seawater, which are good candidates for FS.

The draw solution contained individual salts or blended mixes of KNO_3 and $(NH_4)_2HPO_4$ (DAP), purchased from Scharlab. These salts were chosen as they are commonly used as fertilizers worldwide (Phuntsho et al., 2012), and already tested in previous FDFO studies, showing their potential for FDFO applications.

2.2. Forward osmosis experimental setup

Experiments were performed with constant feed and draw recirculation, leading to continuous DS dilution and FS concentration. All tests were carried out with DS facing the active layer (within the fibers) because this configuration results in higher water fluxes (Phuntsho et al., 2013; Su et al., 2010). Although this configuration of having the active layer facing the DS may lead to fouling, this negative impact was not expected due to FS nature (without any foulant agent). Additionally, although external concentration polarization may increase with DS facing the active layer, internal concentration polarization would decrease in the proposed experimental setup, since it generally used DI water as FS. The module was positioned vertically with the DS and FS circulating in counter-current (Fig. 1), since operation in counter-current leads to better use of osmotic pressure, achieving a higher dilution rate than in co-current mode (Blandin et al., 2020). The initial volumes were 2 L of DS and 60 L of FS. FS and DS were circulated with a peristaltic pump (Watson-Marlow RS232), with an average flow rate of 34.6 L h^{-1} and 60.7 L h^{-1} respectively, according to the manufacturer's recommendations. The water flux crossing the membrane (J_w, from FS to DS) was determined by measuring the volume extracted from FS to DS thanks to the increase in the mass of the DS with a balance (Kern PCB) and considering 1 kg/L as density of DS.

2.2.1. Module evaluation tests

Prior and throughout the experiments, module integrity and performance were evaluated with 1 M NaCl as DS, DI water as FS, and with the active layer of the membrane facing FS, which were the conditions established by the manufacturer.

2.3. FDFO experimental procedure

To evaluate the FDFO process, different tests were performed (Table 2). Water flux (J_w) in L.m^{-2}.h^{-1} was determined by equation (1):

$$J_w = \frac{\Delta V_{FS}}{A*\Delta t} \tag{1}$$

where ΔV_{FS} represents the decrease in the volume of FS over time in L, A the membrane area (2.3 m^2), and Δt the time variation, in h. Average J_w was calculated considering the total duration of the tests, while initial J_w was calculated as the average of the three values of J_w from the highest J_w value (approximately at 2–3 min). Since operation in batch led to
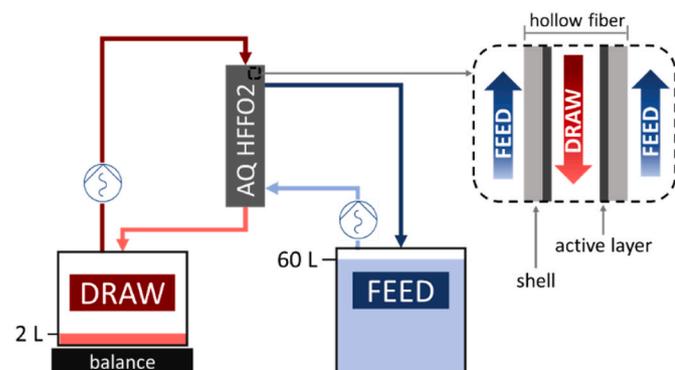
continuous DS dilution and FS concentration, with J_w decreasing along time, initial water flux values served to analyze J_w with the maximum osmotic pressure gradient between DS and FS.

Reverse salt fluxes (J_s) of each ion (from DS to FS), in mmol.m^{-2}.h^{-1} were calculated by equation (2):

$$J_s = \frac{C_{FSf}*V_{FSf} - C_{FSi}*V_{FSi}}{A*\Delta t} \tag{2}$$

where C_{FSf} and C_{FSi} represent final and initial ion concentrations in FS (mmol.L^{-1}), respectively; and V_{FSf} and V_{FSi} represent final and initial FS volume (L), respectively. Forward solute fluxes (J_{sf}) of each ion (from FS to DS), in mmol.m^{-2}.h^{-1} were calculated by equation (3):

$$J_{sf} = \frac{C_{DSf}*V_{DSf} - C_{DSi}*V_{DSi}}{A*\Delta t} \tag{3}$$

where C_{DSf} and C_{DSi} represent final and initial ion concentrations in DS (mmol.L^{-1}), respectively; and V_{DSf} and V_{DSi} represent final and initial DS volume (L), respectively.

Nutrient losses from DS to FS were evaluated by analyzing final concentrations in FS (being zero the initial concentration of each nutrient in FS). Since EC increases proportionally to concentration and osmotic pressure (Corzo et al., 2017), it is assumed as a good indicator of osmotic equilibrium. Therefore, osmotic equilibrium was assumed as achieved when the ratio between final EC in FS and DS was between 0.8 and 1.2.

2.3.1. Baseline tests

Baseline tests were performed in duplicates at initial DS of 0.05 M of DAP and KNO_3, alone or blended (Table 2). The aim of these preliminary tests was twofold: to evaluate the differences in terms of flux and ion behavior (when using fertilizers individually or blended) and to serve as a reference for the rest of the tested conditions. Due to setup limitations, it was not possible for DS to extract more than 30 L from FS (*i.e.*, 15 times DS dilution rate). Therefore, initial DS concentrations were designed to achieve adequate nutrient concentrations in the final DS with a 15-fold DS mass dilution. In addition, this configuration was used to evaluate ions behavior under conditions identical or close to osmotic equilibrium between FS and DS.

2.3.2. Effect of draw solute concentration

A set of tests conducted in duplicates, with a concentration 10 times higher than the baseline concentration (*i.e.*, 0.5 M) were carried out to evaluate the effect of DS concentration (Table 2). This concentration was chosen as it was commonly used in previous studies (Irvine et al., 2013; Wang et al., 2020).

2.3.3. Optimal nutrient solution

Four mixes of DAP and KNO_3 as DS were tested in duplicates, with initial concentrations ranging from 0.025 to 0.05 M for DAP and from 0.05 to 0.20 M for KNO_3 (Table 2). The salts were also tested individually and evaluated for their effectiveness to achieve proper DS dilution. It is worth mentioning that this study was designed as a proof of concept, so the objective was to analyze the feasibility of the system to reach certain levels of DS dilution that would lead to nutrient concentration ranges suitable for hydroponics, without focusing on a specific crop or a certain growing stage. Accordingly, the target final nutrient concentrations in the DS were set to range between 100 and 200, 30–60 and 150–300 mg.L^{-1} for N, P and K, respectively.

2.3.4. Effect of salts in the feed solution

To evaluate the influence of feed solutes on reverse fluxes (from DS to FS) and forward fluxes (from FS to DS) close to osmotic equilibrium, four different salts with initial concentration of 6.5 mM in the FS were tested individually. Draw fertilizers were used blended and alone at the previously stated baseline concentration (*i.e.*, 0.05 M, Table 2). The four FS

Fig. 1. Experimental setup.

salts were monovalent ions (NaCl), divalent ions (MgSO$_4$), monovalent cation with divalent anion (Na$_2$SO$_4$) and divalent cation with monovalent anion (MgCl$_2$). Salts were tested separately to analyze the influence of the different pairs of ions in the process.

2.4. Sample collection and analytical methods

Samples from feed and draw solutions were collected at the beginning, after 30 and 60 min, and at the end of each test (generally 24 h). Ion concentrations were analyzed by ion chromatography (ICS 5000 from DIONEX). Electrical conductivity was measured with an EC meter (GLP31+ from Crison).

3. Results and discussion

All tests were characterized by a sharp decrease in ion concentration in the DS during the first 30 min, as already observed in previous studies (Sahebi et al., 2020) and by an increase in the mass dilution of the DS throughout the entire duration of each experiment.

3.1. Module evaluation tests

Water fluxes with 1 M NaCl in DS, DI water as FS and with the FS facing the membrane active layer, were above 15 L m^{-2}.h^{-1} throughout all evaluation tests (avg. 16.2 L m^{-2}.h^{-1}), while specific reverse salt fluxes (J$_s$/J$_w$) were below 0.3 g.L^{-1} in all evaluation tests (avg. 0.21 g. L^{-1}). These results are in accordance with manufacturers' guidelines, (i. e., water flux greater than 12 L m^{-2}.h^{-1}, specific reverse salt fluxes below 0.3 g.L^{-1} under similar operating conditions), hence confirming that the module was working properly. The obtained results remained similar throughout all tests, confirming that no fouling, nor scaling or other issue was compromising the membrane performance. Further module evaluation parameters can be found in Sanahuja-Embuena et al. (2019).

3.2. Effect of draw solute type and concentration

A set of tests using individual or blended draw solutes at 0.50 vs 0.05 M of initial concentrations served to evaluate the effects of DS concentration and composition on the process performance (Table 3). Initial water fluxes were in the same range for all tests using the same DS concentrations and decreased significantly throughout the process due to the dilution of the DS and the consequent loss of osmotic pressure driving force. As expected, water fluxes for 0.05 M DS were low compared to those of the tests with DS at 0.50 M (Table 3) due to the resulting lower difference in osmotic pressure between FS and DS. Working with low DS salinity (0.05 M) not only reduces J$_w$ because of the lower initial flux, but also because of operating near osmotic equilibrium due to dilution over time.

All tests at 0.50 and 0.05 M achieved the targeted dilution rate (around 15 times) except for KNO$_3$ at 0.05 M (Table 3). Osmotic equilibrium was not achieved in any of the tests (except for KNO$_3$ at 0.05 M) indicating that the water extraction capacity of the tested DS was higher than the 15 times dilution rate, which was the limit of the setup. Only when operating with KNO$_3$ as DS at 0.05 M, a higher EC was observed in

the final FS than in the DS, indicating that osmotic equilibrium was reached, at a lower DS dilution rate (6-fold) than the target. This behavior results from the reverse salt flux from DS to FS, which not only leads to fertilizers losses but also limits the dilution capacity of the system.

In tests with 0.05 M of fertilizer salts in initial DS, reverse salt fluxes (J$_s$) did not exceed 1.5 mmol m^{-2}.h^{-1} in any case but were affected by the nature of the ions present in the DS (Table 4). As noted above, the highest J$_s$ were observed when using KNO$_3$, leading to the highest diffusion of both of its ions. NO$_3^-$ has been widely reported as an ion with high reverse fluxes (Gulied et al., 2019), due to its small hydrated radius. K$^+$ passed through the membrane in equal equivalent concentration to balance the charges and keep the ionic equilibrium in both solutions (see supplementary S1).

Reverse fluxes of DAP ions were much lower, confirming results from other studies (Y. Y. Kim et al., 2019; Mirshekar et al., 2021). Phosphate J$_s$ was up to two orders of magnitude lower than the counter ions present in the DS (i.e. K$^+$ and NH$_4^+$). Higher FO membrane rejection of phosphate compared to ammonium and potassium has already been reported due to its bigger hydrated radius, and the stronger electrostatic repulsion with the negatively charged membranes caused by its negative multivalent charge (Achilli et al., 2009; Xie et al., 2015). Consequently, phosphate reverse fluxes through the membrane are generally reported to be minimal regardless of the DS composition and concentration (Majeed et al., 2015; Minier-Matar et al., 2016).

The observed ions J$_s$ were different when using individual fertilizers or blended (MIX 1). In MIX 1, reverse fluxes followed the trend NO$_3$>NH$_4$>K > P (Table 4), which is inversely correlated to their hydrated radii at the same charge type (0.34, 0.25, 0.33, and 0.49 nm for NO$_3^-$, NH$_4^+$, K$^+$, and PO$_4^{3-}$ respectively) (Xie et al., 2015), and in accordance to other studies (Gulied et al., 2019; Zou and He, 2016).

When using blended fertilizers (MIX 1), J$_s$ were found to be lower for K$^+$ and NO$_3^-$, but higher for NH$_4^+$ (Table 4). That could be explained by a lower overall diffusivity due to the presence of two or more ions species in the DS, which also leads to lower reverse diffusion (McCutcheon and Elimelech, 2006). It was also shown that the migration of NH$_4^+$ was favored compared to K$^+$ as counter ion of NO$_3^-$. The smaller hydrated radius of NH$_4^+$ compared with K$^+$ (0.25 vs 0.33 nm) explains the higher diffusion of NH$_4^+$ when salts were tested together in DS (Table 4). In addition, the reverse fluxes of the cations were facilitated by the negatively charged membrane surface, which enhances the cation diffusion to FS (Lotfi et al., 2015; Minier-Matar et al., 2016).

Lower percent solute losses occurred with a higher initial DS concentration (Table 4). This is because the DS concentration gradient drives the passage of both water and salts in opposite directions across the membrane, and higher fertilizer losses could be expected with higher water fluxes (Sahebi et al., 2020). However, it is hypothesized that if tests at 0.50 M would have been closer to osmotic equilibrium, as it happened with tests with 0.05 M, the corresponding losses would have been higher. This increase in losses is due to the longer time of contact between the solutions, which is required to achieve the targeted dilution rate but also enables more solute transport across the membrane. This issue is illustrated by the higher reverse salt fluxes in tests at 0.50 M than in the baseline tests at 0.05 M (Table 4). These results highlight the importance of the setup conditions, as FO performance cannot only be

Table 3

Results of tests with 0.50 and 0.05 M of salts in initial DS and DI in FS. Osmotic equilibrium was considered achieved for (EC$_{FS}$)/(EC$_{DS}$) between 0.8 and 1.2.

DS content	initial DS (M)	initial J$_w$ (L.m^{-2}.h^{-1})	avg. J$_w$ (L.m^{-2}.h^{-1})	Mass dilution	EC in final FS (μS.cm^{-1})	EC in final DS (μS.cm^{-1})	Relation EC final FS/DS
DAP	0.50	14.7	5.7	14	58	5765	0.0
KNO$_3$	0.50	12.1	3.1	15	1683	2725	0.6
MIX 0.5	0.50	17.5	7.1	14	941	8375	0.1
DAP	0.05	3.8	0.5	15	49	638	0.1
KNO$_3$	0.05	2.7	0.2	6	228	242	0.9
MIX 1	0.05	5.5	0.7	15	231	894	0.3

Table 4

Solute losses, reverse fluxes (J_s) and specific reverse salt fluxes (SRSF) for tests with 0.50 and 0.05 M of salts in initial DS and DI water in FS.

Initial DS			% solute losses from DS to FS				J_s (mmol.m^{-2}.h^{-1})				SRSF (J_s/J_w)			
Content	DAP (M)	KNO$_3$ (M)	N–NH$_4^+$	K$^+$	N–NO$_3^-$	P–PO$_4^{3-}$	N–NH$_4^+$	K$^+$	N–NO$_3^-$	P–PO$_4^{3-}$	N–NH$_4^+$	K$^+$	N–NO$_3^-$	P–PO$_4^{3-}$
DAP	0.50		3.1			0.3	12.4			0.6	2.2			0.1
KNO$_3$		0.50		37.5	37.1			42.2	44.4			13.8	14.6	
MIX 0.5	0.50	0.50	8.3	6.9	19.6	0.2	38.1	19.5	53.0	0.5	5.4	2.7	7.5	0.1
DAP	0.05		8.3			2.6	0.3			0.0	0.5			0.1
KNO$_3$		0.05		65.9	70.3			1.5	1.5			7.2	7.5	
MIX 1	0.05	0.05	19.4	17.2	46.9	1.7	0.8	0.5	1.1	0.0	1.2	0.7	1.6	0.1

evaluated in terms of water and solute fluxes, but also in solute losses. Hence, it seems reasonable to look for conditions generating lower ion dilution instead of just considering the J_s values. Since J_s increases with DS concentrations (Majeed et al., 2015), solute losses are expected to also be higher at higher mass dilution. For example, in tests with KNO$_3$ alone, the initial DS concentration of 0.50 M lead to losses of around 37%, while 0.05 M KNO$_3$ lead to losses of up to 70% for both potassium and nitrate to FS. However, looking at reverse fluxes, the J_s was 1.5 mmol m^{-2}h^{-1} for both K$^+$ and NO$_3^+$ at 0.05 M, but 42.2 and 44.2 mmol m^{-2}h^{-1} for K$^+$ and NO$_3^-$ respectively, for tests at 0.50 M of KNO$_3$ in the initial DS (Table 4). This fact is also clear when looking at the specific reverse salt fluxes (SRSF: J_w/J_s), since they indicate the mass of each ion passing through the membrane to the FS per each liter of water recovered. The SRSF of all ions except for phosphate were much higher in tests with high DS concentration than in those with low DS concentration (Table 4). Therefore, since reverse fluxes and consequent nutrient losses increased with increasing DS concentration, tests with higher initial concentration can be expected to result in higher losses if the process had continued to run until osmotic equilibrium was reached.

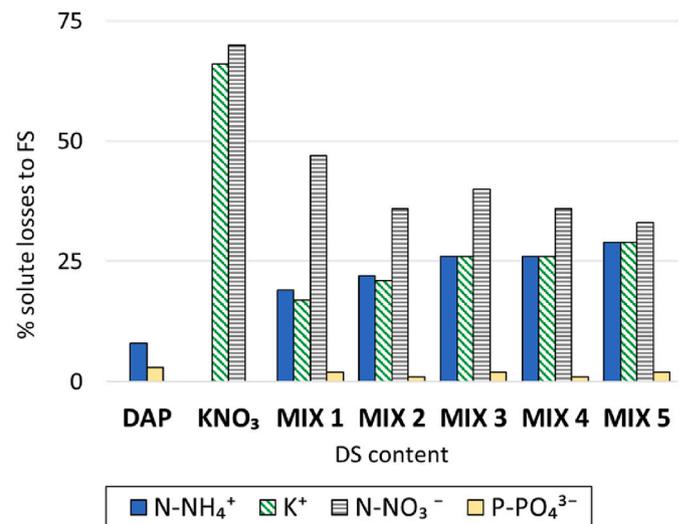### 3.3. Draw dilution aiming at nutrient content adequate for hydroponics

To reach adequate NPK concentrations to nourish hydroponic systems, five KNO$_3$/DAP mixes were tested (Table 2) and compared to the individual fertilizers. All the mixes achieved the target draw dilution (15 times) with initial J_w between 5.5 and 8.7 L m^{-2}.h^{-1} and without reaching osmotic equilibrium (Table 5), which attests the possibility of achieving a higher dilution rate and the benefits of using blended fertilizers instead of individual ones. However, the low average J_w (between 0.6 and 1.0 L m^{-2}.h^{-1}) might limit the feasibility of further diluting the DS, resulting in very large filtration units when the system is operated near osmotic equilibrium, as already mentioned in the previous section.

Similar nutrient losses were experienced for ammonium and potassium (Fig. 2), and they varied analogously along the mixes, (ranging from 19 to 27% for N–NH$_4^+$ and 17–29% for K$^+$), due to the similar nature of both cations. In line with previous tests, phosphate losses were almost negligible (lower than 1.8% in all cases), while nitrate losses were the highest with up to 47%. Such high losses have not been reported before in the literature. However, the other studies addressing this topic (Chekli et al., 2017b; Phuntsho et al., 2012) were carried out

Fig. 2. Nutrient losses (% from mass balance) for tests with DI water as FS and different concentrations of DAP and KNO$_3$ as DS.

using higher DS concentrations with much faster filtration kinetics. This may indicate that the migration of draw solutes through the FO membrane in the current study was promoted by the long filtration time due to the operation at low water flux. Former studies probably underestimated potential losses of the FDFO. Nutrient losses observed in this study might be too large in some cases, depending on the composition of salts and their concentrations in the DS. Additionally, the presence of nitrate or phosphate in the FS could make its management and discharge more complex, as these elements can cause eutrophication (Phuntsho et al., 2012). This issue could be mitigated by treating the FS prior to discharge, as proposed by Wang et al. (2020), who added microalgae in their FS that could benefit from the nutrient fluxes from DS to FS. However, we consider that this approach would increase the costs and make the process more complex. Zou et al. (2019) pointed out the need of system optimization, membrane development, long term evaluation, as well as other cost-effective strategies to reduce the reverse salt fluxes in FO, which is crucial for a proper FO operation. In the same line, our results point out the need to evaluate nutrient losses when regard to DS

Table 5

Tests aiming at achieving a suitable NPK content for hydroponics. Osmotic equilibrium was evaluated with electrical conductivity (EC) and considered as achieved when the ratio between final EC in FS and DS was between 0.8 and 1.2

Solution name	DAP (M)	KNO$_3$ (M)	initial J_w (L. m^{-2}.h^{-1})	avg. J_w (L. m^{-2}.h^{-1})	EC final FS (µS/cm)	EC final DS (µS/cm)	relation EC final FS/DS*	mass dilution	L extracted	L extracted/kg fertilizer
DAP	0.050		3.8	0.5	49	638	0.08	15	28	2092
KNO$_3$		0.05	2.7	0.2	228	242	0.94	6	10	1029
MIX 1	0.050	0.05	5.5	0.7	231	894	0.26	15	28	1178
MIX 2	0.050	0.10	6.7	0.8	401	1105	0.36	16	30	902
MIX 3	0.030	0.08	5.6	0.6	290	798	0.36	15	28	1147
MIX 4	0.050	0.20	8.7	1.0	732	1473	0.50	16	30	564
MIX 5	0.025	0.15	6.7	0.8	500	1165	0.43	15	28	757

dilution close to real conditions of the FDFO application. Designing the system to limit the filtration time like operating in counter-current mode, applying hydraulic pressure, or increasing membrane surface area may help to avoid such important losses of DS ions. In any case, it is essential to develop membranes with higher reverse flux selectivity, as well as finding suitable FS-DS combinations for a more efficient FDFO process.

The tests also showed that using DAP individually as DS led to lower ammonium losses compared to using blended DAP and KNO_3. On the contrary, KNO_3 alone caused greater nitrate and potassium losses (Fig. 2). Therefore, for practical applications, phosphate fertilizers are a promising DS to be used individually, due to their low J_s, while nitrate fertilizers should be used in combination with other salts to reduce their nutrient losses. These results point out the importance of choosing not only the right salts for the FDFO process but also the right combination.

Nitrogen, phosphorous and potassium (NPK) concentrations at the end of the tests are presented in Fig. 3. Values within the target NPK concentration ranges (yellow bands in Fig. 3) were achieved with MIX 5 (119, 40 and 264 mg.L^{-1} for N, P and K, respectively) without the need for further dilution of DS or changes in nutrient concentrations. Other mixes achieved the targeted concentrations for some of the nutrients. MIX 2 and MIX 4 achieved 117 and 158 mg.L^{-1} of N and 179 and 315 mg.L^{-1} for K, respectively, while MIX 3 achieved 60 mg.L^{-1} of P. However, special attention must be paid to P concentration since values higher than 62 mg.L^{-1} could be toxic for the plants (Termaat and Munns, 1986). Thus MIX 2 and 4, although with acceptable concentrations of N and K, are not considered suitable nutrient solutions due to the concentrations of P well above the reported toxicity level.

It should be noted that some of the tested mixes could still serve as nutrient solutions for some growing stages, despite having a N and K content below the previously mentioned target (*i.e.*, MIX 3). For example, the standard Hoagland solution (Hoagland and Arnon, 1950), which is commonly used in hydroponic experiments, has been applied at half strength in some studies (Adrover et al., 2013; Garland et al., 2004; Wiser and Blom, 2016). In contrast to our results, the final DS concentrations obtained in previous studies (Chekli et al., 2017b; Majeed et al., 2015; Xie et al., 2015; Zou and He, 2016) ware way higher than those required for plant growth, so they pointed out the need for substantial dilution prior to application. Other studies showed the potential of FDFO systems to achieve an adequate DS dilution for direct application for plants, but assuming an unlimited FS volume (Phuntsho et al., 2011), which implies not considering the salinity buildup in the FS; or by applying extra pressure in order to increase the nutrient dilution in DS (Chekli et al., 2017a; Jamil et al., 2016; Sahebi et al., 2015). Results of our study indicate that solutions with an appropriate nutrient content for hydroponics can be achieved with FO (deionized water as FS), showing promising applications of FDFO.

MIX 5 setup could be used in small-scale applications, such as homes or small buildings, considering that only 2 L of concentrated fertilizer solution in DS could extract up to 30 L of FS, to be applied directly in small hydroponic systems within the same building. Extrapolating from the volume of water extracted in the experimental setup (28 L extracted with 37 g of fertilizer salts), 757 L could be extracted for kg of fertilizer using MIX 5 (Table 5). This is a much higher volume than indicated by Phuntsho et al. (2011), who tested the performance of FDFO with 9 fertilizer salts and estimated that 1 kg of fertilizer (DS) could extract up to 29 L water from seawater (FS). However, nutrient losses should be considered in the balances, and low water fluxes could compromise the process. As pointed out by Suwaileh et al. (2020) in a recent review about FO, further work is required regarding membrane development in order to increase the efficiency of the FO process. This study confirms that more selective membranes are required to increase the efficiency of FDFO process, by lowering nutrient losses, especially for monovalent ions.

3.4. Effect of salts in feed solution

To evaluate the impact of feed salinity, tests with 6.5 mM of saline solutions in the FS and with 0.05 M of blended or individual fertilizer salts in DS were carried out and compared with baseline tests (DI water in FS) (Fig. 4).

The initial EC for FS ranged between 730 and 1448 μS cm^{-1} and osmotic equilibrium was achieved in all tests (supplementary S2). A good fit of the molar balance for anions and cations in both FS and DS was observed (supplementary S1), showing that even with more ions present in the FS, ions were passing through the membrane to equilibrate the charges (solution diffusion mechanism). Results show that all tests with salts in FS presented lower initial water fluxes compared with the tests with DI water in FS (Fig. 4a), which is in accordance with other studies (Raval and Koradiya, 2016). This is explained by the osmotic pressure present in FS at initial time, related to salt presence (Su et al., 2010), which decreases the net osmotic pressure and thus J_w (Phuntsho et al., 2013). Although J_w were similar along the tests with salts in FS, tests with NaCl in FS showed the best performance in terms of water fluxes and draw dilution (Fig. 4b). A higher difference in osmotic pressures between FS and DS was observed when having NaCl in FS, as its osmotic pressure is lower than those of the other FS salts at the same molar concentration. The results from all tests with salts in FS were far from the target mass dilution and extracted liters and followed the trend of MIX 1>DAP > KNO_3 (Fig. 4c), as it was observed in tests with only DI in FS. All tests performed with KNO_3 alone (except in the case of NaCl) ended up with less DS volume than at the beginning (see negative extracted liters in Fig. 4c), and with the FS having an even higher EC than the DS (supplementary S2). These results show that the presence of salts in FS limits the FDFO performance and application, as the target draw dilution was not achieved in any case. Fig. 5 shows the forward solute fluxes (from FS to DS) as well as the reverse solute fluxes (from DS to FS) for tests with fertilizers alone and blended.

For DAP ions, phosphorous reverse fluxes were not influenced by FS salinity and remained minimal in all tests, including tests with just DI
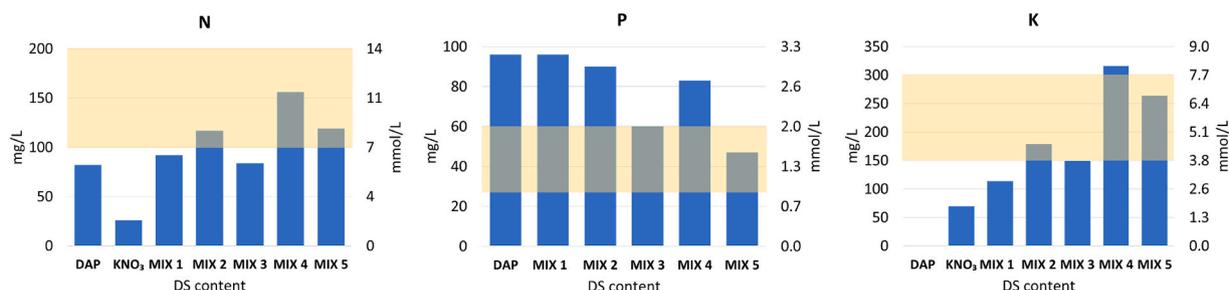

Fig. 3. Final NPK concentrations (blue bars) and desirable NPK ranges (yellow bands) for tests with DI water in FS and individual fertilizers (at 0.05 M initial DS) or blended DAP and KNO_3 in DS (initial DAP concentrations: 0.05 M of for mixes 1, 2, and 4; 0.03 and 0.025 M for mix 3 and 5, respectively; initial KNO_3 concentrations: 0.05, 0.1, 0.08, 0.2, and 0.15 M for mixes 1, 2, 3, 4, and 5 respectively). (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)
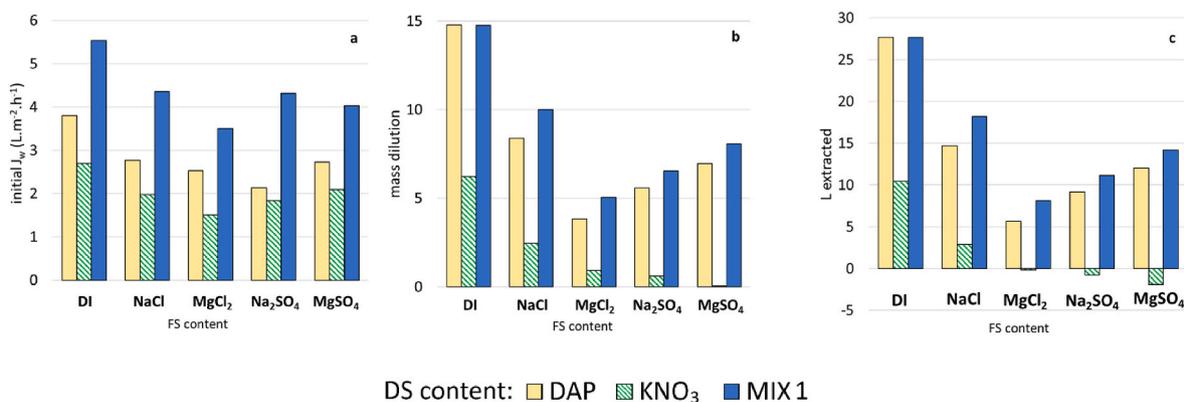
Fig. 4. Initial water fluxes (a), mass dilution of DS (b) and L extracted from FS (c) in tests at 0.05 M initial DS concentration of DAP and KNO$_3$ individually or blended. FS content refers to DI water or 6.5 mM initial FS concentration of individual salts when applicable.
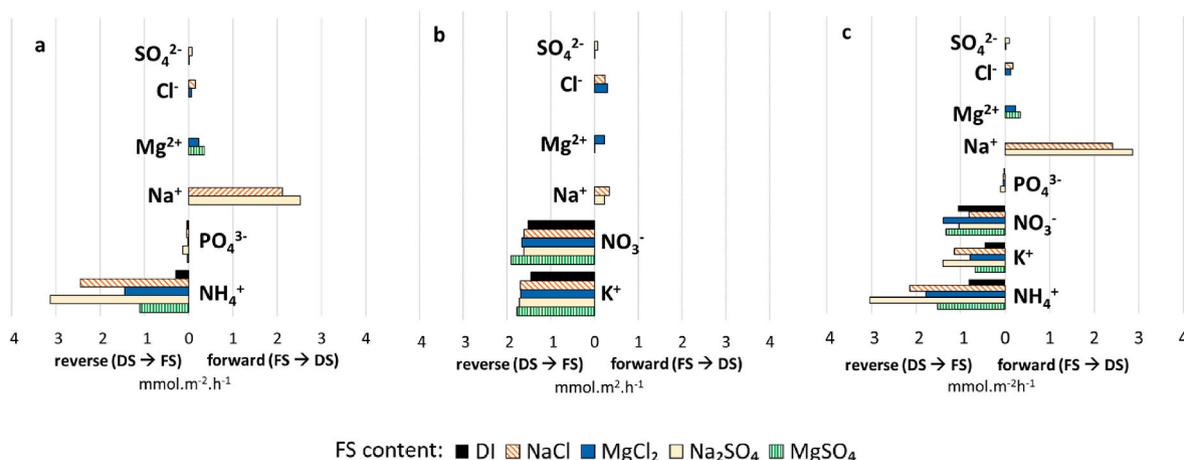

Fig. 5. Solute fluxes (mmol.m^{-2}h^{-1}) for tests with DAP (a), KNO$_3$ (b) and MIX 1 (c).

water in FS (Fig. 5a and c). Ammonium J$_s$ were slightly higher for blended fertilizers compared to using DAP alone (Fig. 5a and c) and showed a similar trend as when using DI water in FS. Results in Fig. 5a and c also show that the presence of salts in the FS had a strong impact on favoring ammonium passage through the membrane, because of the resulting higher J$_s$. Ammonium reverse fluxes were up to one order of magnitude higher with salts in FS than with just DI water, the highest being 3.1 mmol m^{-2}.h^{-1} for tests with Na$_2$SO$_4$ in FS. This effect was less strong in tests with Mg^{2+} due to its better rejection by the membrane (NH$_4^+$ J$_s$ lower than 1.8 mmol m^{-2}.h^{-1} for all tests with Mg^{2+} in FS). For KNO$_3$ ions, nitrate and potassium reverse fluxes were similar for tests with and without salts in FS (Fig. 5b) when tested with KNO$_3$ alone. For blended DAP and KNO$_3$ (Fig. 5c), both reverse fluxes of potassium and nitrate were similar regardless of the FS tested, but smaller than with KNO$_3$ alone. Therefore, it is hypothesized that the J$_s$ of K$^+$ and NO$_3^-$ are more dependent on DS than FS composition. However, it is worth mentioning that when using blended salts, nitrate reverse fluxes were even lower with Na$^+$ in FS than when using only DI water (Fig. 5c) due to lower ion exchange. Thus, the presence more than the type of salt in the FS influenced the J$_s$ of nitrate ions. The obtained results show the complexity of ion interactions because the tested DS ions (ammonium, phosphate, nitrate, and potassium) behaved differently and were influenced, to different degrees, by both the presence of salts in FS and the DS composition.

Forward fluxes (J$_{sf}$, from FS to DS) were minimal for all ions except for Na$^+$ in tests with DAP and blended salts in DS, with J$_{sf}$ ranging from 2.1 to 2.9 mmol m^{-2}.h^{-1} (Fig. 5a and c). As indicated by Hancock and Cath (2009), feed solutes with larger hydrated radii (*i.e.*, Mg^{2+}), had

better FO membrane rejection than monovalent ions (*i.e.*, Na$^+$). High sodium fluxes (imperfect rejection by FO membranes) are commonly reported (Roy et al., 2016), and in this study its presence influenced ammonium reverse fluxes. This is because Na$^+$ has higher diffusivity than Mg^{2+} and therefore its transport to the DS facilitates the ammonium transport to FS and vice versa (ion exchange mechanism). The percentage of mass of ion passage from FS to DS (supplementary S2) showed higher Na$^+$ passages with the pair of monovalent ions (NaCl), because of the retarded sodium diffusion due to the divalent SO$_4^{2-}$ presence in the case of Na$_2$SO$_4$ in the FS (solution diffusion mechanism). Similarly, lower anion (Cl$^-$ and SO$_4^{2-}$) passages were experienced in presence of the divalent Mg^{2+} (supplementary S2). These results point out the importance of FS composition for the performance of the FO process. Concerning draw solutes, divalent ions from FS showed lower forward fluxes, and thus the final FS and DS were less contaminated with ions from the opposite solution when divalent ions were present on both sides of the membrane.

Fig. 6 indicates the distribution of solutes in FS and DS at the end of the tests. A high percentage of draw solutes passed to the feed side, resulting in almost 100% fertilizer losses to FS in some cases (KNO$_3$). Such observations are of utmost importance as they jeopardize the interest of the FDFO concept. This reinforces the fact that KNO$_3$ cannot be used alone as DS for fertigation. Comparatively, using DAP or blended fertilizers reduced the losses, which however were still very high for ammonium, nitrate and potassium in most cases as soon as salts were present in the feed solution.

Ideally, osmotic equilibrium should be achieved by an equal EC between original FS and DS solutes without nutrient losses, and not
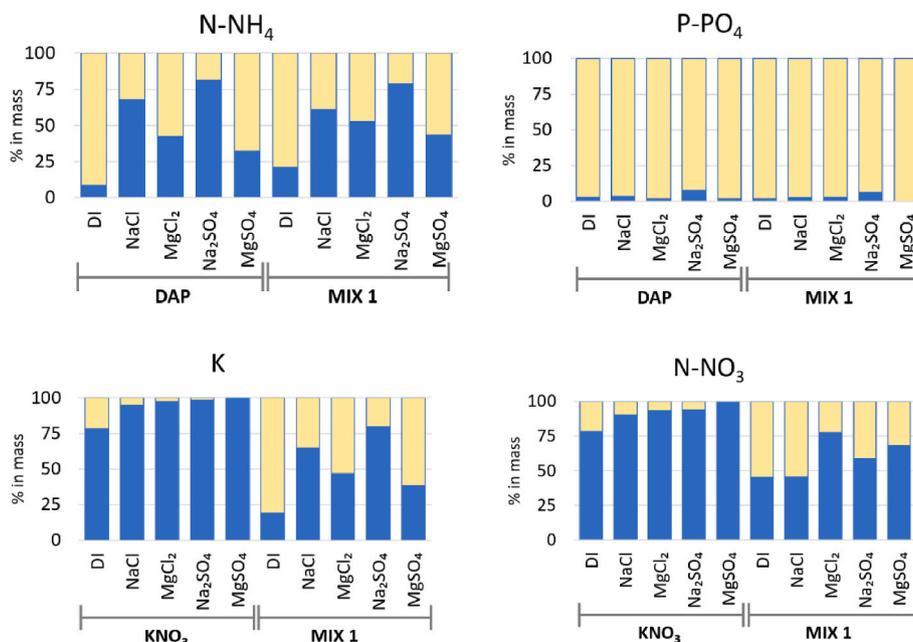
Fig. 6. Percentage of mass of initial DS ions in FS (blue) and DS (yellow) at the end of tests with 0.05 M of initial DS concentration (KNO$_3$, DAP, and blended, *i.e.*, MIX 1) and 6.5 mM of initial concentration for FS salts when applicable. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

because of large reverse flux of DS solutes to FS, limiting the DS dilution. However, the obtained J_s and nutrient losses were very high, osmotic equilibrium was reached, and water fluxes decreased as a consequence of the salinity buildup in FS caused by the reverse fluxes of DS ions. Existing studies are controversial since some of them point out that the solute fluxes from DS to FS are not influenced by the presence of salts in FS (Hancock et al., 2011), while others indicate the opposite (Phuntsho et al., 2013). In this study, while the presence of salts in FS did not influence PO_4^{3-} behavior, it did clearly influence both J_s and losses of NH_4^+. Although the influence of salts in FS was not clear in terms of reverse fluxes of K^+ and NO_3^-, Fig. 6 shows that it strongly influenced the passage of K^+ and NO_3^- ions to the FS.

Adequate nitrogen and potassium dilution for direct hydroponics application was achieved in some tests with magnesium ions in FS (supplementary S2). For the rest of the cases, due to the high reverse fluxes, nitrogen and potassium concentrations were below the target ranges. In contrast, phosphate reverse fluxes, as well as mass dilution were minimal, and phosphate concentrations in the final DS were well above desired concentrations and toxicity levels. Therefore, the presence of salts in FS plays a fundamental role in the final concentrations of NPK in DS. Additionally, one of the main problems of sodium diffusion in FDFO is its final concentration in the DS, since the DS is intended to be used as a nutrient solution for direct application in hydroponics. Sodium concentrations over 50 mg.L^{-1} are toxic for the plants (Raval and Koradiya, 2016).

Average sodium concentrations in the final DS of tests with NaCl and Na$_2$SO$_4$ in FS were above the level of toxicity (supplementary S2). These results indicate that Na$^+$ forward fluxes may compromise the quality of the final DS and the general efficiency of the FDFO process.

4. Conclusions

This study demonstrated that achieving adequate NPK concentrations for hydroponics by extracting water from reclaimed sources and for direct applications with FDFO process was possible. Using different combinations of KNO$_3$ and DAP as DS generated promising results for FDFO applications. However, these results were only reached with DI water in FS, which showed the highest osmotic pressure difference

between FS and DS, as well as minimal concentration polarization and ion exchange effects. Having real (waste)water as FS might cause decreased water fluxes and increased salt fluxes, which are the opposite of desirable conditions for efficient FDFO applications.

The complexity and the limitations of the FDFO process were especially highlighted when considering operating under conditions close to osmotic equilibrium. A main problem was the loss of the key fertilizer components, from DS to FS, especially when using KNO$_3$. Additionally, the presence of salts in the feed water could be a limiting factor affecting the achievable dilution rate of the fertilizer due to osmotic equilibrium limitations. Furthermore, the increased reverse salt diffusion of fertilizer when having salts in the feed solution will highly affect the economic and technical feasibility of FDFO applications. Feed ions and especially sodium passage from FS to DS was influenced by ammonium presence, and vice versa, indicating that both FS and DS composition influences the performance of the FDFO process.

Further validation work for specific crops and growth stages should be carried out and the influence of feed (waste)water quality on the process should be studied. Also, more selective membranes, adequate DS composition and concentrations, FS type, and more detailed relations between FS and DS should be carefully evaluated to design future efficient FDFO processes.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.jenvman.2021.114339.

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