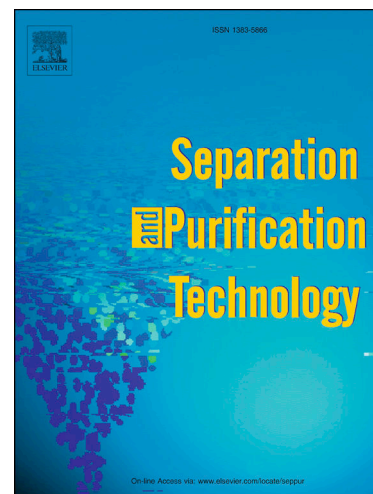# Journal Pre-proofs

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Highly concentrated HCl and NaOH from brines using Electrodialysis with Bipolar Membranes

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Abstract

Electromembrane technologies, such as electrodialysis with bipolar membranes (EDBM) have been widely proposed for the treatment and valorization of seawater reverse osmosis (SWRO) concentrated streams. The EDBM technology applied to brines produces acids (HCl) and bases (NaOH) with just two inputs: electric energy and brine. Thus, the reagents self-supply to the SWRO plant could be achieved, which definitively fits the principles of the Circular Economy. However, previous works have shown low concentration values of the produced acids and bases by EDBM, which undoubtedly hampers the possibility of its use in the SWRO plant.

Therefore, the aim of the present work is to demonstrate the viability of a new strategy to valorise SWRO brine producing commercial HCl at 35% wt.by means of the integration of EDBM technology under constant (galvanostatic, equivalent to the use of electrical energy from the grid mix) and variable current intensity (equivalent to the use of PV solar energy) and azeotropic distillation. Concentrations of HCl and NaOH up to ~ 3.3 mol·L^{-1} and ~ 3.6 mol·L^{-1} are obtained respectively, which are almost 50% higher than any other reported in the literature so far using this technology. The specific energy consumption of the EDBM unit was in the range of 21.8 kWh·kg^{-1} of HCl and 43.5 kWh·kg^{-1} of HCl, being dependent on the average applied current density. These HCl concentrations, although being acceptable for internal use in the SWRO plant, did not reach commercial levels (35% wt. of HCl), so a further concentration stage using distillation has been evaluated through simulation. Minimum values of the overall EDBM plus distillation process specific energy consumption (SEC$_{OV}$) was between ~ 40 kWh·kg^{-1} and ~ 60 kWh·kg^{-1} of HCl.

Moreover, the environmental burdens associated with the energy consumption are quantified in terms of the carbon footprint (CF). Although the SEC$_{OV}$ is slightly higher, the use of PV solar energy instead of the grid mix let the process to provide better environmental performance. The renewable alternative provides values between 1.61 kg CO_2-eq.·kg^{-1} of HCl (full PV solar energy) and 6.97 kg CO_2-eq.·kg^{-1} of HCl (PV solar energy and steam).

Keywords: Concentrates valorization; Bipolar membrane electrodialysis; Photovoltaic solar energy; Acid production; Circular economy

Highlights

- 3.3 mol·L^{-1} of HCl and 3.6 mol·L^{-1} of NaOH were obtained by means of EDBM
- The use of galvanostatic or PV solar energy does not affect the final concentration
- SEC$_{EDBM}$ between 21.8 kWh·kg^{-1} and 43.5 kWh·kg^{-1} of HCl were obtained
- EDBM plus distillation can produce commercial concentrations of HCl
- With a higher SEC$_{OV}$, using PV solar provides better environmental performance

1. Introduction

In recent years, desalination technologies have gained noticeable attention as a critical alternative to address water scarcity issues that exist in regions all over the world. Seawater reverse osmosis (SWRO) is the most widespread technology, accounting for 34% of the global desalination capacity, i.e. 32.4 million m^3·day^{-1} [1]. One of the key drawbacks of the SWRO technology is the high-specific energy requirement per unit of freshwater produced which is in the range of 2.5–6.0 kWh m^{-3}, thus sharing most of the operation cost of the desalination plant, contributing up to 40% in the water cost of seawater desalination [2,3]. Apart from specific locations/facilities, reagent consumption is also a drawback, due to both cost and availability. In this sense, it can be estimated that SWRO plants worldwide spend 815 million €·year^{-1} on reagent purchase, assuming a global average dosages of HCl of ~73 mg·L^{-1} of freshwater and of NaOH of ~60 mg·L^{-1} of freshwater [4]. Recent personal communications with a desalination plant located in the Mediterranean area confirm the requirements of HCl from 0.2 g·m^{-3} of freshwater to 0.5 g·m^{-3} of freshwater and of NaOH from 30 g·m^{-3} of freshwater to 60 g·m^{-3} of freshwater, which are employed at commercial concentrations from 31% wt. to 35% wt. (10.1 mol·L^{-1} to 11.4 mol·L^{-1}) and 50% wt. (18.9 mol·L^{-1}), respectively, being able to employ lower concentrations incurring in greater pumping requirements.

Electromembrane technologies, such as electrodialysis (ED) and electrodialysis with bipolar membranes (EDBM) have been widely proposed for the treatment and valorization of SWRO concentrated streams, the so-called brines, which features NaCl at high concentration thus they can be considered as hypersaline streams. Available literature mainly approaches these processes from a zero liquid discharge (ZLD) point of view [5,6]. This ZLD approach pursues the almost total recovery of products from the SWRO brine thus no real liquid effluent can escape the facility as a waste: this is equivalent to an almost 100% recovery of the involved species. However, other approaches are also useful not entailing the efforts derived from a 100% recovery. In this work it is envisage the brine as a source to create added value by means of the generation of acid and bases using EDBM. Therefore, the brine is used partially rather than totally as in a ZLD approach. The EDBM technology applied to brines produces acids (HCl) and bases (NaOH) with just two inputs: electric energy and brine. Thus, reagents self-supply could be achieved [4,7], which definitively fits the principles of the Circular Economy [8]. The arising question is related to the technical specifications of the obtained products, mainly its concentration, as a very diluted stream would demand too much pumping as previously mentioned. A high concentration of HCl and NaOH is therefore expected. Low

concentration of the products neither is technically interesting due extra energy for pumping nor from a storage point of view due to the potential large occupied volumes.

Table 1 summarizes the maximum concentrations of HCl and NaOH obtained using EDBM technology according to previously published works. The concentrations of obtained products neither did exceed in any of the reported studies ~2.0 mol·L^{-1} for the acid, nor ~2.5 mol·L^{-1} for the base. Therefore, the concentrations of HCl and NaOH with this EDBM technology reported in the literature did not reach the specified commercial ones, 35% wt. (11.4 mol·L^{-1}) for HCl and 50% wt. (18.9 mol·L^{-1}) for the NaOH, requiring potential further concentration stages. Of course, this situation may hamper the economic and environmental competitiveness of this self-supply strategy, making it less attractive. Additionally, it is noteworthy that all previous studies in Table 1 are completed at lab-scale. The literature reviewed also reports operating times usually below 30 hours and the bipolar membrane area does also below 3040 cm^2. Qualitatively, it can be stated that the volume and the initial concentration of the feed to the EDBM process has an influence in the final concentration of the products obtained. In the reported literature, the NaCl feed tends to be depleted with time, thus not allowing the achievement of higher concentrations levels. On the other hand, the use of the EDBM would represent additional energy burdens for the already energy intensive SWRO facility. In order to alleviate environmental burdens such as carbon footprint derived for this extra energy use, the integration of photovoltaic solar (PV) energy is proposed. Indeed, the technical feasibility of HCl and NaOH production by means of EDBM using both constant (grid mix energy) and variable (PV solar energy) current intensity has been previously reported by the authors [9–11].

3

Table 1. Summary of maximum concentrations of products HCl and NaOH obtained using EDBM.

Mode of operation	Initial feed concentration	Bipolar Membrane	Current density (constant)	Voltage (constant)	Max. HCl concentration	Max. NaOH concentration	SEC	Ref.
	mol·L^{-1}		A·m^{-2}	V	mol·L^{-1}	mol·L^{-1}	kWh·kg^{-1} of acid	
Desalination brine								
Batch	NaCl 1.71 and 3.42	-	-	9	1.99	2.14	3.8	[12]
Batch	NaCl ~1.00	Neosepta BP-1	250-1000	-	0.8	1	-	[11]
Semicontinuous	Mainly Na$^+$: 0.377 and Cl$^-$ 0.635	Fumatech	570	-	0.7	1	9.0	[13]
Continuous or semicontinuous	NaCl ~1.00	Fumasep (Fumatech)	220	-	0.98	1.64	7.3	[9]
Batch	Mainly NaCl ~0.02	-	100-900	-	~0.2	~0.2	-	[14]
Batch	TDS>20 g·L^{-1}	FuMA-Tech.	1000	-	1.08	1.30	4.13-5.00	[15]
Batch	Mainly Na$^+$~0.16 and Cl$^-$ ~0.22	He-bei Guang-ya	-	15-25	1.4	-	4.7-8	[16]

4

Batch	Mainly Na$^+$0.45-0.79 and Cl$^-$ 0.49-0.78	Neosepta BP-1 and FBM (Fumatech)	100	-	0.60	0.65	1.6-2.0[a]	[17]
Batch	NaCl 0.6	-	250	-	0.22	0.29	-	[18]
Saline wastewater								
Batch	Cl$^-$/ SO$_4^{2-}$ Low concentration: 0.063 / 0.026 Medium concentration: 0.151 / 0.230 High concentration: 0.497 / 0.840	PC BP (PCCell)	-	9	1.3	2.3	4.2	[19]
Batch	Cl$^-$: 0.35 SO$_4^{2-}$: 0.03 Na$^+$: 0.24 K$^+$: 0.17 Ca^{2+}: 0.02	PBM, PCA (Polymer Chemie Altmeier)	-	7.5	1.6	1.25	4-5	[20]
Semicontinuous	Cl$^-$: 0.2225 SO$_4^{2-}$: 0.0029 Na$^+$: 0.1585 Ca^{2+}: 0.0229 Mg^{2+}: 0.0058	He-bei Guang-ya	-	15-25	1.3	1.3	8	[16]

Batch	Cl$^-$: 1.125 Na$^+$: 0.825	PCA - PolymerChemie Altmeier and Fumatech	-	16	1.8	2	-	[21]
Batch	SO_4^{2-}: 0.88-0.92 Na$^+$: 1.04-1.18	FBM (Fumatech)	300-600	-	1.76[b,c]	2.41[a]	5.5	[22]
Continuous	NaCl 0.048-0.390	Neosepta BP-1	2.6-260	-	0.049-0.303	0.037-0.299	-	[23]
Batch	HCl+FeCl$_2$ eq H$^+$ 0.13 Fe 0.01	Neosepta BP-1	200-400	-	0.51	-	1.68	[24]
Batch	H$_2$SO$_4$+FeSO$_4$ eq H$^+$ 0.14 Fe 0.01	Neosepta BP-1	200-400	-	0.63	-	1.39	[24]
Batch	NaOH 0.05-0.2 NaNO$_3$ 0.24-0.85	Neosepta BP-1	200-700	-	-	1.9	1.30-2.80 [a]	[24]

[a] Referred to NaOH (kWh·kg^{-1} of NaOH).

[b] Expressed has normality. Valence of ionic species is 1, so normality and molarity are equal.

[c] The acid is H$_2$SO$_4$

In the light of recent publications focused in the recovery of NaOH by means of EDBM and additional purification steps, it is clear that the target is to reach commercial concentrations [25]. Therefore, the aim of the present work is to demonstrate the viability of a new strategy to valorise SWRO brine producing commercial HCl at 35% wt. by means of the integration of: 1) an electromembrane technology (EDBM) under constant (galvanostatic, equivalent to the use of electrical energy from the grid mix) and variable current intensity (equivalent to the use of PV solar energy), targeting concentration values higher than those previously reported in the EDBM unit (~2.0 mol·L^{-1} for HCl and ~2.5 mol·L^{-1} for NaOH), and 2) an azeotropic distillation. A simplified model synthetic solution, which replicate the TDS contribution from Na$^+$ and Cl$^-$ ions coming from a real brine, [26] has been used. The HCl stream after the EDBM unit is expected to be further concentrated using distillation in order to reach the aforementioned commercial concentration suitable for the self-supply strategy within a SWRO facility (exemplified here as 11.4 mol·L^{-1} of HCl or 35% wt. of HCl). Additionally, it is of interest to evaluate the specific energy consumption of the produced HCl to benchmarking it against existing references. The overall specific energy consumption of a process in which the EDBM HCl output stream is sent to a distillation unit is included in order to analyse the potential synergistic effects of such integration. Moreover, the environmental burdens associated to the energy consumption are quantified in terms of carbon footprint (CF).

2. Experimental methodology

2.1. Lab scale experimental set-up

All the experiments in this work were performed in a modified PCCell (Germany) bench scale laboratory ED system, composed of different elements as shown in Figure 1. A proper description of the experimental set-up is given in previous works [9].
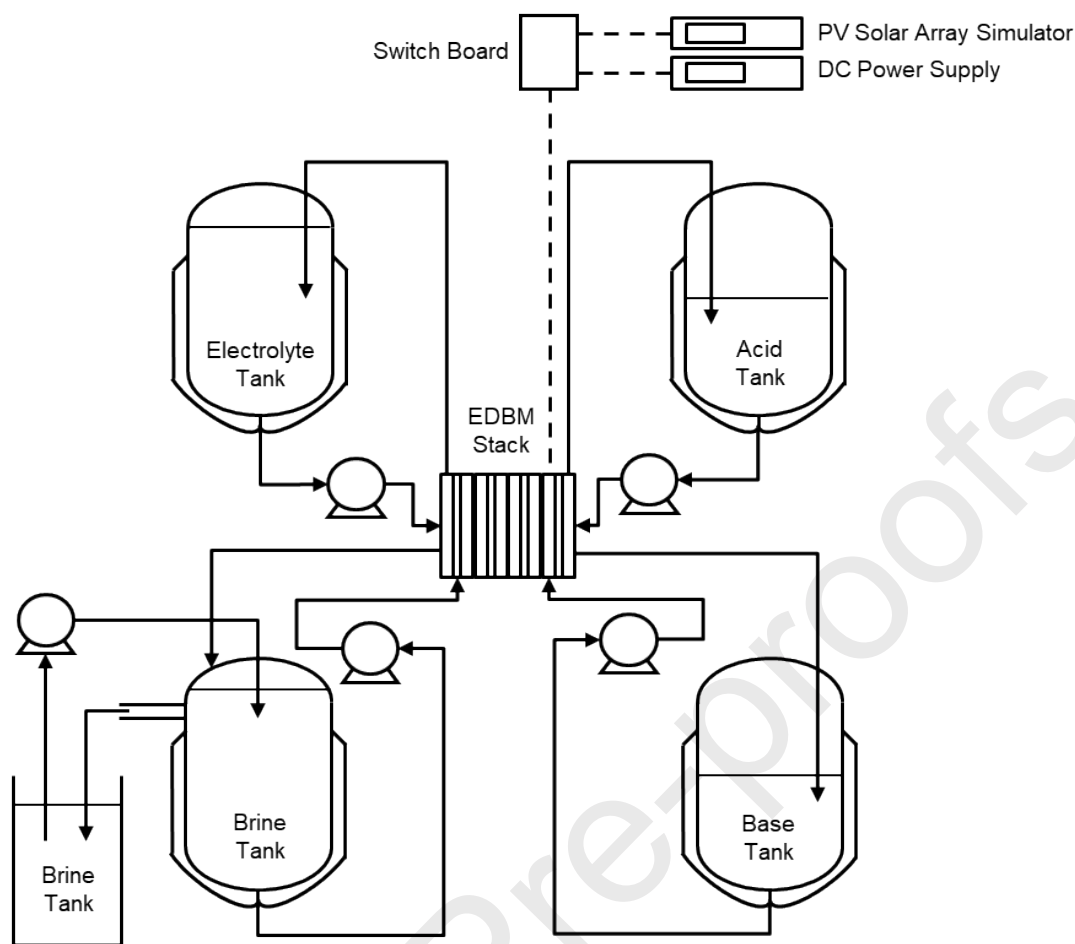
7

Figure 1. Simplified set up of the lab scale EDBM process.

2.1.1. Membrane and cell stack configuration.

EDBM is an electromembrane process requiring a cell in which the different required membranes are configured. In the experimental set-up employed, a commercial electrodialysis cell (Elektrolyse Project, Netherlands) composed of two electrodes made of titanium and coated with ruthenium oxide, with an effective area of 100 cm^2 (square, 10 cm each side) was assembled. Na_2SO_4 has been selected as electrolyte based on previous works [9]. Commercial heterogeneous polyethylene-based anion (AM-PP RALEX) and cation (CM-PP RALEX) exchanges membranes from Mega (Czech Republic) were selected. In addition, the bipolar membranes (Fumasep FBM) were provided by Fumatech (Germany).

The unit cell configuration is AEM/BM/CEM, so the stack configuration follows the next sequence CEM/AEM/BM/CEM/CEM (or abbreviated as –CABCC+ configuration). Once all the experiments were completed, we did not observed any macroscopic damage in the membranes at the operating pH values, thus confirming the expected good performance.

2.1.2. Power supply and solar array simulator

Constant current intensity is provided thanks to a power supply (Statron, Germany) which is integrated into the modified bench scale system. Variable current intensity is provided by means of a PV solar array

8

simulator (Chroma, Netherlands). This power supply is able to simulate the electrical output of a PV solar array, by defining a set of parameters and a given solar irradiation profile. The software is able to provide the current intensity-voltage (I-U) and power-voltage (P-U) operation curves through two inputs: the panel parameters and the irradiation and temperatures curves chosen by the operator. The current intensity (I) and the voltage (U) relation is determined by the total ohmic resistance between the electrodes of the stack, which is obviously not fixed by the simulator. Therefore, the behaviour of the simulator is the same as a real PV solar array. The selected parameters for this case study, based on a real PV panel parameter, are shown in previous works [9,27].

2.1.3. Solutions prepared for the experiments

The brine tank was initially loaded with 20 L of 1.0 mol·L^{-1} NaCl (Scharlau, Spain), which represents a simplified synthetic model SWRO brine [9–11]. The use of Na$^+$ and Cl$^-$ synthetic model solutions avoids the problems derived from divalent ions in real brines. As a result, it helps at the assessment of the system feasibility under favorable operating conditions. As in previous works [10], the initial acid and base concentrations were set up at 0.1 mol·L^{-1} HCl (1 L) and 0.1 mol·L^{-1} NaOH (1 L) instead of 0.0 mol·L^{-1} for both species to avoid the excessive increase in the stack resistance. Both acid and base were ACS grade and purchased from Panreac (Spain). A 0.5 mol·L^{-1} Na$_2$SO$_4$ (ACS grade, Scharlau, Spain) solution was employed as electrolyte. Mili-Q water was used to prepare the solutions.

2.2. Experimental procedure

Six experiments operated in semi-batch mode have been performed (Exp-G500, Exp-G750, Exp-G1000, Exp-PV500, Exp-PV750 and Exp-PV1000). The so-called semi-batch mode refers to an operating mode that simulates a continuous intake from the feed stream, while products are obtained in batch mode. The corresponding experimental conditions for each experiment are summarized in Table 2 and described as follows.

2.2.1. Galvanostatic current intensity experiments (Exp-G500, Exp-G750 and Exp-G1000)

Exp-G500, Exp-G750 and Exp-G1000 experiments were performed along 40 h (4 days, 10 h per day), considering galvanostatic current intensity. A constant current of 5 A (500 A·m^{-2}), 7.5 A (750 A·m^{-2}) and 10 A (1000 A·m^{-2}) provided by the power supply were selected for Exp-G500, Exp-G750 and Exp-G1000, respectively, based on results from previous works [11].

2.2.2. Variable current intensity experiments (Exp-PV500, Exp-PV750 and Exp-PV1000)

Experiments Exp-PV500, Exp-PV750 and Exp-PV1000, lasting 40 h (4 days, 10 h per day), were performed employing a solar array simulator as power supply. The average irradiation profile of July in Almeria (summer in southeast Spain) was obtained from the PV-GIS database [28]. Maximum irradiations of 960 W·m^{-2} are obtained at the central hours of the day in that location in July. Almeria was chosen as an example of a Mediterranean region with potential relevant water scarcity problems [29]. As depicted in Table 2, the

main difference among the experiments carried out under variable current intensity is the average current intensity, which is dependent on the parameters given for the simulation of the PV panel.

Table 2. Description of performed experiments.

Experiment code	Current intensity mode	Average current intensity	PV panel simulated area
		$(A \cdot m^{-2})$	(m^2)
Exp-G500	Constant	500	-
Exp-G750	Constant	750	-
Exp-G1000	Constant	1000	-
Exp-PV500	Variable (Solar irradiation July Almeria, Spain)	493	1.61
Exp-PV750	Variable (Solar irradiation July Almeria, Spain)	734	2.41
Exp-PV1000	Variable (Solar irradiation July Almeria, Spain)	984	3.21

2.3. Analytical methods

Samples were withdrawn from acid, base, feed and electrolyte tanks every two hours. Measurement of conductivity and pH (both by a pHmeter and by acid and base titration using analytical grade reagents) were performed. The concentration of Cl^- and Na^+ were determined by ion chromatography (Dionex ICS-5000 for both anions and cations, Dionex Corp., (USA)). Additionally, control measurements of the concentration of SO_4^{2-} in the electrolyte were carried out through ion chromatography. The use of control samples supported the calculation of the analytical error in the measurement of concentrations of Cl^-, Na^+ and SO_4^{2-} by ion chromatography, being this error less than 5%.

2.4. EDBM specific energy consumption calculation

The energy performance of the EDBM experiments has been evaluated in terms of the specific energy consumption (SEC_{EDBM}). This key performance indicator is the most suitable to understand the energy implications regarding the production of the HCl product [30]. The SEC_{EDBM} metric represents the energy used by the process to produce one unit of mass of the targeted product, in this case HCl. It is noteworthy that this metric is not related with the final obtained concentration, which is different in each experiment. As a result, similar SEC_{EDBM} values from two whatever experiments is not equivalent to reaching similar

final concentrations. The SEC_{EDBM} (expressed in units of kWh·kg⁻¹ HCl) is calculated according to Eq. 1 [9]:

$$SEC_{EDBM} = \frac{\int_{t=0}^{t=te} U \cdot I \cdot dt}{(C_{HCl,\,te} - C_{HCl,\,0}) \cdot V_{HCl} \cdot PM_{HCl}} \tag{1}$$

where U is the total stack voltage (V), I is the current intensity (A), $C_{HCl,te}$ is the concentration of HCl at the end of the experiment (mol·L⁻¹), $C_{HCl,0}$ is the initial concentration of HCl (mol·L⁻¹), V_{HCl} is the volume of HCl (L), PM_{HCl} is the molecular weight of HCl (g·mol⁻¹), te is the total time of the experiment, (h) and t is time (h).

In this sense, SEC_{EDBM} is referred to a unit of mass of HCl, however, the acid it is not produced in an isolated way, but NaOH, which is also employed in SWRO plants, is obtained as coproduct, i.e., HCl and NaOH are simultaneously produced with the same amount of energy.

2.5. Simulation of the product concentration: distillation

Simulations of a dual distillation process for the concentration of a mixture of HCl and water have been carried out using Aspen Plus [31] software. This process would be composed by a first stage of EDBM and a further concentration by means of azeotropic distillation.
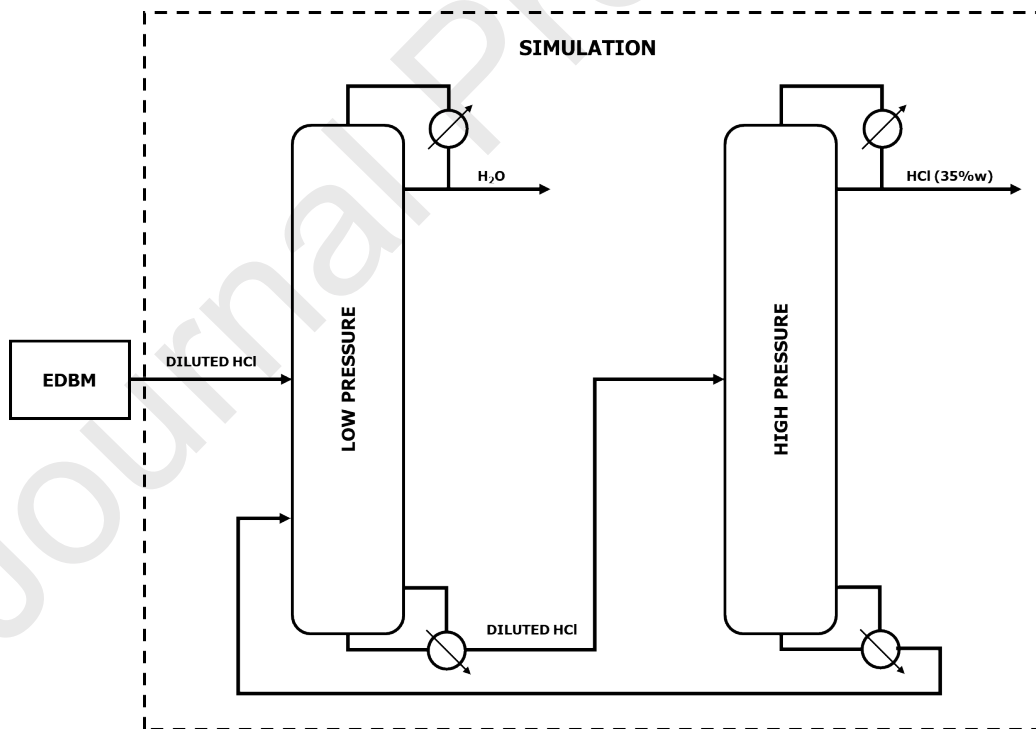

Figure 2. Process block flow diagram used for the concentration of the HCl stream from the EDBM unit.

Figure 2 represents the simulated distillation units. Two distillation columns are considered here, the first one at low pressure (0.25 bara) and the second one at high pressure (3.0 barg), in order to avoid the azeotropic point of the mixture HCl/H_2O. The influence of the concentration of the diluted HCl stream

11

which feeds the low-pressure column in the specific energy consumption of the distillation units SEC_{DIS} has been evaluated, considering concentrations from 0.5 mol·L^{-1} to 3.0 mol·L^{-1} (1.5% wt. – 9.2% wt.). Water is removed as the distillate in the low-pressure column, whereas the pre-concentrated HCl is pumped to the high-pressure column, where a concentrated HCl stream (35% wt.) is produced as the head product. In the high concentration column, the bottoms product is sent back to the low-pressure column. This configuration is typically used for the recovery of diluted HCl streams. An electrolyte thermodynamic model available in Aspen Plus is used here for the azeotrope characterization. Ambient temperature and pressure are set for the feed stream. Both, low-pressure and high-pressure columns have 8 equilibrium stages. The output of the simulation is the total amount of heat that must be added to the separation process to complete the requested concentration. This heat can be translated into the SEC_{DIS}, expressed in kWh·kg^{-1} of HCl (using as equivalence 3.6 MJ = 1 kWh). Once the SEC_{EDBM} and the SEC_{DIS} are available, it is possible to assess the overall specific energy consumption of the integration of the EDBM unit and the distillation unit. This overall SEC (SEC_{OV}) is then the sum of the SEC of both processes, according to Eq. 2:

$$SEC_{OV} = SEC_{EDBM} + SEC_{DIS} \tag{2}$$

2.6. Carbon footprint calculation

The environmental burdens of the production of HCl by means of the EDBM technology coupled to distillation could be represented by the assessment of the carbon footprint (CF) of the overall energy input to the system. In this sense, the CF is calculated according to Eq. 3:

$$CF_{OV} = CF_{EDBM} + CF_{DIS} = SEC_{EDBM} \cdot CF_E + SEC_{DIS} \cdot CF_{STEAM} \tag{3}$$

where CF_E represents the CF of the energy input, if the grid mix is evaluated (Spain as example) a value of 0.338 kg CO_2-eq.·kWh^{-1} will be considered, whereas a value of 0.036 kg CO_2-eq.·kWh^{-1} will be considered if the solar PV energy is chosen [4], and CF_{STEAM} represents the CF for the steam with a value of 0.294 kg CO_2-eq.·kWh^{-1} [32].

The obtained result could be then compared with the benchmark value for the production of HCl reported in the Ecoinvent database [32] based on the H_2 and Cl_2 reaction (1.14 kg CO_2-eq.·kg^{-1} HCl). However, it must be taken into account that the HCl is produced at different concentrations and that the benchmark value does not include the transportation to the desalination facility.

3. Results

Firstly, the results for the production of acid and base under the selected experimental conditions are presented. Once these results are discussed, an evaluation of the SEC referred to the acid (representative product) is assessed. Finally, a study of the most adequate concentration of acid to be obtained by means of EDBM before a concentration stage (distillation) in order to obtain the minimum overall SEC is depicted. Moreover, the environmental burdens associated to the energy consumption are quantified in terms of the carbon footprint (CF).

3.1. Analysis of the production of acid and base in a semi-batch EDBM set-up at lab scale

The evolution of Cl^- concentration in the acid compartment and the Na^+ concentration in base compartment under the experimental conditions used in this study are shown in Figure 3. The operation in a semi-batch mode, as in this work, led to two products, HCl and NaOH, at a higher concentration than the previously reported on the literature (Table 1). In general, it is observed that the temporal variation of the concentration of the ionic species tends to be faster at the beginning of the experiments, while it is shown to be slower as experimental time increases.

Cl^- concentration in the acid compartment of 2.0 mol·L^{-1}, 2.8 mol·L^{-1} and 3.2 mol·L^{-1} are obtained for galvanostatic current densities of 500 A·m^{-2} (Exp-G500), 750 A·m^{-2} (Exp-G750) and 1000 A·m^{-2} (Exp-G1000), respectively. In relation to the Na^+ concentration in the base compartment, values of 2.9 mol·L^{-1}, 3.3 mol·L^{-1} and 3.6 mol·L^{-1} are obtained for galvanostatic current densities of 500 A·m^{-2} (Exp-G500), 750 A·m^{-2} (Exp-G750) and 1000 A·m^{-2} (Exp-G1000), respectively. This means that a 2-fold current intensity increase is able to produce up to 1.6-fold concentrated products in the current densities used in this study.

A similar behaviour is observed when the experiments with variable current density are compared (Exp-PV500, Exp-PV750 and Exp-PV1000). Both the concentrations of Cl^- in the acid and the concentrations of Na^+ in the base are higher when the average current density used is higher. Thus, for the average intensity of 493 A·m^{-2} (Exp-PV500) a concentration of Cl^- in the acid of 2.0 mol·L^{-1} and Na^+ at the base of 2.9 mol·L^{-1} is obtained, for the average intensity of 736 A·m^{-2} (Exp-PV750) a concentration of Cl^- in the acid of 2.7 mol·L^{-1} and Na^+ at the base of 3.3 mol·L^{-1} is obtained, and when an average intensity of 984 A·m^{-2} (Exp-PV1000) is used, a concentration of Cl^- in the acid of 3.2 mol·L^{-1} and Na^+ at the base of 3.6 mol·L^{-1} is obtained.

Almost even concentrations of both Cl^- in the acid compartment and Na^+ in the base compartment are obtained when similar average current intensity values are employed, not depending on the current density profile (constant versus variable). So, experiments Exp-G500 (500 A·m^{-2}) and Exp-PV500 (493 A·m^{-2}), Exp-G750 (750 A·m^{-2}) and Exp-PV750 (736 A·m^{-2}), and Exp-G1000 (1000 A·m^{-2}) and Exp-PV1000 (984 A·m^{-2}) could be considered equivalent.
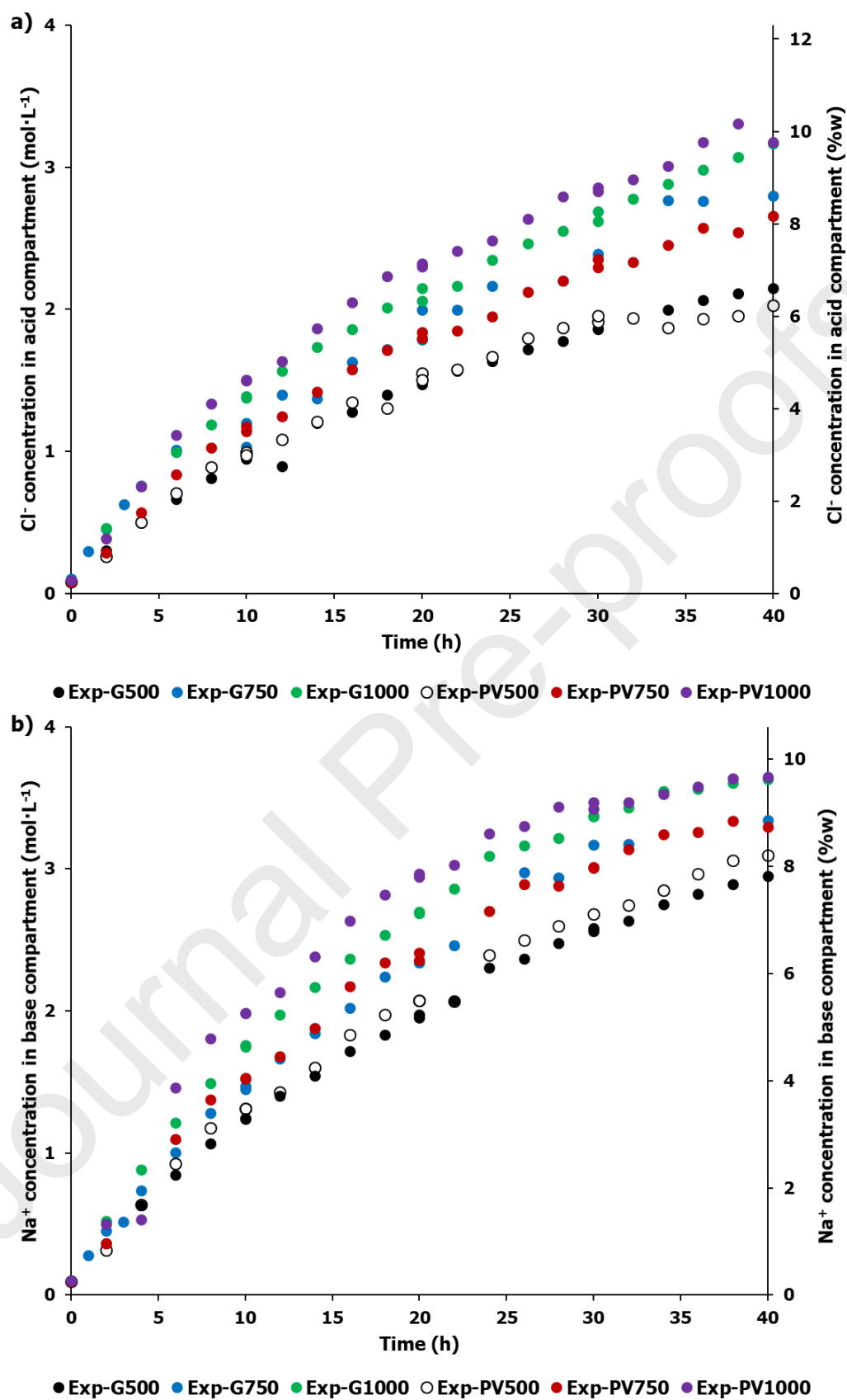
Figure 3. Temporal evolution of: a) Cl⁻ concentration in the acid compartment, and b) Na⁺ concentration in the base compartment.
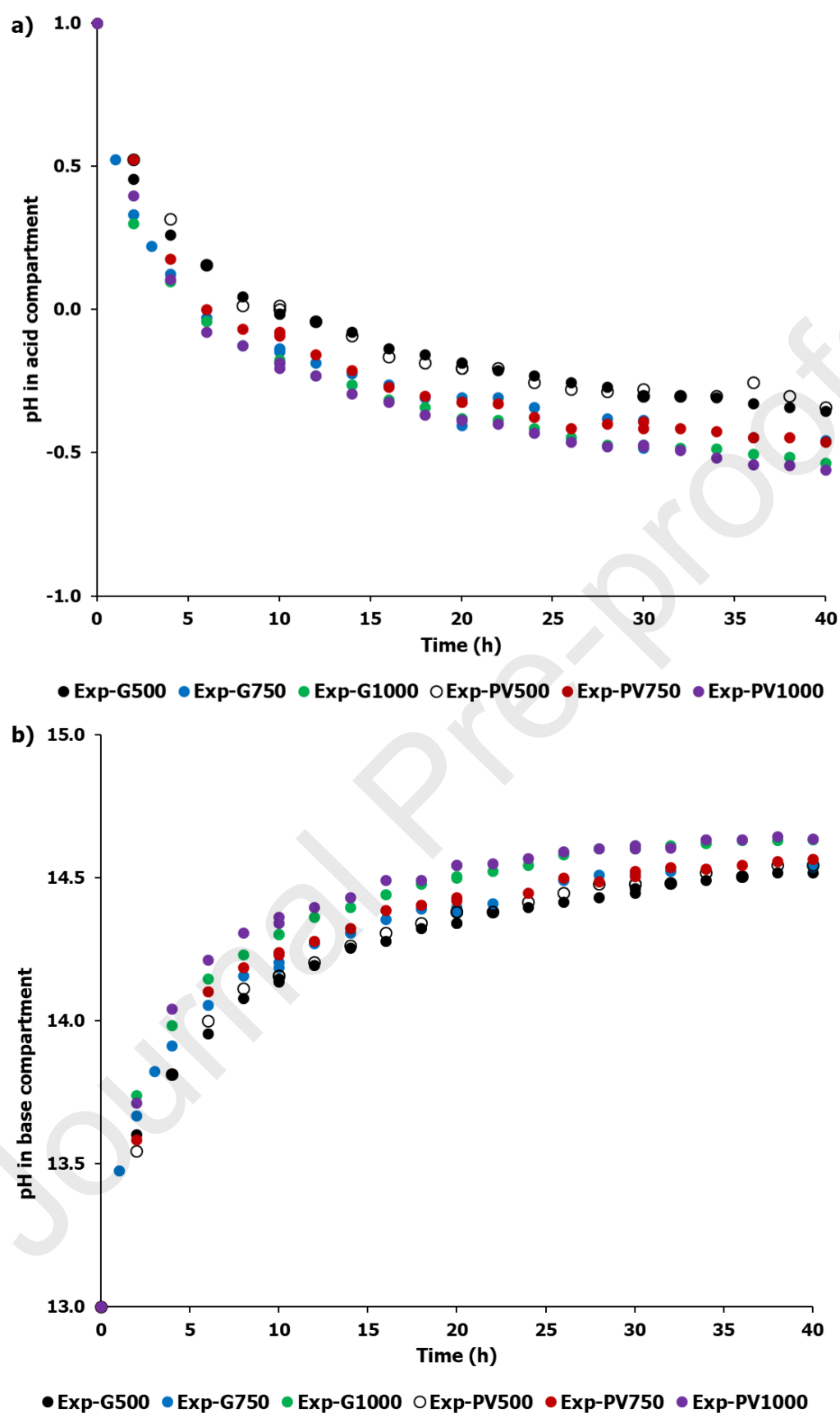
Figure 4. Temporal evolution of the pH values in: a) the acid compartment, and b) the base compartment.

15

Regarding the pH in the acid and the base, which is intrinsically related to H^+ concentration in the acid and OH^- concentration in the base, same behaviour than in the Cl^- in acid and Na^+ in base concentrations is reported (Figure 4). Initial pH value of 1.0 for acid and 13.0 for base are registered, which correspond to 0.1 mol·L^{-1} of HCl and NaOH, respectively.

Taking into account the above mentioned, Table 3 summarizes the maximum product concentrations for the six performed experiments, As a result, it can be stated that the variation in the final concentration of HCl and NaOH is affected by the average current density (from ~500 A·m^{-2} to ~1000 A·m^{-2}), but not by the use of galvanostatic or variable current density. Maximum reported concentrations in this work for HCl were 3.3 mol·L^{-1}, which are really almost 1.7 times the values previously reported in the literature (~2.0 mol·L^{-1} for HCl), which are technically acceptable for self-supply in the desalination plant; however, still far away from those used for commercial distribution (10.1 mol·L^{-1} to 11.4 mol·L^{-1}). Similar conclusions can be withdrawn for NaOH production. Larger concentration values are unlikely to be reachable due to crossover effects. The small size of the protons combined with a concentration gradient promoting the back-diffusion prevent any significant additional increase in the compartment concentration neglecting the additional injected energy [17,18].

Table 3. Summary of maximum product concentrations for the four performed experiments in this work.

	HCl		NaOH	
	mol·L^{-1}	% wt.	mol·L^{-1}	% wt.
Exp-G500	2.15	6.59	2.95	7.81
Exp-G750	3.07	9.41	3.63	9.63
Exp-G1000	3.17	9.72	3.63	9.62
Exp-PV500	2.03	6.22	3.10	8.21
Exp-PV750	2.66	8.16	3.34	8.84
Exp-PV1000	3.31	10.15	3.65	9.66

3.2. SEC evaluation and comparison

SEC_{EDBM} values are dependent on the product concentration and on the current intensity profile applied. Table 4 reports the SEC_{EDBM} values considering the final HCl concentration of each experiment. As depicted in Table 4, maximum SEC_{EDBM} values between 21.8 kWh·kg^{-1} of HCl and 43.5 kWh·kg^{-1} of HCl are obtained under the conditions used in this work. The SEC_{EDBM} value increased as the average current intensity did. However, employing a variable current intensity profile, as in Exp-PV500, Exp-PV750 and Exp-PV1000 leads to slightly higher SEC_{EDBM} values. Values reported in Table 1 show that a conservative approach gives a minimum SEC_{EDBM} value of ~4 kWh·kg^{-1} of HCl. This very relevant difference is explained by the fact that the target of this work is the production of HCl and NaOH as concentrated as possible rather checking the overall efficiency, which is intrinsically related to a lower current density. However, a roughly estimation can deliver here additional insights. Table 1 shows that the minimum SEC_{EDBM} value of ~4 kWh·kg^{-1} of HCl corresponds to a concentration of ~1 mol·L^{-1} of HCl. The upper bound for this SEC_{EDBM} value can be set up at twice that value thus ~8 kWh·kg^{-1} of HCl for the same

concentration of ~1 mol·L^{-1} of HCl. As our HCl product has three-fold concentration, the estimated SEC$_{EDBM}$ value would be around ~24 kWh·kg^{-1} of HCl. This last value is then not far away from those reported in Table 4, especially considering that this process is not energetically optimised. Although the SEC$_{EDBM}$ value is slightly higher in Exp-PV750 than in Exp-G750, the use of PV solar energy instead of grid mix energy is going to result into obvious environmental improvements, as it reduces the environmental burdens associated to the energy consumption, such as in terms of carbon footprint (CF).

Table 4. Comparison of SEC$_{EDBM}$ values and maximum product concentration.

	HCl	NaOH	SEC$_{EDBM}$
	mol·L^{-1}	mol·L^{-1}	kWh·kg^{-1} HCl
Exp-G500	2.1	2.9	21.82
Exp-G750	3.1	3.6	31.1
Exp-G1000	3.2	3.6	41.0
Exp-PV500	2.0	3.1	26.7
Exp-PV750	2.7	3.3	37.2
Exp-PV1000	3.3	3.6	43.5

Table 5 reports the extrapolation of the SEC$_{EDBM}$ values for different final HCl concentrations with the aim of being able to compare different current densities profiles without the dependence on the product maximum concentration achieved. Moreover, SEC$_{DIS}$ values for different HCl input concentrations to the distillation unit are also included (later discussed). As it can be observed in Table 5, it is obvious that a higher concentration at the output of the EDBM unit demands a higher SEC$_{EDBM}$ values due to the concentration of HCl in the semi-batch mode. A linear regression shows a step of 4.1 (kWh·kg^{-1} HCl)·(mol·L^{-1} HCl)$^{-1}$ to 7.1 (kWh·kg^{-1} HCl)·(mol·L^{-1} HCl)$^{-1}$ depending on the applied current density. Therefore, this means that an increase of 1.0 mol·L^{-1} of HCl requests an increase in the SEC$_{EDBM}$ between 4.1 kWh·kg^{-1} and 7.1 kWh·kg^{-1} of HCl.

Table 5. Influence of the HCl concentration in the SEC$_{EDBM}$ and the SEC$_{DIS}$ values.

HCl Concentration		SEC$_{EDBM}$						SEC$_{DIS}$
								kWh·kg^{-1} HCl
mol·L^{-1}	% wt.	kWh·kg^{-1} HCl						
		Exp-G500	Exp-G750	Exp-G1000	Exp-PV500	Exp-PV750	Exp-PV1000	
0.50	1.54	14.5	17.8	24.3	14.7	24.5	23.3	58.0
1.00	3.07	16.5	20.8	27.5	17.8	28.1	26.7	34.7
1.50	4.61	18.5	23.8	30.6	20.8	31.6	30.0	25.7
2.00	6.14	20.6	26.7	33.8	23.9	35.2	33.4	20.8
2.50	7.68	-	29.7	36.9	-	38.8	36.8	17.6
3.00	9.21	-	32.7	40.1	-	-	40.1	15.4

3.3. Selecting an acid concentration for the distillation unit

As previously shown, the EDBM reaches a final output concentration between 3.17 mol·L^{-1} and 3.31 mol·L^{-1} of HCl at current densities of 1000 A·m^{-2}. These values are far away from the concentration values of 10.1 mol·L^{-1} or 11.4 mol·L^{-1} of HCl used in the SWRO facility. Consequently, it is necessary to concentrate further the output stream. Technically, this concentration of the diluted HCl stream is completed by distillation units, leading to commercial concentrations.

A study of the best possible concentration at the diluted HCl output stream of the EDBM unit regarding the overall process of EDBM and distillation has been carried out. The calculation of the SEC$_{OV}$ has been calculated through the experimental data obtained from experiments Exp-G500, Exp-G750, Exp-G1000, Exp-PV500, Exp-PV750 and Exp-PV1000, and through a simulation of two distillation columns.

For each studied current density, and for each value of the HCl concentration at the outlet stream of the EDBM (which is the inlet to the distillation), it is possible to assess the contribution to the SEC$_{OV}$ due to the EDBM, namely SEC$_{EDBM}$, and the distillation process, namely SEC$_{DIS}$. The inset of Figure 5 shows only selected discrete values for a proper visualization of the contribution of the two processes for the case of Exp-G750 as example. Figure 5 shows the SEC$_{OV}$ for each experiment when the full range of HCl concentration is used instead of discrete values. As a result, Figure 5 shows the variation of the SECov (due to EDBM and azeotropic distillation) for different HCl concentrations in the input feed to the first distillation column for the different current densities and energy sources (Exp-G500, Exp-G750, Exp-G1000, Exp-PV500, Exp-PV750 and Exp-PV1000). It can be seen that concentrations between ~2 mol·L^{-1} and ~3 mol·L^{-1} of HCl displays a minimum value for the SEC$_{OV}$, for all the current densities and energy sources used, so no further concentration by means of EDBM will lead to a reduction of the SEC$_{OV}$.

The energy demand for the azeotropic distillation goes down if the concentration of HCl is increased in the EDBM. The reduction is very relevant at low concentration values (under 1 mol·L^{-1} of HCl), while it is less significant once the concentration of HCl is increased (over 1 mol·L^{-1} of HCl). As the SEC$_{EDBM}$ increases linearly with concentration, more electrical energy is needed to break more H$_2$O molecules. For this reason, the composed curve of the SEC$_{OV}$ shows a minimum value in the previously mentioned range between ~2 mol·L^{-1} and ~3 mol·L^{-1} of HCl. Therefore, additional technical efforts for increasing the concentration in the EDBM are not justified. There is no need to go beyond the concentrations obtained in this work. Even if the membrane is engineered to avoid crossover effects, the improved concentration does not help with a lower SEC$_{OV}$. In this sense, the distillation provides better performance, that is, the better use of a single unit of energy relies on the distillation rather than in the EDBM. As an example, for Exp-G750, the share of the distillation is 78% at the lower bound of HCl concentrations (0.5 mol·L^{-1} of HCl) and 25% at the upper bound (3 mol·L^{-1} of HCl). This fact may lead to a suitable HCl concentration value for the EDBM output stream (distillation input stream) in which the value of the SEC$_{OV}$ is potentially minimized.
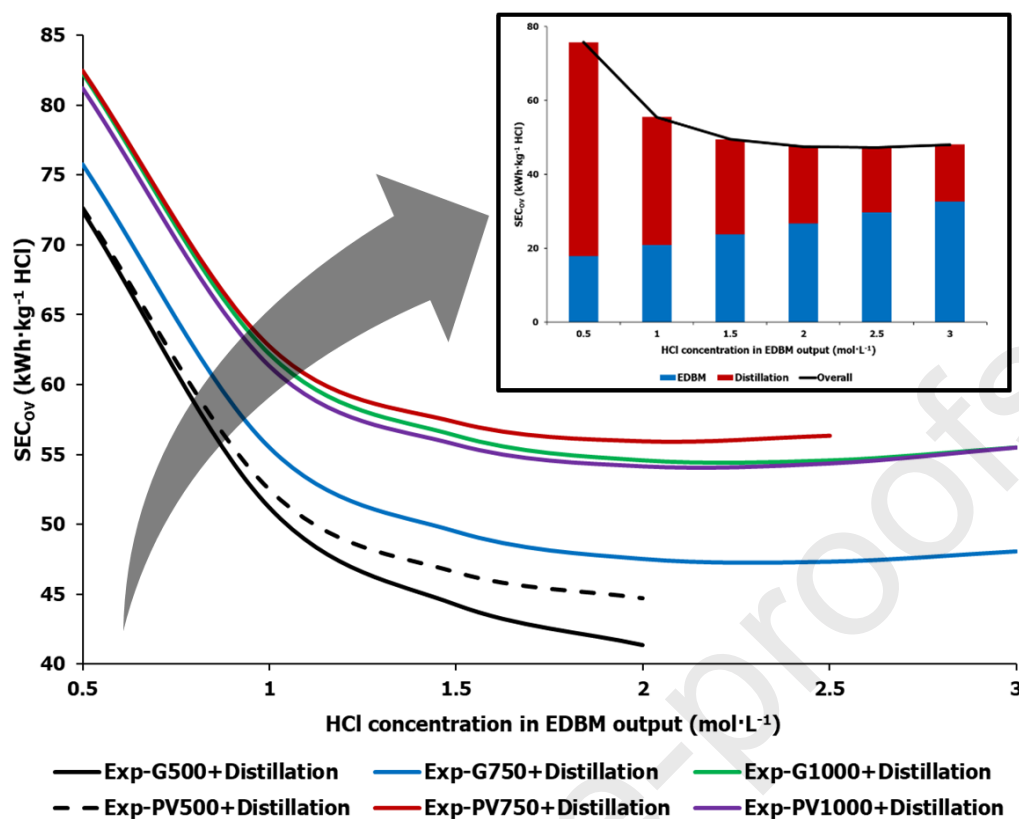
Figure 5. Influence of the HCl concentration at the output stream of the EDBM in the SEC_{OV}.

3.4. Carbon footprint

It is more than evident that using PV solar electricity instead of the grid mix is beneficial in environmental terms. A better CF than using the grid mix can be obtained when PV solar is used as source of electricity. In this sense, the CF when the grid mix (Spanish grid mix, as example) and when PV solar energy are employed have been evaluated. Maximum values of CF_{OV} are obtained for the highest average current density in galvanostatic mode, whereas minimum values are obtained for the lowest average current density given by the PV solar energy. On the other hand, the evaluation of the CF of the distillation is made considering the CF of the steam. Therefore, values between 6.97 kg CO_2-eq.$\cdot kg^{-1}$ HCl (Exp-PV500+Distillation) and 17.66 kg CO_2-eq.$\cdot kg^{-1}$ HCl (G1000+Distillation) are obtained.

Benchmarking against the production of HCl reported in the Ecoinvent [32] database based on the H_2 and Cl_2 reaction (1.14 kg CO_2-eq.$\cdot kg^{-1}$ HCl) does gives lower values than using PV solar energy and steam (6.97 kg CO_2-eq.$\cdot kg^{-1}$ HCl). Anyway, a more in-depth discussion is encouraged for a proper fair comparison. If it is assumed that the steam used in the distillation stage is produced by the PV solar energy, the CF_{OV} would be reduced to 1.61 kg CO_2-eq.$\cdot kg^{-1}$. It is worthy to mention that this range of values is obtained without an allocation procedure, as it is evident that the energy is also used for the simultaneous production of NaOH. Moreover, the benchmark value reported by Ecoinvent database does not include the transport of the HCl from the production sites to the desalination facility.

4. Conclusions

19

In this work the technical feasibility of using EDBM to produce HCl and NaOH up to $\sim$3.3 mol·L^{-1} and $\sim$3.6 mol·L^{-1} respectively using models solutions of SWRO brine at lab scale was demonstrated. The acid and the base concentration levels reached under the experimental conditions used in this work are, to the best of our knowledge, almost 50% higher than any other reported in the literature so far using this technology. The final concentration values of the acid and the base are a function of the average applied current density: the larger the current density, the higher the final concentration. The use of constant, galvanostatic, or variable, PV solar energy, does not affect the final concentration as long as the average current density remains in the same value. The semi-batch operated process showed here a SEC_{EDBM} (related to HCl) corresponding to the range between 21.8 kWh·kg^{-1} and 43.5 kWh·kg^{-1} of HCl, being dependent on the average applied current density. In turn, this SEC_{EDBM} is almost linear with respect to the concentration, between 4.1 kWh·kg^{-1} and 7.1 kWh·kg^{-1} of HCl for every increase of 1.0 mol·L^{-1} of HCl.

The integration of EDBM and distillation can produce commercial concentrations of HCl (11.4 mol·L^{-1} or 35% wt. of HCl) featuring a minimum value of the SEC_{OV} (between $\sim$40 kWh·kg^{-1} and $\sim$60 kWh·kg^{-1} of HCl) for a fixed average current density. This range of values corresponds to HCl concentration values at the output of the EDBM unit which are slightly below the range of HCl concentrations obtained under the experimental conditions of this work ($\sim$3.3 mol·L^{-1} or 10.1% wt. of HCl). As result, in order to produce HCl at commercial concentrations (11.4 mol·L^{-1} or 35% wt. of HCl), it can be concluded that there is no need to improve the concentration of HCl beyond the values obtained here in the EDBM unit ($\sim$3.3 mol·L^{-1} or 10.1% wt. of HCl). Assuming that a larger value could be technically feasible, it will not be of interest, as the distillation is more efficient at concentrating HCl that EDBM per unit of energy used once the HCl concentration belongs to the range between $\sim$2 mol·L^{-1} and $\sim$3 mol·L^{-1} of HCl (6.1% wt. to 9.2% wt. of HCl).

Although the SEC_{OV} is slightly higher, the use of PV solar energy instead of the grid mix let the process to provide a better environmental performance. The renewable alternative provides values between 1.61 kg CO_2-eq.·kg^{-1} of HCl (full PV solar energy) and 6.97 kg CO_2-eq.·kg^{-1} of HCl (PV solar energy and steam), which are still over the value of 1.14 kg CO_2-eq.·kg^{-1} HCl considered the average industrial reference. Therefore, the integration of PV solar is justified in terms of providing a more competitive environmental product than using the electricity from the Spanish grid mix, but still fails when compared with the average industrial production of HCl by the reaction of Cl_2 and H_2. Nevertheless, the CF associated to the commercial HCl transport from the production sites to the desalination facility is not considered in the value reported by the reference LCA database, neither has been considered in this work.

5. Acknowledgements

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21

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Graphical abstract
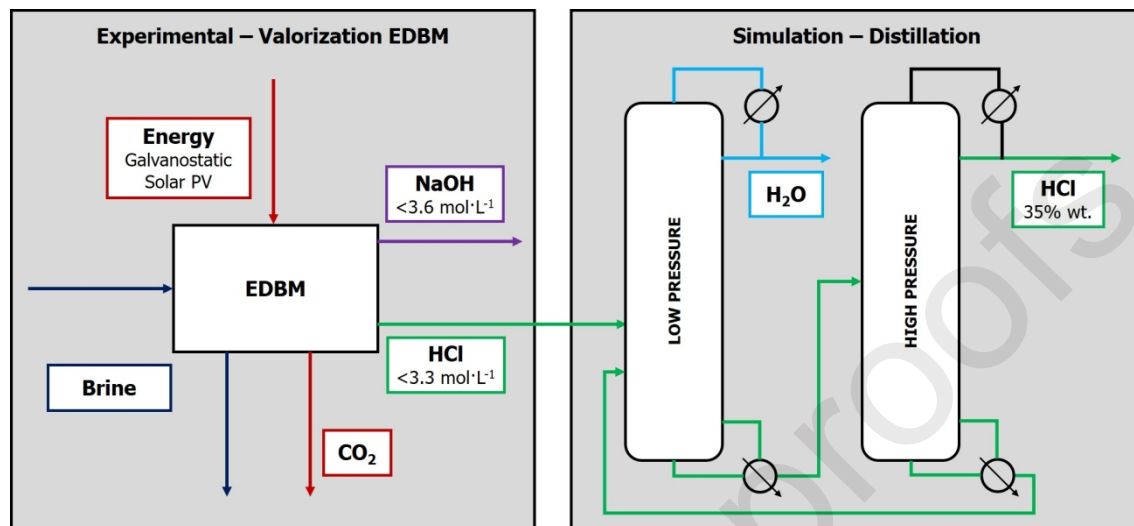
Highlights

- 3.3 mol·L^{-1} of HCl and 3.6 mol·L^{-1} of NaOH were obtained by means of EDBM
- The use of galvanostatic or PV solar energy does not affect the final concentration
- SEC_{EDBM} between 21.8 kWh·kg^{-1} and 43.5 kWh·kg^{-1} of HCl were obtained
- EDBM plus distillation can produce commercial concentrations of HCl
- With a higher SEC_{OV}, using PV solar provides better environmental performance

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Declaration of interests

☒ The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

☐The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: