IWA World Water Congress & Exhibition
11-15 SEPTEMBER 2022 | COPENHAGEN, DENMARK | www.worldwatercongress.org

Reagent recovery from dairy industry wastewater through membrane processes

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Abstract: From 2014 to 2019, India was the world's faster growing major economy, specially at dairy. Thus, the development of more circular production strategies is the key for a continuous and sustainable growth.

The INDIA-H_2O project aims to develop and assess an optimal train of technologies for managing dairy wastewater effluents. This approach fits in a zero-waste discharge philosophy, where the objective is to recover some of the chemicals involved in the activity of the industries, reducing the reagents consumption, and finding more sustainable processes both economical and environmentally.

The train of technologies is composed by Forward Osmosis as a pre-treatment to remove solids and other harmful agents, follow by a bipolar membrane electrodialysis for recovering acids and bases in the form of HNO_3 and NaOH, respectively, which can be reused in the same industry as a cleaning reagent.

Keywords: Dairy wastewater; Forward Osmosis; Electrodialysis; Reagent recovery.

India's industrial sector has undergone great development that boost the country's economy towards never-before-seem heights, with some outstanding industries in terms of Gross Domestic Product generated such as the dairy industry. In a few decades, India has become the world's largest milk producer with nearly 190 million tonnes in 2019, reaching 20% of world's milk production (FICCI, 2020).

In this context, it is necessary to be committed with environmental sustainability as well as the continuous improvement of the system in terms of technical and financial efficiency. One of the more important points in this context is the Clean-In-Place system (CIP), where reagents (acids and bases) are used to clean vessels, pipes, equipment, and other industry facilities. These reagents, present in industry wastewater beside other compounds come from the production process. The compounds can negatively affect areas where they are poured if they are not treated properly (Veiga *et al.*, 2022).

In this context, several studies have shown the effectiveness of Bipolar Membrane Electrodialysis (BMED) for compound separation in a complex matrix. This technology has emerged as the more environmentally friendly option for water reclamation and reagents recovery by using ion exchange membranes driven by electric currents (zhang *et al.*, 2019; Song *et al.*, 2021). The initial concentration in both draw solutions facilitate the separation of the different reagents.

Bipolar membranes are expensive and delicate, so the use of pre-treatments for removing solids and reduce the number of harmful compounds is highly recommended. One of the technologies used for this purpose is Forward Osmosis (FO), which is a pressure-driven process where the osmotic pressure difference between two solutions (feed and draw solutions) generates the pass through a semi-permeable membrane of water, from the less concentrated solution to the other side

(Haupt *et al*., 2018). The draw solution, once diluted by the water extracted from the feed solution can be recovered to be used again.

In this study, a train of technologies (FO – BMED) for reagent recovery from dairy industry was tested by using synthetic solutions with the composition provided by a dairy company. The initial concentration of salts in the FO stage was 0.019 mg L^{-1}, in the case of BMED, the initial concentrations were between 7.5 and 9.5 g L^{-1}. different concentrations and solute in draw solution (Table 1.1) were used as a draw solution in order to concentrate salts in the case of FO and reagents in BMED present in samples. In Figure 1.1 and Figure 1.2 are shown the results obtained in FO and BMED, respectively. The voltage used in BMED to extract both reagents was 9.02 V. The experiments were carried out separately for both systems.

As was shown in the previous figures, FO reported better results with an initial draw solution concentration of 0.5 M $NaNO_3$ (d), obtaining an increase of salts concentration (NO_3^-, Cl^-, SO_4^{2-}, Ca^{2+}, Mg^{2+}, K^+) in the feed solution from 0.02 to 0.32 mg L^{-1}. Also, the experimentation time was the lowest as it reported the highest osmotic pressure in the membrane, which allowed to extract water from feed side more efficiently. By using this draw solution, it was extracted the 90% of water from a 5L sample to the draw solution side in 20 minutes.

Regarding BMED tests, the best performance, in terms of reagent extraction, was reported for an initial concentration of 0.05 M for both acid and base as draw solution (HNO_3 and NaOH respectively), (a) concentrating them until 62.38% and 42.86% of the initial concentration, respectively. The concentration of $NaNO_3$ in the feed was also measured along the experiments and was found that, with 0.05 M of extractant, the concentration was reduced by 75%.

The work done so far, yields promising results in the concentration and extraction of cleaning reagents for reuse in dairy industry. The optimal conditions for reagent extraction in terms of efficiency were set.

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ACKNOLEDGEMENT

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No 820906 and the Indian Department of Biotechnology.

Table 1.1 Draw solutions used in the experiments of the study.

Stage	Solute in DS	Solute concentrations			
FO	$NaNO_3$	0.005M	0.125M	0.250M	0.500M
BMED	HNO_3	0.005M	0.100M	0.200M	
	NaOH	0.005M	0.100M	0.200M	

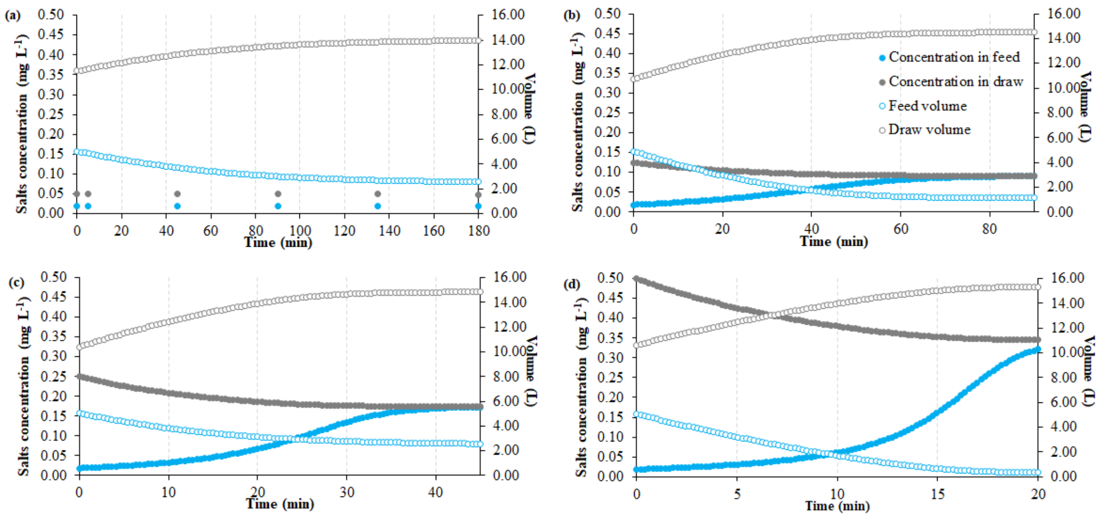

Figure 1.1 Performance of FO system in terms of salts concentration in feed and $NaNO_3$ concentration in draw solution. Initial concentration of $NaNO_3$ in draw solution: (a) 0.005M; (b) 0.125M; (c) 0.25M and (d) 0.50M of $NaNO_3$.

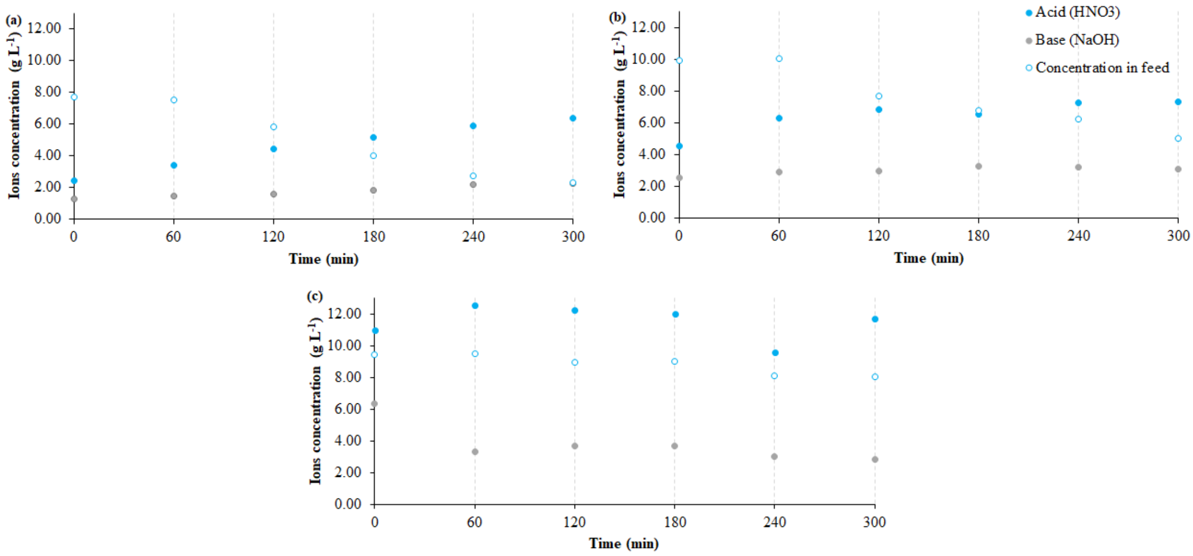

Figure 1.2 Performance of BMED system in terms of acid (HNO_3) recovery and base (NaOH) recovery in draw solutions. Initial concentration for acid and base in draw solutions: (a) 0.05M; (b) 0.1M and (c) 0.2M of HNO_3 and NaOH.