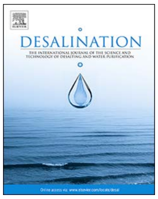
Desalination 436 (2018) 56–62

Contents lists available at ScienceDirect

Desalination

journal homepage: www.elsevier.com/locate/desal



Freeze desalination by the integration of falling film and block freeze-concentration techniques

A. Zambrano[a], Y. Ruiz[a], E. Hernández[b], M. Raventós[b], F.L. Moreno[a,*]

[a] Engineering Faculty, Universidad de La Sabana, Campus Universitario del Puente del Común, Km 7 Autopista Norte de Bogotá, 250001 Chía, Cundinamarca, Colombia
[b] Agri-Food Engineering and Biotechnology Department, Universidad Politécnica de Cataluña (UPC), C/Esteve Terradas, 8, 08860 Castelldefels, Barcelona, Spain

ARTICLE INFO

Keywords:
Cryoconcentration
Seawater
Freezing
Falling film

ABSTRACT

Block and falling film freeze concentration are two technologies that separate water by freezing, with the potential to desalinate seawater. In this study, the integration of two freeze concentration techniques as an alternative to obtain potable water was analysed. Water with 0.5%–8% NaCl was freeze-concentrated by the falling film technique. The ice from each stage was fractionally thawed to recover the solids retained in the ice. The diluted fractions of the thawing stage were freeze-concentrated using the block technique to increase water purity. Falling film freeze concentration was effective to separate the salt from the solution, even at high salt concentrations. Block freeze concentration was effective to increase the water purity until drinkable water was obtained. A multistage process with the integration of these techniques was proposed to obtain 74% of the amount of the initial solution at 0.05% of salt, and 26% at 13.4% of salt. With this process, a salt removal efficiency of 98.5% was achieved. The energy consumption was analysed. The integration of these techniques results in water that meets the requirements for drinkable water and demonstrates the technical feasibility of the process.

1. Introduction

The availability of drinking water is a global necessity [1–3]. According to a UNICEF report, 780 million people lack access to this resource and about 40% of the population cannot afford sanitation [4]. Potable water and irrigation water are among the basic needs of humans, and unfortunately expected to decline due to population growth and climate change. Meanwhile, approximately 50.5% of the population lives at a distance < 10 km from the sea. Thus, the desalination of seawater is an interesting alternative to generate potable water from an abundant resource.

Desalination can be achieved using technologies based on the principle of evaporation of water such as multiple-effect evaporation, membrane distillation, pervaporation, or solar distillation. Other technologies are membrane technologies, such as reverse osmosis [5]. Evaporation technologies have some disadvantages such as the high cost associated with the latent heat of evaporation of water. Membranes have a good yield separation but must be periodically changed due to the phenomenon of solute obstruction called ‘fouling’. An alternative that has been explored in an attempt to reduce operating costs is freeze concentration [6].

Freeze concentration is a method of removing water from a solution through the formation and separation of ice crystals of high purity [7]. Maintaining a solution at temperatures below the freezing point generates the phenomena of elution mass transfer and heat that can separate a liquid phase with a higher solute concentration relative to the solid phase; even under suitable conditions, it is possible to remove all the solutes present and have pure water [8]. In terms of water purification, the freeze concentration technique has proved to be viable for removing highly toxic metal ions like Chromium VI present in natural waters such as ocean water [9]. Although the freeze concentration process has several advantages over other techniques of concentration, there are still problems associated with the separation yield that do not yield a highly pure effluent.

Freeze concentration can be carried out by three techniques: suspension, falling film freeze concentration (FFFC), and block freeze concentration (BFC). Suspension is a technology available worldwide in the food industry [10]. Other techniques are being studied, such as block and falling film for food applications, biotechnology, and water treatment processes; these demonstrate high efficiencies compared with the suspension technique and require simple and inexpensive equipment [8], [10–14].

In falling film freeze concentration (FFFC), the solution is in contact with a cooled plate upon which the ice forms as a single layer [15].

---

[*] Corresponding author.
E-mail address: leonardo.moreno@unisabana.edu.co (F.L. Moreno).

https://doi.org/10.1016/j.desal.2018.02.015
Received 27 September 2017; Received in revised form 12 January 2018; Accepted 8 February 2018

Nomenclature

C	concentration (% w/w)
CI	concentration index (unitless)
f	mass fraction of ice or liquid (unitless)
K	average distribution coefficient (unitless)
m	mass
RE	removal efficiency (%)
COP	coefficient of performance

Subindex

0	initial
ice	ice (diluted fraction)
liq	liquid (concentrated fraction)

Superindex

F	falling film freeze concentration (FFFC)
T	fractionated thawing (FT)
B	block freeze concentration (BFC)

Flesland [16] proposed a multi-stage FFFC coupled with reverse osmosis for water desalination, which afforded efficient water elimination. More recently, the recovery of solutes from sucrose solutions retained in ice was attempted by the fractionated thawing of ice [17,18]. In the block freeze concentration technique (BFC), the solution is frozen and partially thawed to separate diluted and concentrated fractions [11]. The viability of this technique was primarily demonstrated for low solid concentrations [7]. However, there are no viable commercial processes for the application of FFFC or BFC to desalination. The future of the freeze desalination depends on the study of new hybrid systems that enables the profitable operation of falling film and block freeze concentration [6]. The aim of this work is to study the use of falling film concentration coupled with block freeze concentration for water desalination and to propose an integrated process of FFFC, fractionated thawing, and BFC to obtain desalinated water.

2. Materials and methods

2.1. Materials

The solutions were prepared from commercial grade salt (Refisal, Colombia) and distilled water at 20 °C, and stirred for 10 min at 300 rpm. The samples were refrigerated to achieve a temperature of 0 °C. The solid concentration was expressed in mass concentration (C), which is defined as the mass percentage of solute per unit mass of solution. The conductivity of the samples was measured using a portable conductivity meter, CM-135 (Crison, Spain). The relationship between conductivity and C is represented by the equation, C = 6.69 E-2* k (g/g), ($R^2 = 0.998$). The calibration curve was obtained from the solutions at 1.10, 5.17, 11.17, 16.89, 22.13, 28.43, 34.17, 39.97, 98.97, 162.60, and 231.00 mg/L, and measuring the mass fraction of salt using the method of weight loss proposed by Mandri et al. [19]. The measurements were performed in triplicate.

2.2. Methods

Two techniques of freeze concentration and one technique of solute recovery were studied following the flowchart of freeze concentration tests reported by Moreno et al. [8]. The initial solution was freeze concentrated by the falling film freeze concentration (FFFC) technique, and the resulting ice was melted in ten fractions to study the recovery of the retained solutes. Finally, the diluted fractions obtained during the thawing process were freeze concentrated by the block freeze concentration (BFC) technique in order to increase the amount of pure water. Each technique was studied individually and based on the results a global process was proposed.

2.2.1. Falling film freeze concentration tests

In each test, 800 mL of saline solutions of different concentrations (0.5, 1.5, 2.5, 3.5, 6.0, and 8.5% (w/w)) was concentrated by the falling film freeze concentration technique according to the protocol reported by Moreno et al. [20]. The experimental setup is shown in Fig. 1a). The solution flows as a falling film on a refrigerated plate (1), inside which circulates an aqueous solution of ethylene glycol at −20 °C provided from a circulated bath (Polystat, Cole Parmer, USA). The bath was temperature controlled at an interval from −35 °C to 150 °C ± 0.01 °C. The bath pumped the heat exchange fluid to the plate. The solution was collected in a tank (3) and again circulated by a peristaltic pump, VGC-400 (Seditesa, Spain), with a frequency meter (VFD007L2 Seditesa, Spain) (2) to control the speed of the pump. The saline solutions flux was fixed at $8 \times 10^{-5}\,\mathrm{m^3{*}s^{-1}}$. The ice produced (4) was collected to be later recovered fractionally. Each experiment was performed between 40 and 80 min (less time was spent at lower initial concentrations) to obtain an ice sheet between 290 and 340 g, which correspond to an ice width between 12 and 14 mm. The salinity of the concentrated solution was measured every 20 min during the experiment and at the end of the process, by a portable conductivity meter (CM-135, Crison, Spain). The experiments were carried out in triplicate at room temperature around 20 °C. The energy consumption of the cooling stage was measured by a bifilar single phase meter (@meter, Colombia).

2.2.2. Fractionated thawing tests

The thawing experiments were performed according to the method described by Gulfo et al. [17]. The plates obtained in the previous step (as the product of the seven different initial concentrations of FFFC in triplicate) were used to carry out the fractional thawing. One sample of 30% of the ice sheets was taken and thawed according to the configuration in Fig. 1b). The experimental configuration consisted of a cubic thermally insulated chamber (volume: 0.5 m^3) (2). The camera had a temperature control system (1) (Pie Electro Dit, model 11,551,

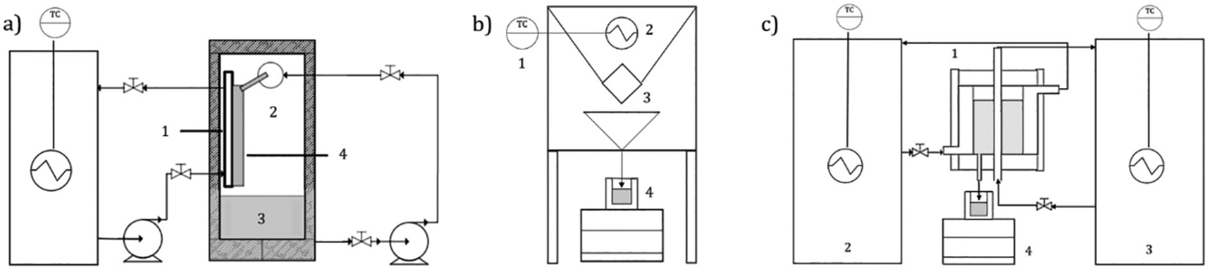


Fig. 1. Experimental setup for freeze concentration tests. (a) Falling film freeze concentration; (b) fractionated thawing and (c) block freeze concentration.