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Evaluation of lactic acid purification from fermentation broth by hybrid short path evaporation using factorial experimental design

Andrea Komesu [a,*], Patrícia Fazzio Martins [a,b], Betânia Hoss Lunelli [a], Johnatt Oliveira [a], Rubens Maciel Filho [a], Maria Regina Wolf Maciel [a]

[a] School of Chemical Engineering, University of Campinas (UNICAMP), 13083-970 Campinas, SP, Brazil
[b] Departamento de Ciências Exatas e da Terra, Universidade Federal de São Paulo (UNIFESP), 09972-270 Diadema, SP, Brazil

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ABSTRACT

This work describes the evaluation of lactic acid purification from fermentation broth by hybrid short path evaporation. The proposed hybrid purification process consists of an evaporation system composed by a cylindrical wiped film evaporator with two condensers, one located internally and other externally to the evaporator. Through factorial experimental design, the influence of operation conditions as feed flow rate, agitation, condenser and evaporator temperature on residue and distilled percentages, lactic acid purity and recovery were studied. Models were developed in order to describe the response of interest as function of operating conditions. The results showed that with a high operating pressure (in terms of short path evaporation), with a pressure of 1000 Pa, and one step of separation, lactic acid purity around 89.7% was obtained which was about 18 times lactic acid concentration higher than the initial content in raw material.

1. Introduction

An increasing interest for discovering new environment-friendly sources of chemicals has been observed due to the current concerns related to the cost and environmental impact of using traditional petrochemical processes. One important technological biomass-based platform is the biotechnological process for lactic acid production by fermentation that potentially offers several advantages: low substrate costs, production temperature and energy consumption [1].

Lactic acid has a wide variety of applications such as cosmetics, pharmaceutical products, chemistry, food and more recently in the medical area. The presence of two adjacent functional groups in the lactic acid (hydroxyl and carboxyl) in a small molecule with only three carbon atoms shows its high reactivity, as well as their tendency to decompose at high temperatures.

The development of an efficient method of lactic acid separation and purification from fermentation broth is very important, because, these steps can reach up to 50% of the total costs [1,2] and it is still difficult to recover it with high purity for the reasons: high affinity with water, decomposition at elevated temperatures and complex and energy intensive recovery technology [3]. A considerable number of methods for the recovery of lactic acid from fermentation broth, such as solvent extraction [4–7], separation with membranes [8–10], reactive distillation [11–13] and others have been studied.

Conventional molecular distillation (or short path evaporation) had been used to recovery lactic acid with purity up to 95–96% [14–16]. The operating pressure is usually below 0.1 Pa and two or more steps of refining are required. To keep the high-vacuum a mechanical force-pump and diffusion pump should be used simultaneously [14], which is in direct conflict with energy-saving. Each additional step in the downstream represents an increase in the total operating costs.

Hybrid short path evaporation [17–19] is an alternative separation process with potential for the recovery and concentration of thermally unstable molecules such as lactic acid. It has been recognized as a promising technology mainly because of its low evaporation temperature and short residence time, which minimize problems with thermal decomposition [20].

In previous work, our research group studied the technical feasibility of lactic acid concentration from synthetic mixture of water: lactic acid (36 wt% of lactic acid) using hybrid short path evaporation system [20]. The experimental results showed that carrying out the lactic acid concentration by using evaporative system is technically feasible and advantageous. Based on the

* Corresponding author. Tel.: +55 19 35213971, +55 19 983764925; fax: +55 19 35213910.
E-mail address: andrea_komesu@hotmail.com (A. Komesu).

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preliminary results with synthetic mixture, new experiments were carried out with fermentation broth. By the fact that fermentation broth is a mixture more complex than synthetic mixture, by the presence of residual sugars and other organic acids, preparation and analysis of the raw material was different as well as the performance of the separation process.

Bearing all this in mind, the objective of this paper was to evaluate the lactic acid purification from fermentation broth by hybrid short path evaporation system using factorial experimental design. It allows the determination and evaluation of the relative significance of operational parameters on the process, even in the presence of complex interactions [19]. In the studied process higher operating pressure (1000 Pa), compared to that usually employed in conventional molecular distillation, and one step of refining were used, which make this technique more suitable for lactic acid purification than the literature published works.

2. Experimental

2.1. Raw material preparation

Fermentation process was carried out in a 7 L New Brunswick Scientific BioFlo 415 bioreactor. Sugarcane molasses (48% sucrose w/w) without pretreatment, typical of large scale industrial mills (from Costa Pinto Mill, Piracicaba, Brazil) was diluted with distilled water in order to obtain an initial sucrose concentration of 32 g/L approximately. The fermentation medium was enriched with 4 g/L of yeast extract to attend the nutritional requirements of the bacterium. The *Lactobacillus plantarum inoculum*, from Fundação Tropical de Pesquisa e Tecnologia André Tosello (Campinas, Brazil), with adequate preparation [21] was added to the fermenter. The temperature was maintained at 37 °C, pH at 5.0 by adding 4 M NaOH and agitation speed at 200 rpm. A pulse of diluted molasses (32 g/L) was carried out after the sucrose had been completely consumed in order to avoid the inhibition of the cell growth by high sucrose concentration as well as to increase of lactic acid end concentration [21]. The total time of fermentation was approximately 30 h. The fermentation product containing about 5% (w/w) lactic acid was vacuum filtered and centrifuged (5000 rpm for 15 min at room temperature) to removal of *Lactobacillus*. The sediment was discarded and in the fermentation product (supernatant liquid) was added sulfuric acid to convert sodium lactate to lactic acid and used as raw material for the investigation of the evaporation system.

2.2. Chromatographic analysis

Analyses of the raw material and products were performed in an equipment of high performance liquid chromatography (HPLC), Agilent model 1260, equipped with UV detector (UV/vis) connected in series with the chromatography column Bio-Rad Aminex, model HPX-87H (300 × 7.8 mm). The equipment was controlled through OpenLab software. Sulfuric acid solution with 5 mM was used as mobile phase at flow rate of 0.6 mL/min. The column temperature was kept constant at 37 °C. In each run, an injection volume of 25 μL was used. For lactic acid detection and quantification, the wavelength of 215 nm was used in the UV detection system. The lactic acid concentrations were determined using the calibration curve (regression coefficient of 0.99987) obtained with standard solutions of DL-Lactic acid 90% supplied by Sigma–Aldrich (St Louis, Missouri, EUA). The identification of the substances peaks in the chromatogram profiles was performed by comparison of their retention times with standard substances.

2.3. Hybrid short path evaporation system for lactic acid concentration

Lactic acid was concentrated in an evaporation system composed by a short path evaporator, Model Pope 2 Wiped Film Still, manufactured by Pope Scientific Inc. (Saukville, WI, USA). An external condenser was associated to the evaporation system which was named hybrid short path evaporation. The distance between the evaporator and the internal condenser is 17 mm. The evaporator has an evaporation surface of 0.033 m^2 (or 0.35 ft^2) and it is jacketed with an electric heating system. A schematic diagram of the apparatus is shown in Fig. 1 [20]. From tests carried out, it was realized that for pressures lower than 1.33 kPa a considerable amount of volatile material moved toward the trap, hindering the equipment operation at this condition, because the separated material in the trap returned to the evaporator [18]. In order to allow the use of lower pressures, in this work an external condenser was attached to the equipment.

During the experiments, the external condenser was fixed at −5 °C. A trap was coupled to an external condenser which was continuously fed with liquid nitrogen (−196 °C), freezing and avoiding the volatiles migration to the pump and its oil contamination. The transfer of raw material (about 40 g at room temperature) to the equipment was conducted by using a peristaltic metering pump Cole Palmer Masterflex model 77200-60. The vacuum system was composed of a mechanical pump, keeping the pressure at 1 kPa. At lower pressures a considerable amount of volatile material moved toward the trap, which is undesired.

In this system, it was possible to collect 03 streams: light stream, residue and distillate as identified in Fig. 1. The substances of higher vapor pressure were collected predominantly in the light stream, while substances with intermediate vapor pressure in the distillate and substances with lower vapor pressure in the residue.

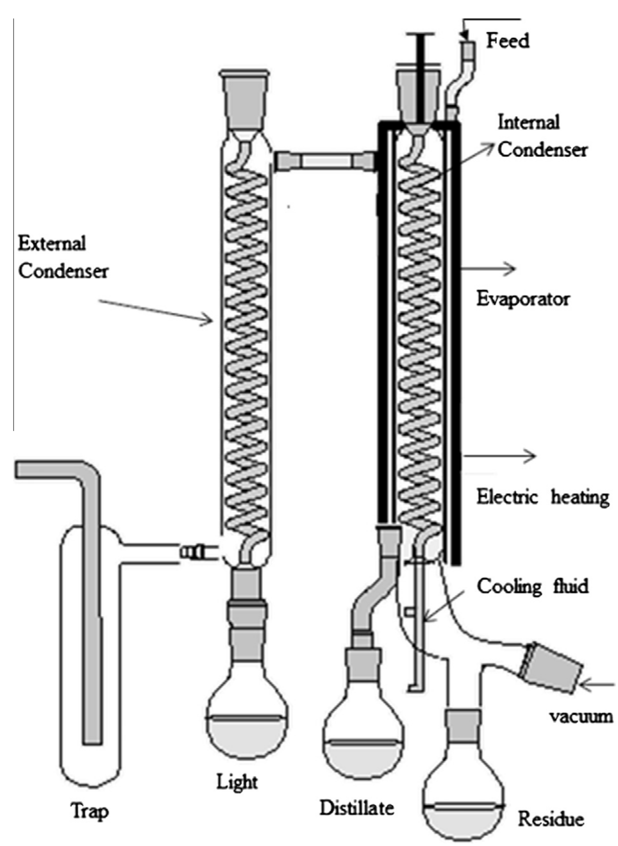

Fig. 1. Schematic diagram of evaporator [20].