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Lactic Acid Purification by Hybrid Short Path Evaporation

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In this work, the evaluation of lactic acid concentration from synthetic mixture of water: lactic acid (36 wt% of lactic acid) was carried out using hybrid short path evaporation system. The system allowed to employ lower temperatures of operation due to the use of vacuum and presented short residence time, avoiding the tendency of lactic acid decomposition. The following operating conditions were used: feed flow rate of 8 mL/min, stirring of 750 rpm, pressure of 1 kPa and the evaporator temperature varied from 30 to 170 °C. The experimental results showed that maximum lactic acid concentration was obtained using evaporator temperature at 50 °C.

1. Introduction

Currently, fossil resources are used to produce the vast majority of chemicals. However, the use of fossil resources causes serious environmental problems. Discovery of new environment-friendly sources of chemicals has captured the attention of researchers. Different building-block intermediates have been produced from biomass via biotechnological routes (Gao et al, 2011). Special attention is given to biotechnological process for lactic acid production that offers several advantages: low substrate costs, production temperature and energy consumption (Datta and Henry, 2006).

Lactic acid or 2-hydroxypropionic acid (CAS 50-21-5) was discovered in 1780 by the Swedish chemist Scheele from sour milk (Datta e Henry, 2006). It has two optically active forms: L (+) - lactic acid and D (-)-lactic acid, and a wide variety of applications: in cosmetics, in pharmaceutical products, in chemistry and in food. Furthermore, it is used as precursor of several other products, such as propylene oxide, acetaldehyde, acrylic acid, propionic acid, 2,3-pentanedione, ethyl lactate and poly-lactic acid. The presence of two adjacent functional groups in the lactic acid (acid and alcohol) in a small molecule with only three carbon atoms show its high reactivity, as well as their tendency to decompose at high temperatures (Lunelli, 2010).

The development of an efficient method of separation and purification of the lactic acid from fermentation broth is very important, since these steps are responsible for 50 % of the production costs (Wasewar et al., 2002). A considerable amount of researches have done a great deal of work on the purification procedures (Chen et al, 2012). Recently, various separation technologies have been reported, such as solvent extraction with kerosene, tributyl phosphate or hexane (Alkaya et al, 2009; Gao et al, 2009, Matsumoto et al, 2003, Malmary et al, 2000), separation with membranes such as nanofiltration and electrodialysis (Pal et al, 2009, González et al, 2008, Hábová et al, 2004), reactive distillation (Mo et al, 2011, Edreder et al 2011, Kumar et al, 2006) and others. But many drawbacks such as high equipment costs, the need for solvent recovery, high energy cost, and others, still limit the application of these technologies on industrial scale.

Hybrid short path evaporation (Martins et al, 2012a; Martins et al. 2012b) is an alternative separation process with potential for recovery and concentration of thermally unstable molecules such as lactic acid. It

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has been recognized as a potential method because of its low evaporation temperature and short residence time that minimize thermal decomposition problems.
So, in this work, the objective was to evaluate the lactic acid concentration from a synthetic mixture of water: lactic acid (36 wt% of lactic acid) using hybrid short path evaporation system.

2. Materials and Methods

2.1 Materials

Lactic acid 85 % supplied by Ecibra (São Paulo, Brazil) was used to prepare synthetic solution water: lactic acid. The DL-lactic acid 90 % standard supplied by Sigma-Aldrich (St Louis, Missouri, EUA) was used to build the calibration curve (from 1 to 60 g/L) for lactic acid quantification (R^2=0,99987).

2.2 Preparation of synthetic solution

The synthetic solution (feed solution) used in this work was prepared with commercial lactic acid 85 % diluted in distilled water under stirring. Solution with 36.14 % mass concentration of lactic acid was prepared.

2.3 Hybrid short path evaporation system

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) associated with an external condenser. Because of the addition of the external condenser, the system was named hybrid short path evaporation. Table 1 describes the characteristics of the evaporation system and the schematic diagram of the apparatus given in Figure 1.

Table 1: Characteristics of the evaporation system

Evaporator type	Condenser	Agitation	D* (mm)	Evaporation surface (m^2)	Heating system
Hybrid short path	External and internal	Yes	17	0.033	Electric

D* = Distance between evaporator-internal condenser (mm)

2.4 Operating conditions of lactic acid concentration

Table 2 describes the operating conditions used in the experiments. In this evaporation system, a trap is coupled to an external condenser that is continuously fed with liquid nitrogen (-196 °C), freezing and avoiding the volatiles migrate to the pump and contaminate the oil. The transfer of raw material (about 40 g of synthetic solution at room temperature) to the equipment was conducted by using a peristaltic metering pump (Cole Palmer Masterflex model 77200-60) and a vacuum system, which is composed by a mechanical pump.
In this system, it was possible to collect 03 streams: light, residue and distillate as shown in Figure 1. The substances of higher vapour pressure were collected predominantly in the light stream, while substances with intermediate vapour pressure in the distillate and substances with lower vapour pressure in the residue. The streams (light, residue and distillate) were collected in glass flasks and analysed by liquid chromatography to determine lactic acid concentration.

Table 2: Operational conditions

Evaporator temperature (°C)	Internal condenser temperature (°C)	External condenser temperature (°C)	Flow rate (mL/min)	Agitation (rpm)	Pressure (kPa)
30-170	10	-5	8	750	1

2.5 Chromatographic analysis

The concentration of lactic acid was 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 x 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 obtained with standard solutions of DL-Lactic acid 90 %.

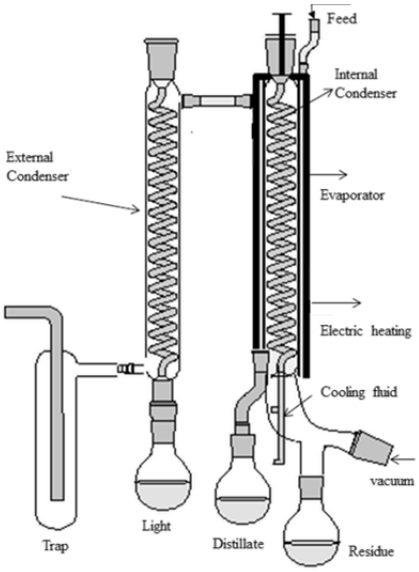

Figure 1: Schematic diagram of evaporator (Komesu et al., 2012).

3. Results and discussions

3.1 Distillate, residue and light streams percentage evaluation

Figure 2 shows the experimental mass percentage of distillate (% D), residue (% R) and light (% Light) streams at various the evaporator temperature (Tevap). The mass percentage of distillate, residue or light was calculated according to equation 1.

$$Percentage\,(\%) = \frac{m_{residue,\ destillate\ or\ light}}{m_{total}} \times 100 \quad (1)$$

in which $m_{residue,\ distillate\ or\ light}$ is the amount of material collected in the residue, distillate or light streams (g) and m_{total} is the sum of residue, distillate and light mass after evaporation (g).

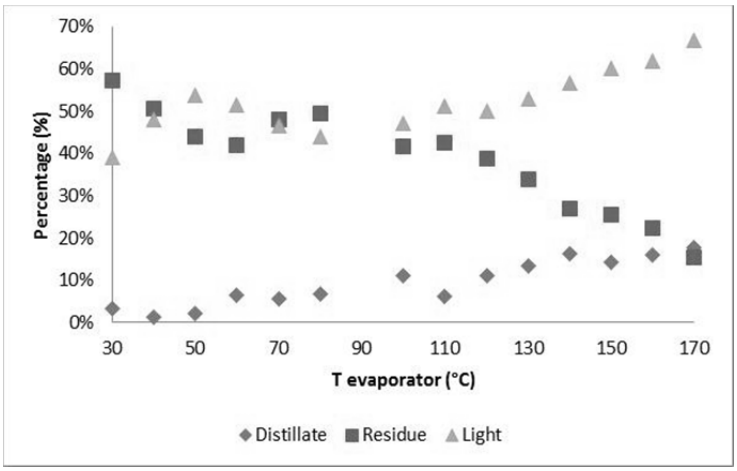

Figure 2: Percentage of distillate, residue and light as function of evaporator temperature (T evaporator).

The amount of residues decreased with increase in temperature because increasing of the temperature promotes the evaporation of a larger amount of material, therefore the percentage of light and distillate increased.

3.2 Lactic acid mass concentration evaluation

Figure 3 shows the mass concentration of lactic acid in distillate, light and residues streams as a function of the evaporator temperature.

At temperatures from 30 to 60 °C, lactic acid concentration in the residue stream increased due to water evaporation. At temperatures higher than 70 °C its concentration were kept approximately constant because of its evaporation for distillate and light streams.

The maximum concentration obtained at residue was 88.3 % (931.66 g/L) at 50 °C. The initial feed concentration was 36.2 % (330.86 g/L). The concentration process was responsible by producing a solution 2.4 times more concentrated.

From 40 °C it was observed that lactic acid tends to migrate to distillate stream, and from 110 °C lactic acid concentration in the distillate was approximately the same found in the residue stream.

The maximum lactic acid concentration (82.18 % (780.89 g/L)) was obtained in distillate at 170 °C, concentrating the solution in 2.27 times.

The lactic acid concentration in light stream increased with higher temperatures, since it promoted the evaporation of lactic acid to other streams. The higher concentration of lactic acid in light was 11.8 % (92.48 g/L) at 160 °C.

The results obtained from these experiments are promising because with one step of evaporation and low temperature (50 °C) was possible to achieve a lactic acid concentration close to the concentration of commercial lactic acid standard 85 %.

Under operating conditions studied in this work, it is more interesting to get lactic acid in the residue stream than in the distillate stream because of using lower evaporator temperatures (50 °C) to reach approximately the same concentration level, saving the energy to heat the evaporator and avoiding thermal decomposition of lactic acid.

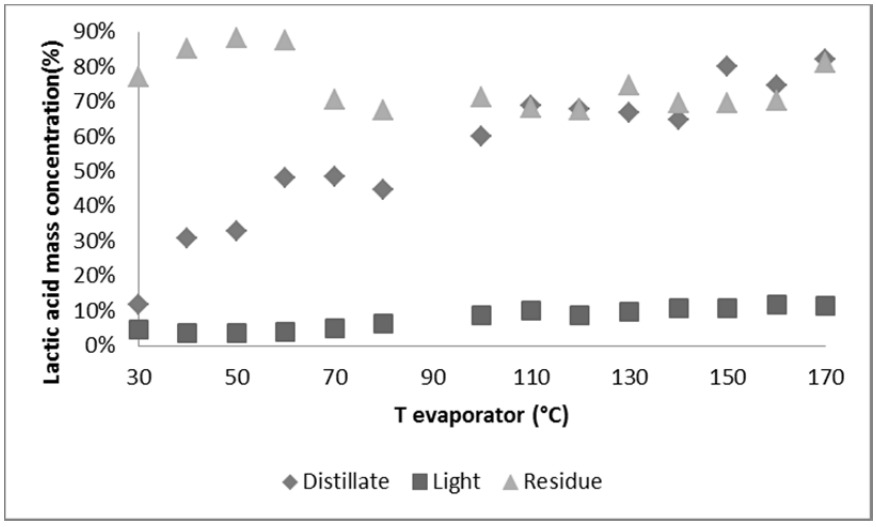

Figure 3: Mass concentration of lactic acid in distillate, light and residue as a function of evaporator temperature (T evaporator).

3.3 Lactic acid recovery analysis

Besides concentration, another important process parameter to evaluate the separation process is the lactic acid recovery. Lactic acid recovery in residue, distillate and light streams varying the evaporator temperatures is defined by equation 2:

$$\mathrm{Re}\,c(\%) = \frac{m_i \times \%AL_i}{\sum_{i=1}^{3} m_i \times \%AL_i} \times 100 \quad (2)$$

In which i is the index for residue or distillate or light, m is the residue, distillate or light mass (g) and %AL is the fraction of lactic acid in residue, distillate and light.

Although similar lactic acid concentrations were obtained in residue stream at 50 °C (88.3 %) and distillate stream at 170 °C (82.2 %), recovery values were quite different. Lactic acid recovery in residue was approximately 94 % and in the case of distillate, it was about 40 %, testifying that the best option to concentrate lactic acid under the studied operating conditions is to use lower temperatures to concentrate lactic acid in the residue stream. In addition to higher recovery obtained in residue stream, the temperature used was lower than one used in more concentrated distillate stream, which is beneficial from viewpoint of energy consumption in evaporation.
One strategy to align high recovery and increase the lactic acid concentration would work with milder temperatures and make multiple evaporations in the residue stream.

4. Conclusions

The influence of evaporator temperature in the purification process of lactic acid from a synthetic mixture water: lactic acid was studied in this work. The evaporator temperature was varied from 30 to 170 °C keeping the other operating parameters fixed (feed flow rate of 8 mL/min, stirring of 750 rpm and pressure of 1 kPa).
The experimental results showed that it is possible to concentrate the lactic acid using the evaporation system proposed in this work. Lactic acid was concentrated in residue stream in about 2.4 times with a recovery of 94 % at low temperature (50 °C).
Therefore, carrying out the lactic acid concentration by using hybrid short path evaporation system is technically feasible and advantageous because the use of low temperature helps to avoid lactic acid decomposition and it contributes to save energy evaporator.

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