Journal of the Taiwan Institute of Chemical Engineers 000 (2017) 1–9

Contents lists available at ScienceDirect

Journal of the Taiwan Institute of Chemical Engineers

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

Optimizing ethanol recovery in a spinning cone column

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ARTICLE INFO

Article history:
Received 28 June 2017
Revised 27 October 2017
Accepted 24 November 2017
Available online xxx

Keywords:
Wine dealcoholization
Beer dealcoholization
Distillation
Optimization
Modeling

ABSTRACT

Spinning cone column (SCC) is a separation technology used in the food industry to recover natural aromas. It outperforms plate and packed columns given its enhanced mass transfer and capability to process slurries. Limited SCC design and operating guidelines are available, since theoretical studies characterized the SCC hydrodynamics with single components, while experimental studies with complex mixtures do not consider process modeling and optimization. Distillation of a water–ethanol mixture of 14.8% (v/v) in a lab scale SCC was optimized, varying the stripping rate, feed rate and temperature. A first principles model was developed to explain the results in terms of internal variables. Three operating regions were identified. At low stripping rates, the operation was unstable and low performing. At high stripping rates, ethanol recovery and concentration decreased due to a sharp increase in the heat loss that overweighed the mass transfer enhancement. To maximize recoveries, SCC should operate at intermediate-high stripping rates, high feed and intermediate temperatures. A high-performance region was found with ethanol recoveries higher than 94% and ethanol concentrations in the distillate over 70%. Our proposed guidelines improve the competitiveness of wine and beer dealcoholization using SCC technology, increasing the yield and marketability of both dealcoholized product and surplus distillate.

1. Introduction

Spinning cone columns (SCC) are direct contact mass transfer devices in the category of thin film liquid evaporators. SCC technology has been widely used in the food industry for the recovery of tea and coffee aromas, dealcoholization of wine and beer, and the extraction of essential oils [1]. The high gas/liquid contact area available in SCC makes them very efficient. In several applications, SCC performs better than plate and packed distillation columns. Their low residence times, small liquid hold-ups as well as their capability to handle viscous materials, solutions with suspended solids, and slurries make them a superior technology [2]. Due to the presence of rotational parts and the complex hydrodynamics occurring inside SCC, the theoretical work has been focused on understanding the fluid dynamics in lab scale SCC operating with one component.

The first theoretical work of SCC was the development of a hydrodynamic model for a one stage column [3]. Later, the same group extended their work developing models for pressure drop, flooding limits and mass transfer inside the column [4,5]. In their analysis, these authors used experimental data from a steam/water system operating at 100 °C and 500 rpm. These hydrodynamic models were validated using Computational Fluid Dynamics (CFD) simulations of a one stage experimental column, using water as working fluid. More recently, Symons developed a model of a conical centrifuge that incorporated a detailed description of boundary conditions [6] and measured the film thickness on a single spinning cone [7], finding good agreement between experimental results and model predictions.

There are few experimental studies dealing with the impact of operating variables on the performance of multistage SCC processing complex mixtures. The effect of solute dilution and rotation speed in rosemary essential oil isolation in a SCC was explored [8] concluding that the highest extraction yield was achieved at low dilution (1/40) and moderate rotation speed (570 rpm). SCC vacuum distillation is widely used as an ethanol removal process in wine and beer dealcoholization. Catarino and Mendes [9] studied a process where beer was first dearomatized by pervaporation, and then dealcoholized in SCC, concluding that SCC distillation under vacuum operation (50 mbar) was an effective process to remove ethanol from beer. Belisario-Sánchez et al. [10] explored the impact of the inlet wine flow rate and aromatic extraction percentage (the ratio of the distillate product over the amount of fed wine) of the aroma recovery in dealcoholization of wines. To obtain high ethanol concentrations in the aromatic fraction, the authors found that the raw wine flow rate should be high, and the aromatic extraction percentage presented a maximum at 1%.

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https://doi.org/10.1016/j.jtice.2017.11.030
1876-1070/

The complexity of the natural mixtures used in these studies limited the experimental exploration to a small set of operating variables within a relatively narrow range of values. For the isolation of essential oil [8], a 2^2 design with two replications was used. On the other hand, for wine dealcoholization [10], a less standard design was used to evaluate 9, 4 and 7 different combinations of aromatic extraction percentages and raw wine flow rates for red, rose and white wine, respectively. In addition, no first-principles models were used to analyze the experimental data. Therefore, limited conclusions and operating guidelines for SCC were drawn.

SCC is especially effective to dealcoholize alcoholic beverages given its high productivity and its possibility to produce zero alcohol beers [11]. In addition, the distillate obtained from wine and beer dealcoholization is a valuable by-product that can be used to produce aromatic spirits [12]. The integrated process of SCC dealcoholization with the valorization of the distillate has a lower environmental impact and consumption of natural resources than reverse osmosis or evaporative pertraction [13].

There are few operating guidelines to optimize performance of SCC applications in the wine and beer industry [14]. In this work, the operation of a multistage lab scale SCC was optimized to distill a binary ethanol–water mixture (14.8% v/v) which simulates a wine with high ethanol content. This kind of wines could be dealcoholized partially or totally, to increase consumer's preference or produce dealcoholized wines, respectively. In this work, we defined three objectives: (i) reduce the ethanol concentration in the bottom below the legal threshold to ensure the marketability of the dealcoholized wine (1.2% in the EU); (ii) maximize ethanol recovery to increase the benefit of selling the distillate by-product and (iii) keep ethanol concentration in the distillate above 70% to guarantee its marketability.

Since this is a simple mixture, many experiments can be performed reproducibly, shedding light on how to improve SCC operations. The operating variables studied were liquid mixture inlet flow rate (L), stripping rate (S) and liquid mixture temperature (T), while the observed variables were (i) ethanol recovery, (ii) ethanol concentration in the distillate and (iii) ethanol concentration in the bottom product.

The response surface methodology (RSM) was applied to find the optimal operating conditions that maximize ethanol recovery, constrained to a given minimum ethanol concentration in the distillate and a given maximum ethanol concentration in the bottom. Empirical models applied in the process optimization were statistically analyzed to establish parameters significance. To explain experimental results in terms of internal variables, an additional model, based on mass and energy balances, was developed and fitted to experimental data. Operating guidelines for high performance operations were developed.

2. Materials and methods

2.1. SCC system

A lab scale multistage spinning cone column (SCC), designed and built in our lab, was used in the experiments. Each stage consists of a static cone fixed to the body of the column and a rotatory cone fixed to a rotatory shaft. The column is made of stainless steel, comprises 37 stages and is 1.41 m tall with a diameter of 180 mm. The apex angles of both the static and rotatory cones are 47°. Between the 20th and 21st stages, the column has a flow diffusor. The rotation speed was fixed at 297 rpm. SCC design details can be found in supplementary material S2.

The liquid mixture is fed from the top of the column and the stripping gas from the bottom. In each stage, the liquid enters through the fixed cone and descends by gravity towards the rotary cone. At this point, the liquid ascends by centrifugal force until it leaves the rotary cone towards the next stage. In the ascending and descending zones, the liquid forms a thin film whose thickness and velocity depends on the liquid flow rate and the rotating speed of the shaft. In addition, the liquid films disintegrate into droplets at the inlet and outlet zones of the cones. The disintegration generates a dispersed liquid phase with a high surface area that exceeds the film area by an order of magnitude [5].

2.2. Experimental design

The 14.8% v/v ethanol–water mixture was prepared from ethanol 95% (v/v) (OXIQUIM, Chile) and distilled water in 35 L plastic vessels. The prepared mixture was poured into an open 35 L stainless steel tank, and heated indirectly by a water heated bath. The mixture was stirred at 300 rpm with a flat blade turbine stirrer of 1 kW power (SIEMENS, D91056, Czech Republic). A peristaltic pump with a 15 mm silicon hose (Masterflex®, XX 80 002 30, USA) was used to impulse the mixture and control the flowrate. The liquid flow rate was measured just before each experiment, collecting the liquid feed for 30 s and then weighing it with a scale (Jadever, JWE-30K, Taiwan). The steam source was an electric boiler of 9 kg/h capacity (RENE LEON, steam iron 366, Chile) fed with distilled water by a centrifugal pump (Pedrollo, CP 132, Italy). Steam input was controlled by a manual valve in each experiment. A preparation time of 30 min was required to get saturated steam in the boiler and to achieve thermal equilibrium between the ethanol–water mixture and the water in the heated bath. The environmental temperature was kept at 28 °C for all experiments.

After preparation, the SCC and peristaltic pump were turned on, and the steam valve opened at the corresponding conditions for each experiment. Each run lasted 32 min, preceded by 20 min of transient operation until the system reached steady state. The temperature of the mixture was measured with an electronic thermometer (Hanna Instruments, HI 98,501-1, Italy) at the beginning, the middle and at the end of the experiment; the average of the three measurements was recorded and its variation was within ± 0.5 °C. The liquid flow rate was measured just before each experiment, collecting the liquid feed for 30 s and then weighing it with a scale (Jadever, JWE-30K, Taiwan). The steam flow rate was measured using a scale (Jadever, JWE-30K, Taiwan) to weigh the water in the reservoir of the boiler before and after each experimental run. The distillate was condensed in a tube and shell condenser using tap water as cooling agent. Then, the distillate was collected in two 1 L decanters and a 2 L glass bottle. The bottom product was collected in a 60 L plastic vessel. Distillate and bottom product masses were measured with a scale (Jadever, JWE-30K, Taiwan) at the end of each run and divided by the experimental time to get the respective mass flow rates.

At the end of each experiment, 25 mL samples of the fed mixture, distillate and bottom product were taken. Then, the samples were cooled to 20 °C in a water bath. Sample density was measured using a 10-mL pycnometer (Witeg, 3900 010, Germany). Each measurement consisted in drying the pycnometer, filling it with the sample and finally weighing it using a semi-analytical balance (Boeco, BAS31, Germany). Three measurements were performed for each sample and its average was reported. Fig. 1 displays a diagram of the experimental setting. Alcoholic strength was calculated from density and temperature measurements using a spreadsheet programmed with the International Alcoholometry Tables [15].

SCC ethanol-water distillation was assessed through ethanol recovery and ethanol concentration (% v/v) in the distillate (C^D_{EtOH}) and in the bottom product (C^B_{EtOH}). Ethanol recovery was defined as the total mass of ethanol in the distillate over total mass of ethanol fed:

$$Re_{EtOH} = \frac{D \cdot w_D}{L \cdot w_L} \quad (1)$$

Please cite this article as: F. Huerta-Pérez, J.R. Pérez-Correa, Optimizing ethanol recovery in a spinning cone column, Journal of the Taiwan Institute of Chemical Engineers (2017), https://doi.org/10.1016/j.jtice.2017.11.030