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Experimental study of the production of high purity ethanol using a semi-continuous extractive batch dividing wall distillation column

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ABSTRACT

In this paper, the production of high purity ethanol using an experimental dividing wall distillation column using glycerol as entrainer is studied considering batch and semi-continuous operations. In the semi-continuous distillation, an ethanol-water mixture of composition 92% wt. ethanol was introduced in the pot and the glycerol was supplied through a continuous feed near to the top of the distillation column, it was possible to produce a distillate with a composition higher than 99% wt. ethanol. This result is important since it is possible to use this ethanol mixed with gasoline in the current motor vehicles operated with gasoline.

1. Introduction

Despite the process used to obtain bioethanol, a diluted stream of bioethanol is obtained (around 10% wt. of ethanol) and fed to a process separation in order to recover the bioethanol [1]. Several methods have been used to obtain high purity bioethanol, usually a first conventional distillation column is used to remove the water as bottoms product and the distillate contains most of the bioethanol [2]. This first distillation column plays an important role since as the distillate reaches the azeotrope composition of ethanol-water the energy required in the reboiler increases exponentially, thus a negative net energy gain can be obtained in the cycle of production of bioethanol. For that reason, the composition reached in this distillation column is really important as explained in the work of Kiss et al. [1]. In order to obtain high purity bioethanol the preferred industrial method is the extractive distillation using several entrainers; for instance, ethylene glycol, glycerol, ionic liquids and others [3]. From an economic point of view, the glycerol is the cheapest and is obtained as a side product in the production of biodiesel.

This extractive distillation process can be improved by using a dividing wall distillation (DWDC) that can save both energy and capital costs [4]. The DWDC is thermodynamically equivalent to the Petlyuk distillation column when no heat transfer occurs through the wall (Fig. 1). Both complex distillation columns can be used for the separation of a ternary mixture (ABC), where component A is obtained as top product, component B as side stream and component C as bottoms products.

It has been proven the effective use of DWDCs in the purification of bioethanol [5], reactive distillation to produce biodiesel and esterification reactions [6], but most of the studies have been carried out using process simulators, so that the main contribution of this paper is in the experimental production of high purity bioethanol using a DWDC and glycerol as entrainer since the use of ethylene glycol can be forbidden in the near future due to its toxicity [7]. It is important to highlight that in those applications, authors have reported energy savings of around 30% in contrast to the conventional distillation sequences. For that reason, we are interested in using DWDCs in the purification of bioethanol using glycerol, since distillation and dehydration can represent up to 37% of the total energy involved in the cycle of production of bioethanol [2]. Then, the reduction of the energy required in the purification stage is an important opportunity to improve the whole process of production of bioethanol.

2. Details of the experimental DWDC

We have designed, implemented and operated a DWDC that consists of three packed sections of Teflon™ Raschig rings, the sections are numbered from the pot to the condenser (see Fig. 2),

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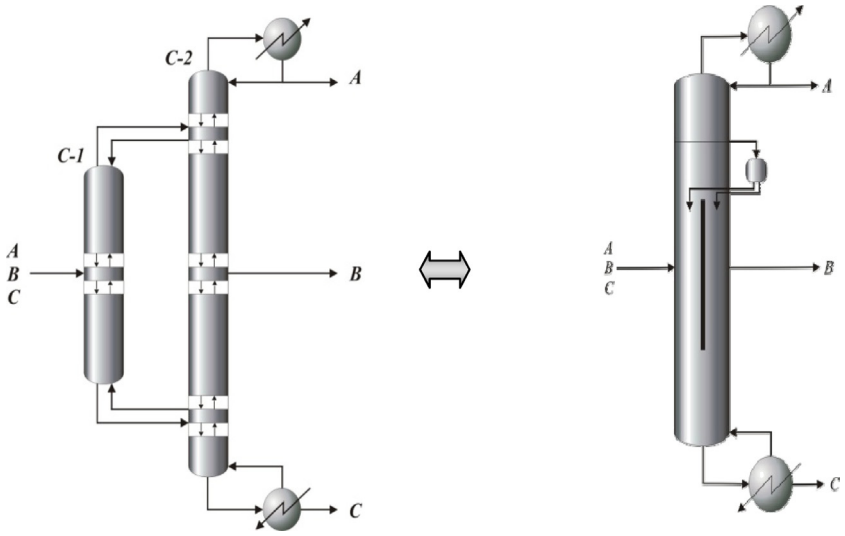

Fig. 1. Petlyuk distillation column and dividing wall distillation column (DWDC).

and six thermocouples are distributed along the column [8,9]. The DWDC has an internal diameter of 17 cm and a total packed height of 2.5 m. Details of the DWDC can be found in the previous published papers [8,9], but it is important to highlight that was implemented using stainless steel 316 L because originally was used to carried out esterification reactions using sulfuric acid as catalyst. In the bottoms part of the distillation column an immersed heating coil supplies the energy required to achieve the separation. In the top of the distillation column, a total condenser is used to provide the reflux and the distillate product. This condenser is a double heating coil operated by cooling water. The design of the DWDC was conducted in two stages, in the first one, total stages and recycle stages were assigned for the two distillation columns of the Petlyuk system (thermodynamically equivalent to the DWDC), and in the second stage the interconnecting streams were varied until the minimum energy consumption was obtained in the reboiler according to the method detailed in Hernández and Jiménez [10].

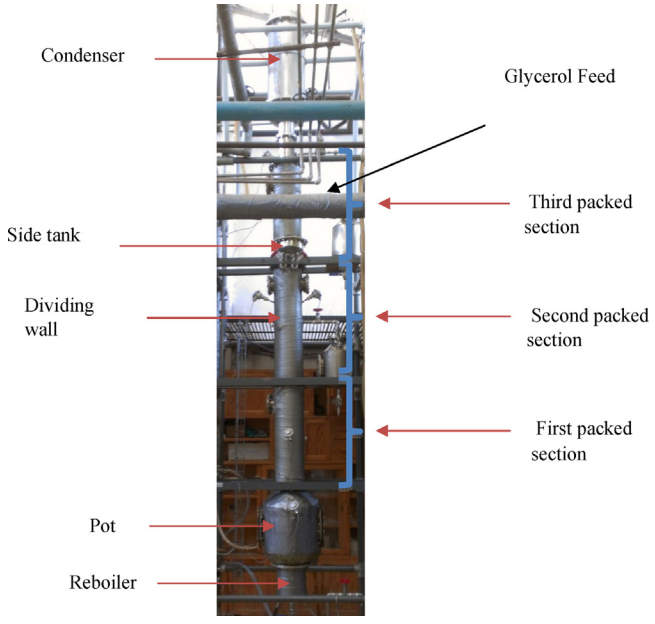

Fig. 2. Experimental DWDC used for the production of high purity bioethanol.

The key part of the DWDC is the middle section, where a dividing wall is implemented in order to have two separated sections in the same shell. As mentioned above, the DWDC contains three packed sections, each section is equipped in the top with a liquid distributor tray and in the bottoms part a support tray is required.

During the operation, the liquid leaving the third section is collected in a side tank that is used to split the liquid to both sides of the wall of the middle section. This middle section is the key part of the DWDC, and the dividing wall permits to have two sections equivalent to prefractionator and main section of the Petlyuk distillation column (Fig. 1).

Regarding the dividing wall, it is important to mention that the minimum energy required in the DWDC depends on the flows of liquid and vapor to both sides of the dividing wall, as a result, this side thank is used to manipulated the liquid flows required to operate close to the optimal conditions.

The vapor that leaves the first packed section continues to the second section and is distributed freely to both sides of the wall of the middle section. During the operation, it is much more difficult to manipulate the vapor split than the liquid split.

Finally, in the third packed section a continuous feed of glycerol can be introduced in order to break the azeotrope of ethanol-water. It is well known that the mass separation agent must be supplied near to the top of the distillation column when the azeotrope presents a minimum boiling point, just like the ethanol-water system.

3. Experimental procedure

The first class of experiments was carried out in batch mode according to the following procedure:

1. An initial liquid mixture of ethanol-water-glycerol was introduced into the pot of the DWDC. For instance, for the first experiment 11225.57 gr of the model mixture of ethanol-water (92% wt. of ethanol) was charged into the pot with the same amount of anhydrous glycerol (JT Baker).
2. After the mixture of ethanol-water-glycerol was poured into the pot of the RDWDC, the hot utility (steam water) in the reboiler is activated in order to start the stabilization of the distillation column.
3. The experimental temperature profile was measured using six thermocouples and registered on a laptop using SIMATIC WinCC of SIEMENS.