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Design alternatives and control performance in the pilot scale production of isoamyl acetate via reactive distillation

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ABSTRACT

In the present work, two different reactive distillation (RD) designs for the pilot scale production of isoamyl acetate are studied by means of steady state simulations. In the first design near stoichiometric amounts of acetic acid and isoamyl alcohol are fed directly to the reactive and rectifying sections of the RD column. On the second design, acetic acid in excess and isoamyl alcohol are first fed to a continuously stirred tank reactor (CSTR) and its product stream is fed to the reactive section of the column. Among different optimization variables, the overhead vapor flowrate appears to be the most influencing variable in the energy requirements, and its value and composition are responsible for the two liquid phase formation in the condensate. This variable also determines the global performance of the process schemes yielding a >99% conversion and >99% isoamyl acetate mass purity of the bottoms product. Having a CSTR upstream to the RD column allows an operation with a lower overhead vapor flowrate, thus a lower energy consumption is achieved for this design.

Indirect composition control of the bottoms product in both designs has been implemented by controlling the temperature of one of the reactive stages. For the design comprising a CSTR, a better dynamic response and process stabilization has been observed for a change in the production rate. This control strategy also showed a good disturbance rejection in the dynamic behavior of the main process variables.

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1. Introduction

Esters production have always been of major interest for applications in reactive distillation (RD) processes due to the substantially lower energy requirements and capital cost saving that it can represent. The evolution of the Eastman Kodak process for methyl acetate production is the preferred example. Formerly, methyl acetate was produced by means of a chemical reactor and a series of conventional and extractive distillations to overcome the separation difficulties of the different azeotropes of the system, resulting in a plant of up to ten pieces of equipment (Schoemakers and Bessling, 2003a). Nowadays it is produced feeding almost stoichiometric amounts of reactants to a single RD column using an acidic catalyst (Agreda and Partin, 1984). For the

esters formed by different alcohols and acids rather than methanol and acetic acid (i.e. methyl formate, isopentyl acetate), RD has extensively been considered as a more sustainable, energy and cost efficient process (Luyben and Cheg-Ching, 2008; Yang et al., 1998; Zhang et al., 2013).

The RD process scheme in the production of esters may vary significantly depending on the reactive system and its mixture properties (Luyben and Cheg-Ching, 2008). In this sense, the feasibility of RD for a specific process and its design can be outlined by analyzing primarily the reaction rate relative to the residence time needed in distillation processes and the relative volatility of the components involved (Schoemakers and Bessling, 2003b). Nowadays process syntheses including RD are carried out with the aid of specialized software that uses a heuristic-numeric approach letting the process engineer

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rapidly identify between feasible and attractive designs from non-feasible options (Harmsen, 2007). Nonetheless, control performance of different designs is not easily analyzed by these methods.

The control structure of a RD process is of major importance and its study for a particular case may determine whether a design is economically and dynamically practical. In a process that integrates reaction and separation, multiple input and output steady states may occur and can sometimes be influenced in a lesser extent on the process design rather than on the separation phenomena occurring on the reactive system (Kienele and Marquardt, 2003). As a consequence, sustained oscillations in key process variables and steady state shifting caused by disturbances are examples of drawbacks that can occur if a good control strategy is not engineered for a RD process (Reder et al., 1999). The best way to achieve this and to obtain reliable control designs is by using dynamic process simulators.

The most common applications of isoamyl acetate are found in the food and cosmetic industries for its characteristic banana fragrance and in the chemical industry mainly as a high boiling solvent. This ester is produced by the esterification of acetic acid with isoamyl alcohol. The latter is mainly obtained as a by-product of the ethanol industry and constitutes up to 60% of its weight. This high boiling cut from the ethanol distillation is commonly known as Fusel Oil and its production yield from the whole process can be as high as 0.1% (Kosaric et al., 2001). Very efficient methods for the purification and production of this alcohol have been proposed earlier and are focused mainly on the atmospheric distillation and extraction of the fusel oil (Ferreira et al., 2013).

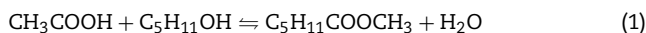
Acetic acid and isoamyl alcohol esterification is a reversible acid catalyzed reaction. When heterogeneously catalyzed by solid acid catalysts, minor equipment corrosion and almost absolute selectivity and ease of separation can be achieved in comparison with homogeneous catalysis. For these reasons, its production is a suitable case of application of RD (Osorio-Viana et al., 2013a).

The aim of this paper is to compare on the one hand the steady state performance, and on the other hand, the dynamics and controllability of two different process arrangements for the production of isoamyl acetate by means of RD at a pilot plant scale.

2. Reactive distillation process

2.1. Reaction kinetics

The esterification of acetic acid and isoamyl alcohol follows the elementary reaction,



Activity based pseudo homogeneous kinetic model using ion-exchange resin Amberlyst 35 has been previously reported (Bastidas-Jiménez and Rodriguez, 2016). The rate of reaction can be described by the following expression,

$$r = k_0 * \exp\left(-\frac{E_a}{RT}\right) \left(a_A a_B - \frac{a_C a_D}{K_{Eq}}\right) \quad (2)$$

where r is the mixture molar based rate of reaction, a_i is the activity of component i in the mixture, k_0 is the pre-exponential factor and E_a is the activation energy as follows:

$$E_a = 6.20 \times 10^4 \frac{\text{J}}{\text{mol}} \quad (3)$$

$$k_0 = 8.88 \times 10^5 \frac{\text{kmol}_i}{\text{kmol}_{\text{mix}} * \text{s}} \quad (4)$$

The term K_{Eq} refers to the equilibrium constant and is expressed as:

$$K_{Eq} = 2.94 \times 10^2 \exp\left(-\frac{552.18}{T(\text{K})}\right) \quad (5)$$

2.2. Thermodynamic model of phase equilibrium

For this esterification system, both reactants, isoamyl alcohol and acetic acid are intermediate boiling components, whereas both products, isoamyl acetate and water, are high and low boiling point components respectively. At atmospheric pressure, the boiling points of the reactive system species are:

Acetate	Alcohol	Acid	Water
142.2 °C	131.1 °C	117.9 °C	100.0 °C

The characterization of the Vapor-Liquid-Liquid Equilibrium (VLLE) of the system has been presented previously by Osorio-Viana et al. (2013b) reporting new sets for the NRTL-HOC activity parameters. They also reported the presence of four different azeotropes including three heterogeneous. Even more, residue curve maps for the quaternary system were simulated, yielding isoamyl acetate as the only stable node.

Due to the non-idealities of the system and the dimerization of carboxylic species in the vapor phase, the most appropriate thermodynamic model is the NRTL-HOC. On another work, Bastidas-Jiménez and Rodriguez (2016) regressed binary experimental VLLE data in order to complement available NRTL-HOC data present in the Aspen Plus data base. This information is shown in Table 1.

Residue curve maps of all the ternary sub-systems at 1 atm of absolute pressure are presented in Fig. 1. As it can be seen, 6 azeotropes are present in the system, of which four are binary and two are ternary. It is also worth to notice that all azeotropes involving water are heterogeneous. This is in good agreement with the previously reported information about the heterogeneous azeotropic behavior of the system (Osorio-Viana et al., 2013b; Cho et al., 1984; Cepeda, 2010).

Fig. 1C shows the VLLE behavior of the acetate-alcohol-water sub-system. In the case of a RD column attaining high acid conversion, the composition of the vapor distillate could be close to the azeotropic boundary of high water composition. Thus, a liquid-liquid separation

Table 1 – Binary NRTL parameters reported by Bastidas-Jiménez and Rodriguez (2016).

Component i Component j	Alcohol Water	Acetate Water	Alcohol Acetate	Acid Alcohol	Acid Acetate	Acid Water
Aij	2.7511	0	0	0	3.2596	-1.9763
Aji	4.1788	0	0	0	-6.9036	3.3293
Bij	-11.6046	511.8316	700.2695	542.4083	-997.4399	609.8886
Bji	-255.8964	2082.929	-379.4592	-340.6272	3085.1691	-723.8881
α	0.4186	0.2	0.3	0.3	1.5864	0.3
Source	Experimental regression	Aspen LLE	Experimental regression	UNIQUAQ predictive	Experimental regression	ELV-HOC

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