12TH INTERNATIONAL SYMPOSIUM ON PROCESS SYSTEMS ENGINEERING AND 25TH EUROPEAN SYMPOSIUM ON COMPUTER AIDED PROCESS ENGINEERING

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Amsterdam – Boston – Heidelberg – London – New York – Oxford
Paris – San Diego – San Francisco – Singapore – Sydney – Tokyo

Elsevier
Radarweg 29, PO Box 211, 1000 AE Amsterdam, The Netherlands
The Boulevard, Langford Lane, Kidlington, Oxford OX5 1B, UK

British Library Cataloguing in Publication Data
A catalogue record for this book is available from the British Library

Library of Congress Cataloging-in-Publication Data
A catalog record for this book is available from the Library of Congress

ISBN (Part B): 978-0-444-63577-8
ISBN (Set): 978-0-444-63429-0
ISSN: 1570-7946

Printed and bound in Great Britain

14 15 16 17 10 9 8 7 6 5 4 3 2 1

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Contributed Papers
T-3: Cyber-Infrastructure, Informatics and Intelligent Systems

Contributed Papers
T-5: Process Dynamics, Control and Monitoring

Krist V. Gernaey, Jakob K. Huusom and Rafiqul Gani (Eds.), 12th International Symposium on Process Systems Engineering and 25th European Symposium on Computer Aided Process Engineering.
31 May – 4 June 2015, Copenhagen, Denmark

Optimization of a Fusel Oil Separation System Using a Dividing Wall Column

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Abstract

Fusel oil is a valuable byproduct of the fuel ethanol industry that can be used to isolate C6 alcohols and other fragrances and flavor ingredients. In this work we propose two distillation schemes for obtaining isoamyl alcohol from fusel oil: a conventional distillation sequence and a dividing wall column. After validation of thermodynamic models to accurately describe phase equilibria of fusel components (VLE and LLE), a dividing wall column (DWC) separation scheme was developed. Both traditional sequential distillation and DWC system were optimized using differential evolution and tabu list coupled to ASPEN PLUS. Results indicate that significant energy and economic savings can be obtained in the purification of fusel oil (a non ideal mixture) using a DWC scheme compared to conventional configuration.

Keywords: Optimization; fusel; dividing wall column

1. Introduction

Through the years, the production of ethanol by fermentation has increased due to the biofuels demand, and consequently generation of by-products of this industry such as fusel oil has increased as well. Fusel has been characterized as a liquid waste and used as fuel in the boilers within distilleries. It is a mixture obtained as a side cut during ethanol distillation, mainly composed of i-amyl alcohol, water, *iso*butanol, ethanol and other short chain alcohols (C3-C5). These alcohols can be considered valuable raw material for various products of industrial interest such as: biosolventes, extractants, flavors, fragrances, pharmaceuticals and plasticizers, among others. According to different characterizations, major components of fusel oil are isoamyl alcohol, water and ethanol, accounting for ~98% of the total mixture. In this context, two distillation schemes to purify isoamyl alcohol and ethanol were developed. Such schemes were constructed taking into account continuous distillation sequences currently used in ethanol distilleries. A traditional scheme (Figure 1) is comprised of three distillation columns where most ethanol and water are separated in the top of the first column. This stream is sent to a subsequent column where azeotropic ethanol can be extracted on the top and water at the bottoms product. Bottoms product from first column is sent to a third distillation column connected to a decanter, where isoamyl alcohol is obtained as a bottoms pure product.

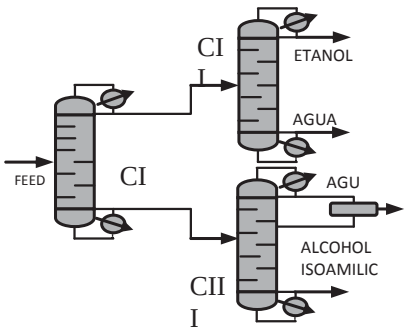

Figure 1. Conventional distillation system for fusel purification.

Dividing wall column has been used in the purification of ternary systems with significantly savings in energy consumption and capital costs over conventional simple column configurations (Hernández et al., 1996). Specifically, the dividing wall column (DWC) has been successfully implemented in many industrial separations (Olujic et al., 2003). In the dividing wall distillation (DWC) scheme proposed in this work (Figure 2), azeotropic ethanol is obtained as the top product of the main column, while isoamyl alcohol is removed from the bottoms product. Water is removed using a side stream connected to a decanter taking advantage of the liquid phase immiscibility

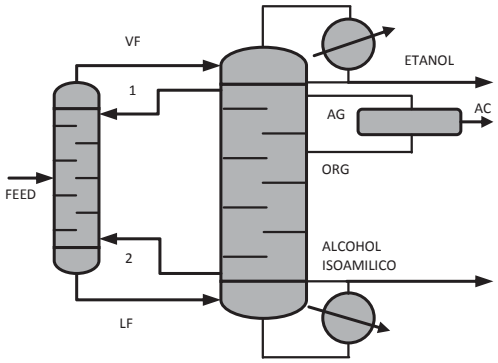

Figure 2. DWC system for fusel purification.

2. Case study

Composition of fusel oil was established according to the analysis performed to six different samples from different sugar mills in Colombia. Samples were characterized by gas chromatography coupled to mass spectrometry. The following components were detected: ethanol, water, n-butanol, isobutanol, isoamyl alcohol and 2-ethylhexanol. The composition of the mixture of study is presented in Table1.

Table 1 Composition of the mixture of study.

T= 25ºC P = 1 atm	
Component	composition % mol
Etanol	0.1258
Water	0.143
isoamyl alcohol	0.7047
n-butanol	0.0139
isobutanol	0.012599
2-ethyl-hexanol	1x10-6

Thermodynamic of phase equilibria was validated by regression of binary and ternary equilibrium literature reports using a NRTL-HOC activity-based model. To obtain the corresponding thermodynamic parameters, NIST equilibrium data incorporated within ASPEN PLUS were used. Using the regression tool included in the same software, VL and LL data were simultaneously fit to accurately model the phase behavior within the distillation system

3. Optimization problem

During optimization of distillation schemes, total annual cost (TAC) minimization was used as objective function. This function takes into account energy consumption, utilities, and column size. The minimization of this objective is subjected to the required recoveries and purities of each product stream, i.e.:

$$\text{Min (TAC)} = f\left(N_{tn}, N_{fn}, R_{rn}, F_{rn}, D_{cn}\right) \quad (1)$$

$$\text{Constrain: } \vec{y}_m \geq \vec{x}_m$$

where Ntn and Nfn are the total number of stages and the feed stages of column respectively, Rrn is the reflux ratio, Frn is the distillate flow, Dcn is the column diameter, ym and xm are the obtained and required purities vectors of the m components, respectively. This minimization implies the manipulation of 14 continuous and discrete variables for each route process, where 5 variables are used for the design of each column. Note that since the product streams flows are manipulated, the recoveries of the key components in each product stream must be included as a restriction for the optimization problem. During each optimization, a Pareto front was generated, plotting the objective function (total annual cost) against the flow of impurities. Impurities flow in each product stream is the sum of the components which are not present in high purity in out flowrate. A list of manipulated variables for both schemes are listed in Table 2.

Table 2 Manipulated variables during process optimization

Manipulated variable	Conventional scheme			DWC	
	CI	CII	CIII	PREF	PETLYUK
number of stages C_n, N_{Cn}	x	X	x	x	x
feed stage in column C_n, $N_{F,Cn}$	x	X	x	x	x
distillate streamflow of column C_n, R_{Cn}	x	X			x
reflux ratio in column C_n	x	X	x		
bottoms streamflow of column C_n			x		x
feed stage of liquid interconnection flow FL in column C_i, N_{FL},C_i					x
feed stage of liquid interconnection flow FV in column C_i, N_{FL},C_i					x
feed stage of liquid interconnection flow FL in column C_i, N_{FL},C_i					x
vapor interconnection flow, FV					x
liquid interconnection flow, FL					x

4. Results

In this section, we present the set of optimal designs in Pareto front, for both schemes: conventional (Figure 3) and divided wall column (Figure 4).

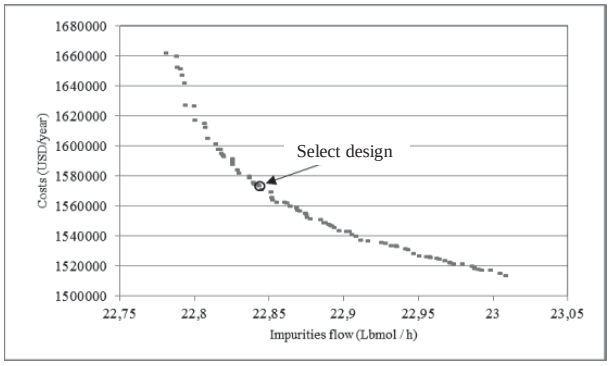

Figure 3. Pareto front of the conventional scheme.

In the conventional configuration, in column CI the number of stages and reflux ratio determine the value of the objective function (TAC) as it is where is performed the separation of water from alcohols including ethanol which forms a minimum temperature azeotrope. Overall, this column represents over 80% of energy consumption scheme. In column CII there is a close relationship between energy consumption, the purity of ethanol and the reflux ratio, due to the higher reflux, there is greater demand for energy and it results in greater purity of ethanol. This was seen as a high purity of ethanol increases energy consumption by 12%.In column CIII was observed the higher flowrate in the bottom stream, the reboiler duty is increased.

Figure 3 shows the Pareto front obtained after optimization, it is clear that both objective functions evaluated are in conflict each other. The Pareto front is formed for several points that accomplish all purity and recovery restriction (84 %, 70 % and 99 % mol). In a general way, the Pareto front consists in three sections, one section where are located designs which have preferably a high number of stages and a high reflux ratio. On the other hand at the end of Pareto front are located some sequences which showed a minor economic impact due to these designs include relative few stages. At the middle of the Pareto front is located a feasible zone, where are designs with suitable values of TAC and impurities flows comparing extremes of plot.

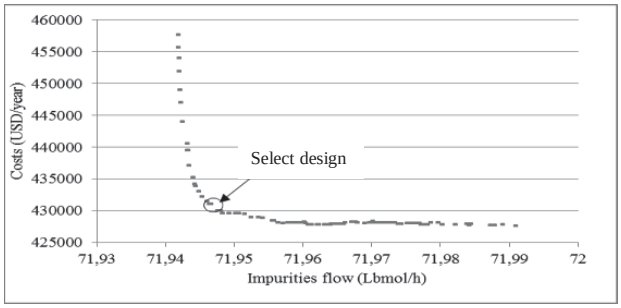

Figure 4. Pareto front Petlyuk scheme.

For the divided wall column, a clear relationship is showed between the reboiler duty and TAC, and high reflux ratio presents a high reboiler duty. Besides, when divided wall column (Figure 4) was evaluated with the same objective functions, and the same restrictions purity of the components of interest, from the Pareto front we appreciate a section with high cost associated to high reflux ratio to achieve a low impurities flow.

Table 3. Design specifications of proposed schemes.

Design specifications						
Equipment		Pref	Petlyuk	CI	CII	CIII
Stages		10	26	16	21	7
Feed stage		8	22	11	15	5
Distillate flow (Lbmol/hr)			13	20	8	---
Reflux ratio			6	6	6	---
Bottom flow (Lbmol/hr)			---	---	---	5
Feed stream	FV		17			
	FL		24			
	ORG		22			
	ETHANOL		1			
	ISOAMYL ALCOHOL		26			
Feed products	AC		22			
	1		16			
	2		25			
Flow interconnection (Lbmol/hr)	AC		60			
	1		12			
	2		95			
Composition	Ethanol		0.84		0.84	
	Water		0.71		0.72	
	Isoamyl		0.99			0.99
Energy consuption (Btu/h)		8´130,642.04		10´214,657.14		
Costs (USD/year)		430,231.31		1´573,677.43		

Characteristics of the design highlighted in Figure 4 are presented in Table 3. Notice that this design includes few stages considering 50 stages as limit. Nevertheless all purity restrictions were accomplished. As well, Table 3 shows general characteristic of design highlighted in Figure 4 such as stages, reflux ratios and interconnection flows values. This design satisfied in the same manner all restrictions. When comparing both schemes, savings of 73% in the total annual costs (TAC) and a 20% saving in energy consumption are obtained. In order to know purity values through stages in divided wall column, Figure 5 shows a concentration profile, which confirms the high isoamyl alcohol and ethanol purities obtained in this column. This behavior of divided wall column projecting itself as a good alternative in fusel purification due to energy and costs savings.

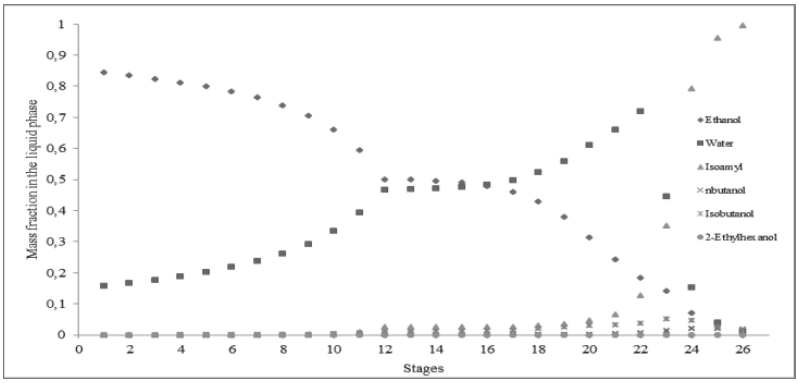

Figure 5. Composition profile for the DWC scheme.

5. Conclusions

In this work, an optimization procedure, based in differential evolution and tabu list coupled to ASPEN PLUS, was proposed for the optimization of complex distillation columns. The results show that divided wall column represents a good option for fusel oil distillation due to the benefits that would be obtained in terms of reduction in total

annual cost (TAC) over the traditional distillation sequence used in industry. The separation of isoamyl alcohol at a high purity and low cost using a dividing wall column represents an excellent option for purification of fusel oil in industry. These results represent a new vision in regard to highly nonideal mixture, where heterogeneous azeotropes are present in multicomponent systems and they are purified in a DWC.

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