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Effect of ionic liquids as entrainers on the dynamic behavior of ethanol-water extractive columns

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Abstract

Ionic liquids (ILs) have been recently considered as potential entrainers for extractive distillation. The use of ILs may affect the vapor-liquid properties to aid the separation of azeotropic mixtures. In particular, their effectiveness has been observed for ethanol dehydration, showing promising perspectives for their industrial implementation. However, there is still a lack of information about the effect of ILs on the system controllability. The objective of this work is to explore the dynamic implications of the use of two types of ionic liquids on the ethanol dehydration process. An equimolar feed mixture of ethanol and water was considered, and different IL concentrations were tested. The results show that changing the IL concentration affect the degree of stabilization of the product stream, even when smooth dynamic responses were in many cases observed.

Keywords: Distillation, Extractive distillation, Ethanol dehydration, Ionic liquids, Process control

Introduction

The ethanol dehydration process has recently received renewed attention because of the sudden increase of bioethanol production as an alternative source of energy. The environmental advantages of bioethanol have been evaluated by different performance indexes such as life cycle analysis, net energy produced, greenhouse gas emissions and agricultural impact [1–3]. Relevant factors such as net energy value (fuel energy minus energy used for production) and carbon footprint depend on two main parameters, namely the nature of the feedstock and the production process. Therefore, in order to make bioethanol a feasible alternative, the optimization of process design and operation variables becomes particularly important.

Traditional extractive distillation systems have been studied for ethanol purification. Hoch and Espinosa [4] presented an evolutionary optimization procedure based on a superstructure and its formulation as a mixed integer nonlinear programming problem. They considered the use of extractive distillation with ethylene glycol, followed by pervaporation membranes for the final ethanol dehydration. Feitosa de Figueiredo et al. [5] presented a systematic procedure for the optimal design of an extractive distillation system for ethanol dehydration with ethylene glycol as solvent, including a sensitivity analysis to analyze the influence of process parameters on the separation task. Kiss and Suszwalak [6] studied the feasibility of new distillation technologies for enhanced bioethanol dehydration by using divided wall columns for extractive and azeotropic distillation, considering ethylene glycol and n-pentane as entrainers. Roth et al. [7] considered hybrid processes for ethanol dehydration, in which they evaluated the effect of four membrane-assisted configurations, based on a preconcentration step by distillation followed by a dehydration process with membranes. These types of works show that distillation (either as an isolated process or combined with new technologies) continues to be the first choice for ethanol dehydration, even when it is an energy-intensive operation.

Another relevant variable for the design of ethanol dehydration systems is the selection of an entrainer that provides a suitable separation. Solvent feasibility can be evaluated by mean of residue curves maps, distillation lines or by relative volatility at infinite dilution. The

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solvent selection should facilitate the desired separation and minimize energy consumption [8, 9]. For instance, Ravagnani et al. [10] evaluated the solvent selection for the production of anhydrous ethanol considering ethylene glycol and tetraethylene glycol as entrainers; their results showed that using tetraethylene glycol may provide an effective separation, but the design showed a higher energy consumption than that required with the use of ethylene glycol.

In recent years, the use of new compounds known as ionic liquids (ILs) has been considered for ethanol dewatering. Several authors have reported that such solvents promote significant changes on the vapor-liquid equilibrium (VLE) properties of the ethanol-water system, improving the ethanol dehydration process with larger separation factors at low ethanol concentrations [11–13]. Some design methodologies have been suggested for extractive distillation columns for ethanol dehydration using ILs as entrainers, such as those proposed by Chavez-Islas et al. [14], Roughton et al. [15], Ramírez-Corona et al. [16] and Zhu et al. [17] Some experimental works have also evaluated the feasibility of using such solvents at a pilot plant scale that show promising perspectives for their application [18–20]. ILs have also been analyzed for the separation of other mixtures, such as methyl acetate-methanol [21] and those that need desulfurization tasks [22]. However, although the use of ILs seems like a feasible technology for the separation of ethanol-water mixtures, there is still a lack of information about their effects on the system controllability.

Distillation control has been the subject of numerous works in the literature; a useful summary on rules for the control of conventional distillation columns can be found in Skogestad [23]. In the case of extractive distillation, an additional variable arises with the addition of a solvent. In this case, solvent to feed flow ratios are typically considered for the implementation of control strategies [24]. In the last decade, some authors have explored the simultaneous optimization of process and control design, showing how this approach can contribute to improve both economic and operational goals [25–28]. The dynamic performance of more complex distillation systems for ethanol recovery has also been studied. Mauricio-Iglesias et al. [29] evaluated the influence of heat integration in the ethanol-water distillation process, showing that the energy savings are achieved at the expense of a deterioration in the process controllability. Ramírez-Marquez et al. [30] analyzed the dynamic behavior of alternative separation processes for ethanol dehydration by extractive distillation, considering different intensified arrangements based on thermally coupled distillation systems. They also analyzed the use of two different solvents, glycerol and ethylene glycol, and their results suggested that solvent selection affects the optimal choice of control structures for such complex columns.

Most of the control studies for extractive distillation have been carried out with the selection of traditional solvents for the separation, without considering the effect of the solvent on the system controllability. In order to highlight the importance of this factor, Luyben [24] explored the influence of three different solvents for the acetone/methanol separation, showing that although all systems were controllable, the product quality depended on the solvent selection, and that the best dynamic behavior was observed for the solvent with the most favorable VLE properties.

As for the use of ILs for ethanol dehydration, we showed in a previous work that the IL concentration has a direct effect on the design parameters for extractive distillation systems [16]. Additionally, the effectiveness of the IL depends not only on its concentration, but also on the type of anion, type and length of cation, and the composition of the mixture to be separated, in this work the ethanol-water mixture. For instance, for ILs based on Imidazolium cations, larger separation factors have been observed at low ethanol concentration [13]. Regarding the effect of the cation length on the VLE, the smaller the IL chain the stronger IL-water interactions, although such an effect is reduced at low ethanol concentrations [13]. It is clear that if the use of different IL concentrations affect the design at steady state, they may also impact the dynamic performance of the process.

The aim of this work is to evaluate the dynamic implications of using ionic liquids as entrainers for ethanol dehydration via extractive distillation. Two ionic liquids with different chain lengths are considered, and the effect of feed composition and solvent concentration on the dynamic performance of the system is analyzed.

Ionic liquids selection and control strategies

The two ionic liquids considered in this work for the separation of ethanol-water mixtures offer different azeotrope-breaking capabilities. Their chemical structures contain the anion Cl^- with different chain lengths in the organic cation. These ionic liquids are 1-methylimidazolium chloride ([mim]Cl), and 1-butyl-3-methylimidazolium chloride ([bmim]Cl). An equimolar ethanol-water feed mixture with a flowrate of 1000 kmol/h is taken as a case study.

The separation was studied considering four different ionic liquid concentrations (X_{IL}) of 0.10, 0.15, 0.20 and 0.30. X_{IL} is defined as the molar fraction of ionic liquid in the solvent feed stage. Under the assumption of constant molar flows, the composition can be given by,

$$X_{IL} = \frac{IL}{IL + RD} \quad (1)$$

where IL is the ionic liquid flowrate, R the reflux ratio and D is the distillate flowrate. Figure 1 shows the effect of each ionic liquid under low concentrations of 0.1 on the vapor-liquid equilibrium curve. One can observe how the separation is aided by the displacement of the equilibrium curve beyond the azeotrope under these IL feed conditions; as shown in Ramírez Corona et al. (2015), higher concentrations of the ionic liquids further enhance the separation away from the azeotrope. The design approach used in this work was based on the one reported by Ramírez-Corona et al. [16].

One of the main advantages of using ionic liquids as entrainers is that they remain in the homogeneous liquid phase and yield lower energy consumption. However, although low concentrations of ILs may be sufficient to break the azeotrope, their high viscosity may affect the operation of the distillation column. As reported by Pereiro et al. [13], the viscosity of a solution containing ILs is a critical factor for extractive columns, and should be maintained below 100 mPa s in order to make the separation feasible. Viscosity values of several ionic liquids are available in the IL Thermo database of NIST [31]. The viscosity of pure 1-methylimidazolium chloride at 351 K is 93 mPa s, while that for 1-butyl-3-methylimidazolium chloride is about 150 mPa s. It is important to highlight that ILs viscosities decrease significantly with temperature and depend highly on their composition in the water-ethanol solution; for instance, a solution of 1-butyl-3-methylimidazolium chloride (the larger cation considered here) in water, with a molar fraction of $X_{IL} = 0.29$ (the larger concentration considered here) at standard conditions has a viscosity of 1.03 mPa s, well below the limits recommended for the operation of extractive columns.

In order to initialize steady state parameters, each case was simulated with the Aspen Plus™ process simulator. Since ILs are not included in the process simulator, their properties were implemented based on their molecular structure. The pure component properties were estimated by group contribution methods, and the NRTL model was used for equilibrium calculations [16]. The binary interaction parameters for the NRTL equation were taken from Shen et al. [12] for 1-methylimidazolium chloride and from Geng et al. [11] for the 1-butyl-3-methylimidazolium chloride.

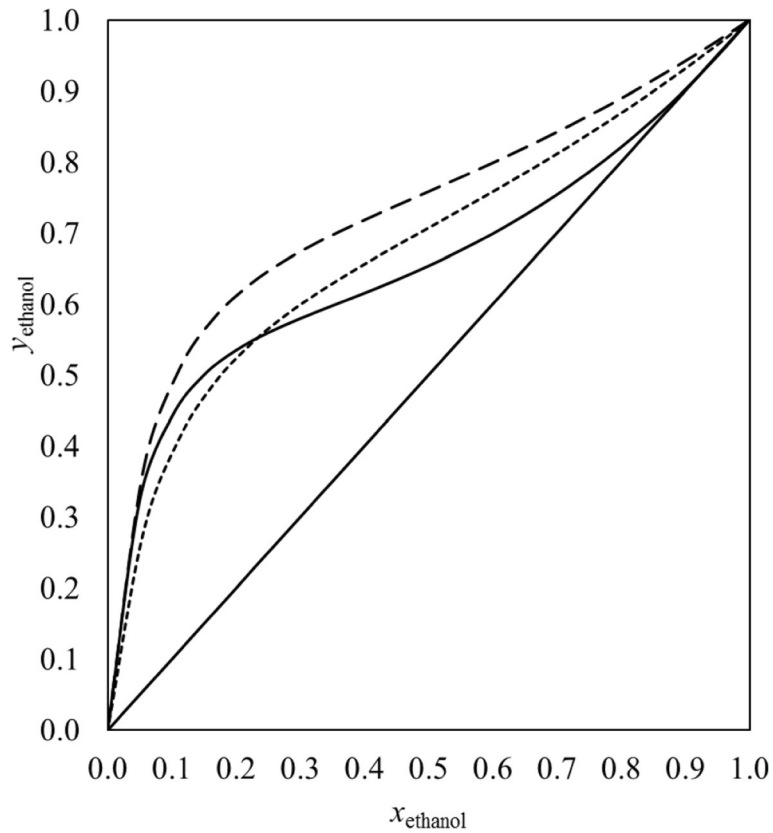

Fig. 1. Effect of ionic liquid at X_{IL} =0.1 on VLE of ethanol-water mixture, solvent free basis. Ethanol-Water ——— , Ethanol-Water-[mim]Cl – – – – – , Ethanol-Water-[bmim]Cl ·····

A sensitivity analysis was carried out first to detect the best location of the IL feed and the operating conditions needed to provide a 0.995 purity of ethanol in the distillate product. The results for each ionic liquid concentration are presented in Table 1. Column diameter and liquid holdups in the accumulator vessels were sized so as to provide a 10-min residence time. All simulations were then exported to Aspen Plus Dynamics™ and the inventory controllers were selected according to an LV standard configuration, where drums levels are controlled by products flowrates and the column pressure by the condenser heat removal.

A set of open-loop tests were first conducted for an initial exploration of the dynamic effect of key operational variables on the response of the column, particularly on the composition of the ethanol top product. Positive and negative disturbances of 5% in reflux flowrate, IL flowrate and reboiler heat load were conducted, and changes from the design steady for ethanol mole fraction of 0.995 were recorded. Figure 2 shows the responses for the two extremes of the IL composition considered here, 0.1 and 0.3. One can observe that when the ionic liquid was more concentrated, the effect of changing the reflux rate was more noticeable than the effect of changing the IL flowrate. On the other hand, for low IL concentrations, the effect of changes in the reboiler heat duty was more significant, particularly for positive disturbances. The shape of the response curves in most cases resembles that of a first-order system, which provides a good expectation in terms of the control behavior of the extractive column.

Closed-loop results

The control structure for the closed-loop tests was implemented by regulating the IL to feed ratio, for which single temperature controllers for indirect composition control were used, with the reboiler heat load as manipulated variable; the tray for temperature control was located following the slope criterion [23, 32]. The resulting control structure is shown in Fig. 3. Since the objective of this work is not centered on the control structure but rather on the effect of IL concentration on the dynamic behavior of the system, simple PI controllers were implemented for all control loops, with the same values for the controller parameters for both systems (see Table 2). The performance of each arrangement was evaluated against process disturbances in feed flowrate and feed composition.

The first set of closed loop responses was obtained under regulatory tests by applying disturbances of + 10% in the feed flowrate. Figure 4 shows the responses of the extractive distillation system against a positive feed flowrate disturbance for each ionic liquid and for each IL concentration. When 1-methylimidazolium chloride ([mim]Cl) was tested as entrainer, the separation showed similar behavior under any of the IL concentrations considered here, with a good disturbance rejection and fairly short settling times. The separation system achieved the temperature set points in less than 30 min, with a higher product purity than that specified by design. Lower IL concentrations provided a slightly higher degree of over-purification.

When 1-butyl-3-methylimidazolium chloride ([bmim]Cl) was considered, the dynamic responses showed larger fluctuations, such that the system was unable to keep the desired purity under high IL concentrations, reaching a new steady state with an ethanol purity below 0.98. The best dynamic behavior was observed under the use of low IL concentrations; in those cases, the system showed a good disturbance rejection, even providing a slight degree of over-purification when the lowest IL concentration of 0.1 was used.

Figure 5 shows the dynamic responses that were obtained when negative disturbances in the feed flowrate were considered. When [mim]Cl was used as entrainer, the distillate composition stabilized at a slightly lower value than the one of the initial steady state for low IL concentrations. The under-purification became less noticeable as the IL concentration increased, with the best response provided by the highest IL concentration of 0.3, in which case the response was smooth, keeping the same purity as specified by design.

The dynamic behavior of the separation system was again more sensitive when [bmim]Cl was analyzed. The ethanol purity was also favored at higher IL concentrations; the under-purification observed for the dilute IL

Table 1 Summary of design variables and temperature set points

IL mole fraction (X_{IL})	Design Variables											
	1-methylimidazolium chloride ([mim]Cl)						1-butyl-3-methylimidazolium chloride ([bmim]Cl)					
	Theoretical stages	Feed stage location	IL flowrate (kmol/h)	Reflux ratio	Selected tray for TC	Temperature set point (K)	Theoretical stages	Feed stage location	IL flowrate (kmol/h)	Reflux ratio	Selected tray for TC	Temperature set point (K)
0.10	23	19	116.0	2.187	22	360.88	29	25	170.12	3.30	28	376.51
0.15	26	16	108.6	1.289	25	361.50	21	17	184.31	2.25	20	371.55
0.20	24	14	115.1	0.964	23	361.94	23	19	161.84	1.38	22	370.29
0.30	20	10	166.0	0.811	19	363.24	26	22	204.18	0.97	25	373.80

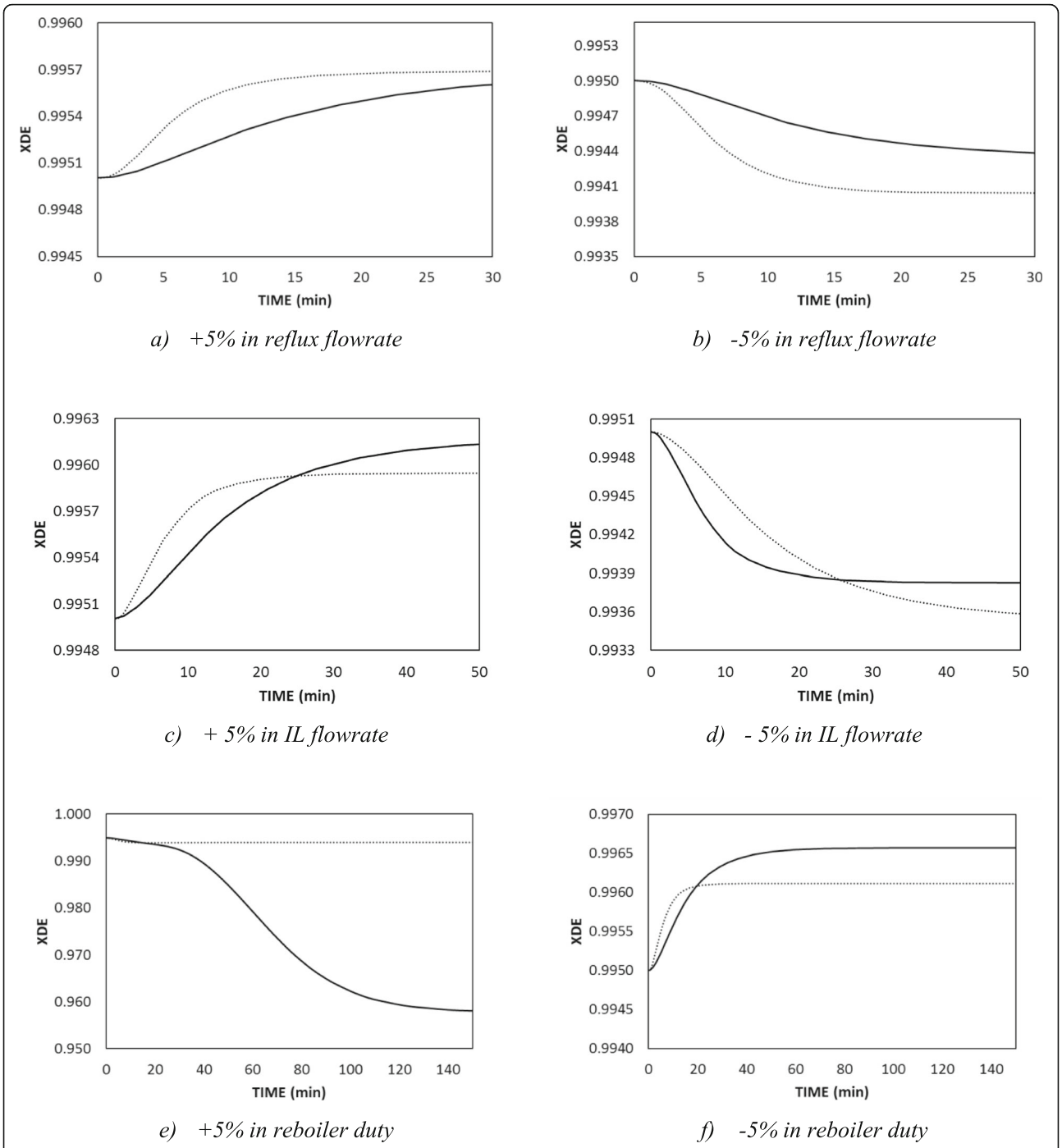

Fig. 2. Open loop responses against changes in reflux flowrate, ionic liquid flowrate and reboiler heat duty for [mim]Cl ionic liquid. [— X_{IL}=0.10, ····X_{IL}=0.3]

cases was overcome to the point that even a higher purity than the target design value was obtained when the highest concentration of 0.3 was tested.

Disturbances in ethanol feed concentration were then analyzed. Figure 6 reports the responses obtained when a + 10% change in ethanol concentration was implemented. When [mim]Cl was tested as entrainer, the dynamic responses showed a steady behavior, with no apparent dependence on IL concentration. Therefore, low IL concentrations could be considered in these cases for implementation.

When the separation was based on [bmim]Cl as entrainer, the disturbance in feed concentration caused some deterioration in the product quality, with the most

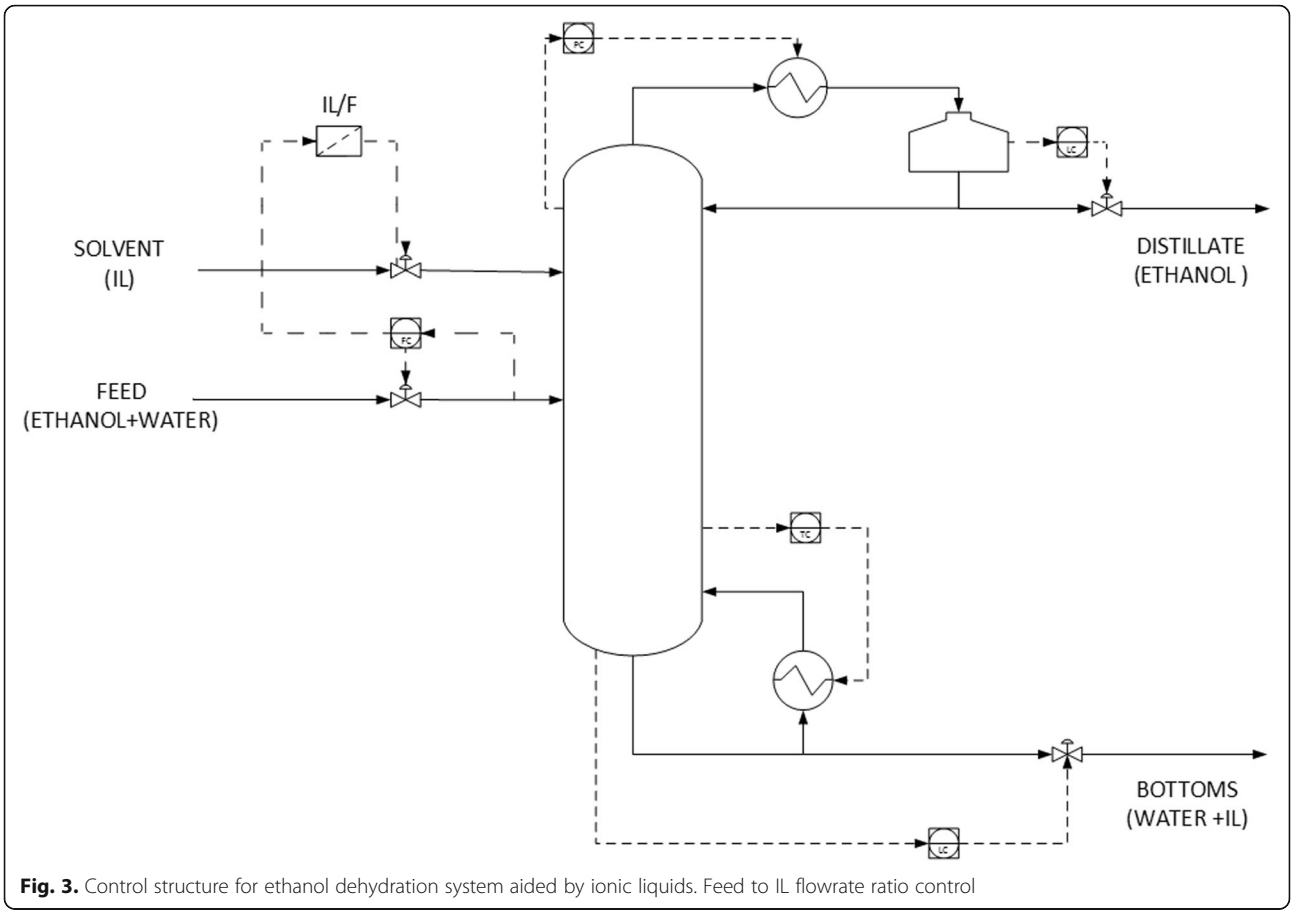

Fig. 3. Control structure for ethanol dehydration system aided by ionic liquids. Feed to IL flowrate ratio control

significant degree of under-purification obtained with the highest IL concentration.

The responses under negative disturbances in ethanol feed concentration are shown in Fig. 7. The distillate stream for the ethanol-water separation with [mim]Cl as entrainer was practically unaffected, while the use of [bmim]Cl produced a slightly more sensitive behavior, causing some degree of over-purification that became more noticeable as the IL concentration was higher.

The results of these tests showed that most of the responses were rather smooth, with typical settling times between 50 and 100 min. There were no cases in which overshoots, undershoots or inverse responses were observed.

Implementation of a double-ratio controller

An important feature during the implementation of the ratio controller used in the previous section between feed and distillate flowrates is that reflux ratios vary during the operation (since the distillate rate is manipulated to control the level of the condenser vessel). Although IL flowrate was ratioed to the feed stream rate, the IL concentration depends directly on the internal flowrates,

Table 2 Controller parameters

Controller	Controller variable	Input variable	Output variable	Controller parameters		
				K_c (%/%)	τ_i min	Controller action
C_PC	Column pressure	Column pressure	Condenser duty	20	12	Reverse
CD_LC	Reflux drum level	Reflux drum level	Valve position (distillate flowrate)	10	60	Direct
CB_LC	Base level	Sump level	Valve position (bottoms flowrate)	10	60	Direct
C_TC	Temperature tray	Temperature tray	Reboiler duty	1	20	Reverse

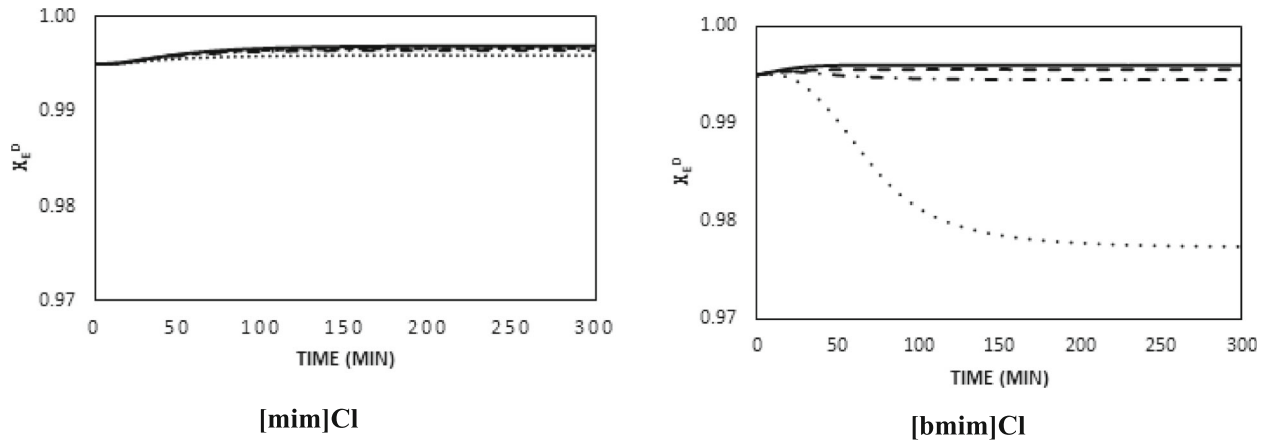

Fig. 4. Dynamic performance for positive disturbances (+10%) in feed flowrate, [— X_{IL}=0.10, — —X_{IL}=0.15, —·— X_{IL}=0.2, ···· X_{IL}=0.3]

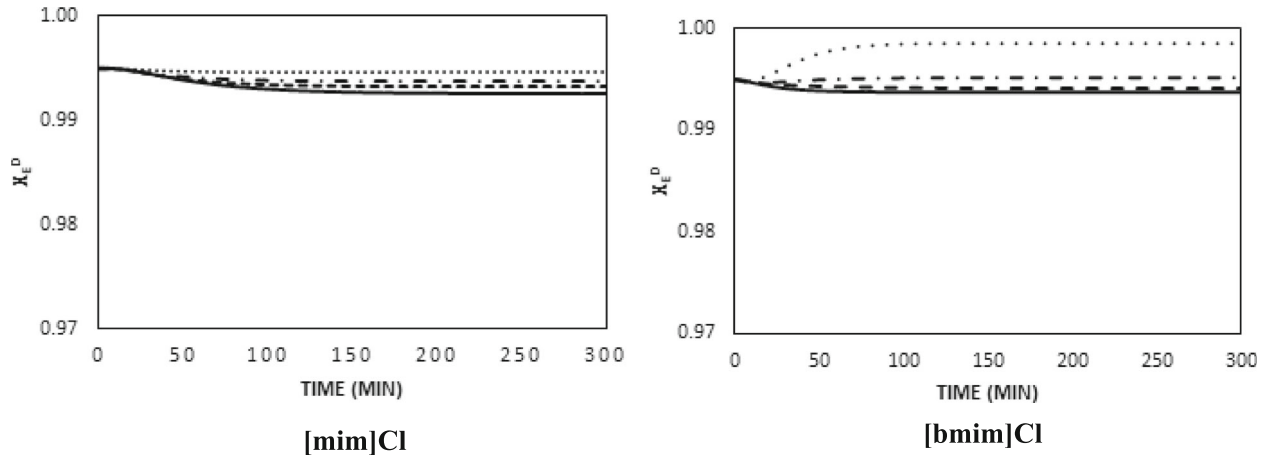

Fig. 5. Dynamic performance for negative disturbances (-10%) in feed flowrate, [— X_{IL}=0.10, — —X_{IL}=0.15, —·— X_{IL}=0.2, ···· X_{IL}=0.3]

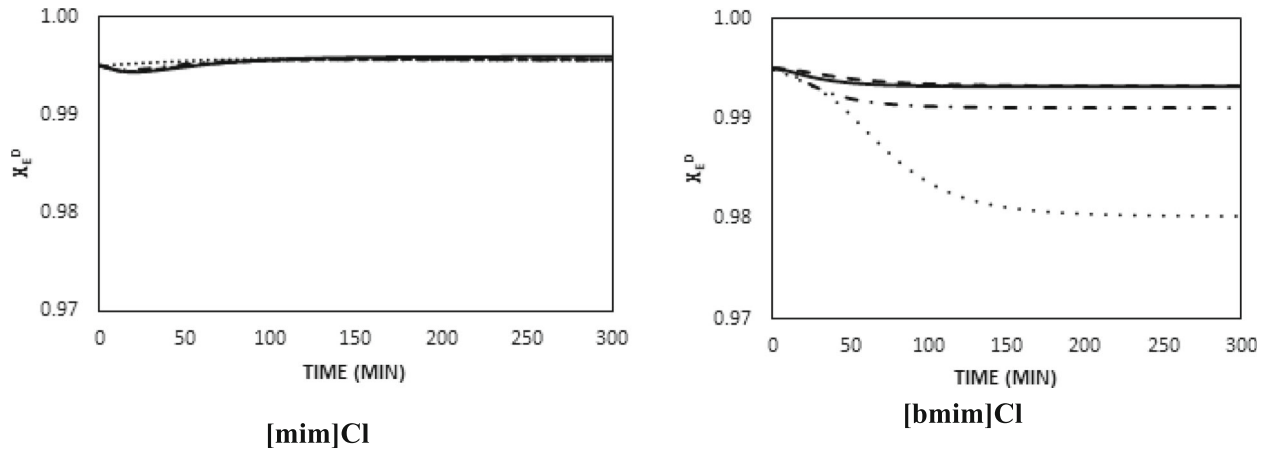

Fig. 6. Dynamic performance for positive disturbances (+10%) in ethanol composition in feed, [— X_{IL}=0.10, — —X_{IL}=0.15, —·— X_{IL}=0.2, ···· X_{IL}=0.3]

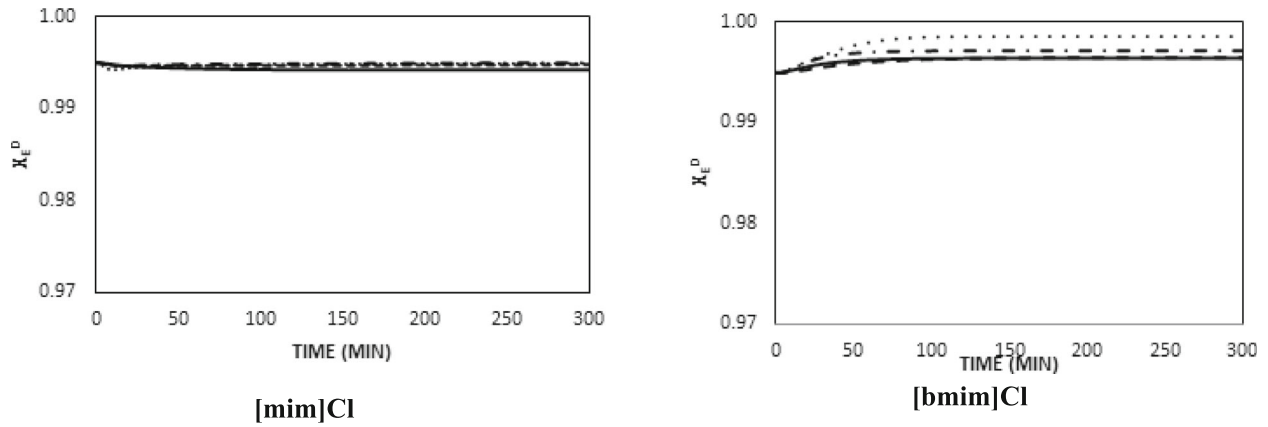

Fig. 7. Dynamic performance for negative disturbances (-10%) in ethanol composition in feed, [— X_{IL}=0.10, — —X_{IL}=0.15, —·— X_{IL}=0.2, ···· X_{IL}=0.3]

in such a way that any variations on the reflux ratio also affect the IL performance for the separation. To explore the potential benefits of a double-ratio controller, a control strategy was implemented by relating the reflux rate to the distillate rate (Fig. 8). The IL concentration was taken at its most concentrated option of 0.3, which showed the most sensitive responses under a single-ratio control scheme. The effect of the new control implementation on the final steady states achieved by the column regarding top product concentration, IL concentration and reflux ratio under plus/minus 10% changes in feed flowrate and ethanol feed concentration are reported in Table 3, where they are also compared to the values obtained when only one ratio controller was used. Under the effect of the [mim]Cl ionic liquid, the double-ratio control scheme kept or improved the concentration of ethanol in the distillate with respect to the action of the single-ratio controller. One can notice how IL concentration and reflux ratio adjusted to provide an effective control task. When the [bmim]Cl ionic liquid was tested, the ethanol purity improved under positive changes in feed flowrate and ethanol

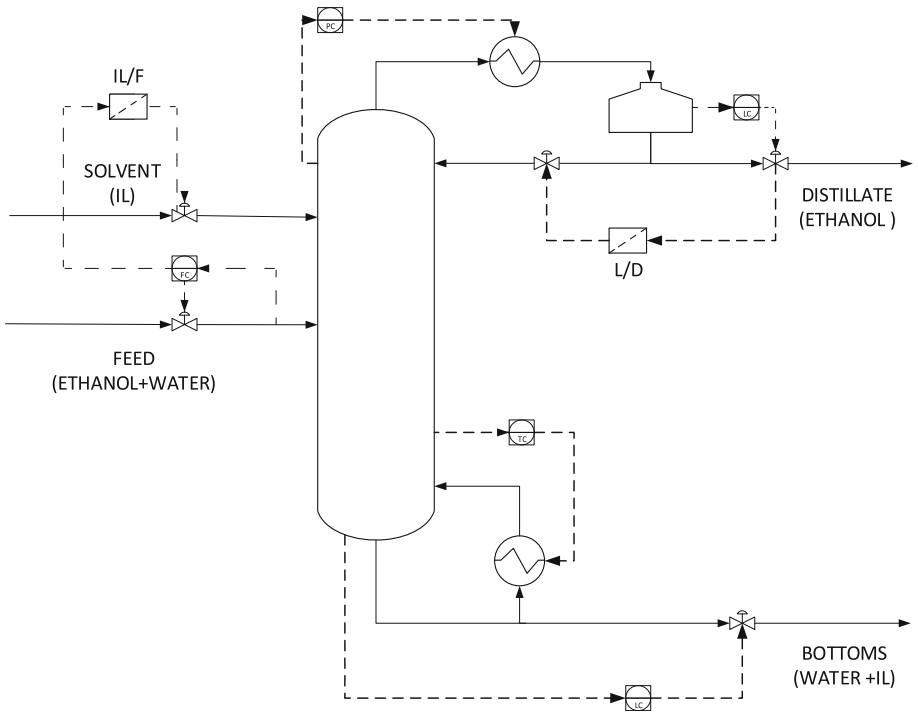

Fig. 8. Double ratio control structure for ethanol dehydration system aided by ionic liquids. Feed to IL flowrate and reflux to distillate ratio controllers

Table 3 Final steady state values for output variables: product purity, IL concentration and L/D ratio

Process variable	[mim]Cl		[bmim]Cl	
	Only IL/F	IL/F and L/D	Only IL/F	IL/F and L/D
Feed flowrate (+ 10%)				
X_E^D	0.995	0.996	0.975	0.996
X_{IL}	0.329	0.320	0.317	0.304
L/D	0.774	0.811	0.885	0.970
Feed flowrate (−10%)				
X_E^D	0.995	0.995	0.998	0.994
X_{IL}	0.285	0.303	0.279	0.301
L/D	0.881	0.811	1.081	0.970
X_E^{Feed} (+ 10%)				
X_E^D	0.993	0.994	0.981	0.996
X_{IL}	0.309	0.292	0.299	0.284
L/D	0.742	0.811	0.875	0.970
X_E^{Feed} (−10%)				
X_E^D	0.996	0.996	0.998	0.989
X_{IL}	0.307	0.330	0.298	0.324
L/D	0.904	0.895	1.099	0.970

concentration in the feed with respect to the use of a single-ratio controller, but deteriorated when negative disturbances were assumed. As a result, [mim]Cl proved again to be a better option as entrainer under a double-ratio controller, an implementation that improved the performance previously obtained under the single-ratio action.

Figure 9 shows the details of the transient responses obtained when [mim]Cl was used. In addition to recovering or improving steady state values, one can observe the smooth responses obtained against the four disturbances that were implemented. Also, a remarkable improvement in settling times was observed with respect to the use of the one-ratio controller, since the column stabilized in about 10 min for each case, as opposed to the times of 50 to 100 min observed under the action of the one-ratio controller. Therefore, from dynamic considerations, [mim]Cl as entrainer was shown to be a better option than [bmim]Cl, which could be associated to the more favorable opening of the ethanol-water equilibrium curve, particularly at high ethanol concentrations.

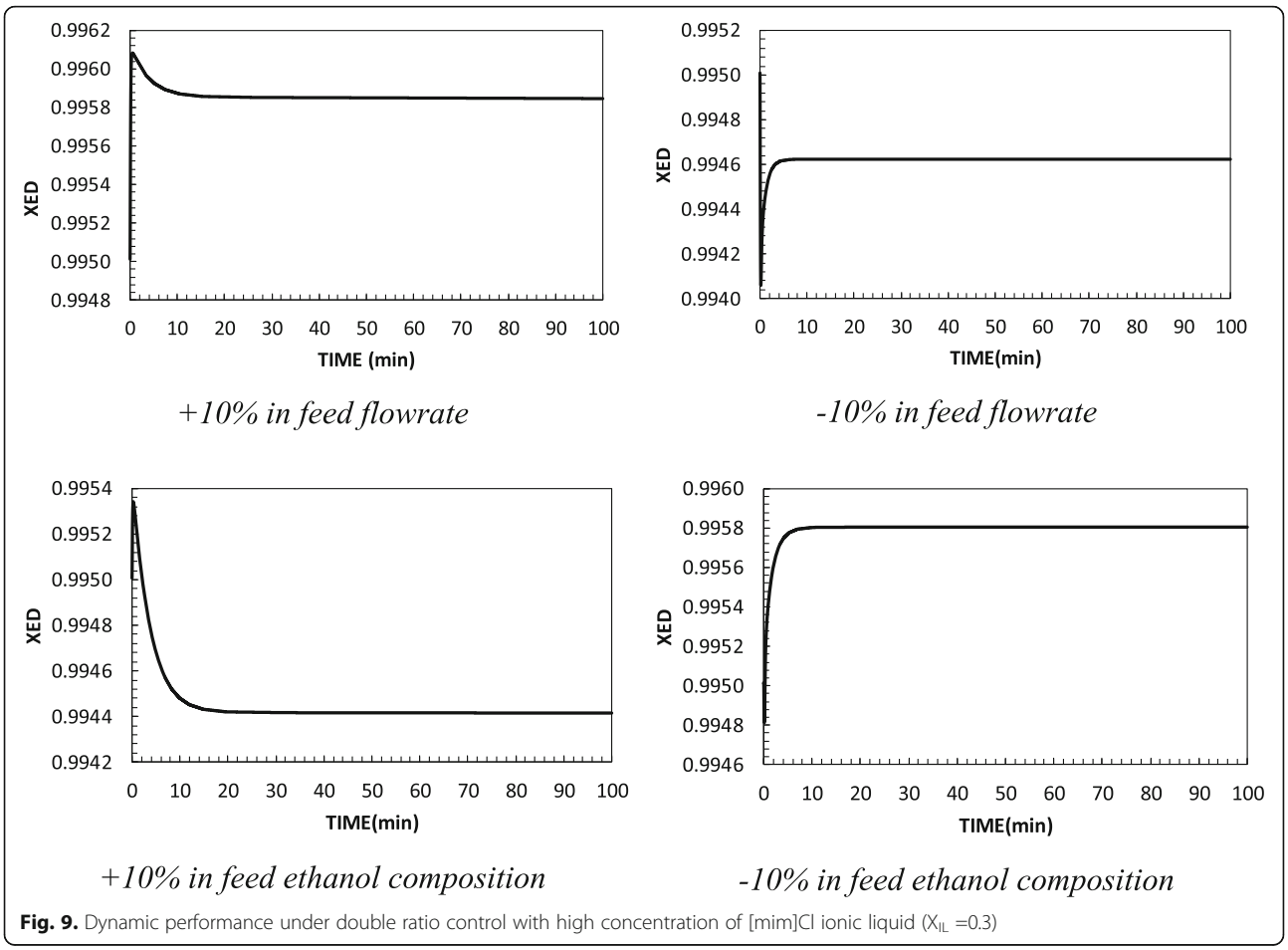

Fig. 9. Dynamic performance under double ratio control with high concentration of [mim]Cl ionic liquid (X_{IL} =0.3)

Conclusions

A control analysis for an ethanol-water extractive distillation system under the implementation of two types of ionic liquids used as entrainers, [mim]Cl and [bmim]Cl, has been presented. The analysis was conducted for an equimolar ethanol-water feed mixture, while four ionic liquid concentrations were considered. After column designs were obtained, dynamic tests were implemented to assess the response of the system in the face of feed flowrate and composition disturbances. The dynamic responses under a single-ratio controller showed that although both entrainers provided good behavior, the [bmim]Cl ionic liquid showed a significant sensitivity when high concentrations were used. On the other hand, [mim]Cl provided smooth responses for all the IL concentrations that were considered. The implementation of a double-ratio controller scheme further improved the dynamic responses of the extractive column, providing smooth responses with low settling times; under this type of controller, the use of [mim]Cl stood out again with respect to the option of using [bmim]Cl as entrainer. Overall, the results of this work show that [mim]Cl offers an interesting potential for its implementation as entrainer for ethanol dehydration extractive columns.

Abbreviations

IL: Ionic liquids; VLE: Vapor-liquid equilibrium; X_{IL}: Ionic liquid concentrations

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Authors' contributions

NR-C supervised the project, did simulations in Aspen and worked on the manuscript. AR-L carried out dynamic simulations in Aspen Dynamics. SR-L carried out dynamic simulations in Aspen Dynamics. AJ-G supervised the project and worked on the manuscript. All authors read and approved the final manuscript.

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Availability of data and materials

Data are available by contacting the authors.

Competing interests

The authors declare that they have no competing interests.

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