SEPARATION OF METHANOL AND ACETONITRILE MIXTURE VIA EXTRACTIVE DISTILLATION USING IMIDAZOLIUM-BASED IONIC LIQUIDS AND CONVENTIONAL SOLVENTS AS ENTRAINERS: A SIMULATION APPROACH

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Abstract

In this work, a comparative simulation study of extractive distillation using imidazolium-based ionic liquids and conventional solvents for the separation of methanol and acetonitrile was conducted using Aspen HYSYS. The ionic liquids selected were 1-ethyl-3-methylimidazolium bis(trifluoromethylsulfonyl)-imide or [EMIM][BTI], 1-butyl-3-methylimidazolium or bis(trifluoromethylsulfonyl)-imide [BMIM][TFSI], and 1-butyl-3-methylimidazolium acetate or [BMIM][OAc], while the conventional solvents were aniline, ethylene glycol, and glycerol. A case study on the solvent feed conditions was conducted to determine the optimum operating temperature and pressure for the extractive distillation columns. The simulation results show that the application of ionic liquid as the entrainer is more effective in eliminating the azeotropic point of methanol and acetonitrile as compared to the conventional solvents under the same specifications for the extractive distillation column and solvent recovery column.

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1.0 INTRODUCTION

The distillation process, one of the major separation processes in the chemical industry, accounts for 10-15% of the total energy consumption in chemical plants (Ma *et al.,* 2019). Despite its extensive energy consumption in the chemical industry, distillation remains the most widely used method in separating a mixture into pure components (Sinnot *et al.,* 2019). The principle of distillation is based on the differences in volatility between the components present in the liquid mixtures (Ma *et al.,* 2019). Acetonitrile and methanol are two of the most widely used chemicals in the chemical and pharmaceutical industries due to their high solubility in organic materials (Joshi *et al.,* 2019). For example, large amounts of acetonitrile and methanol are utilised during the manufacture of peptide drugs (Hintzen *et al.,* 2014). Therefore, the separation of mixtures consist of methanol and acetonitrile must be carried out to minimise the environmental impacts, as well as recycling the methanol and acetonitrile (Zhu *et al.,* 2016).

Unfortunately, the methanol and acetonitrile mixture cannot be separated via conventional distillation processes, as the mixture forms a minimum boiling homogeneous azeotrope at 336.7 K and atmospheric pressure, containing 81% methanol (Luyben, 2013). Several distillation technologies, including pressure-swing distillation, reactive distillation, extractive distillation, and azeotropic distillation, have been proposed to separate azeotropic mixtures (Ma *et al.,* 2019). Pressure-swing distillation is commonly used for the homogenous azeotropic mixtures that are sensitive to changes in pressure (Sinnot *et al.,* 2019). On the other hand, reactive distillation involves a complex separation process, as it separates the components in the mixture while performing the chemical reaction (Ma *et al.,* 2019). Extractive distillation involves the addition of separating agent or entrainer to alter the relative volatility between the key components without forming an azeotrope (Coker, 2010). Extractive distillation is similar to azeotropic distillation; however, no azeotropes are formed upon the addition of the entrainer, and the entrainer added is essentially non-volatile (Coker, 2010).

The entrainer used for the separation processes typically possesses a significantly higher boiling point than the mixture components and can induce a salting-out effect, thereby reducing the volatility of the component that interacts with the entrainer (Wankat, 2012). The remaining component present in the mixture is then become relatively more volatile and easier to be separated and removed as the distillate (Wankat, 2012). Apart from affecting the relative volatilities of the key components, several characteristics are important for selecting a suitable entrainer. These include low latent heat, non-reactivity, non-corrosiveness, non-toxicity, and solubility with the components in the mixture (Coker, 2010). Several conventional solvents have been used as entrainers, such as aniline, dimethylformamide (DMF), glycerol, and ethylene glycol. These solvents have also been studied for the separation of azeotropic mixture of methanol and acetonitrile (Li *et al.,* 2012). However, these entrainers have several disadvantages, such as causing environmental pollution due to volatile emissions and presenting significant challenges in recycling due to their complex separation and purification processes (Cai *et al.,* 2013).

Ionic liquids (ILs) possess various unique physic-chemical properties to replace the conventional solvents, including good chemical and thermal stability, excellent dissolving capacity, and non-volatility (Seiler *et al.,* 2014). Numerous studies have been conducted to investigate and review the utilisation of ILs as entrainers for extractive distillation (Shen *et al.,* 2023: You *et al.,* 2022; Martínez-Galmiche *et al.,* 2022; and Vilas-Boas *et al.,* 2024). Ionic liquid consisting of cations such as imidazolium and alkyl quaternary coupled with anions such as hydrogen sulphate, halogen, and acetate are commonly used to separate polar-polar systems, including alcohol/water, nitrile/water, and alcohol/nitrile mixtures (Pereiro *et al.,* 2012). On the other hand, ILs consisting cations such as imidazolium and pyridine coupled with anions such as tetrafluoroborate (BF4), bis(trifluoromethyl-sulfonyl)imide (NTf2) and dicyanamide are commonly selected to separate nonpolar-nonpolar systems, including alkane/alkene and aliphatic/aromatics mixtures (Pereiro *et al.,* 2012). While ILs consisting of imidazolium cation and sulphate anion are commonly used to separate polar-nonpolar systems, including alcohol/aliphatic mixtures (Pereiro *et al.,* 2012).

A study was conducted by Li *et al.* (2016) to investigate the effects of 1-octyl-3-methylimidazolium tetrafluoroborate, [OMIM][BF_4], and 1-ethyl-3-methylimidazolium tetrafluoroborate, [EMIM][BF_4], on the vapor-liquid equilibrium behaviour of the methanol-acetonitrile system. It was reported that [OMIM][BF_4] has a stronger salting-out effect on methanol compared to [EMIM][BF_4]. However, ILs with the [BF_4]- anion are difficult to synthesise, and corrosive hydrogen fluoride could be emitted upon contact with water (Cai *et al.,* 2013). On the other hand, Zhang *et al.* (2016)] investigated the performance of separating methanol and acetonitrile mixture using 1-ethyl-3-methylimidazolium bis(trifluoromethylsulfonyl)imide or [EMIM][NTf_2]. It was found that the azeotropic phenomenon could be eliminated using this ionic liquid. However, the current production cost of ionic liquids containing [NTf_2]- is economically unfavourable, making them less viable for large-scale use (Nasirpour *et al,* 2020).

Another study by Z. Zhang *et al.* (2018) compared the performance of 1-butyl-3-methylimidazolium chloride or [BMIM][Cl], 1-butyl-3-methylimidazolium bromide or [BMIM][Br] and 1-butyl-3-methylimidazolium acetate or [BMIM][OAc] as entrainers to separate methanol and acetonitrile at atmospheric pressure. The finding reveals that these three ionic liquids produced a significant salting-effect and enhanced the relative volatility of acetonitrile to methanol. A directly proportional relationship between the amount of ILs and the salting-out effect was determined in the study (Zhang *et al.,* 2018). It was also concluded that the [BMIM][OAc] has the best separation performance compared to [BMIM][Cl] and [BMIM][Br].

In this work, three ionic liquids, namely 1-ethyl-3-methylimidazolium bis(trifluoromethylsulfonyl)-imide or [EMIM][BTI], 1-butyl-3-methylimidazolium bis(trifluoromethylsulfonyl)-imide or [BMIM][TFSI], 1-butyl-3-methylimidazolium acetate or [BMIM][OAc] were tested as entrainers to separate binary mixture composed of methanol and acetonitrile via simulation approaches using Aspen HYSYS V11. The effects and performance of these ILs and three conventional solvents were compared and evaluated to determine the optimum operating temperature and pressure for the extractive distillation column. The solvents studied include aniline, ethylene glycol and glycerol. The results obtained from simulation and case studies aim to corroborate the feasibility of utilising ILs as entrainers to separate methanol and acetonitrile.

2.0 METHODOLOGY

The non-random two-liquid (NRTL) model (Renon *et al.,* 1968) was selected as the thermodynamic package to evaluate the vapor-liquid equilibrium of the mixture in Aspen HYSYS V11 software. The hypothetical functions available in Aspen HYSYS were utilised to estimate the properties of ionic liquids. The performance of these ionic liquids and conventional solvents were compared using the same columns specifications including the number of stages, solvent to feed ratio, feed stage as well as the solvent feed stage for both columns. These specifications were adapted from the study conducted by Wang *et al.* (2019). Fig. 1 shows the flowsheet for the extractive distillation process whereas Table 1 shows the specifications for both extractive distillation column (EDC) and solvent recovery column (SRC).

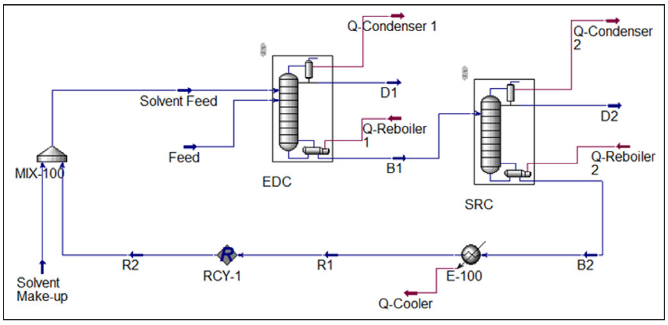

Figure 1: Process flowsheet for extractive distillation of methanol and acetonitrile

Table 1: Specifications of extractive distillation column (EDC) and solvent recovery column (SRC)

Specifications	EDC	SRC
Number of Stages	48	8
Solvent to Feed Ratio	1	-
Feed Stage	4	-
Solvent Feed Stage	33	5

The operating conditions for solvent feed including temperature and pressure were optimised by conducting individual case studies on each of the parameters. The effect of each parameter on the separation performance was investigated through case studies. The best settings for both columns were evaluated based on the purity of methanol and acetonitrile recovered.

3.0 RESULTS AND DISCUSSION

3.1 Simulation of Extractive Distillation Process

The results obtained from the simulation of extractive distillation for methanol and acetonitrile using conventional solvents and ionic liquids are tabulated in Table 2 and Table 3.

Table 2: Simulation results for extractive distillation of methanol-acetonitrile using conventional solvents

Parameters	Unit	Aniline		Ethylene Glycol		Glycerol	
		EDC	SRC	EDC	SRC	EDC	SRC
Reflux Ratio	-	0.205	0.450	1.458	2.060	2.092	0.004
Distillate Flow Rate	kmol/h	85.0	16.0	85.0	16.0	85.0	15.1
Bottom Flow Rate	kmol/h	116.0	100.0	116.0	100.0	115.1	100.0
Purity of Methanol/ Acetonitrile	%	94.0	86.7	94.0	86.9	94.0	92.1
Purity of Solvent Recovered	%	-	99.0	-	96.0	-	99.9

Table 3: Simulation results for extractive distillation of methanol-acetonitrile using ionic liquids

Parameters	Unit	[EMIM][BTI]		[BMIM][TFSI]		[BMIM][OAc]	
		EDC	SRC	EDC	SRC	EDC	SRC
Reflux Ratio	-	1.176	0.047	1.176	0.047	1.176	0.047
Distillate Flow Rate	kmol/h	85.0	15.0	85	15	85.0	15.0
Bottom Flow Rate	kmol/h	115.0	100.0	115	100	115.0	100.0
Purity of Methanol/ Acetonitrile	%	94.8	97.1	95.2	99.5	94.8	97.1
Purity of Solvent Recovered	%	-	100.0	-	100.0	-	100.0

According to the simulation results tabulated in Table 2, the use of glycerol as the entrainer for extractive distillation achieves the best separation performance among the conventional solvents. However, the comparison between the simulation results for both conventional solvents and ionic liquids (Table 3) shows that the use of ionic liquids as an entrainer can achieve higher purity for methanol, acetonitrile, as well as the solvent recovered. It is found that the highest purity of the binary mixture in extractive distillation column is achieved using [BMIM][TFSI] with 95.2% purity. This increase in relative volatility may be attributed to the properties of ionic liquids, including their ability to modify the thermodynamics and molecular interactions within the system. By introducing [BMIM][TFSI] into the extractive distillation process, the separation between the two components becomes more pronounced, resulting in higher selectivity and purity of acetonitrile. Furthermore, the addition of ionic liquids shifts the azeotropic point towards higher methanol concentrations similar to study by Boli *et al.* (2020). This means that the presence of ILs can alter the composition of the azeotrope formed by the methanol-acetonitrile mixture, allowing for easier separation by extractive distillation. Shifting the azeotropic point is a desirable outcome as it overcomes the limitations imposed by azeotropic behaviour (Sofea *et al.,* 2022).

On the other hand, a higher loss of solvents through the distillate of the extractive distillation column and the solvent recovery column is observed for the extractive distillation process that utilises conventional solvents (Table 2) as an entrainer compared to ILs (Table 3). Consequently, a larger amount of make-up solvent is required for the process, resulting in higher operating costs (Wang *et al.,* 2023).

These findings emphasise the advantageous role of ionic liquids, such as [BMIM][TFSI], in extractive distillation processes. Their unique properties and interactions with the mixture components contribute to enhanced separation performance, higher selectivity, and the ability to modify azeotropic behavior. However, it is important to consider factors such as the cost, availability, and environmental impact of ionic liquids when evaluating their suitability for large-scale industrial applications (Berton *et al.,* 2022). Further research and experimentation are necessary to fully explore the potential of [BMIM][TFSI] and other ionic liquids in extractive distillation and other separation processes.

It is worth noting that these results were fully based on simulation data, and further experimental investigations are required to validate these findings. Nonetheless, comparative analysis provides valuable insights into the potential benefits of employing ionic liquids in extractive distillation processes, emphasising their superior performance in terms of both separation efficiency and cost-effectiveness.

3.2 Optimisation of Solvent Feed Conditions

The optimum operating temperature and pressure of the solvent for the extractive distillation process using [BMIM][TFSI] as entrainer were further determined by using the case study function available in Aspen HYSYS. The determination of the optimum operating temperature and pressure for the extractive distillation process employing [BMIM][TFSI] as an entrainer can be accomplished by utilising the case study function available in Aspen HYSYS, a widely used process simulation software. This function allows for the exploration of various operating conditions and provides valuable insights into the process of performance. Fig 2 and Fig. 3 show the results determined from the case studies.

According to the results obtained from the case studies on solvent feed temperature and pressure, there is no appreciable reduction observed (almost constant) in the molar flow of methanol and acetonitrile recovered from the distillate of the extractive distillation column and solvent recovery column as the solvent feed temperature and pressure increase. However, it is important to note that lower feed temperature and pressure are generally more favoured to reduce the cost of utilities and equipment, as a higher solvent feed temperature and pressure will require more heating utilities and additional equipment such as pump and compressor. Further investigations on the top temperature, bottom temperature of both columns, as well as economic analysis on the capital and operating costs can be carried out to optimise the extractive distillation using [BMIM][TFSI] as entrainer (Wang *et al.,* 2023). These may involve exploring the optimal top and bottom temperatures of both columns, as well as performing an economic analysis to evaluate the capital and operating costs associated with the chosen operating conditions. Such analyses can provide a comprehensive understanding of the process and guide the decision-making process in designing an efficient and cost-effective extraction system.

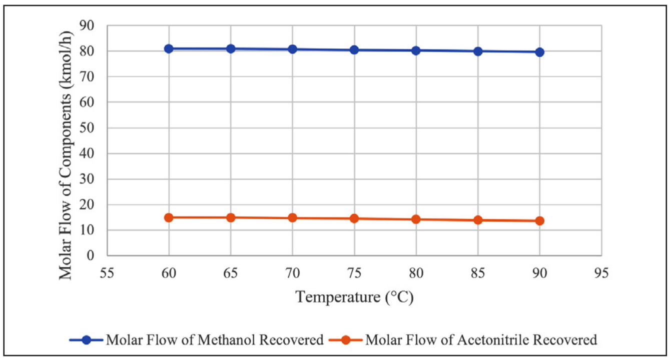

Figure 2: Case study on the solvent feed temperature

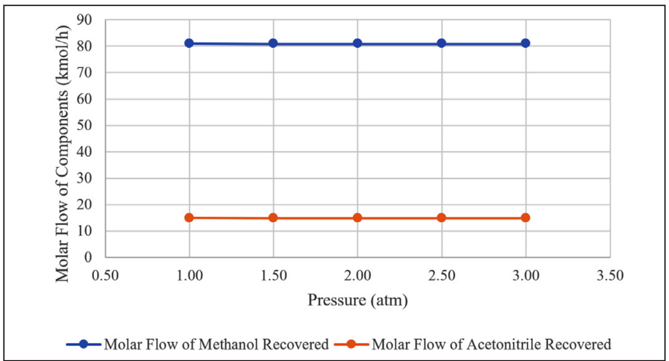

Figure 3: Case study on the solvent feed pressure

4.0 CONCLUSION

The Aspen HYSYS simulation using the NRTL model has been performed to simulate the extractive distillation process for the azeotropic mixture containing methanol and acetonitrile. According to the simulation results, 1-butyl-3-methylimidazolium bis(trifluoromethylsulfonyl)-imide [BMIM][TFSI] is the most efficient solvent for the extractive distillation process. Besides that, the simulation results also showed that the application of ionic liquids as entrainer for the extractive distillation of methanol and acetonitrile are feasible. It is generally more desirable to operate the extractive distillation column at lower temperature and pressure to reduce the operating cost and capital costs. Further improvements can be made by integrating heat integration technology and heat pump technology to reduce the utilities cost for the extractive distillation process. Apart from that, life cycle analysis can be conducted to further evaluate and compare the environmental impacts of the extractive distillation process using conventional solvents and ionic liquids.

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AUTHOR CONTRIBUTION

- **Zhi Ting Ang:** Data Curation, Formal Analysis, Methodology, Visualisation, Writing (Original Draft)
- **Ianatul Khoiroh:** Conceptualisation, Resources, Funding Acquisition, Supervision, Writing (review & editing). ■

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PROFILES

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