

A study on selection of separation agent for Chloroform - Methanol, Ethanol - Water and Methylacetate - Methanol systems

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Abstract:

Distillation is the most common separation operation in chemical engineering industries. Formation of azeotropes by close boiling point compounds present in mixtures complicates the separation process. Chloroform - Methanol, Ethanol - Water and Methylacetate - Methanol systems form azeotropes and are subject to experimental investigation by employing Othmer VLE still to identify potential separating agents to break the azeotropism. The experimental observations are reported in the study. The results show that water, toluene and ethylene glycol can be used as promising agents for the systems Chloroform - Methanol, Ethanol - Water and Methylacetate - Methanol respectively. Also it is inferred that water and ethylene glycol act as solvent whereas toluene plays role as a entrainer for the respective mixture concerned.

INTRODUCTION

Separation operations are achieved by the creation of distinct coexisting zones which have varying temperature, pressure, composition, and/or phase state. In distillation operations these coexisting zones have same temperature and pressure conditions in the vapor and liquid phases (Seader and Henley, 2006). The feed material consisting the mixture is separated between the distillate and the bottoms. The degree of separation depends mainly on the relative volatilities of the components which in turn affects the number of contacting plates / trays. This decides ultimately the height of the distillation column required for given task (Sivaprakash and Manojkumar, 2019). The relative volatility should be far from unity for easier separation. But for azeotropes it is nearly equal to unity and hence needs special methods of distillation. Organic mixtures generally form nonideal systems. Excessive

deviation from ideality (Raoult's Law) leads to the formation of azeotropes (Salam et al, 2015). Azeotropism occurs in systems where the compounds have some specific functional groups, particularly polar groups like oxygen, nitrogen, chlorine and fluorine (Stuart et al, 2015). As the fractional distillation methods prove to be very inefficient for handling the azeotropes in terms of efficiency, energy consumption and initial investment, separation of azeotropic mixtures is a topic of great practical and industrial interest. All liquid mixtures have strong forces of intermolecular attraction which make them to form liquids rather than gases (Manojkumar and Sivaprakash, 2018c). When two or more components are present as a mixture the molecular interactions among them makes the mixture to converge to certain "inseparable" compositions where the vapor and liquid compositions at equilibrium are equal within a given pressure and temperature range (Tom et al, 2013). These specific mixture compositions are called azeotropes. Azeotropy is a commonly

occurring phenomena in industrial distillation operations (Manojkumar and Sivaprakash, 2018d). Existence of strong molecular interactions like hydrogen bonding among the constituents of mixtures leads to highly nonlinear phase equilibrium (Gudjonsdottir and Infante Ferreira, 2016, Manojkumar and Sivaprakash, 2018e). This leads to the deviation from ideal behavior of many systems. The nature of deviation from ideality is dependent on the physiochemical forces between identical and distinct components. In positive deviation from Raoult's law, the attraction between identical molecules is stronger than that between different molecules leading to the formation of a minimum-boiling azeotrope (Salomon and Mauricio, 2014). If the attraction between the different molecules are stronger than the like molecules it leads to negative deviation from Raoult's law and the mixture becomes a maximum-boiling azeotrope. Thus the tendency of a mixture to behave azeotropic depends not only on the difference in the pure component boiling points but also on the degree of nonideality. Literature report (Gustavo et al, 2015, William, 2013) and researches show that more than 90 % of the known azeotropes exhibit positive azeotropism and also more than 80 % of them are homoazeotropic. Azeotropic mixtures are separated by distillation using a third component (usually liquids). This agent can either dissolve one of the two components or can form a new azeotrope with one of the components (Giuseppe and Antonio, 2014). Former is called extractive distillation (Fonny, et al, 2011) and the third component is called as a solvent. Latter is called azeotropic distillation wherein the separating agent is called as an entrainer. Distillation columns generally utilize enormous energy and this comes to nearly 90% of the total industrial consumption. Hence design of an energy efficient distillation column is very much essential. The role of chemical engineers in reducing the energy requirements and hence the capital costs and running costs in distillation column is a major concern. In the present investigation three azeotropic systems, namely, Chloroform - Methanol, Ethanol -

Water and Methylacetate - Methanol have been chosen for the selection of separating agent in terms of recovery.

Chloroform - Methanol mixture

The chloroform - methanol mixture is an organic waste of pharmaceutical and biotechnology industries (Van Kaam et al, 2008). Chloroform is widely used as an effective solvent for bioactive substances. Chloroform cannot be separated from methanol by using conventional distillation columns as they exhibit azeotropism at 1 atm pressure. Henley (2013) introduced design and control of an innovative pressure - swing distillation process for separation of chloroform-methanol. However due to high energy demand, low recovery and high capital cost that incurs in this process makes it least attractive on industrial scale.

Ethanol - Water mixture

Separation of the ethanol - water mixture is of great industrial interest because ethanol is potentially used as a renewable source of energy and as an additive for gasoline. Ethanol is relatively clean burning fuel as it can reduce the amount of pollution emitted to the air. Ethanol is usually found as a mixture with water during its production and hence their separation is an important process in industries (Vicente et al, 2015). Various processes were investigated to separate ethanol - water mixture. Shenfeng et al (2015) proposed isooctane and heptane as entrainers for separating ethanol - water azeotrope. Adnan and Al- Ameer (2000) examined polymeric entrainers for separation of ethanol - water mixture. However these methods failed to break the azeotropic behavior of ethanol - water mixture in a simpler manner. Therefore an essential alternative is needed to separate the ethanol - water mixture.

Methyl acetate - Methanol mixture

Azeotropic mixture of methylacetate - methanol is widely found in manufacturing process of poly vinyl alcohol. Major use of methyl acetate is as a solvent in glues, paints, and nail polish removers. The separation of methyl acetate and methanol mixture is a challenging task due to formation of minimum

boiling homogeneous azeotrope at atmospheric pressure. Amir and Arshad (2014) proposed various techniques to separate methyl acetate-methanol mixture such as azeotropic distillations, reactive distillation, selective, adsorption and ionic liquids. However these methods are not in practice. The extractive distillation is the most popular method and selection of the extractive agent is of high importance.

MATERIALS AND METHOD

CHEMICALS AND REAGENTS

Reagent grade Benzene, Toluene and methyl acetate were supplied by National Scientific Limited Pondicherry and the fractions with physical properties (boiling point, refractive index and density) closely reproducing literature values were used. Research grade Methanol, Ethanol, 1-Butanol and Chloroform were supplied by Indian scientific Limited Chennai. Ethylene glycol from Aldrich chemicals, were directly used.

EXPERIMENTAL

Othmer VLE stills are the generally used in VLE estimation using experimental methods. The circulation of the vapour phase only or the circulation of both the vapour and the liquid phases of the boiling mixture is the principle of operation (Sivaprakash and Manojkumar, 2019). The Othmer apparatus circulates the vapour phase efficiently it had some advantage such as the location of temperature probe, the hold-up of condensate receiver was large, complete condensation of vapour on the wall of boiling flask, and flashing of vapour rich in the less volatile component. The apparatus chosen this study is the recirculation VLE still as shown in Figure 1. Because of the ease of operation and design features, the Othmer VLE recirculation still provides accurate vapor-liquid equilibrium data in a relatively short experimental time. Also Othmer VLE still is noted for its simplicity of design and operation and for giving consistent data. The Othmer VLE Still was made of Pyrex glass, thus the system was not subject to thermal shock and was closely monitored to avoid running the risk of cracking the

still. Insulation also surrounded the still to help keep it warm and avoid condensation and reflux.



Fig. 1 Vapour Liquid Equilibrium Still

EXPERIMENTAL PROCEDURE

Othmer VLE still was employed to determine vapour liquid equilibrium data. The capacity of the still is about 200 ml and it is outfitted with reflux condenser. Binary liquid mixture of known composition of Component A 50 ml and Component B 50 ml and entrainer are dissolved in the mixture at different measurements of 5, 10, 15 and 20 ml was charged at the top of the VLE still and distilled using electrical heating. The distillate (vapour form) richer in more volatile compound enters the condenser with cold water circulation and is collected at the top. The residual product (liquid) richer in less volatile compound can be collected from the bottom. The still is equipped with a quartz thermometer to measure the azeotropic distillation temperature. After equilibrium was established (indicated by a constant reading in the thermometer), heating was stopped and the contents of the top and bottom products were allowed to cool and analyzed.

EXPERIMENTAL ANALYSIS

Gas chromatography is employed as the analytical technique in much of the experimental work. The

Agilent model 6890N gas chromatograph with hydrogen flame ionization detector is used for the purpose (Fig. 2).



Fig.2. The ALIGENT Technologies Series (6890N) Gas Chromatography System

Chloroform-Methanol-Water; Chloroform-Methanol-1-Butanol; Ethanol-Water –Benzene; Ethanol-Water- Toluene

The samples were analyzed using DB-624 GC fused with silica column (75 m × 0.53 mm ID × 3µm). The components were separated using Nitrogen as carrier gas at a constant flow of 6 ml/min. The injector temperature was set at 230°C during the chromatographic run. One µL of extract sample was injected into the instrument and the oven temperature was at 50°C(5 min), followed by 210°C at the rate of 20°C min⁻¹ where it was held for 6 minutes

Methylacetate-Methanol-Ethylene glycol

The samples were analyzed using DB-624 GC fused with silica column (30 m × 0.53 mm ID × 3µm). The components were separated using Nitrogen as carrier gas at a constant flow of 0.2 ml/min. The injector temperature was set at 250°C during the chromatographic run. One µL of extract sample was injected into the instrument and the oven temperature was at 40°C (8 min), then to 80°C at the

rate of 5°C min⁻¹ where it was held for 2 minutes followed by 200°C at the rate of 20°C min⁻¹ where it was held for 10 minutes. Whereas for EG samples were analyzed using DB-624 GC fused with silica column and packed with (30 m × 0.53 mm ID × 3µm). The components were separated using Nitrogen as carrier gas at a constant flow of 0.2 ml/min. The injector temperature was set at 180°C during the chromatographic run. One µL of extract sample was injected into the instrument and the oven temperature was at 40°C (8 min), then to 80°C at the rate of 5°C min⁻¹ where it was held for 2 minutes followed by 200°C at the rate of 20°C min⁻¹ where it was held for 1 minute.

Methylacetate-Methanol-Toluene

The samples were analyzed using DB-624 GC fused with silica column and packed with (30 m × 0.53 mm ID × 3µm). The components were separated using Nitrogen as carrier gas at a constant flow of 0.2 ml/min. The injector temperature was set at 250°C during the chromatographic run. One µL of extract sample was injected into the instrument and the oven temperature was at 40°C (8 min), then to 80°C at the rate of 5°C min⁻¹ where it was held for 2 minutes followed by 200°C at the rate of 20°C min⁻¹ where it was held for 10 minutes.

Results and Discussions:

Chloroform - Methanol

The binary mixture of chloroform-methanol shows a minimum-boiling azeotrope with 34 mol% methanol at 327K under atmospheric pressure. Thus chloroform- methanol mixture cannot be separated by ordinary distillation process. In this work, alternative distillation processes is investigated for chloroform-methanol separation using extractive distillation. Distinctively extractive agents referred in literature (Manojkumar and Sivaprakash, 2018a) for separating chloroform-methanol are isopropanol and 4-methyl-2-pentanone. Solubility of isopropanol in chloroform results in less energy to break the hydrogen bonds; hence isopropanol is less soluble in chloroform. In case of 4-methyl-2-pentanone, it is highly flammable and sensitive to air it form explosive peroxide while soluble in methanol. In the

present investigation water and 1-butanol are used as separating agents for chloroform-methanol mixture. The preparation of chloroform-methanol mixture from chloroform 50% and methanol 50% in terms of volume/volume and the entrainer (water / 1-butanol) are dissolved in the mixture at different measurements of 5, 10, 15 and 20 ml. Table 1 and 2 indicate the separation performance of chloroform-

methanol using water and 1- Butanol as an entrainer respectively. The distillate composition is analyzed and the utmost recovery of chloroform component is found to be 86.99 % when 20ml of water is used as an entrainer. Successively the addition of 20ml of 1-butanol achieved the separation efficiency of 75.8 % .

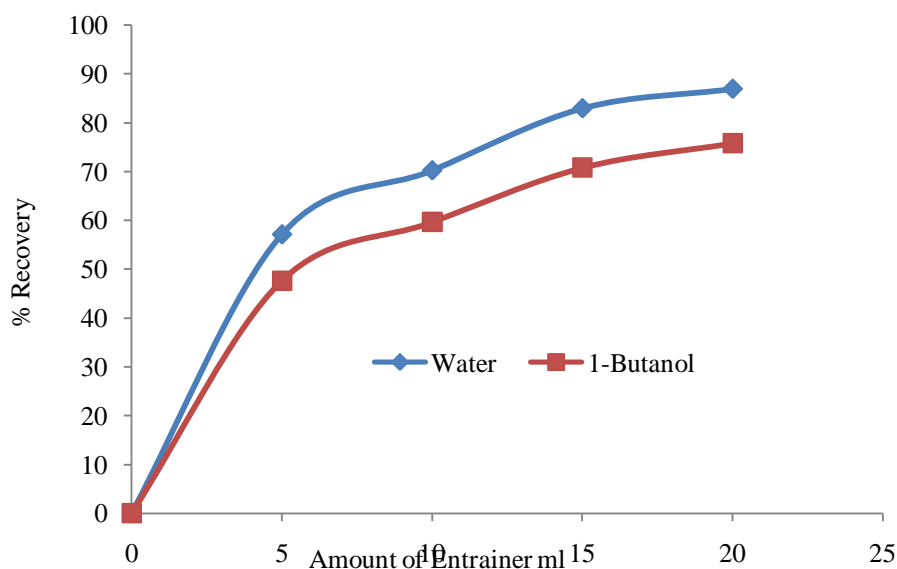


Fig. 3 Separation performance of Chloroform - Methanol mixture using water and 1 - Butanol

Figure 3 represents the comparison of entrainers' water and 1-butanol for chloroform- methanol system. From this figures it is to seen clear that the light solvent water gave the most promising result compared to 1-butanol. This is for the reason that water produces a notable increasing of the relative volatility of the original components than 1-butanol. Besides water have polarity similar to methanol and bring the methanol down the column. When chloroform-methanol mixture is mixed with other agent having low polarity like 1-butanol the efficiency will be reduced (Manojkumar and Sivaprakash, 2018b). Furthermore addition of 1-butanol induces the formation of quaternary mixture which is complicating the separation process. So

water is finally selected as the best entrainer for the separation of chloroform-methanol mixture.

Table 1 Separation Performance of Chloroform - Methanol system using Water

Water (ml)	Recovery Percentage in Distillate			Recovery Percentage in Bottom		
	Chloroform	Methanol	Water	Chloroform	Methanol	Water
5	57.15	64.35	39.05	42.85	35.64	60.94
10	70.28	54.97	29.02	29.71	45.01	70.97
15	83.00	47.66	21.79	17.00	52.33	78.21
20	86.99	40.92	20.09	13.01	59.07	79.91

Table 2 Separation Performance of Chloroform - Methanol system using 1 - Butanol

1 - Butanol (ml)	Recovery Percentage in Distillate			Recovery Percentage in Bottom		
	Chloroform	Methanol	1 - Butanol	Chloroform	Methanol	1 - Butanol
5	47.64	72.85	41.29	52.35	27.15	58.71
10	59.64	56.85	37.17	40.35	43.14	62.82
15	70.78	49.50	45.40	41.26	50.49	54.60
20	75.85	42.08	32.06	24.14	57.19	60.73

Ethanol - Water

Ethanol-water mixture forms an azeotropic condition at a temperature of 78.2°C where the mole fraction of ethanol is found to be 89.4% at one atmospheric pressure. The characteristic of azeotropic condition is observed as minimum boiling azeotropes. Since ethanol and water have close boiling points it cannot be separated into pure components by conventional distillation. In the present investigation extractive distillation is employed to separate ethanol-water mixture. Extractive agents quoted in literature (Adnan and Al-ameer, 2000) to separate ethanol-water mixture are acetic acid, heptane, and diethyl ether. Acetic acid is effective only in acid level pH (pH of 4 or below) and potentially corrosive to metals. Similarly solvent diethyl ether and heptane is highly flammable and can oxidize into an explosive hence these solvents are not used in this work. Our research progress is made through an extractive agents' of benzene and toluene. Preparation of the ethanol-water mixture is from ethanol 50% and water 50% in terms of volume/volume and the entrainer (benzene / toluene) are dissolved in the mixture at different measurements of 5, 10, 15 and 20 ml. The separation performance of ethanol-water using benzene and toluene as entrainer is given in Tables 3 and 4 respectively. The distillate composition is analyzed and the maximum recovery of ethanol component is found to 73.08 % when 20ml benzene is used as an entrainer. Similarly the addition of 20ml of toluene indicates the separation efficiency of 85.05%. Figure 4 represents the comparison of entrainers' benzene and toluene for

shows better recovery of ethanol compared to benzene at different measurements. This is since when toluene is added as an entrainer, the apparent azeotropic point disappears, and the value of relative volatility is larger than one over the entire mole fraction range of ethanol at one atmospheric pressure. When benzene is used as entrainer, a similar disappearance of the azeotropic point is observed at one atmospheric pressure. Therefore, toluene and benzene can be used as entrainers for extractive distillation of the binary azeotropic mixture ethanol-water. When the performances of toluene and benzene are compared, the disappearance of the azeotropic point occurs with a smaller amount of toluene than of benzene. From these results, it can be concluded that toluene is a more selective entrainer than benzene for the separation of ethanol-water by extractive distillation.

Table 3 Separation Performance of Ethanol - Water system using Benzene

Benzene (ml)	Recovery Percentage in Distillate			Recovery Percentage in Bottom		
	Ethanol	Water	Benzene	Ethanol	Water	Benzene
5	39.09	57.01	56.80	60.90	41.87	43.21
10	57.09	49.01	50.80	42.90	50.98	49.19
15	69.08	42.88	54.16	30.93	57.12	45.82
20	73.08	48.11	60.66	26.91	51.89	39.33

ethanol-water system. From this information it is to seen clear that the effect of toluene in the system

Table 4 Separation Performance of Ethanol - Water system using Toluene

Toluene (ml)	Recovery Percentage in Distillate			Recovery Percentage in Bottom		
	Ethanol	Water	Toluene	Ethanol	Water	Toluene
5	57.51	55.16	71.62	42.48	44.83	28.37
10	73.52	47.16	60.81	26.48	52.88	39.18
15	81.54	42.41	67.33	18.46	57.81	32.67
20	85.05	47.48	72.56	14.63	53.51	27.43

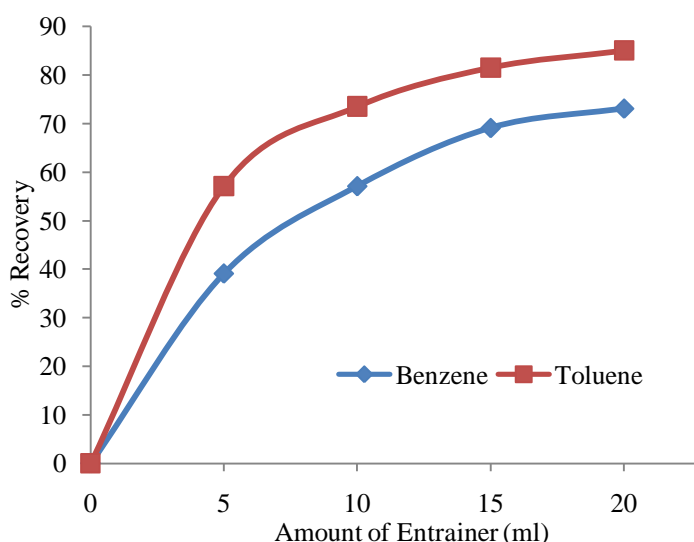


Fig. 4 Separation performance of Ethanol - Water mixture using Benzene and Toluene

Methylacetate -Methanol

Separating agents referred in literature (Perry and Green 1998) for separating this azeotropic mixture are methyl isobutyl ketones, mono ethyl ether etc. Disadvantage of using methyl isobutyl ketone requires high pressure operation and difficult to separate the product from homogenous reaction. Mono ethyl ether is both flammable and explosive it can be used only when special precautions are taken to prevent sparking and combustion. In this work ethylene glycol and toluene are used as solvents to break the azeotropic behavior of methylacetate-methanol mixture. Preparation of the methyl acetate-

methanol mixture is from methyl acetate 50% and methanol 50% in terms of volume/volume and the entrainers ethylene glycol and toluene dissolved in the mixture at different measurements of 5, 10, 15 and 20 ml. Table 5 indicates the separation performance of methyl acetate-methanol using ethylene glycol as an entrainer. The distillate composition is analyzed and the maximum recovery of methyl acetate component is found to 88.02 % when 20ml ethylene glycol is used as an entrainer. Subsequently the addition of 20ml of toluene point out the separation efficiency of 66.11 % it can be seen from the Table 6. Figure 5 represents the comparison of entrainers ethylene glycol and toluene. It is observed that ethylene glycol in the system shows better recovery of methyl acetate compared to toluene at different measurements. This can be due to the solvent ethylene glycol is a proton donor hence it is easily accessible with hydroxyl group present in methyl acetate. Furthermore undergoes strong hydrogen bonding interactions with methyl acetate and alters the relative volatility. When toluene is added with the mixture induced dipole moment exists between the molecules (Manojkumar and Sivaprakash, 2018a). Therefore interaction between the molecules of toluene with methyl acetate-methanol mixture is found to be weaker than ethylene glycol. From these results, it can be concluded that ethylene glycol is a more selective entrainer than toluene for the separation of methyl acetate-methanol by extractive distillation.

Table 5 Separation Performance of Methylacetate -Methanol system using Ethylene glycol

Ethylene glycol	Recovery Percentage in Distillate			Recovery Percentage in Bottom		
	Methylacetate	Methanol	Ethylene	Methylacetate	Methanol	Ethylene

(ml)			glycol			glycol
5	49.00	56.99	35.05	51.00	43.01	64.95
10	71.67	43.47	24.28	28.33	56.52	75.76
15	85.00	29.66	21.85	15.00	70.34	78.15
20	88.02	23.88	16.06	11.98	76.12	83.94

Table 6 Separation Performance of Methylacetate -Methanol system using Toluene

Toluene (ml)	Recovery Percentage in Distillate			Recovery Percentage in Bottom		
	Methylacetate	Methanol	Toluene	Methylacetate	Methanol	Toluene
5	36.15	58.96	40.99	63.85	41.04	59.01
10	51.82	49.06	50.28	48.17	50.94	49.72
15	63.46	37.82	60.93	36.54	62.18	39.07
20	66.11	32.83	67.12	33.89	67.17	32.88

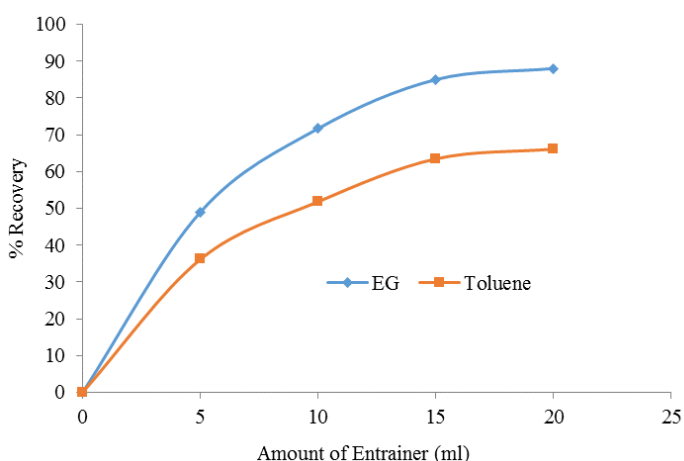


Fig. 5 Separation performance of Methylacetate - Methanol mixture using Ethylene glycol and Toluene

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