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### Comparison of heteroazeotropic and extractive distillation for the dehydration of propylene glycol methyl ether



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#### ABSTRACT

Separation section often is the most energy intensive section of an industrial chemical process, even so when there is an azeotropic mixture needed to be separated. In order to further save energy in a particular azeotropic separation system, various heat integration methods were proposed. However, because of more complex nature of these heat integration methods, the operation and control of such process may be hindered. Since there are various ways which can be used to separate a particular azeotropic mixture, this paper uses an industrial relevant propylene glycol methyl ether dehydration system as an example to demonstrate that selecting the most effective separation method is crucially important in attempting to save energy of the separation system. The drawback of using heterogeneous azeotropic distillation for this separation task can easily be revealed with process understanding. A more effective separation method via extractive distillation is proposed to significantly save steam cost by 39.7% and reduce the total annual cost by 32.7%. Simple feed-effluent heat exchanger can be added in the proposed extractive distillation system to further save energy without much complication of the process design flowsheet.

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#### 1. Introduction

Separation section often is the most energy intensive section of an industrial chemical process, even so when there is an azeotropic mixture needed to be separated. Simple regular distillation cannot be used to achieve complete separation. A recent book by Luyben and Chien (2010) summarizes the feasible ways used in industry to achieve such separation. Although for a particular azeotropic mixture, there may be more than one way to achieve the separation task. However, significant energy saving and reduction of the total annual cost of the separation process can easily be obtained if the most suitable separation method can be selected. In this paper, an industrial relevant propylene glycol methyl ether dehydration system will be used as an example to demonstrate the above claim. Propylene glycol methyl ether (PM) is widely used as a solvent and emulsifier in industrial and consumer products (Chiavone-Filho et al., 1993; Doan et al., 2009; Timofeeva et al., 2014). It can also be used to react with acetic acid via esterification reaction to produce propylene glycol methyl ether acetate, which is one of the most commonly used ester with a high industrial demand in surface coatings (Hsieh et al., 2006; Tochigi et al., 2007; Oh et al., 2015). In the above-mentioned usages, dehydration of PM is an important task in the separation process. PM and water formed a minimum-boiling azeotrope. This azeotropic mixture is customarily separated in industry via heterogeneous azeotropic distillation by adding a light entrainer.

In this paper, the drawback of using heterogeneous azeotropic distillation for this separation system can be

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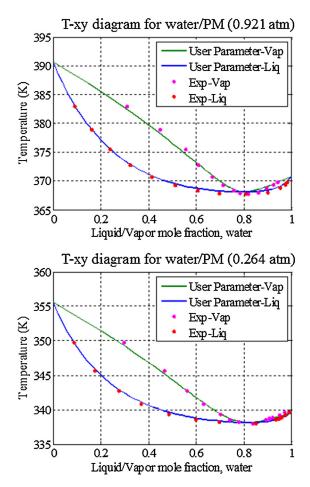
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revealed by simply drawing the material balance lines with the help of simulation tool giving the residual curve maps, liquid-liquid envelope and tie lines. Another design flowsheet via extractive distillation will be proposed for this separation task. By adding a suitable heavy entrainer into the separation system without introducing additional azeotrope, the relative volatility of the original mixture can be greatly enhanced, thus, a two-column system can be designed to separate PM and water. This ternary vapor-liquid equilibrium diagram belongs to a so-called 1.0-1a in Serafimov's classification (Serafimov, 1970). There are large numbers of paper in open literatures proposed to study this 1.0-1a extractive distillation system to separate azeotropic mixtures (e.g. Laroche et al., 1991; Arifin and Chien, 2008; Kossack et al., 2008; Luyben, 2008; Gutiérrez-Guerrz et al., 2009; Rodríguez-Donis et al., 2009; Hsu et al., 2010; Shen et al., 2013; Petlyuk et al., 2015). Some papers also described of adjusting operating pressure in the extractive-distillation system to improve separation efficiency (e.g. Luyben and Chien, 2010; Luo et al., 2014; You et al., 2015, 2016). However, to the best of our knowledge; none of the paper in open literature has investigated into this separation system. Other light or intermediate boiling entrainers may also be added (Laroche et al., 1991), however, a heavy entrainer is most common and will be proposed for this separation task.

## 2. Separation via heterogeneous azeotropic distillation

An industrial relevant separation problem is to obtain propylene glycol methyl ether (PM) from a diluted aqueous solution. The composition of the PM in the fresh feed stream is at



7.8 mol% with the rest to be water. The feed flow rate is assumed to be 1000 kmol/h. The desired product specification of PM is set to be 99.9 mol%.

For the following simulation studies conduct in this paper, The NRTL thermodynamic model is selected to describe the non-ideal vapor-liquid and vapor-liquid-liquid behavior in the studied system and the HOC model (Hayden and O'Connell, 1975) is used to account for non-ideality in vapor phase. The Aspen built-in NRTL parameters for the PM–water pair were not satisfactory to describe the VLE experimental data as published by Tochigi et al. (2007). Therefore, parameter regression was conducted to obtain the suitable NRTL parameters for this pair. It is shown in Fig. 1 that the T-xy plots at three operating pressures (93.3 kPa, 53.3 kPa and 26.7 kPa) are all predicted quite well using the obtained parameters. For other component pairs which do not have any experimental data, UNIFAC group contribution method was used to estimate the NRTL parameters. All NRTL parameters used in this paper are listed in Table 1.

In a particular chemical plant, isopropyl acetate (iPrAc) was used as a light entrainer for this separation system via heterogeneous azeotropic distillation. As shown in Fig. 2, the design flowsheet includes a preconcentrator column and a heterogeneous azeotropic distillation column. The purpose of the preconcentrator column is to remove portions of water in the fresh feed through column bottoms. The limiting distillate composition of this preconcentrator column will approach azeotropic composition is an important design variable to balance the costs associated with the preconcentrator column and the heterogeneous azeotropic column.

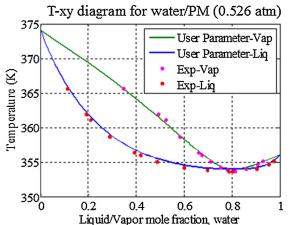


Fig. 1 - T-xy plots of PM-water system at various operating pressures.

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