

Design and control of acetic acid dehydration system via heterogeneous azeotropic distillation using *p*-xylene as an entrainer

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ABSTRACT

In the process of manufacturing terephthalic acid, very small amount of reactant *p*-xylene may enter into the feed stream of an acetic acid dehydration column. Traditionally, acetic esters were used as entrainers to separate acetic acid and water by heterogeneous azeotropic distillation (HAD). However, they can contaminate the process. In the study, the feasibility of using *p*-xylene as the entrainer is evaluated to eliminate the drawbacks of the HAD column using acetic esters as entrainers. An improved column is proposed for the separation of acetic acid and water in the presence of methyl acetate and tiny amount of *p*-xylene. Two configurations of the HAD column using *p*-xylene as the entrainer are designed by using different *p*-xylene purge strategies. The HAD column with a side stream to purge *p*-xylene accumulated in the column gives the most economical design.

A temperature control strategy is proposed for this HAD column to maintain acetic acid compositions in both the aqueous phase of the decanter and the column bottom. The controlled stage temperatures are chosen by singular value decomposition and closed-loop analysis methods. Dynamic simulation results reveal that the proposed control strategy can maintain product purities in spite of feed flow and feed composition changes.

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1. Introduction

In the process of manufacturing aromatic carboxylic acid (such as terephthalic acid or isophthalic acid), aromatic alkyl hydrocarbon such as *p*-xylene (PX) or *m*-xylene (MX) is oxidized with air in the presence of an acetic acid (HAc) solvent and a catalyst system including cobalt, manganese, and bromine. The products from the condensed liquid stream of the oxidation reactor and other dilute HAc streams, comprising mostly the oxidation by-products (methyl acetate (MA) and water), are fed to a dehydration unit to recover high-purity HAc and to remove the oxidation by-products. The recovered HAc is recycled back to the oxidation reactor for reuse as the solvent, while water and MA are sent to a wastewater treatment process. High investment and operating costs are required if conventional distillation is used in the HAc dehydration unit due to a tangent pinch on the pure-water end in the HAc/water system. An entrainer is often introduced into the unit in order to make the separation easier by using a heterogeneous azeotropic distillation (HAD) column.

The entrainer used before 1932 was ethylene dichloride. Now, the most generally used entrainers are acetic esters, such as

n-propyl acetate (NPA), *n*-butyl acetate (NBA), isobutyl acetate (IBA), and ethyl acetate (EA). Siirola [1] used EA as an entrainer to design a complete HAc dehydration process with multiple-effect azeotropic distillation and heat integration. Wasylkiewicz et al. [2] proposed using a geometric method for the optimum design of a HAc dehydration column with NBA as the entrainer. Chien et al. [3] studied the design and control of a HAc dehydration column using three candidate entrainers (EA, IBA, and NBA). A suitable entrainer of IBA was selected by total annual cost analysis. Later, Chien et al. [4] investigated the influence of PX feed impurity on the design and operation of an industrial HAc dehydration column. In normal operation, PX does not leave the column from the top or bottom product. It is accumulated in the decanter and the upper section of the column. Huang et al. [5] and Lee et al. [6] also addressed the design and control of the HAc dehydration column with PX or MX feed impurity. Wang et al. [7] proposed the energy-saving plant-wide design and control of a HAc dehydration system via HAD and divided wall distillation. MA is recovered from the column top of the divided wall distillation and can be recycled to the PX oxidation reactor to inhibit MA formation by the undesired decomposition reaction of HAc in the terephthalic acid production. The product containing entrainer and water is withdrawn from a side stream of the divided wall distillation column and recycled back to the decanter to compensate for the entrainer loss. In these studies, IBA was used as the entrainer. However, there are some drawbacks for the HAD column using IBA,

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Table 1
Comparison of azeotropic compositions and temperatures between experimental data and the data predicted by NRTL model.

Components	Experimental data		NRTL model		Azeotrope type
	Azeotropic composition (mol%)		Azeotropic composition (mol%)	Azeotropic temperature (°C)	
HAc–PX	(82.0, 18.0)	115.3	(80.7, 19.3)	115.0	Homogeneous
H ₂ O–IBA	(61.0, 39.0)	87.5	(63.1, 36.9)	87.6	Heterogeneous
H ₂ O–MA	(12.5, 87.5)	56.3	(11.2, 88.8)	56.2	Homogeneous
H ₂ O–PX	(74.5, 25.5)	92.0	(75.5, 24.5)	92.3	Heterogeneous
HAc–IBA ^a	(62.1, 37.9)	117.0 ^a	(55.3, 44.7)	117.1	Homogeneous
HAc–IBA ^a	(24.2, 75.8)	117.0 ^b	(24.6, 75.4)	117.1	Homogeneous

^a At 101.32 kPa.

^b At 101.13 kPa.

a foreign chemical in the process of producing terephthalic acid, as the entrainer. A small amount of IBA included in the streams of recovered HAc and MA is recycled back to the PX oxidation reactor and then decomposed to methanol, propanol, isobutanol, and carbon oxides. These decomposition products can further react with organic radicals in the reactor and produce a number of impurities, which contaminate process streams and terephthalic acid product. Unreacted decomposition compounds then enter into the HAD column along with the dilute HAc streams. They significantly influence the performance of the HAD column, causing poor separation and higher utility consumption.

To eliminate the drawbacks of the HAD column with acetic ester such as IBA as the entrainer, in the study, we investigate the possibility of using PX component in the feed stream of the HAD column as the entrainer to separate HAc and water. PX is a feedstock used for the production of aromatic carboxylic acid (terephthalic acid). An improved HAD column using PX as the entrainer is designed and a simple temperature control strategy is proposed to reject the disturbances of feed composition and feed rates. PX can form a heterogeneous azeotrope with water. It is more advantageous as an entrainer than IBA because of its higher amount of water entrained in the azeotrope. Consequently less energy is required for the separation of water and HAc. In the study, we will demonstrate that not only the drawbacks of the HAD column with IBA as the entrainer can be overcome but also less energy is used for the column with PX as the entrainer if PX accumulated in the column is withdrawn from the side stream.

2. Design of HAD columns

In this section, two HAD columns with IBA and PX, respectively, as entrainers are designed. Two configurations of the HAD column with different PX purge strategies are also given for the column using PX as the entrainer.

There are five components in the HAc dehydration system. The system is simulated by using ChemCad. Nonrandom two-liquid (NRTL) activity coefficient model was used for the vapor–liquid–liquid equilibrium of this system. Vapor association of HAc due to dimerization has also been included by using the second virial coefficient of the Hayden-O'Connell model [8] in the vapor phase. The ChemCad built-in association parameters were employed to compute fugacity coefficients. NRTL model parameter set for the system can be found in Wang et al. [7] except that the model parameters of HAc–IBA binary mixture are modified to account for the existence of double homogeneous azeotropes in the mixture by regressing the experimental data [9] of HAc–IBA binary vapor–liquid equilibrium. Table 1 shows the comparison of azeotropic compositions and temperatures for the system between experimental data [9,10] and the data predicted by the NRTL model. Six azeotropes, including four homogeneous azeotropes and two heterogeneous ones, may be found in the system. There

are only small differences between these two sets of data. Fig. 1 shows the residue curve maps with the binodal curve of the liquid–liquid equilibrium for HAc–water–IBA and HAc–water–PX

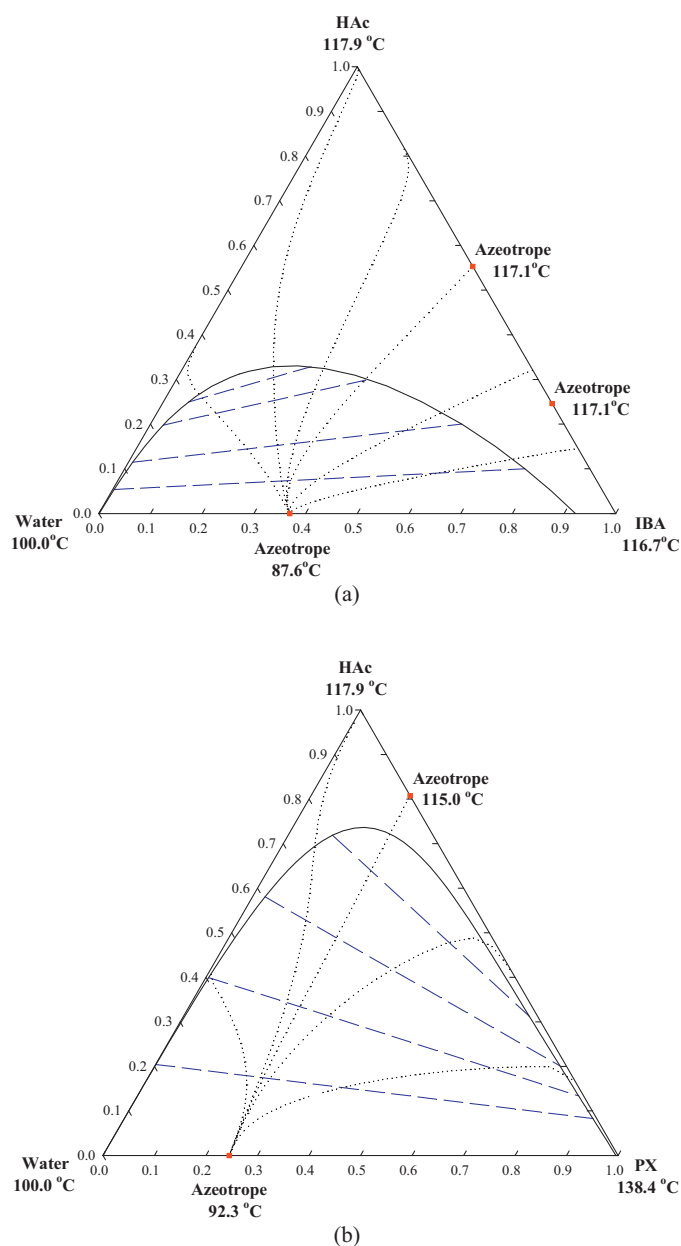


Fig. 1. Residue curve maps with the binodal curve of the liquid–liquid equilibrium for (a) HAc–water–IBA system and (b) HAc–water–PX system.

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