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Solvent Recovery for an Extraction Process

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ABSTRACT

The solvent recovery project involves the recovery of a valuable solute, acetic acid, from a spent solvent stream generated in a laboratory extraction experiment. The majority of this project's efforts center on the simulation of a ten-stage distillation column that will recover the acid. These simulation results will then be used to specify the ancillary process equipment needed for this distillation process.

INTRODUCTION

Description

The solvent recovery project represents a small part of the University of Missouri-Rolla Chemical Engineering Department's efforts to develop a zero-waste laboratory. This laboratory, when completed, will perform the multicomponent separation of an acetic acid/ethyl acetate/water system. The completed laboratory will accomplish the zero-waste goal by integrating three mass transfer unit operations - extraction, distillation, and adsorption. Since the separation of acetic acid from water represents the key step in the zero-waste laboratory, we have decided to focus our study on the distillation process used for this separation.

The relative volatility between acetic acid and water, especially at low acid concentrations, is very close to unity (less than 1.2 below acid mole fractions of 0.1). This makes the purification of the water-rich distillate extremely difficult. The presence of an entrainer, such as ethyl acetate, can potentially increase the separation between the acid and water and thus represents an avenue that we will explore in this paper.

Koch Industries, a manufacturer of a variety of mass transfer process equipment, has pledged a ten-stage, bubble-cap column for the acetic acid/water separation so our design is constrained. To achieve our desired goal of recycling both the acid and water, our simulation studies explore process variables, such as feed location, thermodynamic state of the feed, and reflux ratio subject to this ten-stage constraint. These results will aid in the design of ancillary process heat exchange equipment, such as the reboiler and condenser.

Rationale for Project

We have two main motivations for the zero-waste laboratory. The first is simply the cost associated with offering laboratories that use chemicals. The cost to purchase and dispose of chemical solvents continues to escalate without appreciable increases in the university operating budget to offset this cost. The fact that universities are now subject to substantial fines if they violate EPA regulations for the safe storage and disposal of chemicals only exacerbates the problem. Therefore, if the UMR Chemical Engineering Department is to offer meaningful laboratory courses that deal with real chemical systems, they must develop a program that reduces chemical waste.

The second motivation for the zero-waste laboratory centers on the development of educational experience that actively promotes the reduction of chemical waste in the laboratory. Three

approaches could be taken: the first would be a computer-simulated laboratory where the use of any chemicals is totally eliminated; the second would be a laboratory experience where only innocuous chemicals, such as water and air, are used; a third approach would be a laboratory where the chemicals are recycled. The first two alternatives seriously undermine the educational objectives of UMR's Chemical Engineering Department, which seeks to train engineers who can safely design chemical processes and conduct experimental chemical process research and development. Therefore, we have decided to handle both the health and safety issues and educational objectives by developing new laboratory experiments that process our chemical laboratory waste.

APPROACH

The major thrust of this project was the separation of acetic acid and water using a ten equilibrium stage distillation column. Figure I shows the vapor-liquid equilibrium for the acetic acid/water at 1 atm. These data were obtained from a flash calculation performed using the Aspen Plus simulation package and physical properties package SYSOP07D, which uses the UNIFAC liquid activity coefficient model and Hayden-O'Connell method to describe dimerizable acetic acid in the vapor phase. Figure I shows that at increasing dilute acid concentrations the relative volatility between the acid and water approaches unity.

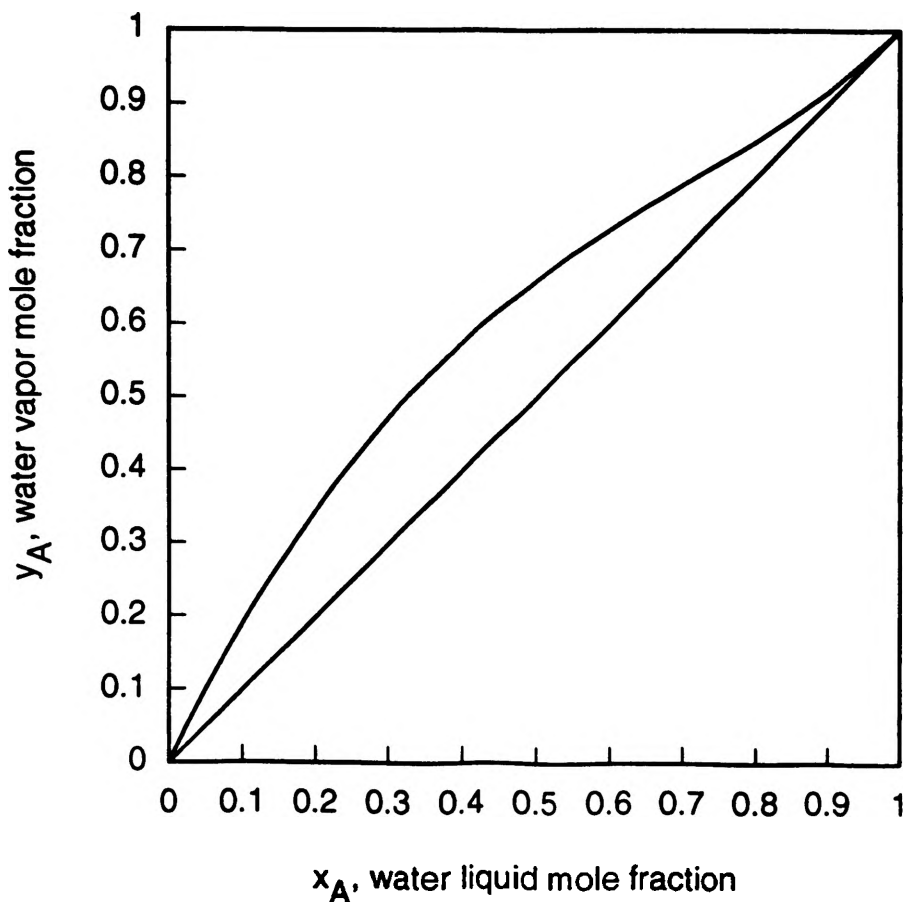


Figure I. Vapor-Liquid Equilibrium for Acetic Acid/Water at 1 atm

Table I shows this relationship more clearly.

Table I. Relative Volatilities Between Acetic Acid and Water at 1 atm as a function liquid water mole fraction. A represents water and B represents the acid.

x_A	α_{AB}
0.0	3.24
0.1	2.09
0.2	2.09
0.3	2.08
0.4	2.02
0.5	1.93
0.6	1.79
0.7	1.60
0.8	1.39
0.9	1.20
1.0	1.31

For the sake of simplicity, we decided to first study the separation of 50/50 mixtures of acetic acid and water and a feed flow rate of 100 lb mol/h. This allows us to establish base cases from we can compare our simulations. Eventually, we will simulate the column using acid mole fractions between 0.15 and 0.05, which more closely represent the waste solvent stream that the extraction process generates.

STUDIES COMPLETED WITH RESULTS

The first case studied involved the separation for a single-feed distillation column. We tested various feed locations to identify the optimum feed location. The optimum feed stage location corresponds to one that gives the highest degree of separation. For the case of 50/50 feed mixture, reflux ratio of four, and bottoms rate of 40 lb mol/h, the optimum feed location is the fourth tray. This case recovered 79 percent of the acid with a bottoms acid mole fraction of 0.9901. The optimum feed location was found to be dependent on the reflux ratio and draw ratio of the distillate and bottoms product. Because of this dependence, we chose a feed stage of four for our base case from which we have made our subsequent comparisons.

The second study tested the effect of an ethyl acetate entrainer at a modest reflux ratio of one. The presence of ethyl acetate dramatically increased the purity of acetic acid as the amount of entrainer mole fraction was increased from 0.0 to 0.2 while maintaining a 50/50 mole ratio of acid to water. We also adjusted the bottoms and distillate product draws to compensate for the increased amount of ethyl acetate. Table II summarizes the effect of the acetate entrainer.

A third case studied the effect of a two-enthalpy feed. Wankat and Kessler [1] suggested that a new method of distillation could increase the separation for a fixed number of stages and constant reflux ratio, especially for 50/50 mixtures. The method should also yield a reduction in energy demand because the reflux ratio can be reduced for the same desired separation as that of the single feed case. We tested the case of a liquid feed at stage five and a vapor feed at stage seven but saw no improvement in separation. As predicted by Wankat and Kessler, we did see a dramatic reduction in the reboiler duty. Table III provides detailed information on the two-enthalpy feed case study.

TABLE II. ENTRAINER CASE STUDY

	Feed Type	Feed Stage	Feed Flow (lbmol/hr)	Mole Frac. AA in Feed	Mole Frac. H2O in Feed	Mole Frac. EA in Feed	Distillate Flow (lbmol/hr)	Bottoms Flow (lbmol/hr)	Reflux Ratio	
1	Liquid	5	100	0.5000	0.5000	0.0000	50	50	1	
2	Liquid	5	100	0.4900	0.4900	0.0200	51	49	1	
3	Liquid	5	100	0.4750	0.4750	0.0500	52.5	47.5	1	
4	Liquid	5	100	0.4500	0.4500	0.1000	55	45	1	
5	Liquid	5	100	0.4000	0.4000	0.2000	60	40	1	
	Recovery Acetic Acid	Recovery H2O	Condenser Duty (Btu/hr)	Reboiler Duty (Btu/hr)	Mole Frac. AA in Distillate	Mole Frac. H2O in Distillate	Mole Frac. EA in Distillate	Mole Frac. AA in Bottoms	Mole Frac. H2O in Bottoms	Mole Frac. EA in Bottoms
1	0.7918	0.7918	-1654510	1671760	0.2081	0.7918	0.0000	0.7918	0.2081	0.0000
2	0.7710	0.7710	-1734360	1739960	0.1897	0.7710	0.0392	0.8025	0.1974	0.0000
3	0.7430	0.7716	-1784510	1791370	0.1903	0.7716	0.0361	0.8212	0.1787	0.0000
4	0.6993	0.7720	-1867740	1877130	0.1915	0.7720	0.0364	0.8547	0.1452	0.0000
5	0.6185	0.7665	-2029670	2045870	0.1661	0.7665	0.0333	0.9277	0.0722	0.0000

TABLE III. TWO FEED EFFECTS ON SEPARATION AND ENERGY REQUIREMENTS

	Feed1 Type	Feed1 Stage	Feed1 Flow (lbmol/hr)	Feed2 Type	Feed2 Stage	Feed2 Flow (lbmol/hr)	Mole Frac. A in Feed	Mole Frac. H2O in Feed	Mole Frac. EA in Feed	Distillate Flow (lbmol/hr)	Bottoms Flow (lbmol/hr)	Reflux Ratio
1	Liquid	5	100	---	---	---	0.5000	0.5000	0.0000	50	50	4
2	Liquid	5	100	---	---	---	0.5000	0.5000	0.0000	50	50	2
3	Liquid	5	100	---	---	---	0.5000	0.5000	0.0000	50	50	1
4	Liquid	5	50	Vapor	7	50	0.5000	0.5000	0.0000	50	50	4
5	Liquid	5	50	Vapor	7	50	0.5000	0.5000	0.0000	50	50	2
6	Liquid	5	50	Vapor	7	50	0.5000	0.5000	0.0000	50	50	1
	Recovery Acetic Acid	Recovery H2O	Condenser Duty (Btu/hr)	Reboiler Duty (Btu/hr)	Mole Frac. AA in Distillate	Mole Frac. H2O in Distillate	Mole Frac. E in Distillate	Mole Frac. AA in Bottoms	Mole Frac. H2 in Bottoms	Mole Frac. EA in Bottoms		
1	0.8707	0.8707	-4254060	4282620	0.1292	0.8707	0.0000	0.8707	0.1292	0.0000		
2	0.8369	0.8369	-2524810	2548410	0.1610	0.8369	0.0000	0.8369	0.1610	0.0000		
3	0.7918	0.7918	-1654510	1671760	0.2081	0.7918	0.0000	0.7918	0.2081	0.0000		
4	0.8724	0.8724	-4256790	3557560	0.1275	0.8724	0.0000	0.8724	0.1275	0.0000		
5	0.8345	0.8345	-2520920	1815770	0.1854	0.8345	0.0000	0.8345	0.1854	0.0000		
6	0.7713	0.7713	-1841560	928207	0.2266	0.7713	0.0000	0.7713	0.2266	0.0000		

CONCLUSIONS

Our work is still very preliminary but we can conclude that the presence of an entrainer substantially improves the acid purity. Also we have reduced the reboiler duty by using a two-enthalpy feed. This method does not necessarily reduce that overall energy demand of the distillation process but it does reduce the temperature at which the energy must be supplied.

FUTURE PLANS

In this work, we used the Aspen Plus properties option set SYSOP07D to estimate the vapor-liquid equilibrium (VLE) for the acetic acid/water/ethyl acetate system. We feel that the description of the ternary VLE can be improved by fitting the experimental binary VLE data [2] to the

UNIQUAC liquid activity coefficient model. Other studies we feel warrant further investigation are:

- the effect of the two-enthalpy feed method at lower acetic acid feed concentrations, and
- the effect of entrainer at lower acetic acid feed concentrations

We hope to achieve substantial improvements in the acetic acid purity over what would result from the standard single feed column.

NOMENCLATURE

AA - Acetic Acid
H₂O - Water
EA - Ethyl Acetate

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