Extractive Distillation of Acetonitrile/Water/Isopropanol mixture using Ethylene Glycol

Kumaran Nagappan K
National Institute of Technology Tiruchirappalli

Background & Description:

Acetonitrile is organic solvent used in refineries, pharmaceutical and electrochemical industries. Isopropanol is an important solvent in pharmaceutical and chemical industry and also used as cleaning agent, intermediate compound in organic synthesis. But separation of acetonitrile and isopropanol mixture after manufacturing processes is difficult because of azeotrope formation. The complexity of process increases when water is present in the mixture which results in formation of three pairs of binary azeotropes and one pair of ternary azeotrope. Hence, extractive distillation is used to separate the mixture. Extractive distillation uses an additional agent called entrainer to break the azeotropes and separate the mixture. In the present study, ethylene glycol is used as an entrainer.

The feed consisting of 30% acetonitrile, 30% isopropanol and 40% water is preheated to 51 ^{o}C. Recycled entrainer (ethylene glycol) at 45 ^{o}C enters the column along with preheated feed at stage 5 and 39 respectively. The first extractive distillation column (EDC-01) operating at 0.35 bar separates acetonitrile as distillate. The bottoms from EDC-01 is heated to 100 ^{o}C and fed to the second extractive distillation column (EDC-02) at stage 31. Recycled ethylene glycol is fed at 75 ^{o}C at stage 5 of EDC-02. The distillate from EDC-02 is enriched with isopropanol. The bottoms are fed to entrainer recovery column (ERC) where water and ethylene glycol is separated in distillate and bottoms respectively. The recovered ethylene glycol is split, cooled and recycled to the two extractive distillation columns.

Flowsheet:

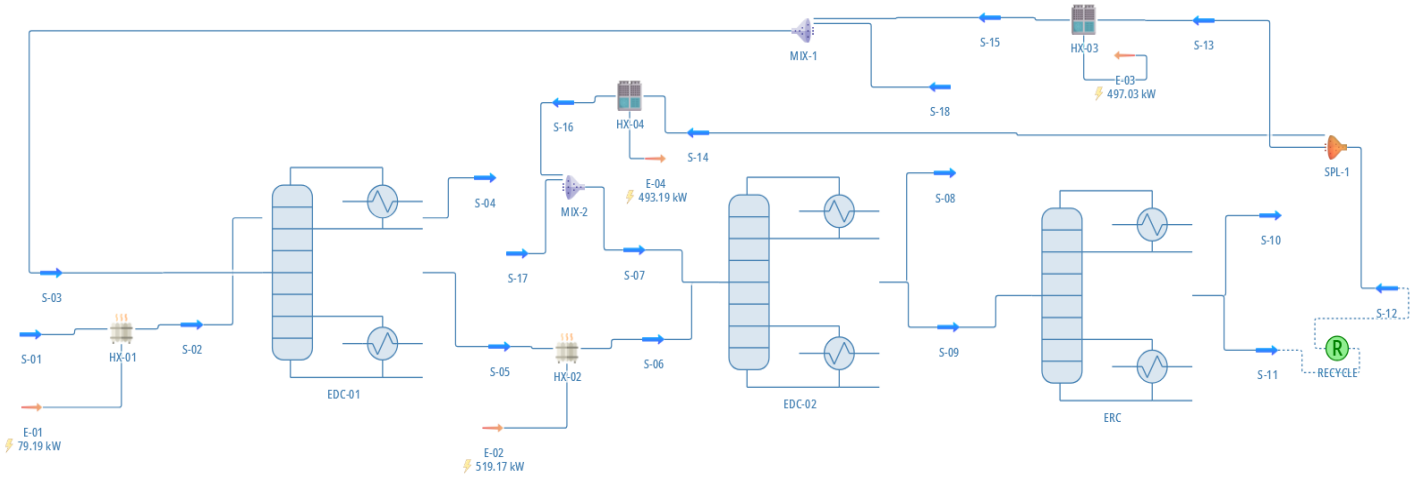

Fig. 1: Flowsheet of extractive distillation process for separation of acetonitrile, isopropanol and water

Results:

The following table shows the properties of streams obtained from DWSIM simulation.

Table 1: DWSIM Simulation Results

Property	S-04	S-08	S-10	S-11	Units
Temperature	50.416	82.420	99.996	196.496	°C
Pressure	0.350	1.000	1.000	1.000	bar
Mass Flow	1216.210	1833.990	742.059	12066.000	kg/h
Molar Flow	29.600	31.400	39.600	194.400	kmol/h
Molar Fraction (Mixture) / Acetonitrile	0.998	0.014	1.16E-04	7.01E-15	-
Molar Fraction (Mixture) / Isopropanol	0.002	0.952	0.001	3.54E-17	-
Molar Fraction (Mixture) / Water	3.84E-13	0.034	0.983	7.66E-11	-
Molar Fraction (Mixture) / Ethylene glycol	1.23E-12	6.66E-10	0.015	1.000	-

The simulated results are compared with ASPEN Plus simulation results reported in Y. Li et al. (2021). The compared results are shown in Table 2. The results from both the software agree with each other.

Table 2 Comparison of DWSIM and ASPEN results

Property	S-04		S-08		S-10		S-11		Units
	DWSIM	ASPEN	DWSIM	ASPEN	DWSIM	ASPEN	DWSIM	ASPEN	
Temperature	50.416	50.900	82.420	81.700	99.996	99.500	196.496	197.900	°C
Molar Flow	29.600	30.000	31.400	30.000	39.600	40.000	194.400	195.000	kmol/h
Molar Fraction (Mixture) / Acetonitrile	0.998	0.999	0.014	8.40E-05	1.16E-04	9.72E-04	7.01E-15	1.14E-04	-
Molar Fraction (Mixture) / Isopropanol	0.002	7.31E-04	0.952	0.999	0.001	1.70E-05	3.54E-17	3.00E-06	-
Molar Fraction (Mixture) / Water	3.84E-13	2.00E-06	0.034	1.00E-06	0.983	0.999	7.66E-11	1.80E-05	-
Molar Fraction (Mixture) / Ethylene glycol	1.23E-12	1.11E-04	6.66E-10	4.29E-04	0.015	7.80E-05	1.000	0.9998	-

Thermodynamics:

NRTL property package is used for calculation of material streams. The columns are simulated using DECHEMA / Ideal Gas Law / NRTL / Antoine / Excess thermodynamic property settings for calculation of K-value / Equation of state / Activity coefficient / Vapour pressure / Enthalpy. NRTL property package is chosen for the simulation study as its results agree well with experimental data and found suitable in predicting Vapour-Liquid equilibrium data as shown by Y. Li et al. (2021).

System of Units:

Temperature	-	ºC
Pressure	-	bar
Mass flow	-	kg/hr
Molar flow	-	kmol/hr

Conclusions and Recommendations

The extractive distillation column process for separation ternary mixture is successfully simulated using DWSIM and found to agree with the results reported in literature. Further work can be done on improving the purity of isopropanol from the second extractive distillation column.

References:

1. https://en.wikipedia.org/wiki/Acetonitrile
2. https://en.wikipedia.org/wiki/Isopropyl_alcohol#Uses
3. Li, Yudong, et al. "Energy-efficient extractive distillation combined with heat-integrated and intermediate reboilers for separating acetonitrile/isopropanol/water mixture." *Separation and Purification Technology* 262 (2021): 118343.