DWSIM Flowsheet Development for Extractive Distillation separation of Ethanol-Ethyl Acetate mixture using Furfural

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Introduction

The boiling point of Ethanol (78.37^{0} C) and Ethyl Acetate (77.1^{0} C) are close and thus providing Ethanol-Ethyl Acetate mixture with a low value of relative volatility. Consequently, simple distillation is inadequate to separate the compounds and extractive distillation is preferred. While there are a wide variety of extractants (Diethylene glycol, Dimethylsulfoxide, Glycerine) [1], for the given problem statement, the process is developed with Furfural as the extractant. The aim is to re-model the existing process flowsheet in DWSIM open source process simulator and reproduce the results effectively.

(This flowsheet development is in accordance for the final round of NCCPS-2018 Online quiz)

Methodology

The saturated feed is the Ethanol-Ethyl Acetate (0.75:0.25 mol%) at 300 kmol/hr is introduced into the Preconcentration Column (PC) to concentrate the mixture. Subsequently, the concentrated distillate from PC and the extractant Furfural are processed into the Extractive Distillation Column (EDC). Ethanol being the light key component is distilled at EDC. The bottom with Furfural and Ethyl Acetate are treated in the Entrainer Recovery Column (ERC) to recover Ethyl Acetate as the distillate. The Furfural is recycled to EDC to complete the entrainer recycle loop. The process re-modelled in DWSIM is shown in Figure 1.

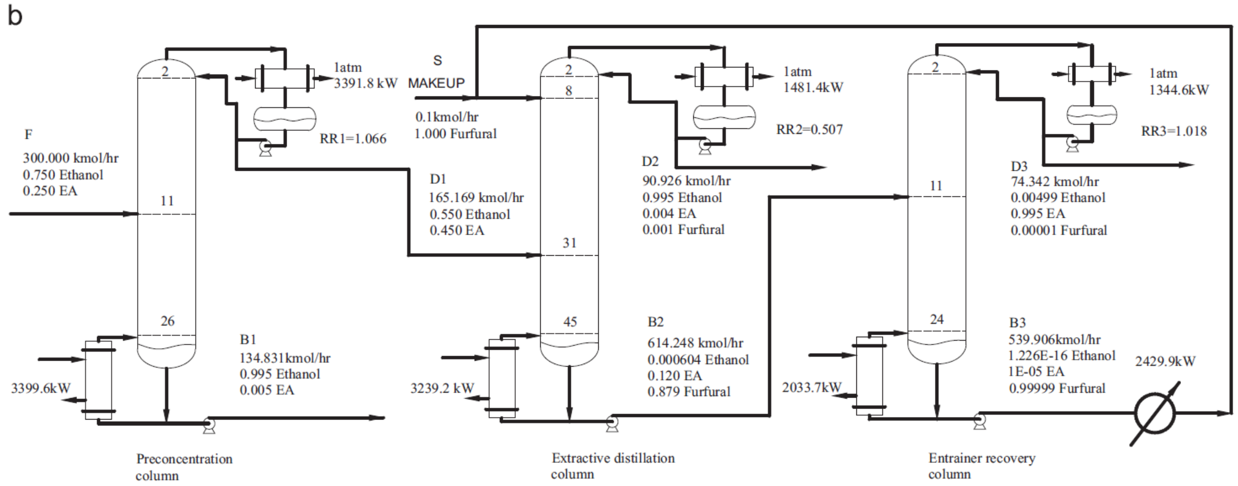

Figure 1 Reference flowsheet for Extractive Distillation of Ethanol-Ethyl Acetate using Furfural

Solutions on Challenges encountered

Though the process is well defined, a considerable amount of limitations is encountered in developing the flowsheet in DWSIM program. Predominantly, NRTL or UNIQUAC are preferred for polar chemicals. With the interaction parameters not well defined for the Ethanol/Furfural system, we fall back on UNIFAC for material streams. Similarly, the distillation columns are developed using CAPE-OPEN (CO) Unit blocks since the standard DWSIM distillation blocks failed at rigorous solving.

In CO-Distillation blocks, the process calculations failed to converge in NRTL/UNIQUAC and, thus UNIFAC is preferred. Nevertheless, for a given reflux ratio and bottoms flow rate (Standard ChemSep recommendations for distillation column design), the recovery yield is quite low for Ethanol and Ethyl Acetate when compared to the desired values (~99.9%). The problem should be primarily due to the Thermodynamic Package selected. Rigorous trail basis testing of different thermodynamic packages is performed to identify the optimum combination, but most of them resulted either with non-convergence issues or in low recovery yield compared to UNIFAC. Thus, currently, UNIFAC is chosen to be optimum for the problem statement.

Nonetheless, to achieve better recovery yields for the components, the process has been improvised. The temperature of the feeds into the columns are regulated to 25^0 C to increase the separation of the components by thermodynamics. Also, reducing feed temperatures conversely change the heat duty of reboiler and condenser. The temperature is regulated via cooler equipment. Moreover finally, to account for the saturated conditions for the material streams, the total stream pressure is reduced to 25^0 C.

Results and Discussions

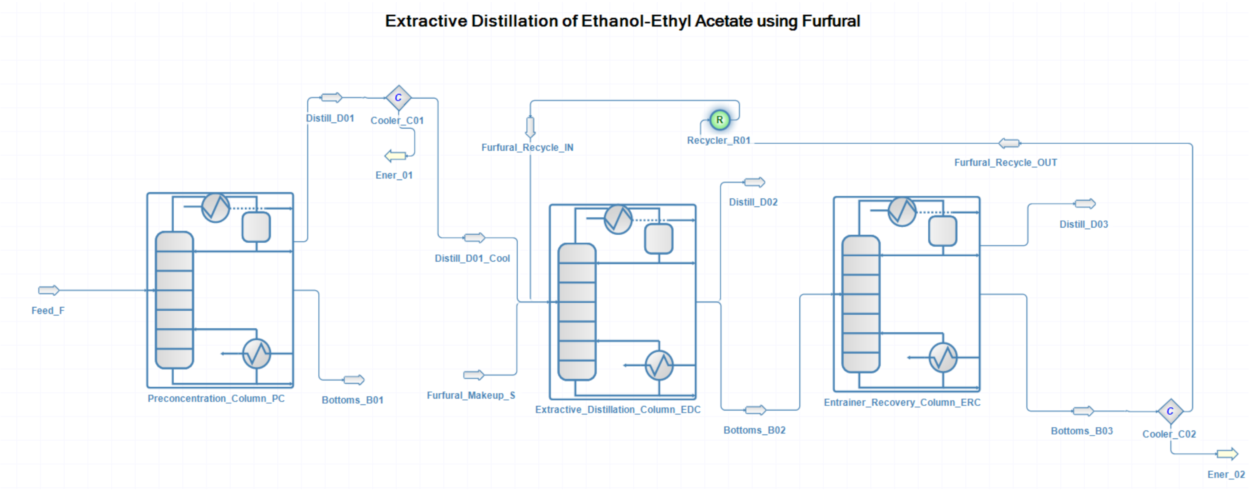

Figure 2 DWSIM Flowsheet for Extractive Distillation of Ethanol-Ethyl Acetate using Furfural

The complete DWSIM flowsheet is shown in Figure 2. It can be noticed on the introduction of two cooler equipment one after the PC and the other after ERC.

From the results in Table 1, the recovery yields of Ethanol and Ethyl Acetate are computed to be 96.59% and 91.74% respectively. Though the recovery yields are still relatively low when compared to the desired values, the range tends to be acceptable. However, with much accurate thermodynamic dataset and package, there should be no necessity of improvisation of the process.

Table 1 DWSIM Flowsheet Results

Object	Feed	Bottoms_B01	Distill_D02	Distill_D03	Furfural_Recycle_OUT
Temperature (^{0}C)	25	78.57	77.41	75.78	25
Pressure (atm)	0.1211	1	1	1	1
Mass Flow (kg/s)	4.715	1.9578	0.9629	1.7953	14.456
Molar Flow (kmol/hr)	300	152.295	73.1973	74.552	541.625
Mole Fraction (-)					
Ethanol	0.75	0.995	0.9695	0.0336	1.54E-13
Ethyl Acetate	0.25	0.005	0.0299	0.9664	1E-05
Furfural	0	0	0.0006	2.45E-07	0.9999

Conclusion

The flowsheet for the proposed Extractive Distillation process of Ethanol-Ethyl Acetate using Furfural is constructed in DWSIM software. Thermodynamic package limitations are encountered, and UNIFAC is chosen to be the most optimum package to handle the system via rigorous trail basis evaluations. However, the recovery yields of the components achieved were low when compared to the desired values. Thus, the process is modified by reducing the temperature of different material streams to 25^0 C. This in turn varies the condenser and reboiler load but results in new recovery yields of Ethanol and Ethyl Acetate are at 96.59% and 91.74% which are relatively close to the desired values. The process development can be improved with better thermodynamic packages or different solving techniques.

Reference

1. Separation of ethyl acetate from ethanol and water by extractive distillation, US06363638, 1982-03-30