용매를 이용한 IPA 탈수를 위한 추출증류공정에서 용매로써 EG과 DMSO에 대한 2기 증류배열과 3기 증류배열 사이의 에너지 소비 비교

조 정호

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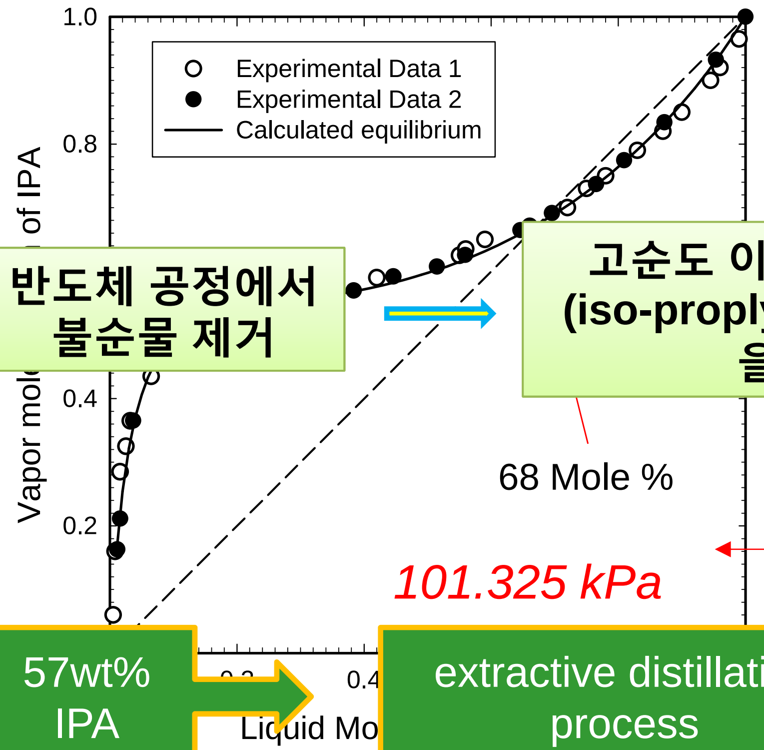
화학공정연구실

목차

Introduction



반도체 공정에서 불순물 제거

고순도 이소프로필알콜 (iso-proply alcohol, IPA) 을 사용

많은 양의 IPA 폐 수용액이 발생

IPA / Water System

...Water는 68mole%(88wt%) 근처에서 공비점을 형성합니다

Using DMSO or EG as a Solvent

57wt% IPA

extractive distillation process

High purity IPA

Reference :
Experimental data 1 : Wilson, A., and E. L. Simons. Ind. Eng. Chem., vol 44 pp. 2214-2219, 1952
Experimental data 1 : Davalloo P., Iran. J. Sci. Technol., 1,279(1971).

Feedstock Conditions

Stream Number	1	
Stream Name	**Feed**	
Temperature (℃)	50	
Pressure (kPa)	250.00	
Vapor Mole Fraction	0.00	
Total Molar Rate (kmol/hr)	99.83	
Total Mass Rate (kg/hr)	**3,000.00**	
	kg/hr	wt%
IPA	1,716.00	**57.2**
WATER	1,284.00	**42.8**

Product Specifications & Utility Conditions

Product Specifications	Value
IPA Purity	> 99.70 wt%
IPA Yield	> 99%
Solvent Content in IPA Product	< 1ppm by weight
IPA Content in Waste Water	< 500 ppm by weight
Utility Conditions	**Value**
MP Steam	180 ℃ Saturated
Cooling water supply & return temperature	32 ℃ / 40 ℃

Proper Thermodynamic Model Selection

- The selection of proper thermodynamic model is very important to the design of highly non-ideal system.

$$\ln \gamma_i = \frac{\sum_j \tau_{ji} G_{ji} x_j}{\sum_k G_{ki} x_k} + \sum_j \frac{x_j G_{ij}}{\sum_k G_{kj} x_k} \left[\tau_{ij} - \frac{\sum_l x_l \tau_{lj} G_{lj}}{\sum_k G_{kj} x_k} \right]$$

$$\tau_{ij} = a_{ij} + \frac{b_{ij}}{T}$$

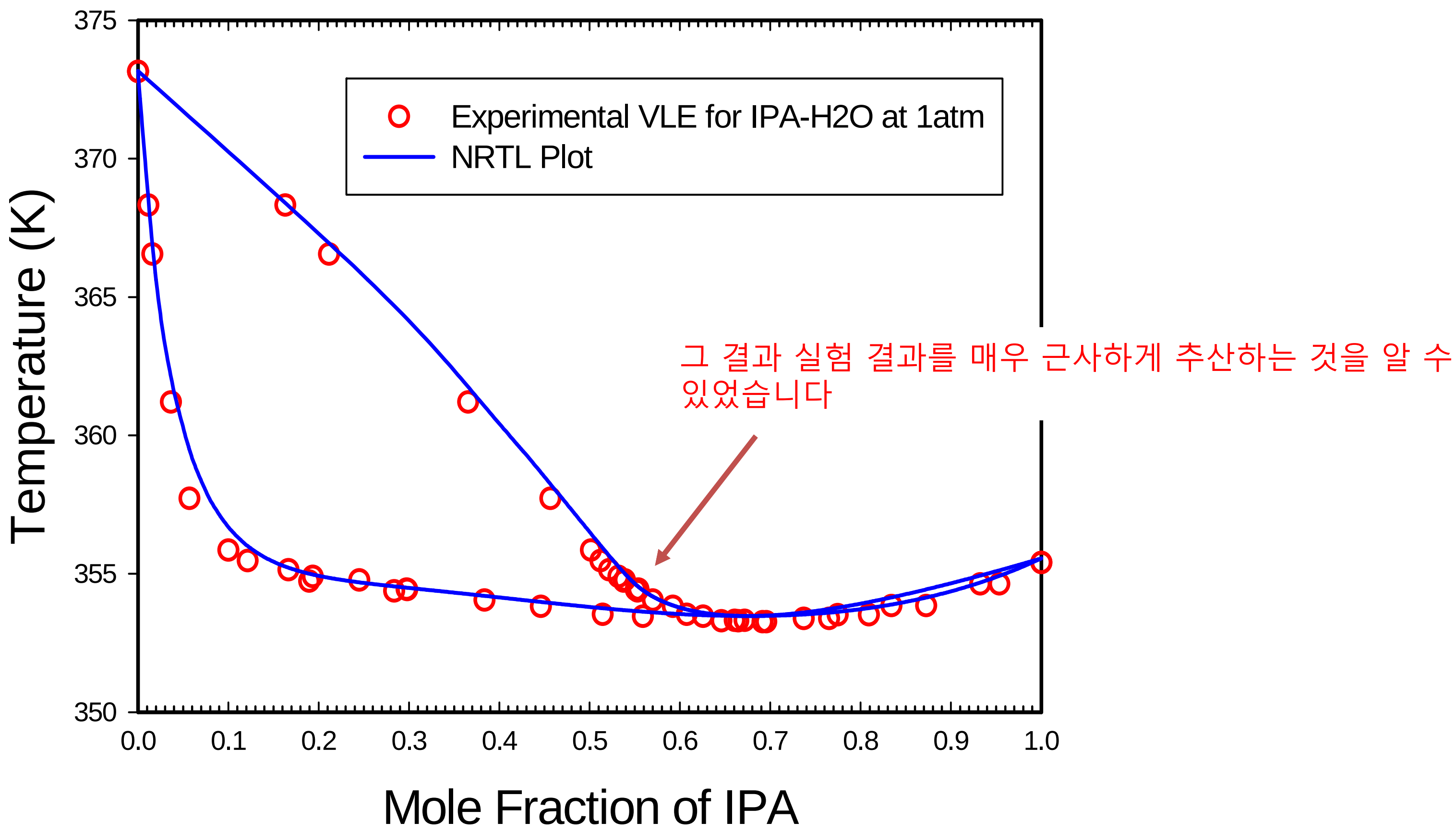
$$G_{ij} = \exp\left(-\alpha_{ij} \tau_{ij}\right)$$

NRTL Binary Interaction Parameters

I	J	a_{ij} a_{ji}	b_{ij} b_{ji}	c_{ij} c_{ji}	α	Units
IPA	H_2O	0.753	-267.132	0	0.3111	K
		1.059	461.719	0		
IPA	DMSO	0.000	697.088	0	0.3000	K
		0.000	-305.335	0		
IPA	EG	0.000	-751.991	0	0.0467	K
		0.000	1158.580	0		
H_2O	DMSO	0.000	1203.770	0	0.6615	K
		0.000	-524.821	0		
H_2O	EG	0.000	1888.750	0	0.0773	K
		0.000	-1445.470	0		
DMSO	EG	0.000	429.425	0	0.3024	K
		0.000	-635.761	0		

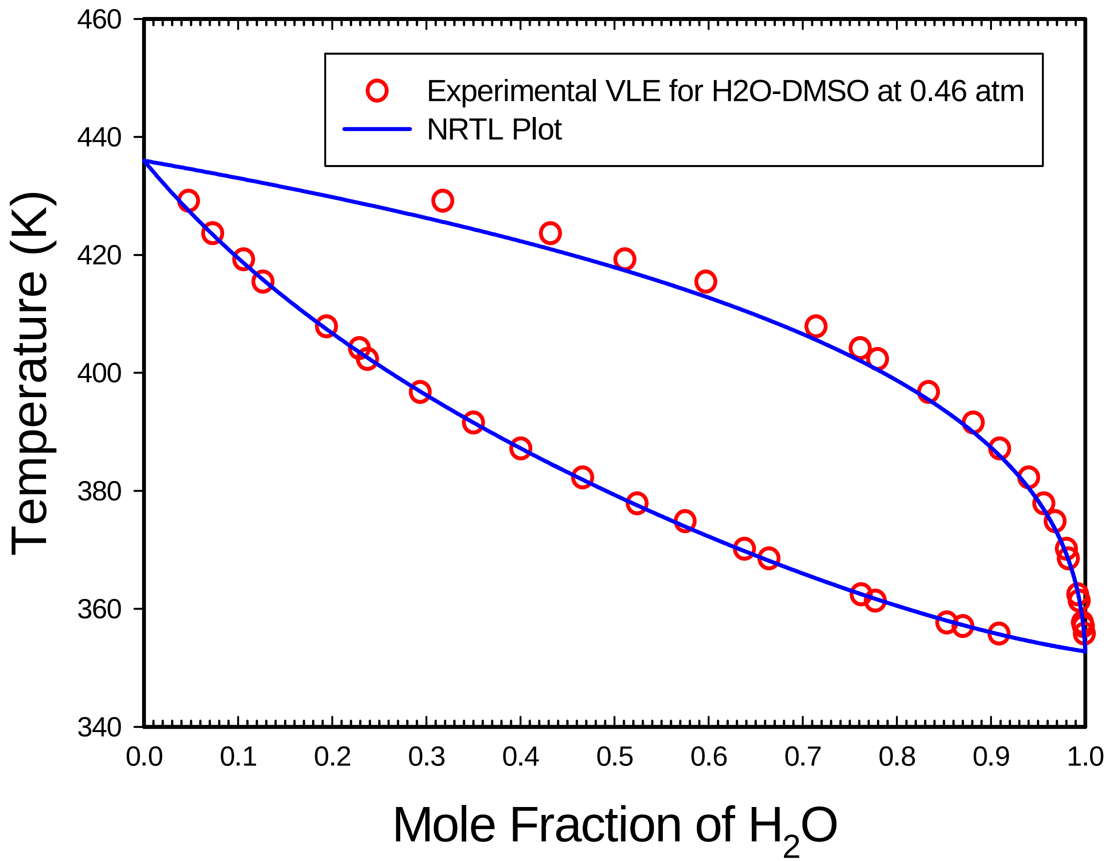
Experimental VLE for IPA-H_2O and NRTL Plot

- Experimental data at 1 atm for IPA-water binary system and its prediction with NRTL model



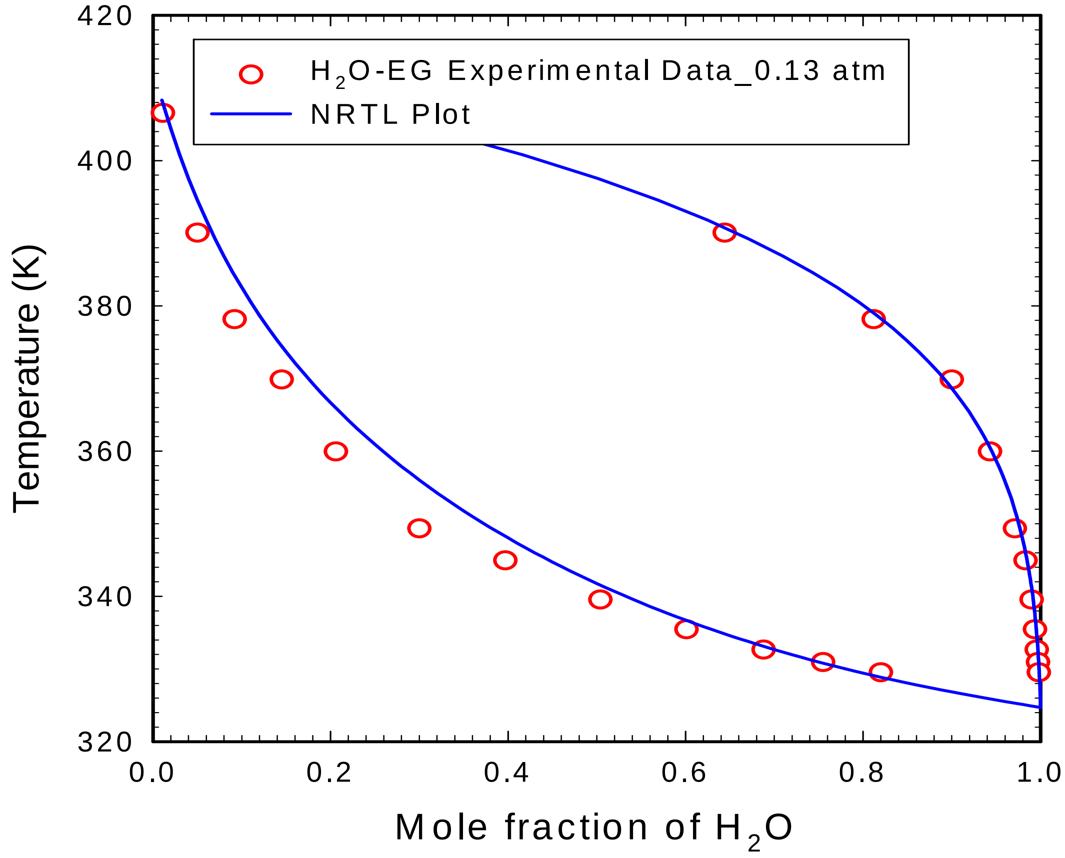
Experimental VLE for H_2O-DMSO and NRTL Plot

- Experimental data at 0.46 atm for Water-DMSO binary system and its prediction with NRTL model



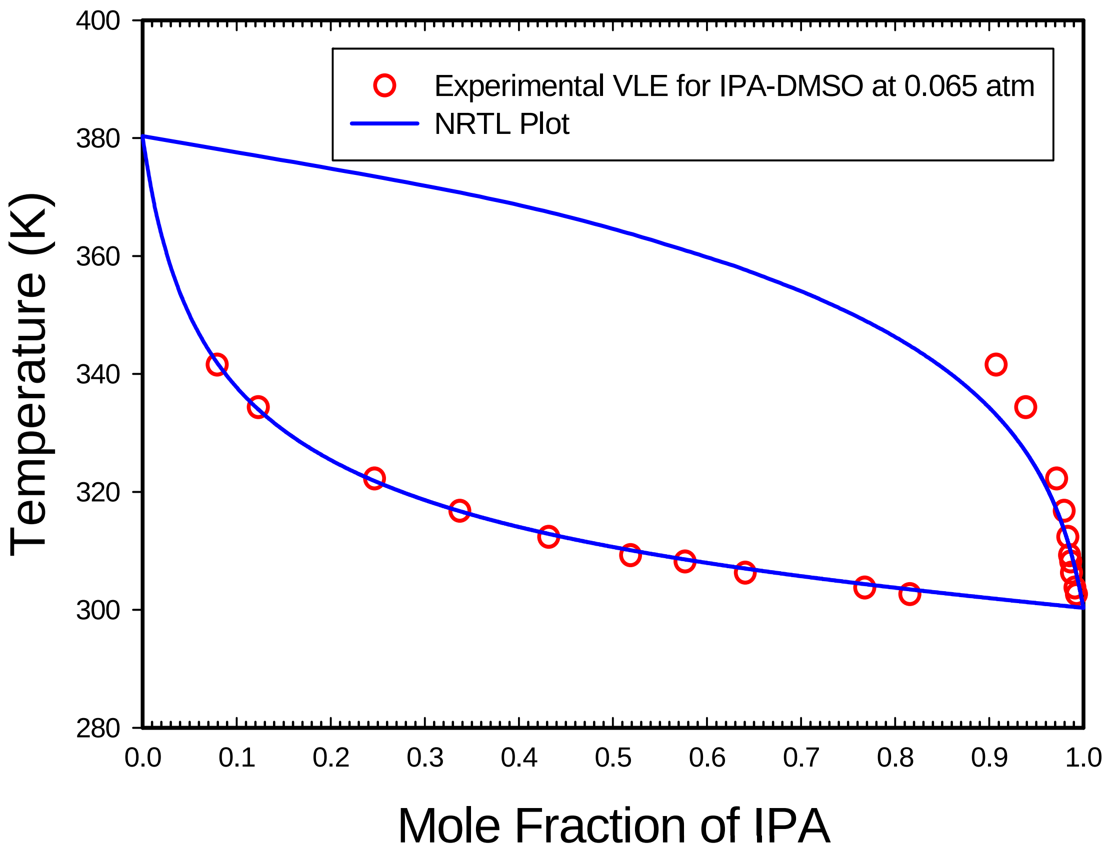
Experimental VLE for H_2O-EG and NRTL Plot

- Experimental data at 0.13 atm for Water-EG binary system and its prediction with NRTL model



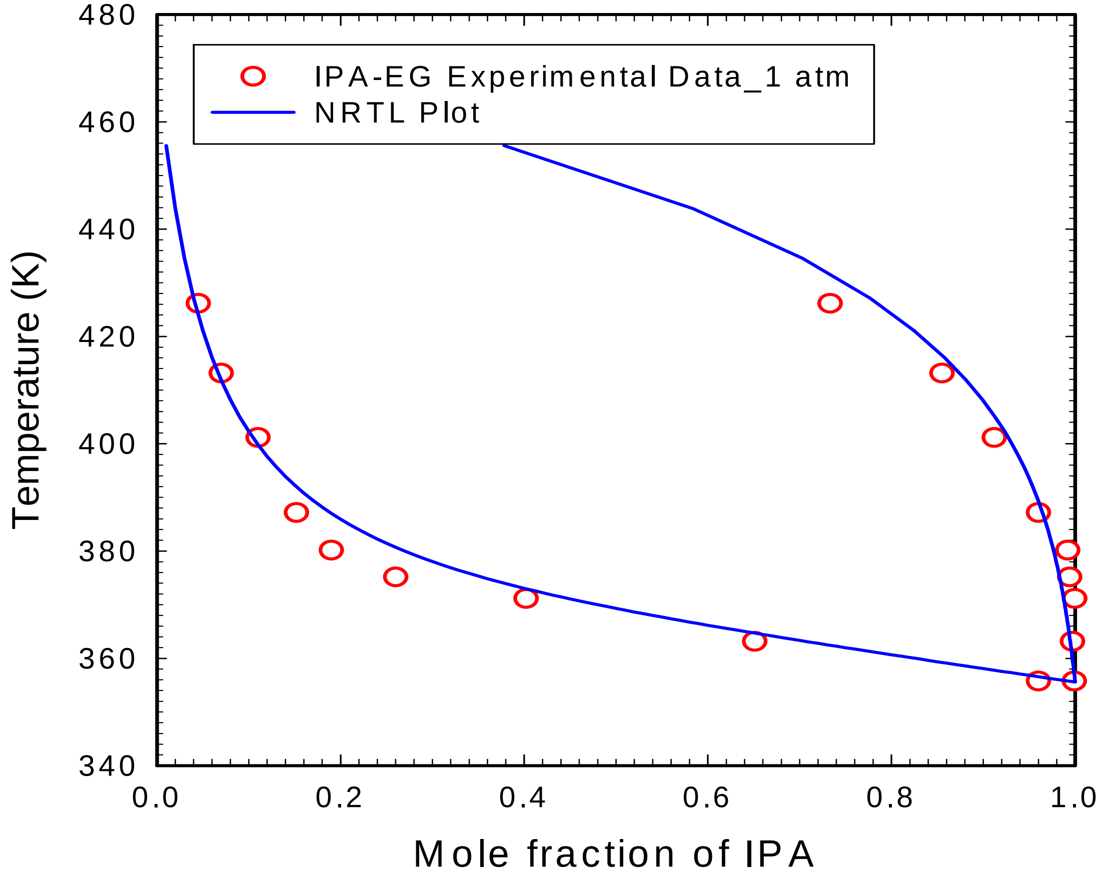
Experimental VLE for IPA-DMSO and NRTL Plot

- Experimental data at 0.065 atm for IPA-DMSO binary system and its prediction with NRTL model

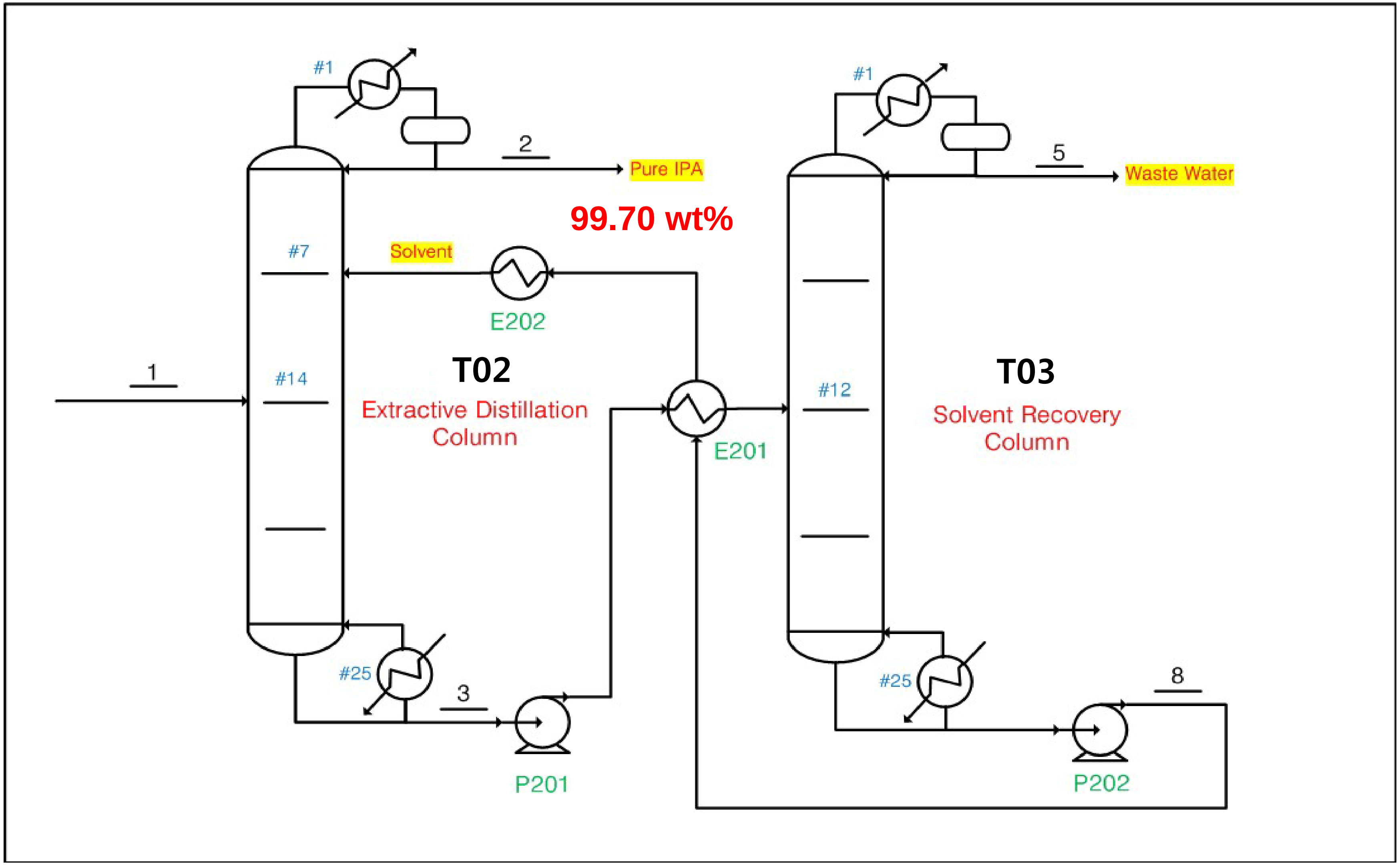


Experimental VLE for IPA-EG and NRTL Plot

- Experimental data at 1 atm for IPA-EG binary system and its prediction with NRTL model

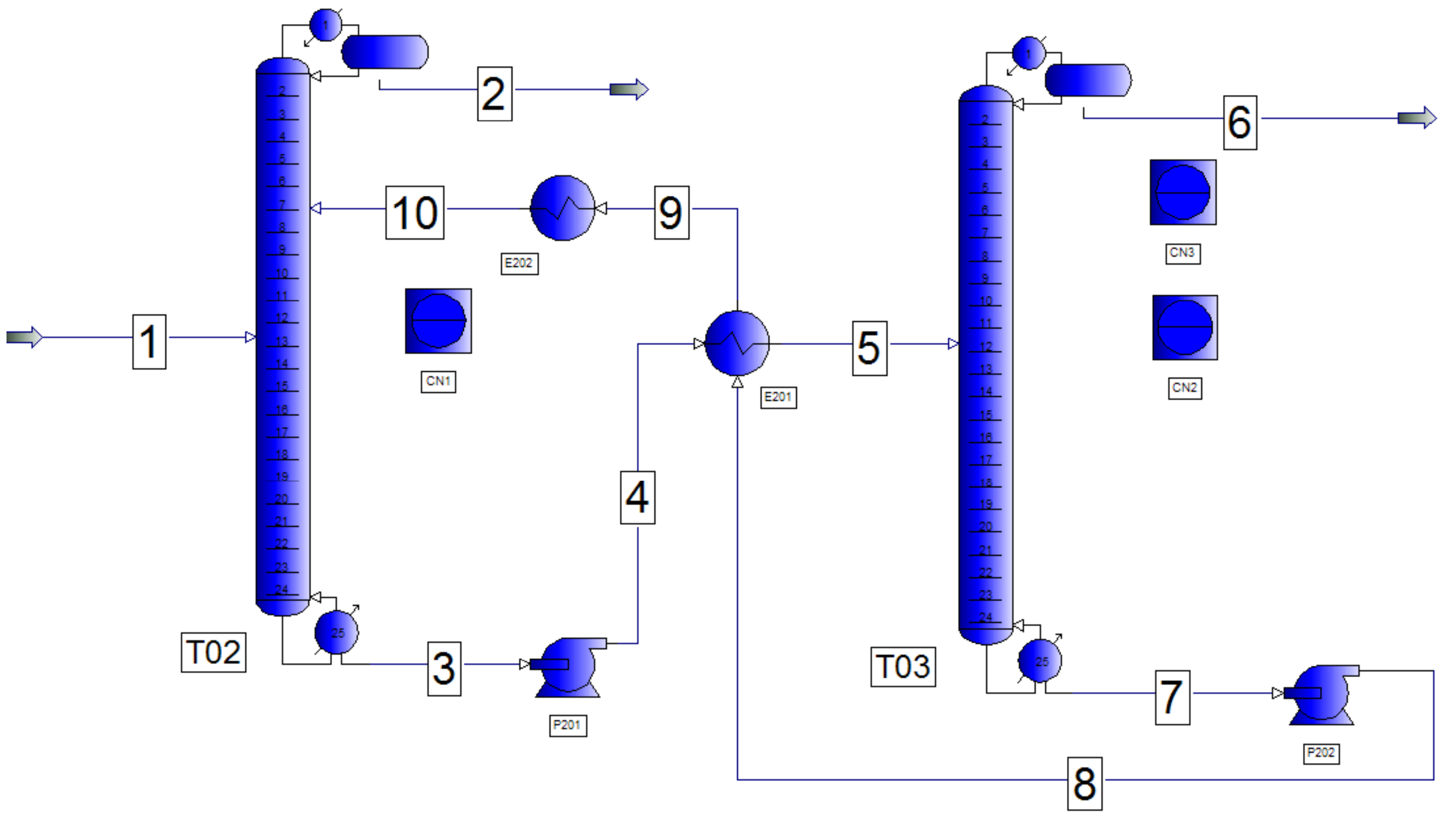


IPA Dehydration Using Two-Column Configuration



IPA Dehydration Using Two-Column Configuration

- Flow sheet drawing for a **two-column** configuration using PRO/II with PROVISION version 9.1



Two-column Stream Summary (EG)

Stream Number	1	2	6	8
Stream Name	T02 Feed	T02 Top (IPA Product)	T03 Top (Waste Water)	T03 Bottom (Solvent)
Temperature (℃)	20.00	45.00	55.22	**150.06**
Pressure (kPa)	250.00	103.00	16.00	250.00
Vapor Mole Fraction	0.00	0.00	0.00	0.00
Total Molar Rate kmol/hr	99.83	28.83	71.00	108.99
Total Mass Rate kg/hr	3,000.00	1,720.24	1,279.76	6,763.11
Comp. Flow Rate (kg/hr)	**kg/hr**	**kg/hr**	**kg/hr**	**kg/hr**
IPA	1,716.00	**1,716.00**	0.40	0.00
WATER	1,284.00	5.16	1,278.84	0.68
EG	0.00	0.00	0.00	6,762.44
Comp. Composition (wt%)	**wt%**	**wt%**	**wt%**	**wt%**
IPA	57.20	**99.70**	**0.03**	0.00
WATER	42.80	0.30	99.97	0.01
EG	0.00	0.00	0.00	99.99

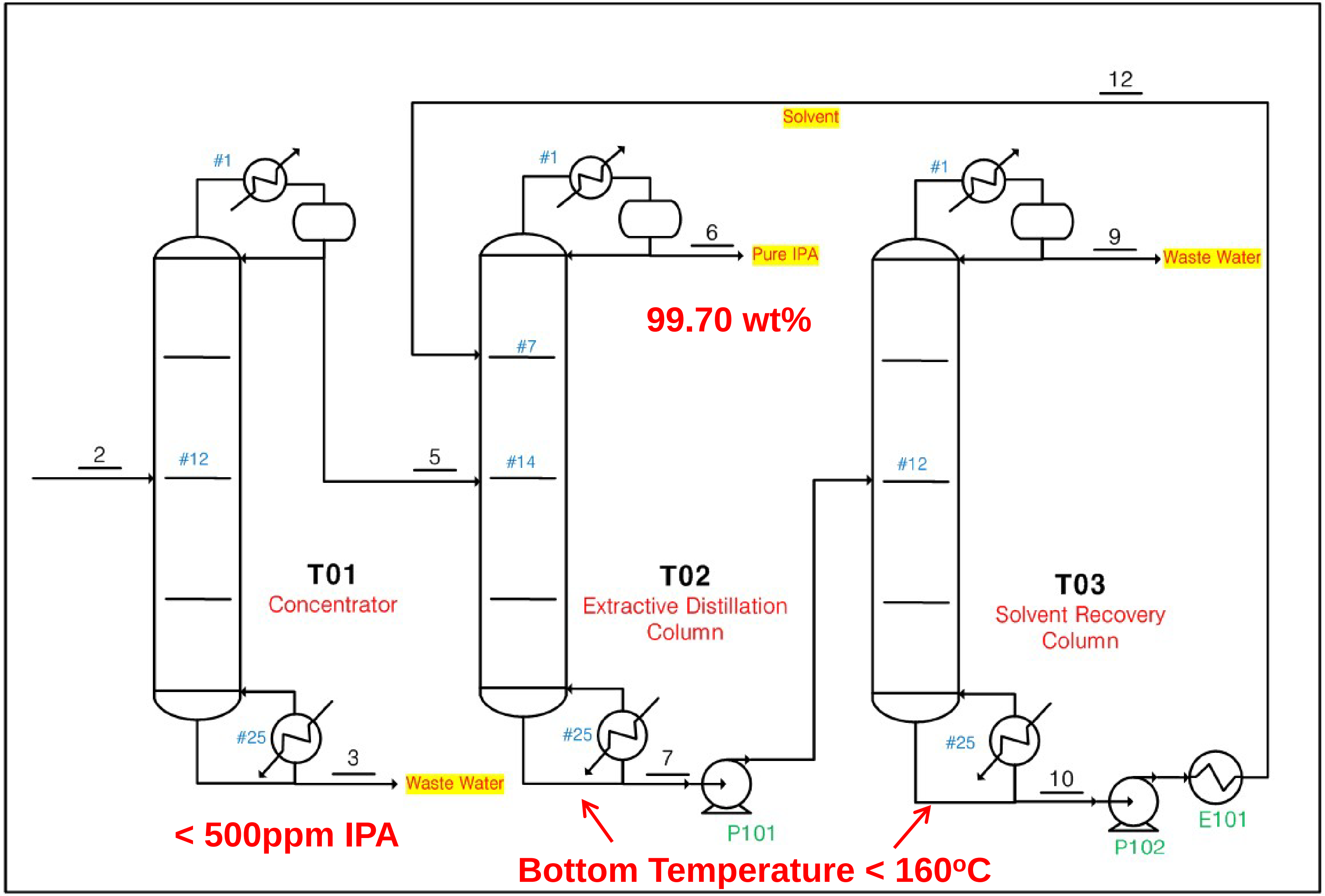
Two-column Stream Summary (DMSO)

Stream Number	1	2	6	8
Stream Name	T02 Feed	T02 Top (IPA Product)	T03 Top (Waste Water)	T03 Bottom (Solvent)
Temperature (℃)	20.00	45.00	55.25	**150.06**
Pressure (kPa)	250.00	103.00	16.00	250.00
Vapor Mole Fraction	0.00	0.00	0.00	0.00
Total Molar Rate kmol/hr	99.83	28.83	71.00	56.29
Total Mass Rate kg/hr	3,000.00	1,720.51	1,279.49	4,396.59
Comp. Flow Rate (kg/hr)	**kg/hr**	**kg/hr**	**kg/hr**	**kg/hr**
IPA	1,716.00	**1,715.35**	0.65	0.00
WATER	1,284.00	5.16	1,278.84	0.44
DMSO	0.00	0.00	0.00	4,396.15
Comp. Composition (wt%)	**wt%**	**wt%**	**wt%**	**wt%**
IPA	57.20	**99.70**	**0.05**	0.00
WATER	42.80	0.30	99.95	0.01
DMSO	0.00	0.00	0.00	99.99

Comparison of Two-column Configurations

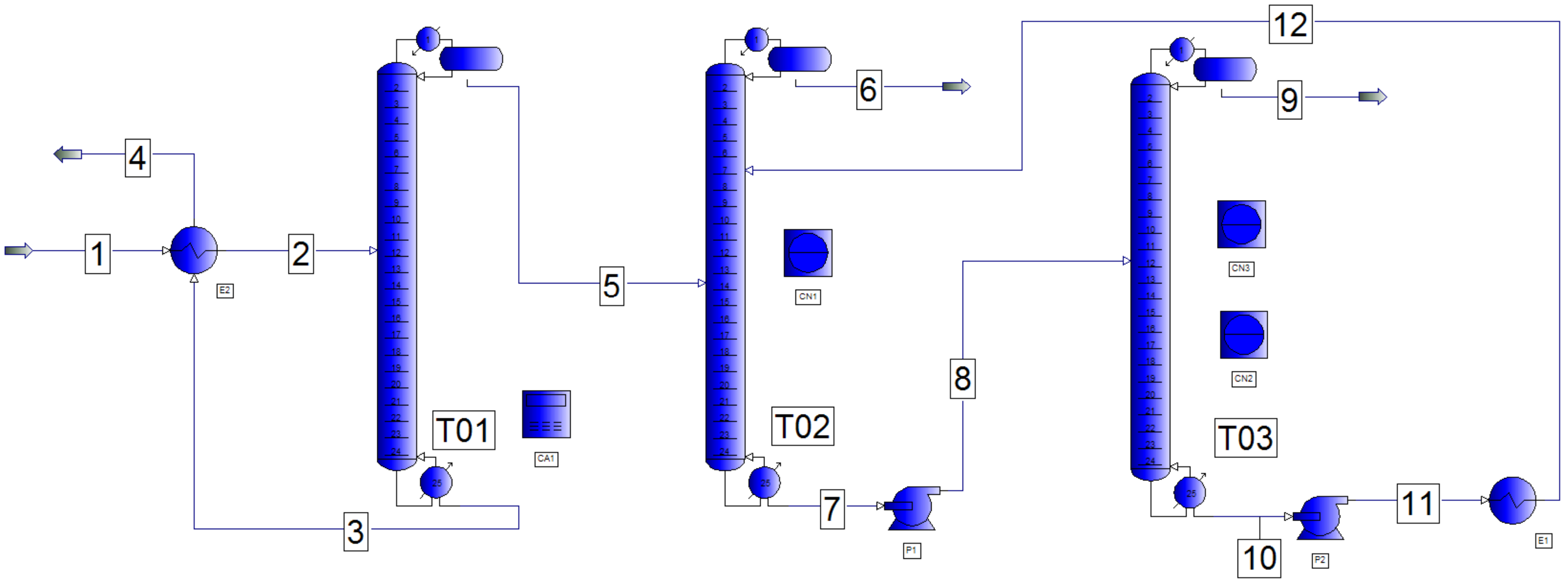
Items	Two-columns(EG)	Two-columns(DMSO)
Condenser duty of T02	0.9656×10^6 kcal/hr	0.9654×10^6 kcal/hr
Condenser duty of T03	2.1573×10^6 kcal/hr	2.1850×10^6 kcal/hr
Total	**3.1229×10^6 kcal/hr**	**3.1504×10^6 kcal/hr**
Reboiler duty of T02	1.5648×10^6 kcal/hr	1.3303×10^6 kcal/hr
Reboiler duty of T03	2.0538×10^6 kcal/hr	2.0851×10^6 kcal/hr
Total	**3.6186×10^6 kcal/hr**	**3.4154×10^6 kcal/hr**
Solvent circulation rate	**6,766 kg/hr**	**4,396 kg/hr**
Cooling water consumption	**390 ton/hr**	**394 ton/hr**
Steam consumption	**7,518 kg/hr**	**7,096 kg/hr**

IPA Dehydration Using Three-Column Configuration

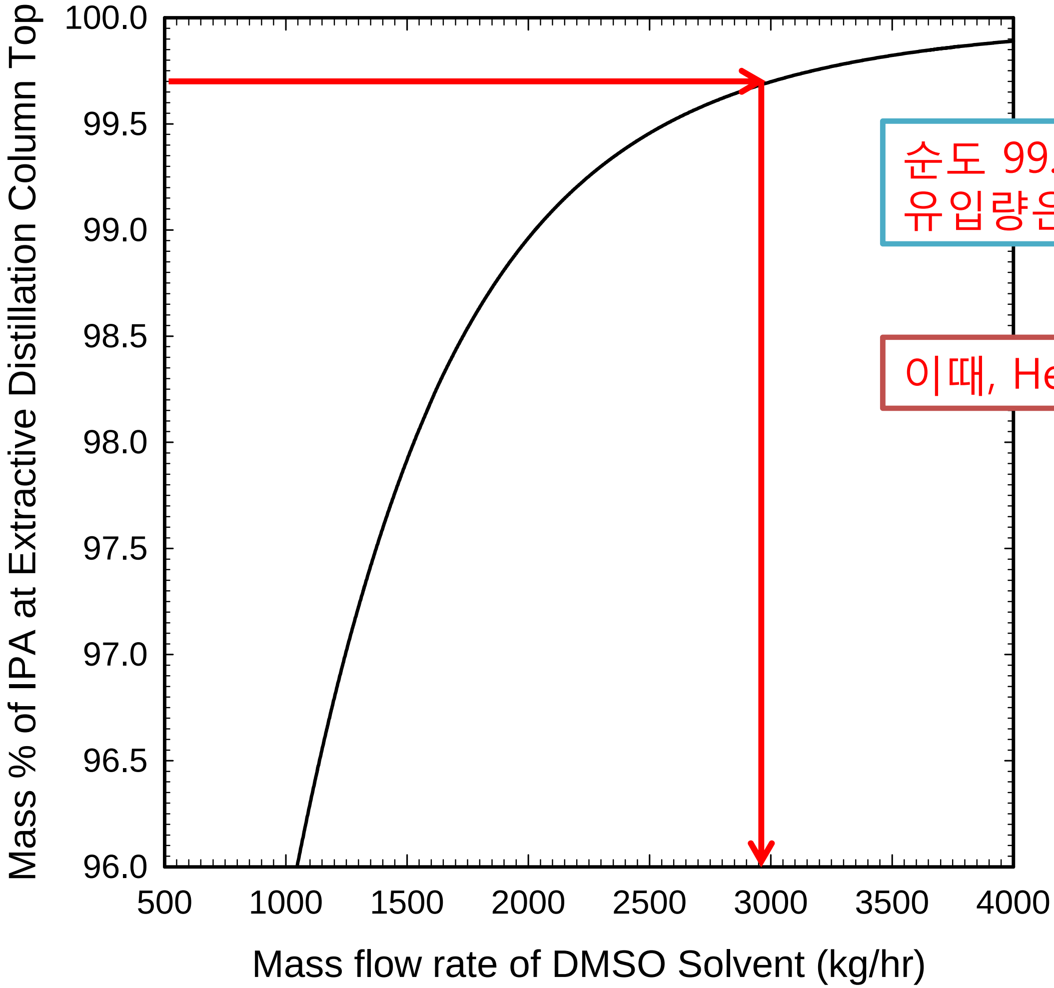


IPA Dehydration Using Three-Column Configuration

- Flow sheet drawing for a **three-column** configuration using PRO/II with PROVISION version 9.1



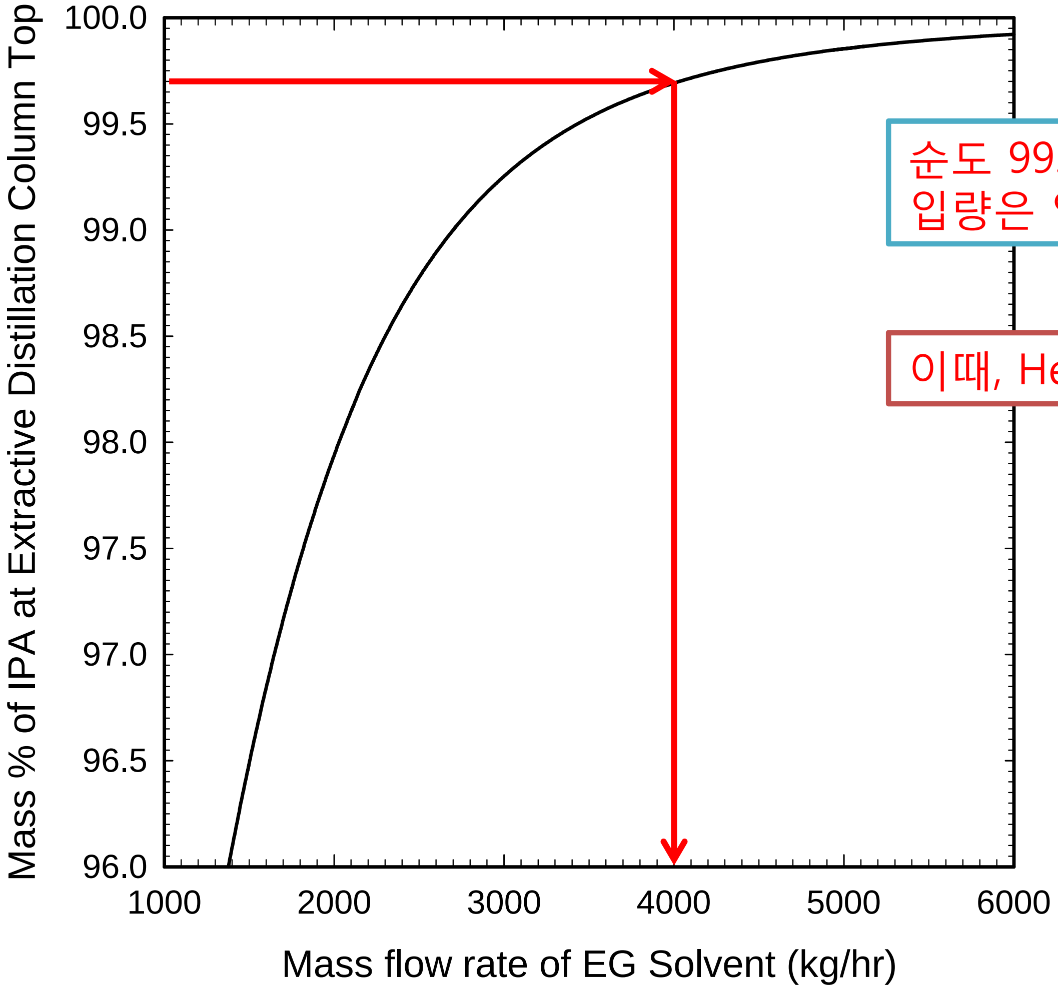
추출증류탑에서 추출용매(DMSO)의 유량에 따른 추출증류탑 탑 상제품에서 IPA순도(wt%)



순도 99.7wt%를 얻기 위한 DMSO용매 유입량은 약 3,000kg/hr정도 되어야 함

이때, Heat Duty는 1.1671×10^6 Kcal/hr

추출증류탑에서 추출용매(EG)의 유량에 따른 추출증류탑 탑상제품에서 IPA순도(wt%)

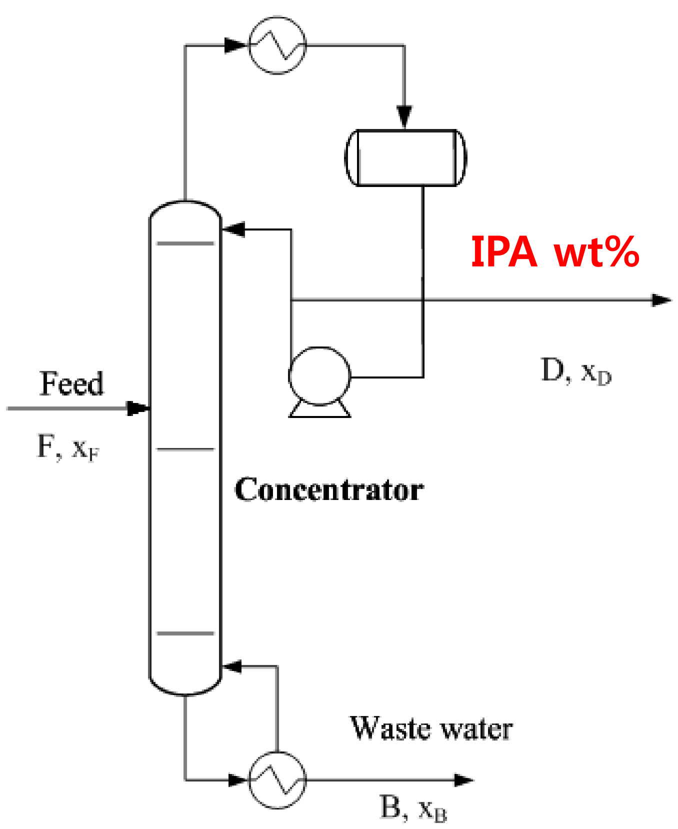


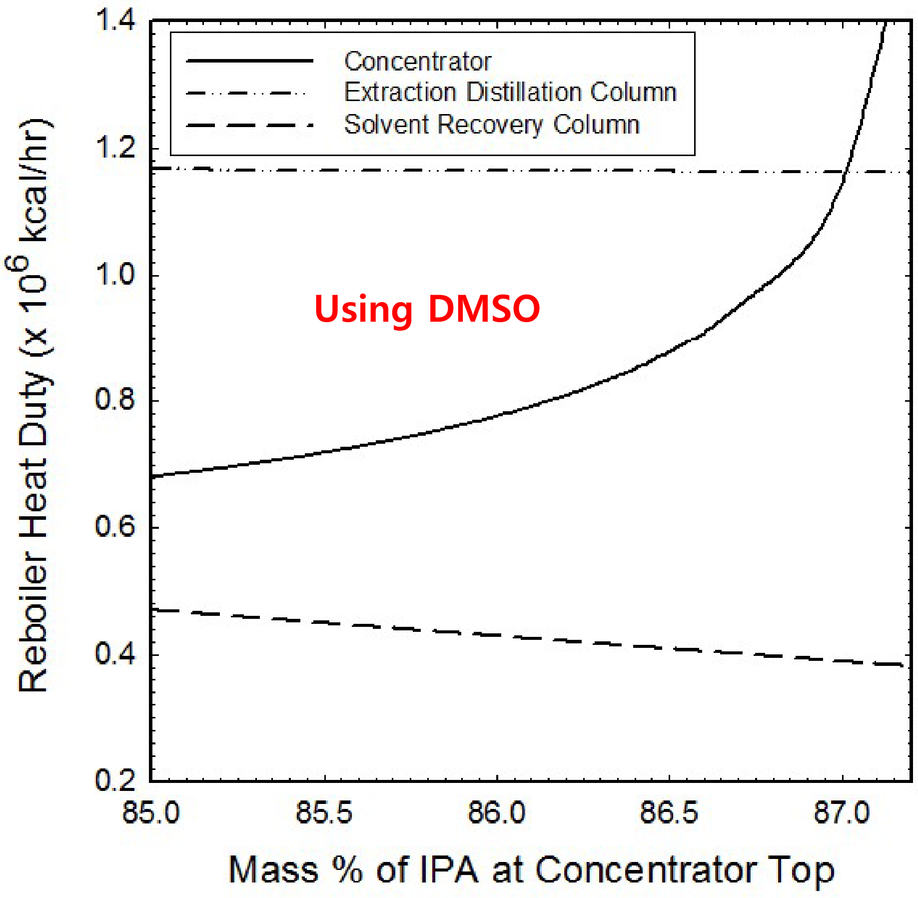
순도 99.7wt%를 얻기 위한 EG 용매 유입량은 약 4,000kg/hr정도 되어야 함

이때, Heat Duty는 1.3093×10⁶ Kcal/hr

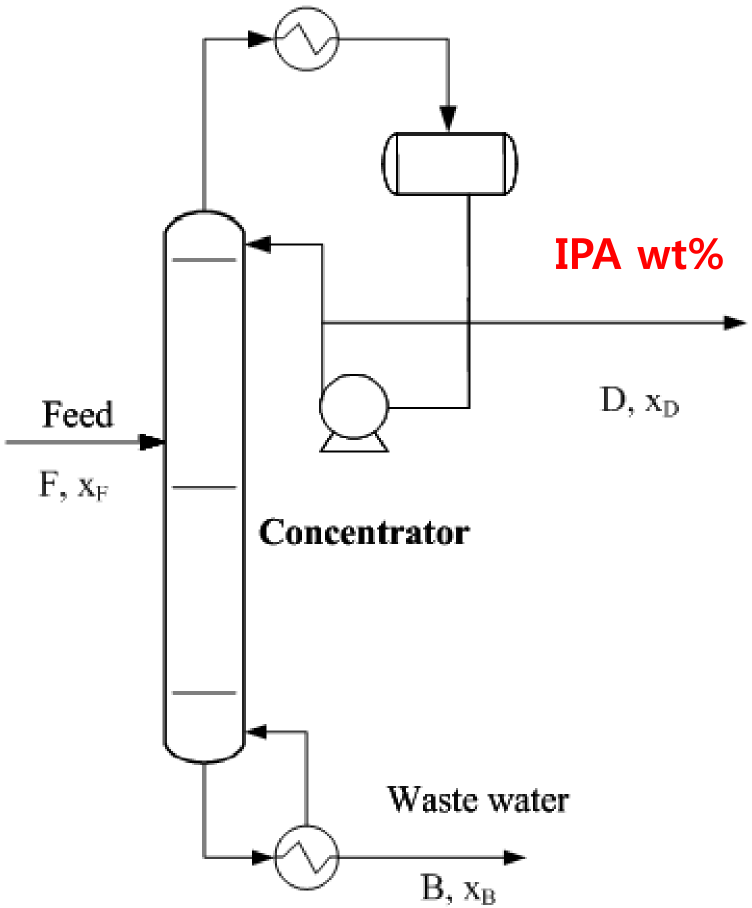
Reboiler heat duty of Concentrator, Extractive Distillation Column and Stripper vs. IPA wt% of concentrator top

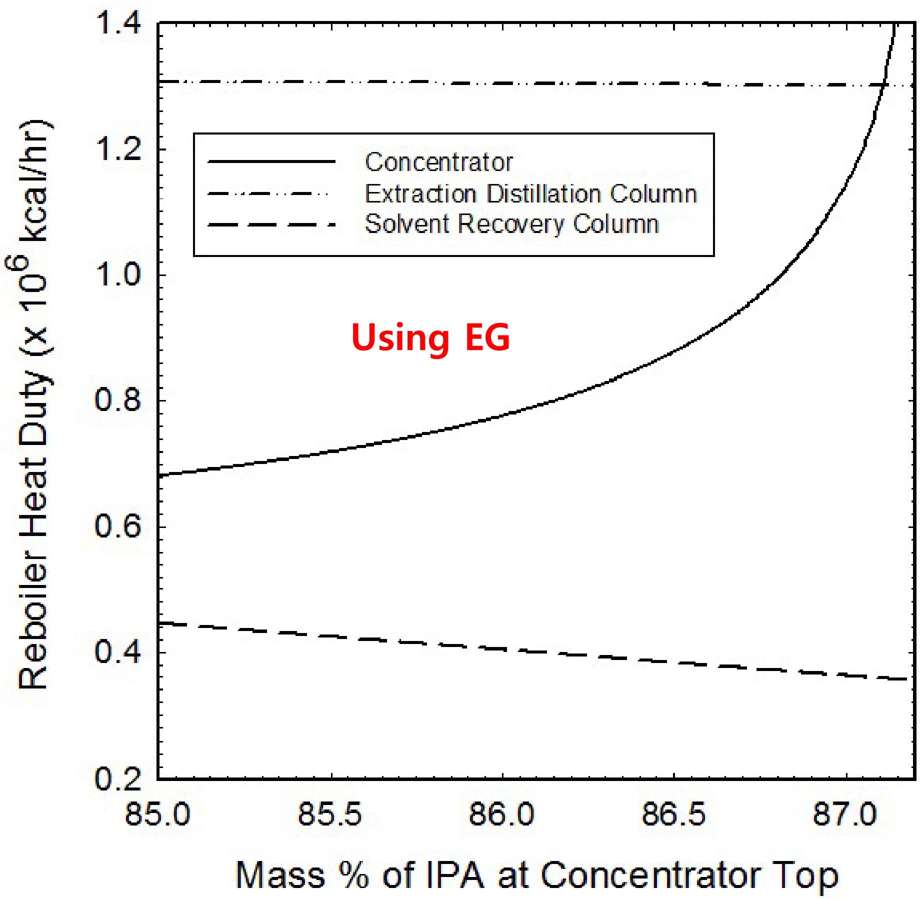
- 농축기 상부에서의 IPA순도(wt%)에 따른 **농축기**와 **추출탑** 그리고 **용매 재생탑**의 **재비기 heat duty** 변화량을 나타낸 결과입니다.





Reboiler heat duty of Cocentrator, extractive distillation Column and Stripper vs. IPA mass% of concentrator top

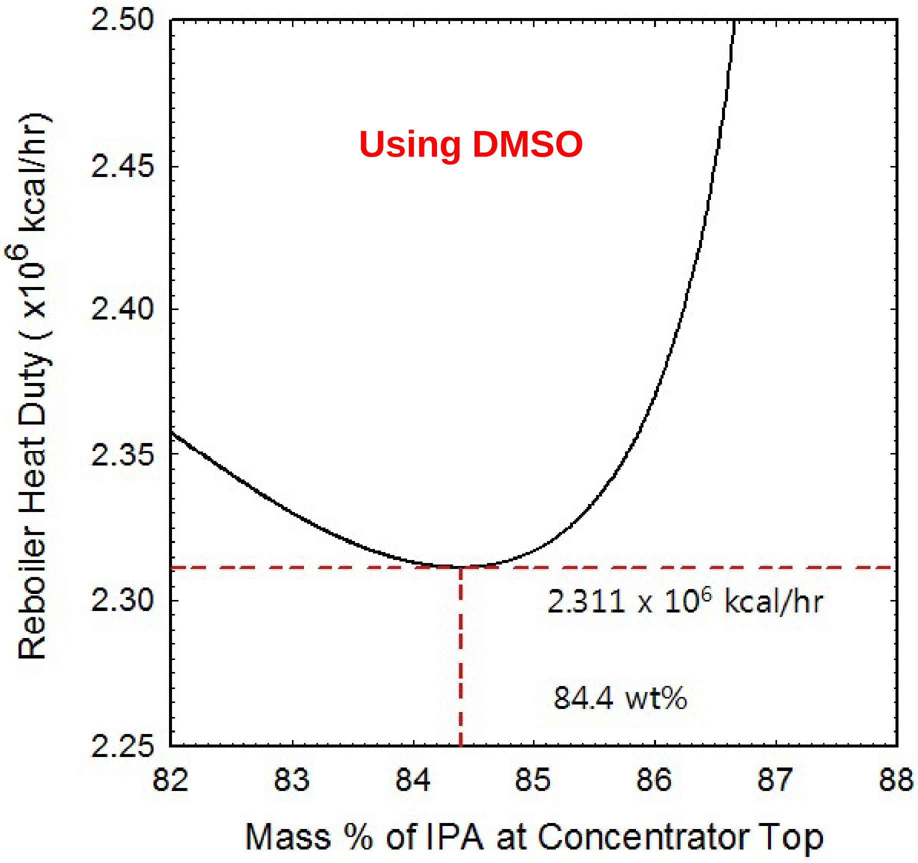


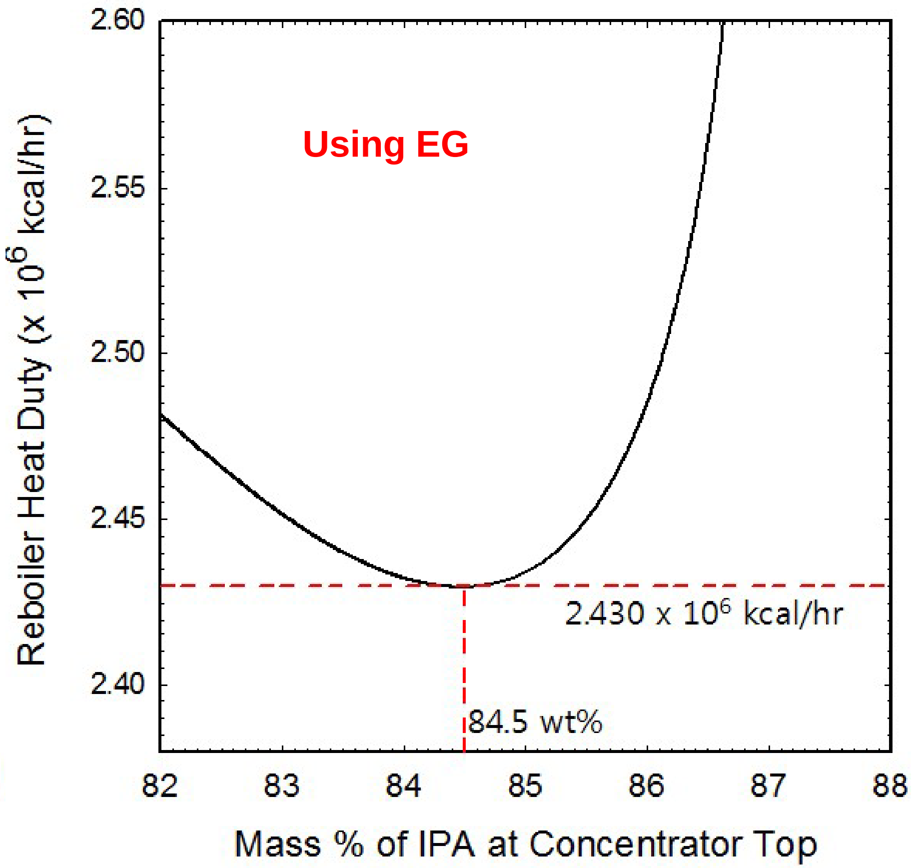


Total Reboiler heat duty in extractive distillation process vs. IPA wt% of concentrator top

DMSO의 경우 농축기 상부에서의 IPA순도 (wt%)가 84.4wt%일 때, 총 재비기 heat duty 값이 가장 낮은 것을 알 수 있습니다.

EG의 경우에는 농축기 상부에서의 IPA순도 (wt%)가 84.5wt%일 때, 총 재비기 heat duty 값이 가장 낮은 것을 알 수 있습니다.

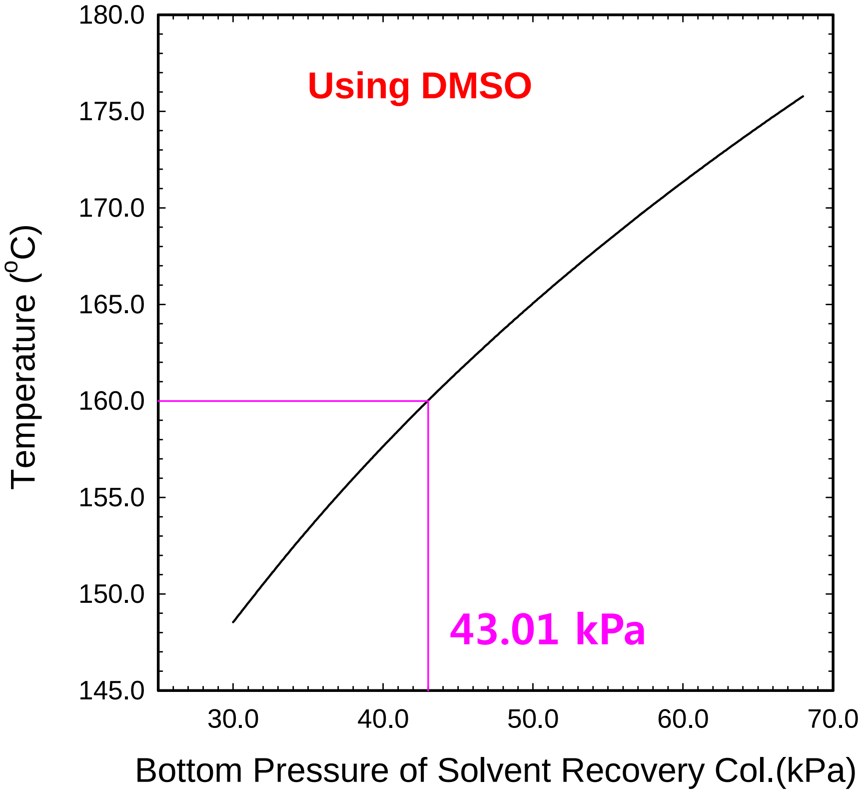


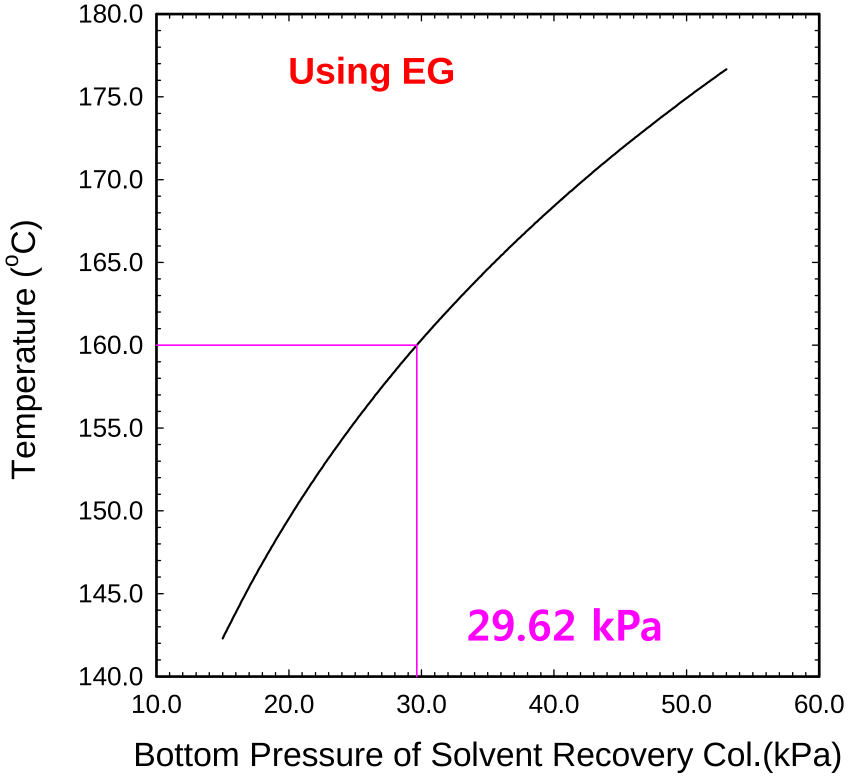


용매 회수탑 하부의 온도가 160℃가 되기 위한 용매 회수탑 하부(재비기) 압력

DMSO의 경우 용매 회수탑(T3) 하부의 온도가 160℃가 되기 위한 재비기 압력은 43.01 kPa정도 됩니다.

EG의 경우 용매 회수탑(T3) 하부의 온도가 160℃가 되기 위한 재비기 압력은 29.62 kPa 정도 됩니다.





Three-column Stream Summary(EG)

Stream Number	2	5	6	9	10
Stream Name	T01 Feed	T01 Top	T02 Top (IPA Product)	T03 Top (Waste Water)	T03 Bottom (Solvent)
Temperature (℃)	66.22	45.00	45.00	45.00	160.00
Pressure (kPa)	250.00	105.00	103.00	25.62	29.62
Vapor Mole Fraction	0.00	0.00	0.00	0.00	0.00
Total Molar Rate kmol/hr	99.83	46.01	28.83	17.18	64.74
Total Mass Rate kg/hr	3,000.00	2,030.20	1,720.52	309.67	4,017.43
Comp. Flow Rate (kg/hr)	**kg/hr**	**kg/hr**	**kg/hr**	**kg/hr**	**kg/hr**
IPA	1,716.00	1,715.52	**1,715.36**	0.15	0.00
WATER	1,284.00	314.68	5.16	309.52	0.40
EG	0.00	0.00	0.00	0.00	4,017.03
Comp. Composition (wt%)	**wt%**	**wt%**	**wt%**	**wt%**	**wt%**
IPA	57.20	**84.50**	**99.70**	**0.05**	0.00
WATER	42.80	15.50	0.30	99.95	**0.01**
EG	0.00	0.00	0.00	0.00	99.99

Three-column Stream Summary(DMSO)

Stream Number	2	5	6	9	10
Stream Name	T01 Feed	T01 Top	T02 Top (IPA Product)	T03 Top (Waste Water)	T03 Bottom (Solvent)
Temperature (℃)	66.20	45.00	45.00	45.00	160.04
Pressure (kPa)	250.00	105.00	103.00	27.01	43.01
Vapor Mole Fraction	0.00	0.00	0.00	0.00	0.00
Total Molar Rate kmol/hr	99.83	46.15	28.83	17.32	38.44
Total Mass Rate kg/hr	3,000.00	2,032.60	1,720.52	312.08	3,002.46
Comp. Flow Rate (kg/hr)	**kg/hr**	**kg/hr**	**kg/hr**	**kg/hr**	**kg/hr**
IPA	1,716.00	1,715.52	**1,715.36**	0.16	0.00
WATER	1,284.00	317.09	5.16	311.92	0.30
DMSO	0.00	0.00	0.00	0.00	3,002.16
Comp. Composition (wt%)	**wt%**	**wt%**	**wt%**	**wt%**	**wt%**
IPA	57.20	**84.40**	**99.70**	**0.05**	0.00
WATER	42.80	15.60	0.30	99.95	**0.01**
DMSO	0.00	0.00	0.00	0.00	99.99

Comparison of Three-column Configurations

Items	Three-columns(EG)	Three-columns(DMSO)
Condenser duty at T01	0.6446×10^6 kcal/hr	0.6410×10^6 kcal/hr
Condenser duty of T02	0.9655×10^6 kcal/hr	0.9655×10^6 kcal/hr
Condenser duty of T03	0.5735×10^6 kcal/hr	0.5466×10^6 kcal/hr
Total	**2.1476×10^6 kcal/hr**	**2.1522×10^6 kcal/hr**
Reboiler duty of T01	0.6537×10^6 kcal/hr	0.6492×10^6 kcal/hr
Reboiler duty of T02	1.3093×10^6 kcal/hr	1.1671×10^6 kcal/hr
Reboiler duty of T03	0.5019×10^6 kcal/hr	0.5147×10^6 kcal/hr
Total	**2.4649×10^6 kcal/hr**	**2.3310×10^6 kcal/hr**
Solvent circulation rate	**4,017 kg/hr**	**3,002 kg/hr**
Cooling water consumption	**268 ton/hr**	**269 ton/hr**
Steam consumption	**5,120kg/hr**	**4,843 kg/hr**

결론

- 2기 배열에서 IPA 99.7wt% 정제를 위해 필요한 추출용매의 순환 유량은 DMSO 용매를 사용한 경우 4,396 kg/hr 였으며 EG 용매를 사용한 경우 6,763 kg/hr로 DMSO에 비해 53.8%정도 더 소요되는 것으로 계산되었다.

- 3기 배열에서 IPA 99.7wt% 정제를 위해 필요한 추출용매의 순환 유량은 DMSO 용매를 사용한 경우 3,002 kg/hr 였으며 EG 용매를 사용한 경우 4,018 kg/hr로 DMSO에 비해 33.8%정도 더 소요되는 것으로 계산되었다.

- 2기 배열에서 DMSO 용매를 사용한 경우가 EG를 사용한 경우에 비해 재비기 Heat Duty가 5.6%정도 더 적게 소요되는 것으로 계산되었다.

- 3기 배열에서 DMSO 용매를 사용한 경우가 EG를 사용한 경우에 비해 재비기 Heat Duty가 4.9%정도 더 적게 소요되는 것으로 계산되었다.

- 3기 배열에서 DMSO 추출 공정의 경우 IPA 순도가 84.4 wt%일 때 재비기 Heat Duty는 2.312×10^6 kcal/hr로 최소화 되었으며, EG의 경우는 IPA 순도가 84.5 wt%일 때 재비기 Heat Duty가 2.432×10^6 kcal/hr로 최소화됨을 알 수 있었다.

감사합니다.