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Separation of Methanol & Toluene From Spent Toluene And Methanol Mixture Azeotropically

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Abstract

There are economic and environmental benefits when we recover solvent for re-use, especially when expensive solvents and large volumes are involved. An industry has been recovering solvents for many decades at facilities. Solvent recovery is a routine practice in the pharmaceutical industry when it is technically and economically viable for the particular waste stream. Various waste streams being generated at an API (Active pharmaceutical ingredient) synthesis plant. The goal is to investigate those streams that could not be most easily recovered with traditional separation and purification processes. As a first step in that analysis, the recovery of toluene & methanol solvent from a waste stream in the synthesis was considered. Here it is very difficult to separate this complex mixture, so that for separation of this complex mixture azeotropic distillation method is to establish by using the water and an entrainer.

Keywords: *Separation between the two azetropic solvent by entrainer*

Introduction

Azeotropic distillation is a commonly used solution to a fractionation problem in which, at whatever pressure, the value of α is too low for the techniques described so far. It is particularly valuable for breaking apart the components of existing azeotropes in a system, but it can also be used when the required separation is a very difficult one. Azeotropic distillation can be done continuously or batch-wise.

In the latter case, enough entrainer should be used so that while it is removing the solvent with which it azeotropes, there should be a low but positive concentration of it in the still kettle. Azeotropic distillation done batchwise is particularly well suited to a hybrid unit, since small amounts of entrainer can be held in the column top while the second product is being removed at the column mid-point. Here separation of the toluene & methanol can be done in the packed column by using water as an entrainer, hence the water is very cheap and environment friendly and easily available, after at the end of the distillation water remain in methanol which can easily disposed in conventional Effluent Treatment Plant. Utility required for this is Cooling water of temperature 25-35°C, Chill water of temperature +5°C, and for heating hot oil required of temperature 100°C, for analyzing the collected solvents GC (Gas Chromatograph) is required for purity, KF (Karl Fisher) for water content & Hydrometer for specific gravity measurement.

Experimental

Clean the 4 Neck round bottom glass flask. Put glass flask in to the heating mental. Connect the vertical packed column in on glass flask. Put the thermometer in thermo well and this thermo-well insert in one of the neck of glass flask. And connect the top vapour outlet in glass coiled type condenser & connect second condenser in series & connect the cooling water in primary & chilling water in the secondary condenser Connect flask to Condensate collection outlet.

Procedure for distillation

Charge 3500 ml to 4000 ml of spent solvent mixture in to the 5000 ml in to the four necks round bottom glass flask. Send the sample of spent solvent mixture for analyzing content, moisture & specific gravity. Then apply the cooling water in primary condenser & chill water in secondary condenser, subsequently start heating and keep the solvent under reflux for 30-60 minutes. Start collection of first fraction (Toluene) from the top of the column, at the same time start feeding of water (Entrainer) from top of the column. (Note : Keep water addition rate as same as reflux ration) after collecting toluene fraction send the sample for complete analysis by GC. Stop the feeding of water from the top of fractionation column. Start collection of second fraction (Methanol) from top of column. Keep the reflux ratio 1:3. Check the water content of Methanol (In process analysis) (Note: If it is in limit start collection of Methanol). If it is out of limit then keep the reflux ratio accordingly. After collecting and separating Methanol fraction completely. Stop heating & cool the remaining residue & unload the residue and measure it.

Table I.

Temperature recorded during the experiment

S.NO.	OBSERVATION	TEMPERATURE (°C)
1	Vapor start at top temperature	62.5
2	Toluene cut collect at temperature	63.7
3	99.5 % Methanol collection start at top temperature	63.9
4	Distillation stop at top temperature	72.6

Experimental Setup

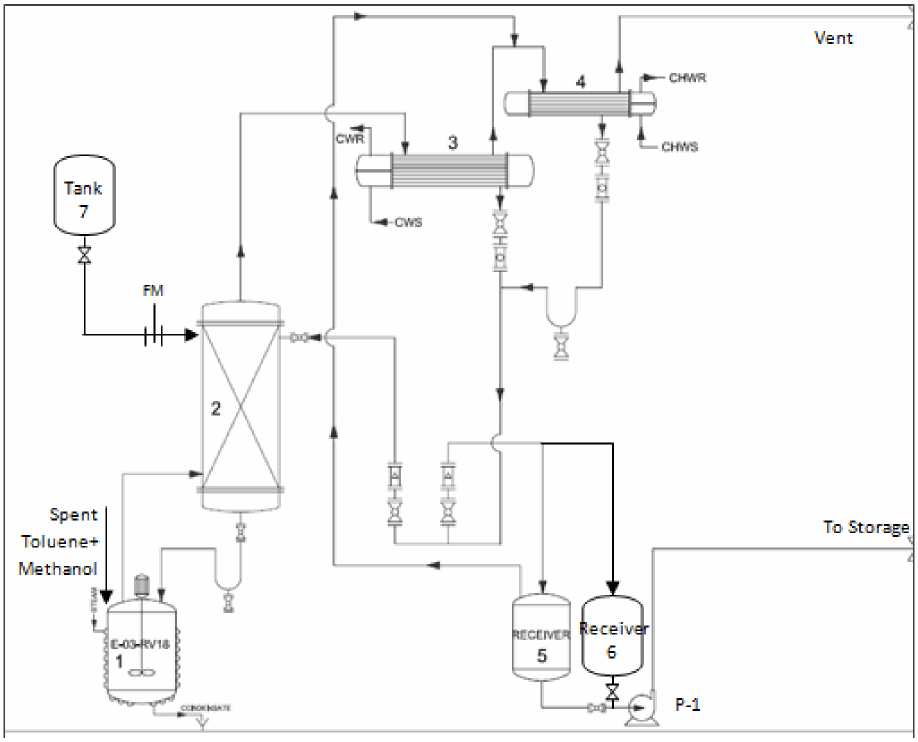

Figure I Typical Commercial distillation unit.

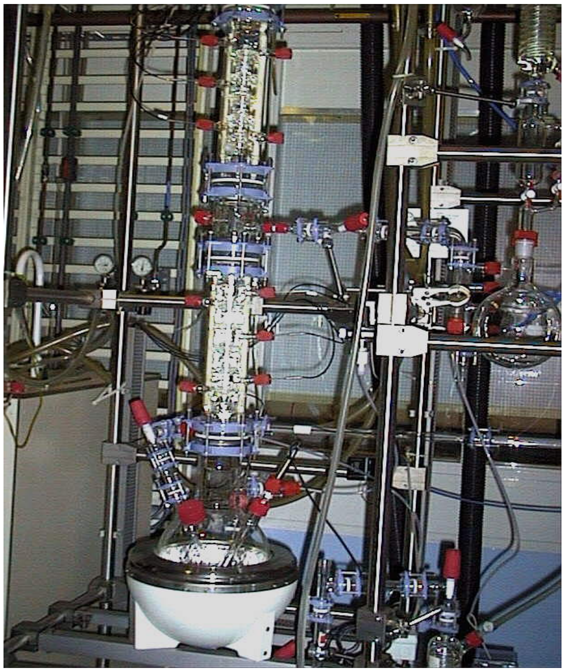

Figure II Lab apparatus for distillation Observation

Table II

Material input output

Components	Unit of Measurement	Expected on 100%	Experiment 1	Experiment 2	Experiment 3
Time	Hour	--	1.2	2.4	2.9
Feed (F)	Qty (Grams)	**1000**	**3640**	**4300**	**4500**
Reflux Ratio (R=L/D)			1.5	1.8	2
Water addition rate (R1=R)			1.5	1.8	2
Water (E)		--	1440	2160	2540
Toluene (D1)	Qty (Grams)	**125**	**402**	**447**	**486**
	%	12.5	11.0	10.4	10.8
Methanol (D2)	Qty (Grams)	**825**	**1832**	**3073**	**3649**
	%	82.5	50.3	71.5	81.1
Residue (W)	Qty (Grams)	**50**	**14.4**	**41.2**	**167.4**
	%	5	0.4	1.0	3.7
Loss	Qty (Grams)	**NA**	**1391.6**	**738.8**	**197.6**
	%	NA	38.2	17.2	4.39
Water (w)	Qty (Grams)	**NA**	**1397**	**2117**	**2464**
Loss	%	NA	43.2	43.2	76

Table III

Experiment result

Components	Specification	Expected on 100%	Experiment 1	Experiment 2	Experiment 3
Toluene (D1)	Purity	100%	63.20%	99.60%	99.72%
	Water Content	0%	2.30%	0.08%	0.06%
Methanol (D2)	Purity	100%	85.23%	99.56%	99.82%
	Water Content	0%	1.20%	0.07%	0.02%

Results & Discussion

Table, I, II & III Show the experiment detail performed in laboratory scale and it also show the input and output of all the material across the distillation operation. These all the input and output shown in the table is shown in percentage as well as in grams.

Experiment number 1 : This experiment started by kipping reflux ratio 1.5, recovery found less in toluene & Methanol & purity also loess & loss are also more.

Experiment number 2 : This experiment started by kipping reflux ratio 1.8, & recovery found near to the expected percentage & purity also not meet as per the requirement.

Experiment number 3 : By studying above tow expiring here gain increased the reflux ratio & kept it 2, in this experiment results are meets to expectation.

Conclusion

The experiment was performed at different reflux ration & water (entrainer) addition rate. It is observed that at the higher water addition rate & at the higher reflux ratio separation of mixture is take place. Spent solvent mixture is consider for experiment it is contain 12.5% toluene, 85.5% Methanol, and 5% contain water and some impurities.

During the distillation in the packed column, start heating keep the reflux ratio 2, maintain reflux for 30 to 60 minutes, when top temperature of column reaches 63.7°C then start addition of water (entrainer) from the top, entrainer addition rate should be keep same as maintain as reflux rate & toluene collected from the top of the column at the temperature 63.7°C, after collection of toluene, then immediately stop the water addition. After removal of toluene, insight vessel water and methanol. Change the reflux ration from 2 to 1.3 and maintain for 30 minute, temperature of vapour at top of column reaches 63.9°C start collection of Methanol and collect methanol in separate container. Send the sample for GC analysis for purity. By this methodology it is observed that percentage recovery of toluene is 84% & methanol recovery is 95%. It is clear that recovery of the both solvent is increased. The result of this study and experimental data shows that separation of the toluene & methanol is possible by using water as an entrainer & distillation carried out by azeotropically in the batch distillation vessel.

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Linear Low-Density Polyethylene Membrane

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