일일 10톤 DME 생산 Demo Plant에서의 분리정제 공정

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SEPARATION AND PURIFICATION PROCESS OF DEMO PLANT FOR 10TON PER DAY DME PRODUCTION

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ABSTRACT

DME(Di-Methyl Ether) is a new clean fuel and an environmental-friendly energy resource, also is recently increasing with an alternative interest because of the industrial use. DME has been shown to have excellent properties as a diesel fuel giving emission level better than ULEV standard. So it has been attracting considerable as an alternative diesel fuel.

In this study, we carried out simulation of separation and purification process of demo plant for 10ton per day DME production, which cause the effect that is important in productivity, from operation results of pilot plant for 50kg per day DME production. The liquefied stream, which was separated by gas-liquid separator after DME reactor, includes CO_2, DME, Methanol and H_2O. We established three distillation columns for separation and purification of the stream. CO_2 was extracted from the stream by first distillation column, DME was extracted by second column and Methanol was extracted by third column. We investigated and analyzed the effect which the actual operation variables cause in efficiency of process and optimized process, finally we got the DME of purity 100%.

Keywords : DME, Distillation column, CO_2 absorber, Methanol absorber, TSA

1. INTRODUCTION

DME(Di-Methyl Ether) is a new clean fuel and an environmental-friendly energy resource, also is recently increasing with an alternative interest because of the industrial use. Been identified as a potential diesel and cooking fuel, DME has many excellent characteristics. It has an oxygen concentration of 34.78% and can be burned without soot emission, while for traditional diesel fuels, one cannot expect simultaneous NOx and soot emission control target. It has a boiling point of −25℃, while is 20℃ higher than LPG and can be liquidized at 0.54 MPa (20℃). Therefore, based on the matured technology of LPG application, there does not exist any problem for the storage, transportation and usage of DME. At present, DME is commercially prepared by dehydration of methanol using acidic porous catalysts such as zeolites, silica-alumina, alumina, etc. It has been reported that using syngas as a starting material, DME can be prepared in a one-step process, which is more thermodynamically and economically favorable than the two-step process of syngas to methanol and further to DME.

The primary function of this system is to accept a mixed feed comprising mostly of DME and separating it away from the other constituents. The target is to produce DME of 99.9% purity but simultaneously limiting the methanol to under 100 ppm and water to under 500 ppm.

2. SIMULATION RESULTS AND DISCUSSIONS

2.1 CO_2 Column

The liquid streams from D-501 and from D-504 are combined and fed into the CO_2 Column CL-601. The CO_2 Column CL-601 serves to remove the CO_2 from the DME. The CO_2 is taken from overhead in the vapor phase along with small quantities of non-condensable gas and the DME along with some methanol and water is taken from the bottom as a liquid. The column functions as a distillation column with an overhead condenser, reflux drum and pump, and a bottoms reboiler. The column has two sections of high efficiency wire mesh packing, one above the feed and one below the feed. The incoming stream is introduced above stage 7 using an appropriate liquid distributor.

The required number of theoretical stages for the column is 18, 6 above the feed and 12 below the feed. Two additional stages are added at each of the two liquid distribution points, one at the feed and one at the top of the column. Therefore the total number of stages required is 8 above the feed and 14 below the feed. The calculated HETP is 0.13 meters.

The overheads from the column are routed through a partial overhead condenser and then enter the CO_2Column Overhead Drum D-601. The condensable fraction is recovered in D-601 and refluxed back to the column under level control. The non-condensable gas stream consisting primarily of $CO_2$is vented from D-601 on pressure control. This stream is returned in part to the Tri-Reformer, on flow control, and vented in part to the vent system, on pressure control.

The bottom of the column is fitted with a liquid sump and has a vertical thermosiphon type reboiler E−601 to supply heat to the column. The bottom product is taken off based on level control of the sump and flows to CL−602.

The operating conditions for CL−601 and the vapor and liquid streams exiting the column are shown in Table 2 below.

Table 2. Operating conditions for CO_2 Column

Parameter		Normal Condition
Temperature (C)		110/−11
Pressure (kg/cm^2)		32
Vapor	CO (Mole Fraction)	0.0422
	CO_2 (Mole Fraction)	0.9454
	H_2 (Mole Fraction)	0.0058
	DME (Mole Fraction)	0.0002
	CH_4 (Mole Fraction)	0.0055
	N_2 (Mole Fraction)	0.0008
	Average MW	42.92
	Total Molar Flow (kmol/h)	8.816
	Total Mass Flow (kg/h)	378.4
Liquid	H_2O (Mole Fraction)	0.0182
	CH_3OH (Mole Fraction)	0.2294
	DME (Mole Fraction)	0.7524
	Average MW	42.34
	Total Molar Flow (kmol/h)	12.03
	Total Mass Flow (kg/h)	509.5

The CO_2 Column is designed for a normal reflux rate of rate of 3.0. The normal duty of the reboiler E−601 corresponding to a normal reflux rate of 3.0 is 529,600 kJ/h. The reboiler design must at least be increased to accommodate the additional reflux ratio. This requires an additional duty of 294,600 kJ/h. This equates to a duty of 824,200 kJ/h. E−601 uses 1500 kPa saturated steam as its heating source.

A vertical thermosiphon is the recommended choice for a reboiler in this application. Due to the relative high operating pressure it is a more economical choice than either a kettle type reboiler or a forced circulation reboiler. Nonetheless, care must be taken in the layout of the equipment and the hydraulic and mechanical design of the piping for a thermosiphon reboiler to operate properly.

From the column sump the bottoms product, primarily DME with a small quantity of methanol and water, flows to the DME column CL−602 on level control of the bottoms sump.

2.2 DME Column

The DME Column CL−602 serves to purify the DME by removing the methanol and water. The DME productis taken from overhead as a liquid and the methanol and water are taken from the bottom as a liquid. The column functions as a distillation column with an overhead condenser, reflux drum and pump, and a bottoms reboiler. The column has two sections of high efficiency wire mesh packing, one above the feed and one below the feed. The incoming stream is introduced above stage 7 using an appropriate liquid distributor.

The column diameter is sized based on an operating pressure of 700 kPa. The required number of theoretical stages for the column is 17, 6 above the feed and 11 below the feed. Two additional stages are added at each of the two liquid distribution points, one at the feed and one at the top of the column. Therefore the total number of stages required is 8 above the feed and 13 below the feed. The calculated HETP is 0.11 meters.

The overheads from the column are routed through a total overhead condenser and then enter the DME Column Overhead Drum D−602. A fraction of the liquid refluxed back to the column under temperature control and the DME product is removed to the DME Storage Tank D−60xxx on level control.

The bottom of the column is fitted with a liquid sump and has a vertical thermosiphon type reboiler E−603 to supply heat to the column. The bottom product is taken off based on level control of the sump.

The operating conditions for CL−602 and the overhead and bottom streams exiting the column are shown in Table 3 below.

The DME Column is designed for a normal reflux rate of rate of 1.2. The normal duty of the reboiler E−603 corresponding to a normal reflux rate of 1.2 is 270,600 kJ/h. The reboiler design must at least be increased to accommodate the additional reflux ratio. This requires an additional duty of 196,500 kJ/h. This equates to a duty of 467,100 kJ/h. E−603 uses 1500 kPa saturated steam as its heating source.

Table 3. Operating conditions for DME column

Parameter		Normal Condition
Temperature (C)		130
Pressure (kPa)		700
Overhead Product	DME (Mole Fraction)	1.0000
	Average MW	46.07
	Total Molar Flow (kmol/h)	9.053
	Total Mass Flow (kg/h)	417.1
Bottom Product	H_2O (Mole Fraction)	0.0733
	CH_3OH (Mole Fraction)	0.9266
	Average MW	31.01
	Total Molar Flow (kmol/h)	2.979
	Total Mass Flow (kg/h)	92.39

A vertical thermosiphon is the recommended choice for a reboiler in this application. Due to the relative high operating pressure it is a more economical choice than either a kettle type reboiler or a forced circulation reboiler. Nonetheless, care must be taken in the layout of the equipment and the hydraulic and mechanical design of the piping for a thermosiphon reboiler to operate properly.

From the column sump the bottoms product, methanol and water, flows to the Methanol Dehydration Columns CL−403 A/B via the DME Bottoms Cooler E−605. This stream combines with the bottoms from CL−402 and the methanol is recovered and reused.

3. CONCLUSIONS

In this study, we carried out simulation of separation and purification process of demo plant for 10ton per day DME production, whichcauses the effect that is important in productivity, from operation results of pilot plant for 50kg per day DME production. The liquefied stream, which was separated by gas−liquid separator after DME reactor, includes CO_2, DME, Methanol and H2O. We established two distillation columns and temperature swing adsorption (TSA) column for separation and purification of the stream. CO_2 was extracted from the stream by first distillation column, DME was extracted by second column and methanol was recovered by TSA. Weinvestigated and analyzed the effect which the actual operation variables cause in efficiency of process and optimized process, finally we got the DME of purity 100%.

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