CO₂ Freezing Area Concept for Improved Cryogenic Distillation of Natural Gas

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Abstract. Sweetening process for natural gas with high content of sour gas (≥ 40% mol of CO₂ & H₂S) is difficult to be profitable. A lot of gas fields in Indonesia has high content of sour gas. New improved cryogenic distillation process of natural gas applying the concept of CO₂ freezing in the middle of the column is promising. To study the feasibility of this technology, comparison with other two methods (Absorption and Membrane process) in technical and economic aspects were elaborated. Process simulation of improved cryogenic distillation steady-state model was developed using Aspen HYSYS. CAPEX of all methods were estimated using Aspen Process Economic Analyzer. Compared to the other methods, this improved cryogenic distillation process showed tendency to have lower CAPEX and OPEX, but have some uncovered operational problems.

1. Introduction

Energy supply is one of many challenging problems many countries face recently. Natural gas is one of important energy source. More than 24% of world energy consumption in 2016 is supplied by natural gas [1]. However, many of gas fields have high content of acid gas. Gas field is classified to have high content of sour gas if that field has more than 40%-v/v total content of CO₂ and H₂S. These impurities need to be removed from natural gas to meet sales gas or pipelines specifications. One of important sales specification is acid gas content, which is < 2% v/v for CO₂ and < 4 ppm v/v for H₂S [2].

Indonesia has lot of field that has high content of acid gas (e.g. Natuna field with 71%-v/v of acid gases, West Java gas fields containing 45% - 75%-v/v of acid gas, and East Java gas fields with 40%-v/v of acid gases) [3]. Most of this field development raise a lot of questions to be profitable. Minimizing CAPEX and OPEX of separation process for this kind of natural gas is the key to make this development profitable. A new improved cryogenic distillation process has been developed by applying the concept of CO₂ freezing in the middle of the column. Feasibility study of this new improved cryogenic distillation process is needed to substantiate advantages of this method.
2. Development Of Gas Field Containing Acid Gases

Separation process of acid gas from natural gas commonly called as sweetening process. Various technologies were commercially available for this sweetening process. They are categorized based on the underlying governing phenomena, such as adsorption, physical absorption, chemical absorption, membrane, and cryogenic distillation. Natural gas sweetening technologies can be classified as shown in Table 1.

| CO₂ Removal Mechanism          | Process Type                        | Technology                  |
|--------------------------------|-------------------------------------|-----------------------------|
| Chemical Absorption            | Regenerative, Continuous            | Amines                      |
|                                | Non regenerative, Continuous        | Potassium carbonate         |
| Physical Absorption            | Regenerative, Continuous            | Sodium hydroxide            |
| Physical-Chemical Absorption   | Regenerative, Continuous            | Physical Solvents           |
| Physical Adsorption            | Regenerative, Continuous (Adsorption/Desorption sequence) | Physical Chemical Solvents |
| Permeation                     | Continuous                           | Molecular Sieve             |
| Distillation                   | Continuous                           | Cyrogenic Distillation      |

Design of an efficient and competitive processing scheme is an important stage in the development of gas fields. Table 2 shows a representative gas stream composition and condition as a basis of feasibility study exercised in this paper.

The first challenge is to select the technology that best fits the project needs. Compilation of best practice technologies in the form of graphics are most commonly used to predict the most proper technology for sweetening natural gas [4]. Important factor that affecting the technology selection are acid gas content in feed, acid gas content in sweet gas product, and gas flowrate.

New improved cryogenic distillation for natural gas was developed, particularly for gas field that has high content of acid gas. The underlying phenomena of this new improved process is the concept of CO₂ freezing in the middle of the column [5]. Applying freezing of CO₂ will improve separation of CO₂ and methane. As comparison to the selected technology, other methods are chosen based on traditional selection guidelines. Based on graphs in Figure 1, two technologies met the feed criteria (feed gas acid gas content = 40%-v/v, outlet sweet gas acid gas content = 2%-v/v, and gas flowrate=189 MMSCFD). Those are technologies based on physical-chemical absorption and hybrid method (membrane + physical-chemical absorption).

3. Comparison Methodology

Comparison of the three technologies was carried out in four steps: flowsheet simulation, preliminary equipment sizing, estimation of CAPEX, and estimation of OPEX.
3.1. Flowsheet Simulation Development

Steady-state simulation models of separation process were developed using advanced process simulation software, Aspen HYSYS v8.8. The software is appropriate to model any process equipment contained in the three technologies evaluated. Figure 2 shows the simulation flowsheet for the new improved cryogenic distillation. This process consists of four sub-processes: condensate extraction and stabilization, main fractionation, CO₂ liquid injection, and refrigeration. The heavy hydrocarbon is extracted from the feed gas by cooling utilizing the cold sweet gas stream. Using cascade propane-ethylene refrigeration unit, the lean feed gas is then further cooled down to just above the freezing temperature of CO₂ and fed to the main fractionation column at the bottom of the controlled freezing section. The liquid CO₂ is taken off the bottom of the column and then injected to underground reservoir. The cold sweet gas is drawn from the top of the column and utilized as cooling medium for the condensate extraction sub-process.
Table 2. Gas Field Composition and Operating Condition

| Operating Condition | Composition (%-mol) |
|---------------------|---------------------|
| 1 Volume Flow       | 189 MMSCFD          |
| 2 Pressure          | 700 psig            |
| 3 Temperature       | 170 oF              |

| No. | Component | Composition (%-mol) |
|-----|-----------|---------------------|
| 1   | Methane   | 55.8                |
| 2   | Ethane    | 1.92                |
| 3   | Propane   | 0.64                |
| 4   | i-Butane  | 0.14                |
| 5   | n-Butane  | 0.18                |
| 6   | i-Pentane | 0.07                |
| 7   | n-Pentane | 0.06                |
| 8   | C6+       | 0.50                |
| 9   | N₂        | 0.32                |
| 10  | CO₂       | 39                  |
| 11  | H₂S       | 1                   |
| 12  | Organic Sulfur | 0.04       |
| 13  | BTX       | 0.33                |

The physical-chemical absorption process has been considered as reported in Figure 3. This process consists of four sub-processes: dew point control unit (DPCU), main acid gas removal unit (AGRU), acid gas injection by compression, and refrigeration. The lean feed gas from DPCU is fed to the absorption column, where it is contacted counter-currently with the lean MDEA solution containing sulfolane. The sweet gas stream is obtained at the top of the absorber and a rich liquid stream is taken from the bottom, containing contaminants to be removed. The rich stream is then flashed to low pressure, heated in the intermediate heat exchanger, and sent to the regeneration column, where acidic gases are stripped from the solvent and obtained as gas at the top, while the lean regenerated solvent is recovered at the bottom of the regeneration column and recycled back to the absorber after being cooled at the intermediate and second heat exchangers. Make up of water, amine, and sulfolane is needed due to leakages during solvent regeneration. The acid gases is finally compressed and injected to the underground storage.

Shown in Figure 4, simulation flowsheet for the hybrid technology is similar to the previous process with the addition of two-stage membrane unit at the upstream side of the physical-chemical absorption sub-process. divided into five sub-processes: dew point control, membrane unit, main absorption, acid gas injection, and refrigeration. The acid gases from both the membrane and absorption sub-processes are finally compressed and injected to the underground storage.

Multiple assumption and process parameter have been chosen and adjusted during development and execution of the simulation models. All feed gas parameter, such as composition and flow rate, and product specification, have been kept at the same values for all technologies evaluated to make sure the results are comparable. Feed stream was saturated by water first before being further simulated. For validation and comparison of simulation results, data was collected from several references with selected parameters as follows.
For all three process technologies:

- Polytropic efficiencies for compressors and pumps are assumed 75%.
- Cooling medium is assumed to be able to cool down the stream to 35 °C.
- Minimum temperature approach in heat exchanger is greater than 2 °C.
- Pressure drop of heat exchanger is 5 psi at tube side, and 2 psi at shell side.

For Improved Cryogenic Distillation:

- Heavier hydrocarbons have to be removed by cooling from the feed gas stream to prevent the occurrence of premature hydrocarbon freezing before entering the main fractionation column.

For Chemical + Physical Absorption:

- Aqueous absorbent with sulfolane content of 10.52 %-w/w and MDEA of 40.48 %-w/w.
- Rich loading for solvent is limited in the range of 0.40 – 0.42 mole/mole.
3.2. Equipment Sizing Method

Equipment sizing was carried out to determine variables required for estimation of equipment price. In Aspen HYSYS, calculated variables differ for every equipment as follows:

- For compressor and pump, process variable is duty and taken directly from simulation results.
- For separator and column, sizing was performed using available vessel and tray sizing tools.
- For heat exchanger, sizing was estimated using Aspen Exchanger Design and Rating tool.
- For membrane equipment, sizing (surface area calculation) was done based on capacity comparison with other installed units.

3.3. Capital Expenditure (CAPEX) Calculation Method

Capital expenditure for main equipment was estimated using Aspen Process Economic Evaluation. Methods used in this CAPEX calculation are as follows.

- The cost for each equipment is calculated based on three factors: bare equipment, material (for piping, instrument, electrical, insulation, and civil), and labor. If equipment is quoted from other references, this price will be inserted as packaged unit. The calculated total cost is known as Total Direct Cost (TDC).
- Indirect cost was added as 46.52% of TDC. Indirect cost covers various components: engineering, site office, permit & license, insurances, taxes, transportation, and overhead.
- Contractor profit and contingency cost are assumed to be 5% and 25% of TDC respectively.
- The final capex was estimated with some adjustment to incorporate the conditions in Indonesia.

3.4. Operating Expenditure (OPEX) Calculation Method

OPEX calculation was based on the consumption figures of supporting materials, electricity, cooling water, hot oil, and hydrocarbon loss. Prices of the components and basis used in OPEX calculations were quoted from price list commonly used in industry. Indicators used in total OPEX calculation can be seen in Table 3.
4. Result and Discussion

Each of simulation flowsheet shown in Figure 2 – Figure 4 was executed for the same evaluation basis as listed in Table 2. The quantitative results of this flow-sheeting calculation are concisely reported as block flow diagram for each evaluated technology as shown in Figure 5 – Figure 7. Each diagram contains complete information to be used for equipment sizing, estimation of CAPEX, and estimation of OPEX.

| Cost Item                              | Typical Range of Multiplying Factors | Value Used   |
|----------------------------------------|--------------------------------------|--------------|
| 1. Direct Manufacturing Cost           |                                      |              |
| a. Raw Material                        | C_{RM}                               | C_{RM}       |
| b. Waste Treatment                     | C_{WT}                               | C_{WT}       |
| c. Utilities                           | C_{UT}                               | C_{UT}       |
| d. Operating Labor                     | C_{OL}                               | C_{OL}       |
| e. Direct supervisory and Clerical Labor| (0.1 - 0.25) C_{OL}                  | 0.18 C_{OL}  |
| f. Maintenance and Repairs             | (0.02-0.1) FCI                       | 0.06 FCI     |
| g. Operating Supplies                  | (0.1 - 0.2) Line 1.F                 | 0.09 FCI     |
| h. Laboratory Charges                  | (0.1-0.2) C_{OL}                     | 0.15 C_{OL}  |
| i. Patents and Royalty                 | (0 -0.06) COM                        | 0.03 COM     |

Total Direct Manufacturing Cost: C_{RM} + C_{WT} + C_{UT} + 1.33 C_{OL} + 0.03 COM + 0.069 FCI

| 2. Fixed Manufacturing Cost            |                                      |              |
| a. Depreciation                        | 0.1 FCI                              | 0.1 FCI      |
| b. Local taxes and insurance           | (0.014 - 0.05) FCI                   |              |
| c. Plant Overhead Costs                | 0.15 Line 1D + Line 1F              | 0.177 C_{OL} + 0.009 FCI |

Total Fixed Manufacturing Cost: 0.708 COL + 0.068 FCI + depreciation

| 3. General Manufacturing Expenses      |                                      |              |
| a. Administration Costs                | 0.15 Line 1D + Line 1F               | 0.177 C_{OL} + 0.009 FCI |
| b. Distribution and Selling Costs      | (0.02 - 0.2) COM                     | 0.11 COM     |
| c. Research and Development            | 0.05 COM                             | 0.05 COM     |

Total General Manufacturing Costs: 0.177 COL + 0.009 FCI + 0.16 COM

Total Costs: C_{RM} + C_{WT} + C_{UT} + 2.215 COL + 0.19 COM + 0.146 FCI + depreciation

CAPEX of the improved cryogenic distillation technology is estimated about $265 Million. This is smaller than CAPEX of the chemical-physical absorption technology (about $371 Million) and Hybrid (Membrane +chemical-physical absorption in series) technology (about $319 Million). Detail of CAPEX estimation for each process section of the improved cryogenic distillation process, chemical-physical absorption technology, and hybrid technology are shown in Table 4 respectively.
CAPEX distribution for each equipment for improved cryogenic distillation, chemical-physical absorption technology, and hybrid technology are shown in Figure 8 respectively. In improve cryogenic distillation technology, CAPEX is dominated by equipment price for column + refrigeration system (63%), while the other two technologies require small cost for column (less than 5%).

Power, hot oil, propane and ethane are used as utilities in improved cryogenic distillation technology. Propane and ethane are used as refrigeration working fluids. In Chemical-Physical Absorption and Hybrid technology, make-up solvent is used as additional utility beside previous mentioned utilities. This utility consumption is used as a basis for OPEX calculation of each technology. OPEX of improved cryogenic distillation, chemical-physical absorption, and hybrid technology is shown in Table 5, Table 6, and Table 7 respectively.

Comparison for three methods based on economical aspect and hydrocarbon losses is shown in Figure 9. Improved Cryogenic Distillation is the lowest cost of all three technologies, followed by hybrid technology and chemical-physical absorption process. Improved cryogenic distillation also shows the lowest hydrocarbon losses compared to two other technologies. This shows that this new improved cryogenic distillation process is promising in economical aspect and hydrocarbon losses.

Figure 5. Block flow diagram of the improved cryogenic distillation process.
Figure 6. Block flow diagram of the physical-chemical absorption process.

Figure 7. Block flow diagram of the hybrid technology.

Table 4. Capital Expenditure Comparison for the Three Technologies Evaluated
Process Section | Improved Cryogenic Distillation | Chemical-Physical Absorption | Hybrid Technology
--- | --- | --- | ---
Acid Gas Compression Unit | $60,065,850.69$ | - | $93,918,325.96$
Acid Gas Injection | $2,386,653.92$ | - | -
Condensate Regeneration | $33,835,049.58$ | - | -
Cooling Water Handling | $4,892,749.93$ | $3,154,966.98$ | $7,958,199.91$
Dehydration Unit | $15,402,525.77$ | $13,269,137.43$ | $13,256,450.12$
Dew Point Control Unit | $-13,814,073.36$ | $13,800,865.00$
Hot Oil Handling | $928,124.33$ | $21,063,993.38$ | $7,650,742.44$
Main Absorption | - | $85,135,039.32$ | $95,184,790.93$
Main Fractionation | $85,985,178.87$ | - | -
Membrane Unit | - | - | $42,668,343.28$
Power Generation | - | - | $37,550,981.27$
Refrigeration Unit | $79,408,466.91$ | $115,199,696.85$ | $7,062,301.08$
Turbine Generation | $42,513,250.68$ | $59,961,241.99$ | -

Total CAPEX | $265,352,000.00$ | $371,664,000.00$ | $319,051,000.00$

Figure 8. Proportion of Equipment Cost for the three technologies evaluated.

Table 5. OPEX Calculation of Improved Cryogenic Distillation
### Table 6. OPEX Calculation of Chemical-Physical Absorption

| Consumption                      | Price   | Sub Total ($/yr) |
|----------------------------------|---------|------------------|
| **Raw Materials (CRM)**          |         |                  |
| Make-up Propane                  | 20.3 ton/h | 16 $/ton | 2,575,076 |
| Make-up Ethylene                 | 11.1 ton/h | 30 $/ton | 2,627,267 |
| TEG                              | 1.0 ton/h | 167 $/ton | 1,354,320 |
| Product for Fuel                 | 6.7 MMSCFD | 10 $/MMBTU | 13,597,793 |
| **Utility (CUT)**                |         |                  |
| Cooling Water                    | 178.7 m3/h | 1.32 $/m3 | 1,863,328 |
| Makeup Hot Oil                   | 0.7 ton/h | 250 $/ton | 1,353,513 |
| **Operating Labor (COL)**        |         |                  |
| 30 person(s)                     | 4200 $/person/yr | 126,000 |
| **Cost of HC Loss (CHL)**        | 21 MMSCFD | 10 $/MMBTU | 42,948,905 |
| **Fixed Capital Investment (FCI)** |        | $265,352,000   |
| **Total OPEX Without CHL**       | 0.18FCI + 2.73COL + 1.23(CRM+CUT) | 76,854,036 |
| **Total OPEX With CHL**          | 0.18FCI + 2.73COL + 1.23(CRM+CUT) + CHL | 119,802,941 |

### Table 7. OPEX Calculation of Hybrid Technology

| Consumption                      | Price   | Sub Total ($/yr) |
|----------------------------------|---------|------------------|
| **Raw Materials (CRM)**          |         |                  |
| MDEA + Sulfolane                 | 1.82E-02 ton/h | 12000 $/ton | 1,725,840 |
| TEG                              | 1.0 ton/h | 167 $/ton | 1,354,320 |
| Make-up Propane                  | 79.6 ton/h | 16 $/ton | 10,084,966 |
| Ethylene Glycol                  | 1.87E-02 ton/h | 1200 $/ton | 5,378 |
| Product for Fuel                 | 10.7 MMSCFD | 10 $/MMBTU | 21,713,219 |
| **Utility (CUL)**                |         |                  |
| Cooling Water                    | 113.9 m3/h | 1.32 $/m3 | 1,187,406 |
| Hot Oil                          | 31 ton/h | 250 $/ton | 61,018,079 |
| **Operating Labor (COL)**        |         |                  |
| 30 person(s)                     | 4200 $/person/yr | 126,000 |
| **Cost of HC Loss (CHL)**        | 21 MMSCFD | 10 $/MMBTU | 42,948,905 |
| **Fixed Capital Investment (FCI)** |        | $371,664,000   |
| **Total OPEX Without CHL**       | 0.18FCI + 2.73COL + 1.23(CRM+CUT) | 186,663,226 |
| **Total OPEX With CHL**          | 0.18FCI + 2.73COL + 1.23(CRM+CUT) + CHL | 223,951,710 |

### 5. Conclusion

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**Consumption**

**Price**

**Sub Total ($/yr)**

| Raw Materials (CRM) |         |                  |
| Make-up Propane     | 20.3 ton/h | 16 $/ton | 2,575,076 |
| Make-up Ethylene    | 11.1 ton/h | 30 $/ton | 2,627,267 |
| TEG                 | 1.0 ton/h | 167 $/ton | 1,354,320 |
| Product for Fuel    | 6.7 MMSCFD | 10 $/MMBTU | 13,597,793 |

| Utility (CUT) |         |                  |
| Cooling Water | 178.7 m3/h | 1.32 $/m3 | 1,863,328 |
| Makeup Hot Oil | 0.7 ton/h | 250 $/ton | 1,353,513 |

| Operating Labor (COL) |         |                  |
| 30 person(s) | 4200 $/person/yr | 126,000 |

| Cost of HC Loss (CHL) | 21 MMSCFD | 10 $/MMBTU | 42,948,905 |

| Fixed Capital Investment (FCI) | $265,352,000 |

**Total OPEX Without CHL**

0.18FCI + 2.73COL + 1.23(CRM+CUT) = 76,854,036

**Total OPEX With CHL**

0.18FCI + 2.73COL + 1.23(CRM+CUT) + CHL = 119,802,941

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**Consumption**

**Price**

**Sub Total ($/yr)**

| Raw Materials (CRM) |         |                  |
| MDEA + Sulfolane    | 1.82E-02 ton/h | 12000 $/ton | 1,725,840 |
| TEG                 | 1.0 ton/h | 167 $/ton | 1,354,320 |
| Make-up Propane     | 79.6 ton/h | 16 $/ton | 10,084,966 |
| Ethylene Glycol     | 1.87E-02 ton/h | 1200 $/ton | 5,378 |
| Product for Fuel    | 10.7 MMSCFD | 10 $/MMBTU | 21,713,219 |

| Utility (CUL) |         |                  |
| Cooling Water | 113.9 m3/h | 1.32 $/m3 | 1,187,406 |
| Hot Oil       | 31 ton/h | 250 $/ton | 61,018,079 |

| Operating Labor (COL) |         |                  |
| 30 person(s) | 4200 $/person/yr | 126,000 |

| Cost of HC Loss (CHL) | 21 MMSCFD | 10 $/MMBTU | 42,948,905 |

| Fixed Capital Investment (FCI) | $371,664,000 |

**Total OPEX Without CHL**

0.18FCI + 2.73COL + 1.23(CRM+CUT) = 186,663,226

**Total OPEX With CHL**

0.18FCI + 2.73COL + 1.23(CRM+CUT) + CHL = 223,951,710

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**Consumption**

**Price**

**Sub Total ($/yr)**

| Raw Materials (CRM) |         |                  |
| MDEA + Piperazin    | 4.29E-03 ton/h | 6000 $/ton | 204,000 |
| TEG                 | 1.0 ton/h | 167 $/ton | 1,354,320 |
| Product for Fuel    | 6.2 MMSCFD | 10 $/MMBTU | 12,673,802 |

| Utility (CUL) |         |                  |
| Air Pendingin     | 293.5 m3/h | 1.32 $/m3 | 3,059,420 |
| Hot Oil           | 10.3 ton/h | 250 $/ton | 20,306,969 |

| Operating Labor (COL) |         |                  |
| 31 person(s) | 4200 $/person/yr | 130,200 |

| Cost of HC Loss (CHL) | 11 MMSCFD | 10 $/MMBTU | 42,948,905 |

| Fixed Capital Investment (FCI) | $319,051,000 |

**Total OPEX Without CHL**

0.18FCI + 2.73COL + 1.23(CRM+CUT) = 104,030,794

**Total OPEX With CHL**

0.18FCI + 2.73COL + 1.23(CRM+CUT) + CHL = 223,951,710

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**5. Conclusion**

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Gas field with high content of acid gases requires preliminary processing technology capable of cost-effectively eliminating high acid gas content and large amounts of gas. Whether the processed acid gas will be re-injected or released into the air after the sulfur content is taken, the three process technologies studied have the potential to be applied, including new improved cryogenic distillation that applying freezing of acid gas in column.

Quantitative technical evaluation of total hydrocarbon consumption shows that Chemical-Physical Absorption technology will require the largest operating cost (OPEX), followed by Membrane Hybrid, and then new improved cryogenic distillation. The same sequence is also obtained when an evaluation is made on the required capital cost (CAPEX).

However, this new improved cryogenic distillation has concern about operability, particularly during start-up and shut down. This can be mitigated by a more specialized review by visiting a demonstration facility to discuss in more detail the operational issues.

![Comparison of the three technologies evaluated for CAPEX and OPEX and Hydrocarbon Losses](image)

**FIGURE 9.** Comparison of the three technologies evaluated for (a) CAPEX and OPEX (b) Hydrocarbon Losses

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