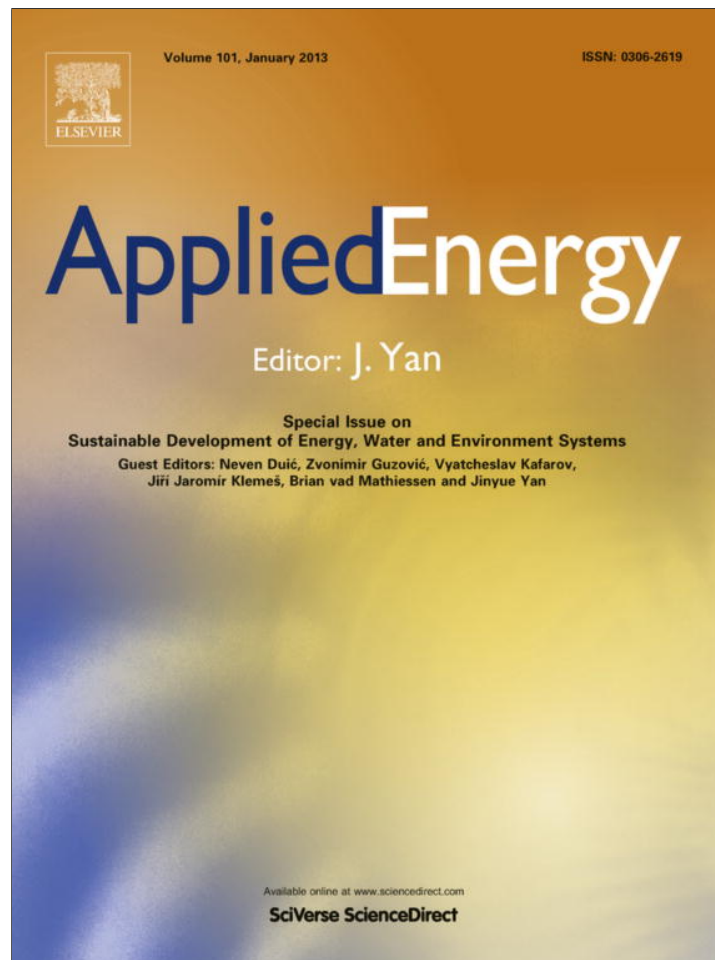


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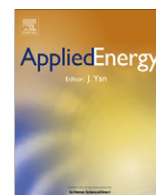
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Analysis, synthesis, and design of a one-step dimethyl ether production via a thermodynamic approach

Hsi-Jen Chen ^{*}, Chei-Wei Fan, Chiou-Shia Yu

Dept. of Chemical and Materials Engineering, Tamkang University, Tamsui Dist., New Taipei City 25137, Taiwan

HIGHLIGHTS

- ▶ We analyze a one-step DME production via a thermodynamic approach.
- ▶ We synthesize a one-step DME process.
- ▶ We design a one-step DME process with pinch technology.

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ABSTRACT

In this work, we have developed a direct one-step process design on an oxygenate production, namely, dimethyl ether (DME). DME can be used as a cetane-number booster for diesel, in addition to being capable of a substitute for liquefied petroleum gas (LPG). In order to analyze the independent chemical reactions involved in the reactor, it is necessary to carry out a study of the chemical reaction stoichiometry. And with a specific syngas feed, the following reactions are found: (1) $\text{CO}_2 + \text{H}_2 = \text{H}_2\text{O} + \text{CO}$, (2) $\text{CO} + 2\text{H}_2 = \text{CH}_3\text{OH}$, and (3) $3\text{CO} + 3\text{H}_2 = (\text{CH}_3)_2\text{O} + \text{CO}_2$. To gain an insight into the reactor design, we have also utilized the concept of thermodynamics, including equilibrium-constant method and the minimization of Gibbs free energy. Additionally, we have also united the pinch technology with the base-case design for heat exchanger network synthesis in order to compare the energy consumption and capital costs of the process with/without heat integration. Two kinds of software were used in the research—Aspen Plus and SuperTarget. The former was used for the process synthesis, design, and simulation; the latter was used to carry out the pinch analysis and the synthesis of heat exchanger network.

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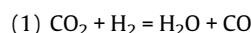
1. Introduction

Dimethyl ether (DME) is a clean energy which can be produced from syngas. Due to its similar properties of LPG, DME can be used as a fuel for home heating and cooking. Additionally, it can be used as transportation fuel due to its high cetane number. Also it can be used as a starting reagent for making special chemicals such as methyl acetate and acetic anhydride. Previous related studies investigate a double integrated reactor for DME synthesis and hydrogen production [1], the optimization of reformer to produce syngas for methanol production [2], a large-scale combined production of methanol and electricity from natural gas from an economic point of view [3]. A comprehensive study from the perspective of thermodynamics has not been performed. This study aims to simulate a dimethyl ether process and its efficient use of energy. Chemical reactor's design is based on the concept of thermodynamics, including equilibrium constant method and the minimization of

Gibbs free energy. The feed contains syngas, with a mole ratio of $\text{H}_2:\text{CO}:\text{CO}_2 = 5:4:1$. Plant capacity is on the order of 45,000 metric tons per year of 99.9 mol% purity DME. Pinch technology [4] is utilized to deal with energy savings of the dimethyl ether plant. Two process software tools, Aspen Plus [5] and SuperTarget [6] were used in this paper. The results of this study may be of interest to researchers interested in the development of clean energy.

2. Chemical reaction stoichiometry

Chemical reaction stoichiometry (CRS) is a branch of chemical stoichiometry dealing with the constraints, in the form of chemical reactions, placed on changes in the composition of a closed reacting system by the requirement for conservation of the amount of each atomic species. Using CRS for the reaction path analysis in the one-step dimethyl ether production, we find the independent equilibrium reactions involved are, given that chemical species H_2 , CO , CO_2 , H_2O , CH_3OH and $(\text{CH}_3)_2\text{O}$, as follows:



^{*} Corresponding author. Fax: +886 2 26209887.

E-mail address: hjchen@mail.tku.edu.tw (H.-J. Chen).

- (2) $\text{CO} + 2\text{H}_2 = \text{CH}_3\text{OH}$
- (3) $3\text{CO} + 3\text{H}_2 = (\text{CH}_3)_2\text{O} + \text{CO}_2$

The reactions are the water–gas shift reaction, the methanol synthesis reaction, the DME synthesis reaction, respectively. It is noteworthy that the reaction formulas concerning DME synthesis [7] are:

- (1) $3\text{CO} + 3\text{H}_2 \rightarrow (\text{CH}_3)_2\text{O} + \text{CO}_2$
- (2) $2\text{CO} + 4\text{H}_2 \rightarrow (\text{CH}_3)_2\text{O} + \text{H}_2\text{O}$
- (3) $2\text{CO} + 4\text{H}_2 \rightarrow 2\text{CH}_3\text{OH}$
- (4) $2\text{CH}_3\text{OH} \rightarrow (\text{CH}_3)_2\text{O} + \text{H}_2\text{O}$
- (5) $\text{CO} + \text{H}_2\text{O} \rightarrow \text{CO}_2 + \text{H}_2$

The afore-mentioned five reactions are not independent and can be reduced to three independent equilibrium reactions.

3. Sensitivity analysis of key thermodynamic parameters

The parametric analysis is achieved to evaluate the effects of key parameters, that is, reaction pressure and temperature, on the system performance. The yields of DME and methanol vs. the key parameters are as shown in Figs. 1–4. We will now use equilibrium constant method as well as free energy minimization method to analyze both temperature and pressure effects on the reaction yields in detail. According to the Gibbs free energy at standard state

$$\ln K_a = -\frac{\Delta G}{RT} \quad (1)$$

Eq. (1) states that the reaction temperature has effect on the equilibrium constant K_a . Therefore, we can calculate the equilibrium compositions under fixed reaction temperature. The following van't Hoff equation can be used to relate temperature to the equilibrium constant

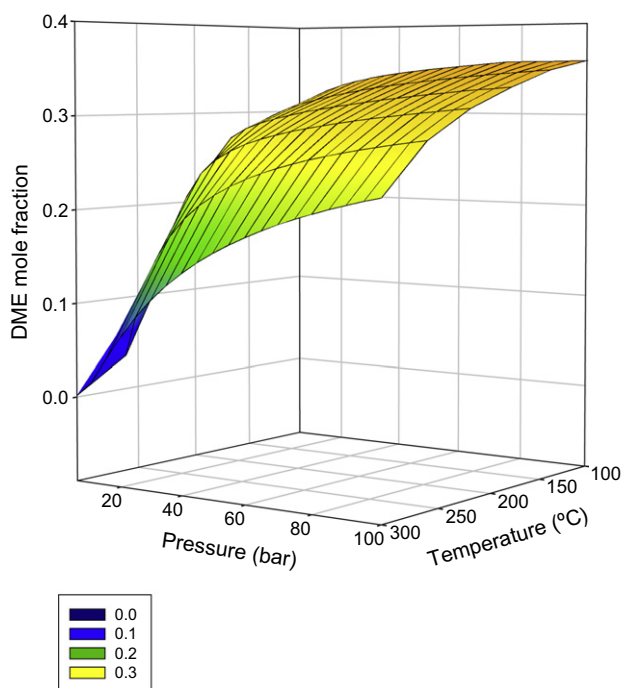


Fig. 1. Reaction yield of DME versus reaction pressure and temperature at a molar ratio of syngas $\text{H}_2:\text{CO}:\text{CO}_2 = 5:4:1$.

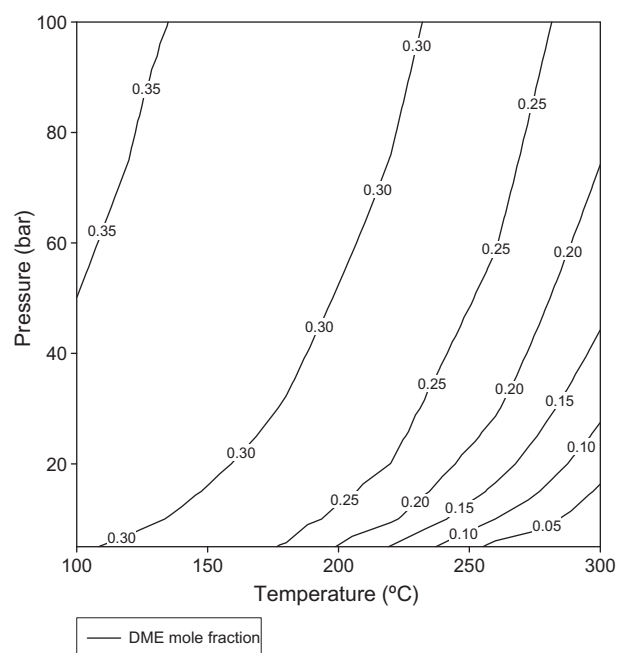


Fig. 2. Contours of DME yield versus reaction pressure and temperature at a molar ratio of syngas $\text{H}_2:\text{CO}:\text{CO}_2 = 5:4:1$.

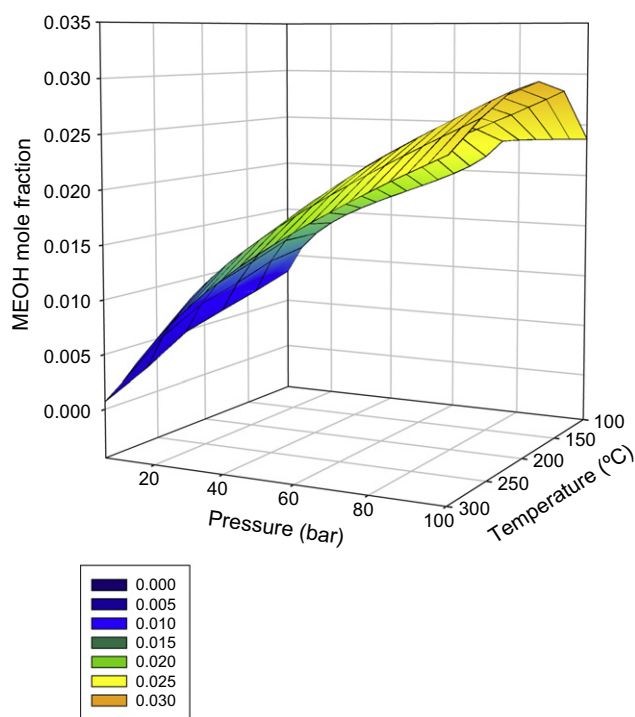


Fig. 3. Reaction yield of methanol versus reaction pressure and temperature at a molar ratio of syngas $\text{H}_2:\text{CO}:\text{CO}_2 = 5:4:1$.

$$\ln \left(\frac{K_a}{K_{aR}} \right) = \frac{\Delta G_R^0}{RT} - \frac{\Delta G_R^0}{RT_R} = -\frac{\Delta H_R^0}{R} \left(\frac{1}{T} - \frac{1}{T_R} \right) \quad (2)$$

where ΔH_R^0 is the reaction at standard state, R the gas constant (8.314 kJ/kmol K), T the system temperature (K) and T_R is the reference temperature (K).

Eq. (2) states that if reaction is exothermic, $\Delta H_R^0 < 0$, the equilibrium constant will decrease as the temperature increases; if the reaction is endothermic, $\Delta H_R^0 > 0$, the equilibrium constant will

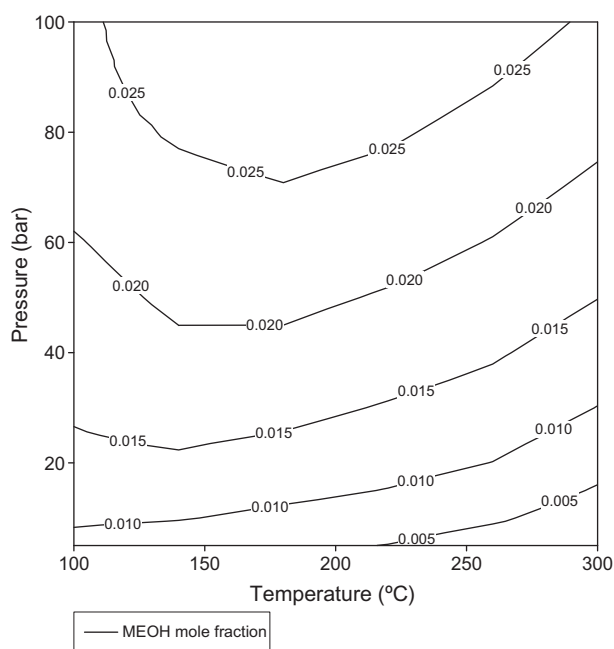


Fig. 4. Contours of methanol yield versus reaction pressure and temperature at a molar ratio of syngas $H_2:CO:CO_2 = 5:4:1$.

Table 1
Free energies and heat of formation at standard state (298 K, 1.013 bar).

Species	ΔG_f^0 (kJ/mol)	ΔH_f^0 (kJ/mol)
H_2	0	0
CO	-136.93	-110.35
CO_2	-393.73	-392.86
H_2O	-228.22	-241.42
Methanol	-162.06	-200.6
DME	-112.62	-183.8

increase as the temperature increases. Table 1 lists the chemical species involved in the direct DME synthesis:

From Table 1, we can calculate the Gibbs free energy, heat of reaction and equilibrium constants in the direct DME synthesis (Table 2).

3.1. Temperature effect

According to the short-cut van't Hoff equation

$$\ln K_a = -\frac{\Delta H_R^0}{R} \left(\frac{1}{T} - \frac{1}{T_R} \right) - \frac{\Delta G_{298}}{RT_{298}} \quad (3)$$

For a feed mole ratio of $H_2:CO:CO_2 = 5:4:1$, we derived the following simultaneous equations corresponding to the three independent reactions

$$x_1(4 + x_1 - x_2 - 3x_3) - K_{a1}(5 - x_1 - 2x_2 - 3x_3)(1 - x_1 + x_3) = 0$$

$$x_2(10 - 2x_2 - 4x_3)^2 - K_{a2}(5 - x_1 - 2x_2 - 3x_3)(4 + x_1 - x_2 - 3x_3) = 0$$

Table 2
Free energies, reaction heats, and equilibrium constants.

Reaction	ΔG	ΔH	K_a
(1) $H_2 + CO_2 = H_2O + CO$	28.58	41.09	9.78×10^{-6}
(2) $2H_2 + CO = CH_3OH$	-25.13	-90.25	25412.46
(3) $3H_2 + 3CO = DME + CO_2$	-95.56	-245.61	5.63×10^{16}

Table 3
Yields vs. various reaction temperatures at atmospheric pressure 1.013 bar.

Species temperature (°C)	H_2O		CH_3OH		$(CH_3)_2O$	
	Mole	Yield	Mole	Yield	Mole	Yield
200	0.009	0.0013	0.015	0.002	0.55	0.07
250	0.015	0.0015	0.004	390 ppm	0.013	0.0013
260	0.018	0.0018	0.003	260 ppm	0.005	470 ppm
300	0.033	0.0033	0.0006	65 ppm	0.0001	10 ppm

Table 4
Yields of component species at various temperatures and pressures.

Temperature (°C)	Pressure (bar)	Yield					
		DME	Methanol	H_2	CO	CO_2	H_2O
200	30	0.287	0.017	0.174	0.017	0.483	0.022
	50	0.296	0.018	0.159	0.007	0.493	0.026
	70	0.306	0.023	0.132	0.007	0.493	0.040
250	30	0.235	0.016	0.234	0.082	0.418	0.015
	50	0.261	0.020	0.198	0.052	0.448	0.021
	70	0.274	0.023	0.177	0.038	0.462	0.026
260	30	0.219	0.016	0.251	0.105	0.395	0.014
	50	0.249	0.020	0.211	0.068	0.432	0.020
	70	0.264	0.023	0.188	0.050	0.450	0.025
300	30	0.137	0.014	0.336	0.217	0.283	0.012
	50	0.184	0.020	0.279	0.156	0.344	0.017
	70	0.210	0.023	0.245	0.122	0.378	0.021

$$x_3(1 - x_1 + x_3)(10 - 2x_2 - 4x_3)^4 - K_{a3}(5 - x_1 - 2x_2 - 3x_3)^3(4 + x_1 - x_2 - 3x_3)^3 = 0$$

where x_1 , x_2 and x_3 represent moles of H_2O , CH_3O and $(CH_3)_2O$, respectively. K_{a1} , K_{a2} and K_{a3} represent equilibrium constant for reactions $CO_2 + H_2 = H_2O + CO$, $CO + 2H_2 = CH_3OH$ and $3CO + 3H_2 = (CH_3)_2O + CO_2$, respectively. Table 3 shows the results for the direct DME synthesis for temperatures of 200, 250, 260 and 300 °C. This table reveals that reaction yield is very low at 1.013 bar for both methanol and DME, especially under high reaction temperature.

Another approach to determine the reaction compositions is to apply the method of free-energy minimization:

$$\frac{G}{RT} = \sum_{i=1}^C n_i \left[\frac{G_i^0}{RT} + \ln \left(\frac{P}{P^0} \right) + \ln \frac{n_i}{\sum n_i} \right] \quad (4)$$

where n_i is the number of moles of individual component, G_i^0 the free energy of component i at standard pressure and temperature T (kJ/mol), G the total free energies of the reaction system (kJ/mol), T the equilibrium temperature (K), C the number of species, P the system pressure (bar) and P^0 is the atmospheric pressure (bar).

The mathematical optimization problem accounting for both temperature and pressure effects on the direct DME synthesis can be formulated as follows:

$$\text{Minimize : } \frac{G}{RT} \quad (5)$$

$$\text{Subject to Oxygen Balance : } 2n_2 + n_3 + n_4 + n_5 + n_6 = 6 \quad (6)$$

$$\text{Hydrogen Balance : } 2n_1 + 2n_3 + 4n_5 + 6n_6 = 10 \quad (7)$$

$$\text{Carbon Balance : } n_2 + n_4 + n_5 + 2n_6 = 5 \quad (8)$$

(note that $n_1 = H_2$, $n_2 = CO_2$, $n_3 = H_2O$, $n_4 = CO$, $n_5 = CH_3OH$ and $n_6 = (CH_3)_2O$).

Table 4 lists reaction yields for various temperatures of 200, 250, 260 and 300 °C, and various pressures of 30, 50 and 70 bar. From Table 4, it is observed that while high pressure and low temperature favor DME's production. However, lower temperatures favor the formation of CO_2 and H_2O which in turn would raise the operating

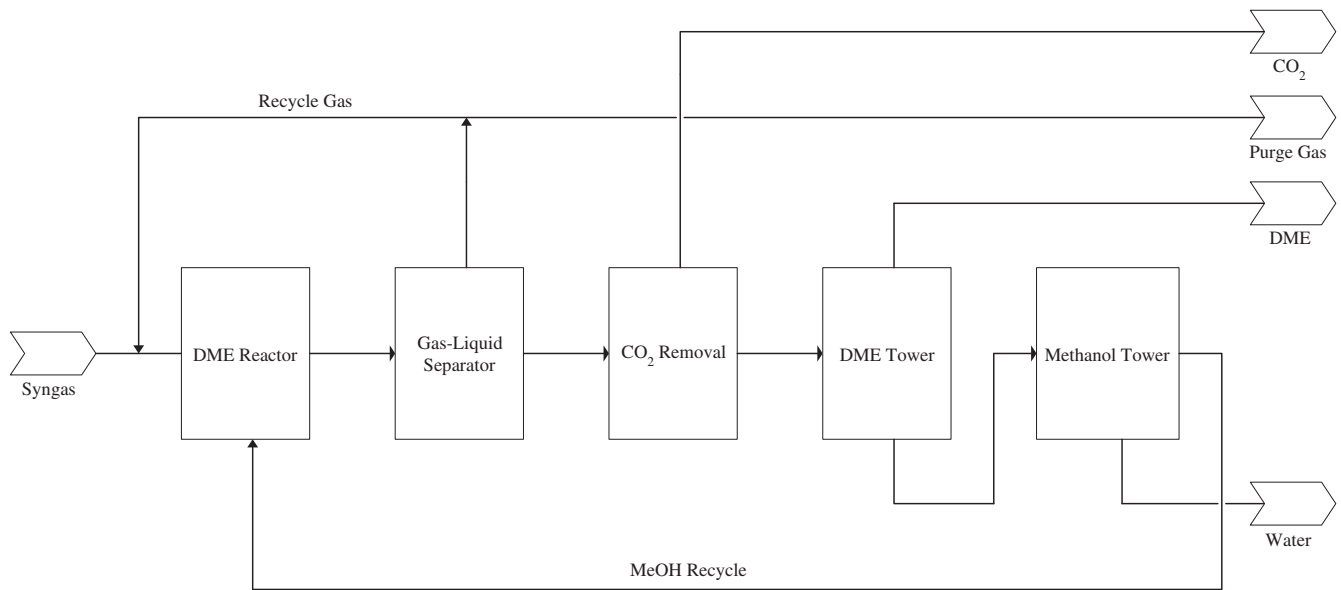


Fig. 5. Block flow diagram for the production of the direct DME synthesis.

costs of both tail gas and waste water. Consequently, we decided to choose 50 bar and 260 °C as our final design parameters.

4. Process synthesis and design

First, a block flow diagram is shown in Fig. 5 for the direct synthesis of DME from syngas. The process features DME reactor, gas and liquid recycle streams, a CO₂-removal tower, two distillation columns. Second, by following the guidelines of design heuristics [8] and hierarchical principle of onion model [9], we are able to

depict a flow diagram of the one-step DME synthesis. Fig. 6 shows a preliminary process flow diagram for the production of 45,000 tonnes DME per year. Referring to Fig. 2, the syngas is supplied at a pressure of 1.013 bar, 120 °C, and is mixed with recycled stream from flash drum V-101. The mixed streams enter a three-stage compressor (50 bar), cooled (260 °C), and then to the one-step-process reactor R-101. The reaction is exothermic; therefore, it can be used to produce high-pressure steam. The reactor effluent, Stream 4, is then cooled to –10 °C and flashed at 25 bar to separate the unreacted syngas from the DME–methanol–water solution. A large portion of the vapor leaving V-101 is recycled to increase the

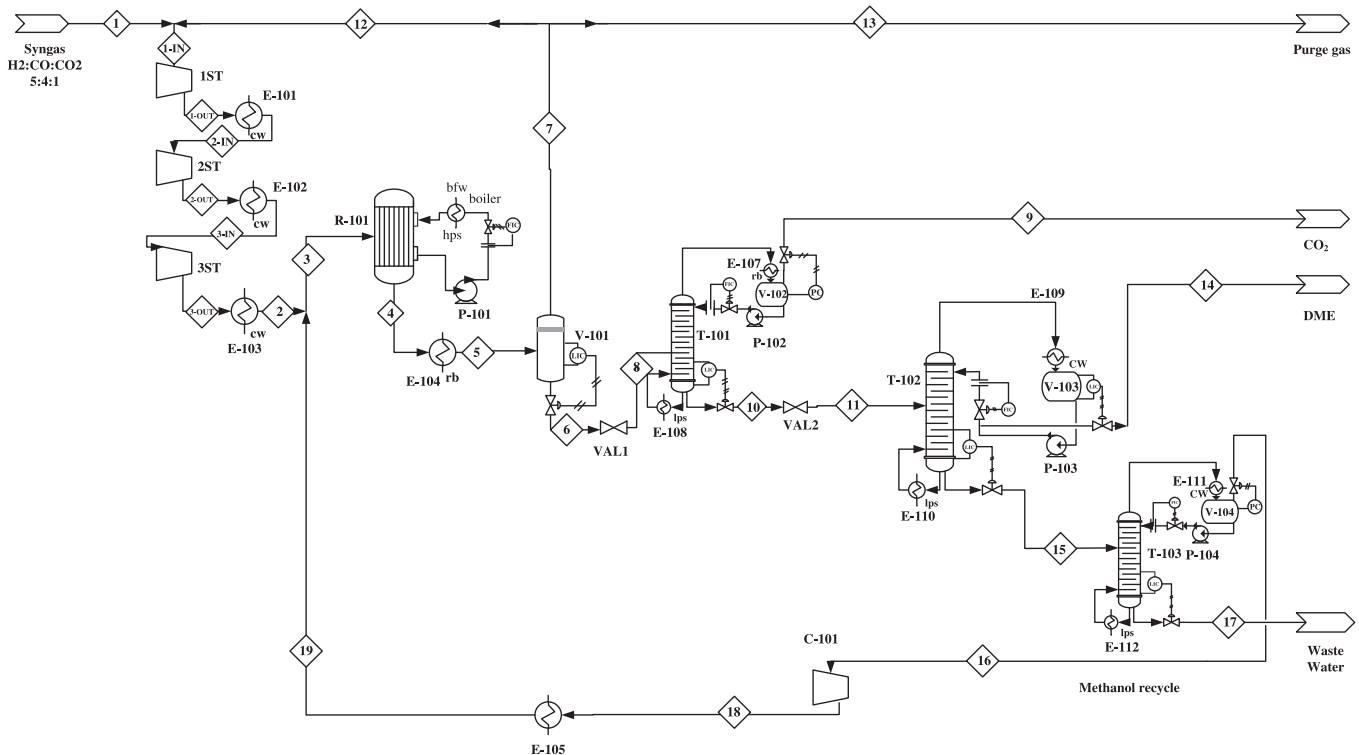


Fig. 6. Preliminary process flow diagram for the production of DME without heat integration.

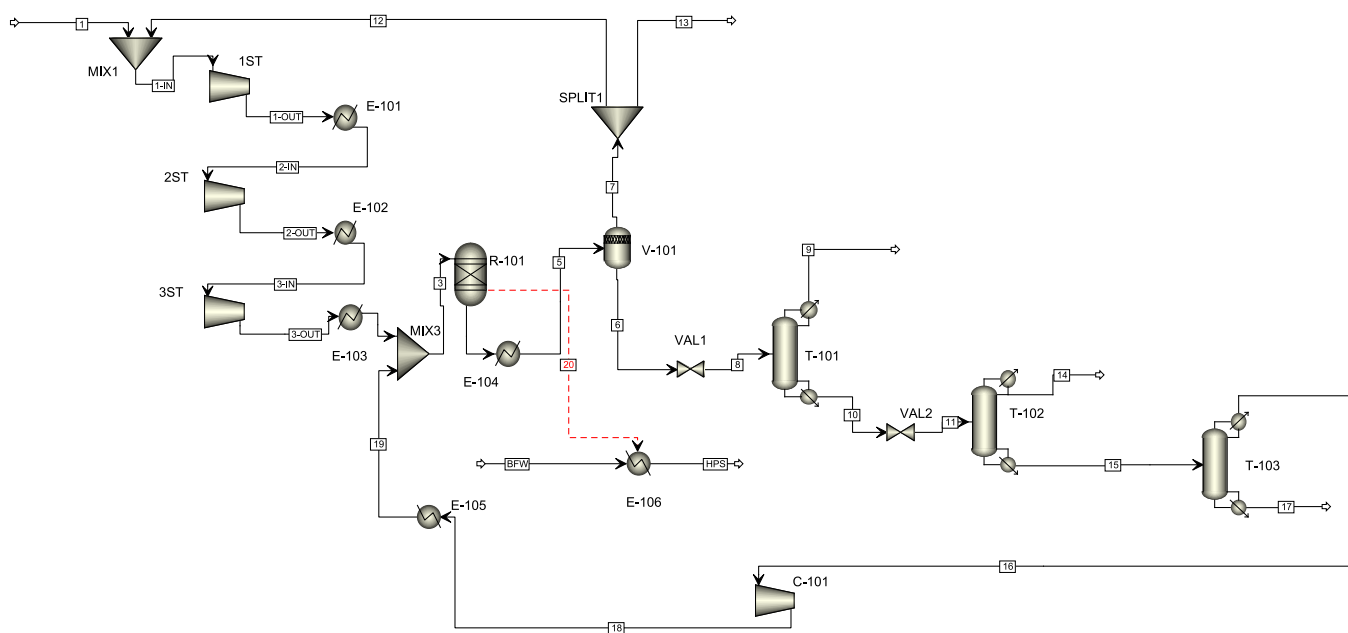


Fig. 7. Flowsheet structure used by Aspen Plus in the simulation of the direct DME synthesis.

overall DME conversion and the remainder purged. The DME–methanol–water containing some residual gas CO/CO₂, Stream 6, is sent to distillation tower T-101 with a partial condenser to remove the residual gas. Stream 10, from the bottom of T-101, is sent to tower T-102 to separate the DME as a top product (with 99.9 mol% purity) from the DME–methanol–water solution. The methanol–water solution, Stream 15, is sent to T-103 to separate water from the methanol. In this tower, a methanol-rich stream is compressed to 50 bar prior to being sent to mix with Stream 2. Fig. 5 represents a base-case design for this study. Note that a base case is a reasonable first estimate of a process design that has not yet been optimized.

5. Process simulation

Corresponding to Fig. 6, a process flowsheet is as shown in Fig. 7. It is worth noting that since the one-step DME process is highly exothermic, we have to recover this heat while maintaining reactor temperature at 260 °C. Using “Heat Stream” in Aspen Plus,

we are able to heat-up the boiler feed water to high-pressure steam at 42 bar and 254 °C with an amount of 754 kmol/h. The simulation result, Stream 14, shows that DME amounts to 5680 kg/h, which gives a stream factor (SF) of 90(=45,000,000/5680/8760). The “UNIQU-RK”, that is, UNIQUAC/Redlich–Kwong equation of state with Henry’s law, thermo-physical property package in Aspen Plus is used for the design of all systems. Also the Aspen “Design Specs/Vary” feature is used to drive overhead composition to desired value (DME purity = 99.9 mol%) by manipulating molar distillate flowrate. A portion of the stream summary is presented in Table 5.

6. Pinch technology

The pinch technology consists of the following steps [10]:

- data extraction;
- pinch analysis;

Table 5
A portion of stream summary for the direct one-step DME process.

	1	3	4	5	14	17	BFW	HPS
Temperature (°C)	120	259.3	260	–10	44.4	179.8	90	254
Pressure (bar)	1.013	50	50	30	10	10	1.013	42.013
Vapor fraction	1	1	1	0.582	0	0	0	1
Mole flow (kmol/h)	1000	1387.09	845.205	845.205	123.298	24.189	753.585	753.585
Mass flow (kg/h)	16613.08	26142.18	26141.29	26141.29	5678.955	436.253	13576.04	13576.04
Volume flow (m ³ /h)	32276.89	1243.831	735.595	358.827	9.131	0.528	14.623	684.976
Enthalpy (MMkcal/h)	–19.275	–34.88	–43.051	–47.058	–5.923	–1.579	–50.564	–42.39
<i>Mole flow (kmol/h)</i>								
CO ₂	100	260.208	371.403	371.403	0.039	0	0	0
H ₂	500	661.965	231.333	231.333	0	0	0	0
CO	400	431.978	49.848	49.848	0	0	0	0
DME	0	15.39	150.794	150.794	123.175	0	0	0
Methanol	0	16.468	16.599	16.599	0.085	0.034	0	0
Water	0	1.08	25.228	25.228	0	24.155	753.585	753.585
<i>Mole fraction</i>								
CO ₂	0.1	0.188	0.439	0.439	0	0	0	0
H ₂	0.5	0.477	0.274	0.274	0	0	0	0
CO	0.4	0.311	0.059	0.059	0	0	0	0
DME	0	0.011	0.178	0.178	0.999	0	0	0
Methanol	0	0.012	0.02	0.02	0.001	0.001	0	0
Water	0	0.001	0.03	0.03	0	0.999	1	1

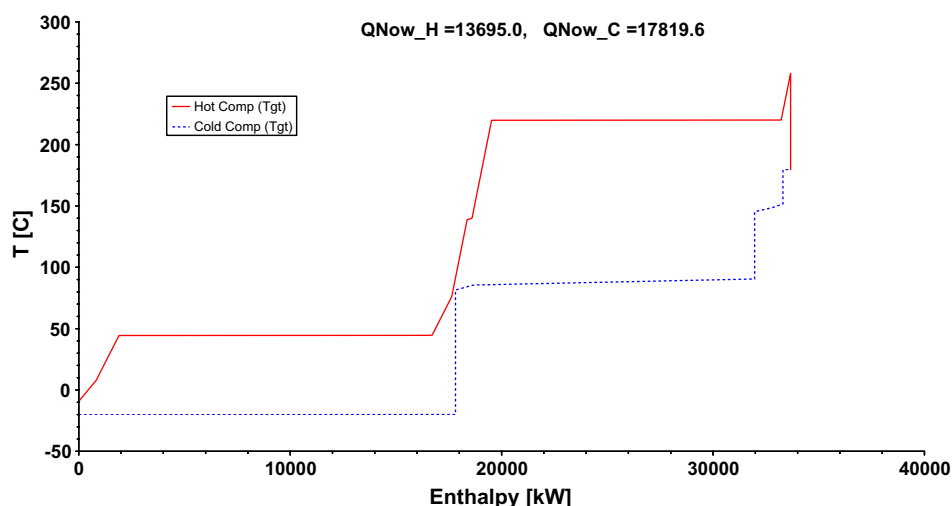


Fig. 8. Composite hot/cold streams curve of the one-step DME process for $\Delta T_{\min} = 10$ °C.

Table 6

Comparisons of various minimum approach temperatures in the synthesis of heat exchanger networks.

Minimum approach temperatures ΔT_{\min} (°C)	Hot utilities Q_H (kW)	Cold utilities Q_C (Kw)	No. of Heat Exchangers (N)	Hot-stream pinch temperature (°C)	Cold-stream pinch temperature (°C)
5	13,640	17,760	8	86.4	81.4
10	13,700	17,820	8	91.4	81.4
15	13,750	17,880	8	96.4	81.4
20	13,810	17,940	8	101.4	81.4
25	13,870	18,000	8	106.4	81.4
Base case	15,850	19,980	7	–	–

- heat exchanger network synthesis; and
- network configuration.

The first step, data extraction, involves translation of flowsheet information into relevant thermal and cost information required for the application of pinch analysis. This step usually calls for a process simulation. The second step, pinch analysis, is to explore various options for process improvement quickly and easily using targeting, without getting into the detail of specific flowsheet changes. This allows quick screening of various options for process improvement such as energy recovery, process modifications, utility system integration, and so forth. Note that given a minimum approach temperature (ΔT_{\min}) of a heat exchanger, the exact amount for minimum utility consumption can be predicted in this step. The key improvement options identified in the pinch analysis stage need to be implemented in design. In this step the focus is on the design aspect of pinch technology. This translates the ideas into the specific synthesis of the heat exchanger network. The third step, heat exchanger network synthesis, is based on pinch analysis principles and is called the pinch design method. There are basically three rules behind the pinch design method: (1) Heat must not be transferred across the pinch, (2) there must be no external cooling above the pinch, and (3) there must be no external heating below the pinch. The method systematically leads the engineer to good network designs that achieve the energy targets within practical limits. The network design procedure uses a special representation for heat exchanger networks called the grid diagram. Note should be made that the fewest number of exchangers is often equal to the number of process and utility streams minus one. The network design procedures are capable for doing both grassroots design and retrofit design. Answer to the step three leads to step four, the final network configuration. For illustrative purpose, only the composite

curve of $\Delta T_{\min} = 10$ °C is shown in Fig. 8. Table 6 shows various minimum approach temperatures versus the utilities requirements. Clearly, the smaller the ΔT_{\min} , the higher the heat recovery is and thus the less utility requirements. As seen from Table 6, the network saves utilities as compared with the preliminary base-case design. We have constructed the heat exchanger network for various ΔT_{\min} by SuperTarget manually by using the design heuristics. Note that for illustrative purpose, only the heat-flow match of $\Delta T_{\min} = 10$ °C is shown in Fig. 9. A final network configuration for $\Delta T_{\min} = 10$ °C is depicted in Fig. 10. A comparison of the network shows that compared with the base-case design (Fig. 6), the number of heat exchangers and the utility requirements has changed. As seen from Fig. 10, the network therefore represents a saving in utilities at the cost of more heat exchangers.

7. Results and discussion

We have demonstrated that the thermodynamic approach can be useful in analyzing, synthesizing and designing a new chemical process. However, there is limitation of thermodynamics. Although thermodynamics may predict the reaction yields at equilibrium, the rates of reaction have yet to be investigated. To this end, catalyst studies are important for catalyst is quite specific. It can accelerate just one reaction, not another; therefore, with the right catalyst we can get the desired reaction to go. Also from pinch technology, it is clear that an infinitely large transfer area will maximize the energy recovery in the streams but at the exorbitant cost. At this point, the profitability analysis that takes into account the time value of money can be incorporated into the projects. To further select the best alternative, we can explore the capital/energy trade-off and estimate equivalent annual operating cost (EAO) [4] of each heat exchanger network.

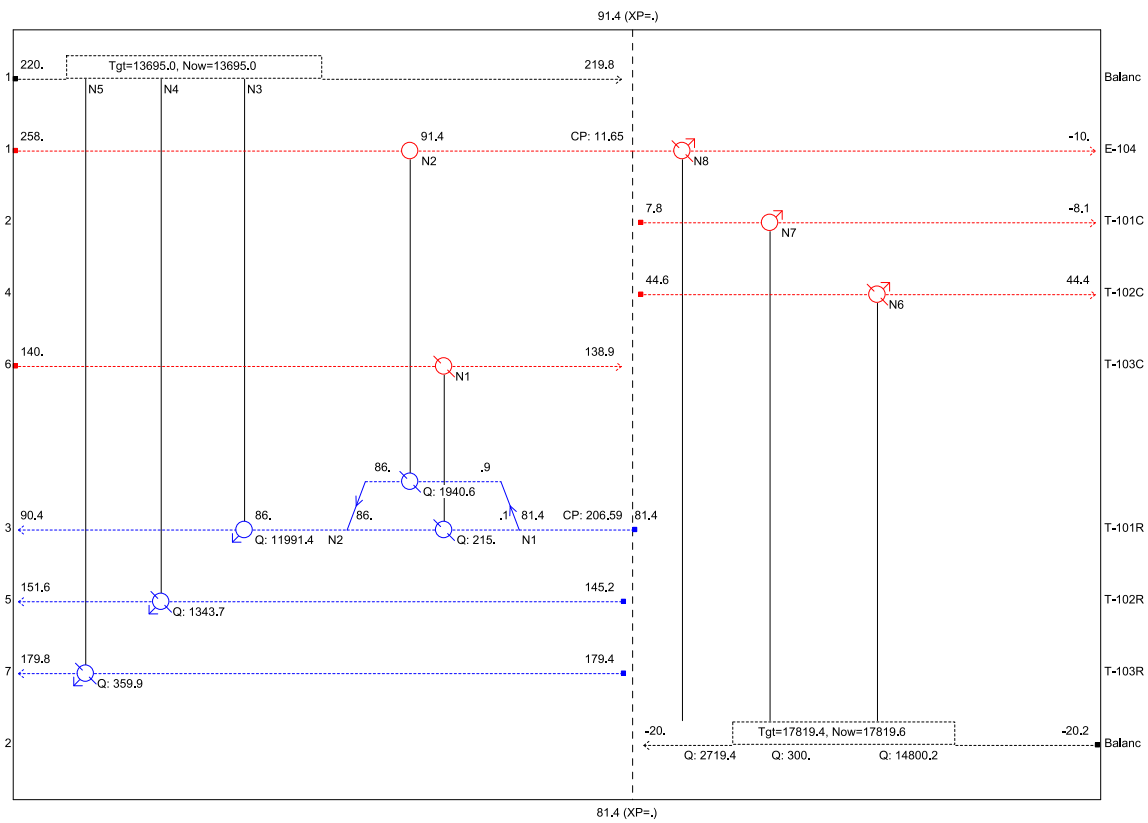


Fig. 9. Grid diagram of the construction of heat exchanger network for $\Delta T_{\min} = 10\text{ }^{\circ}\text{C}$.

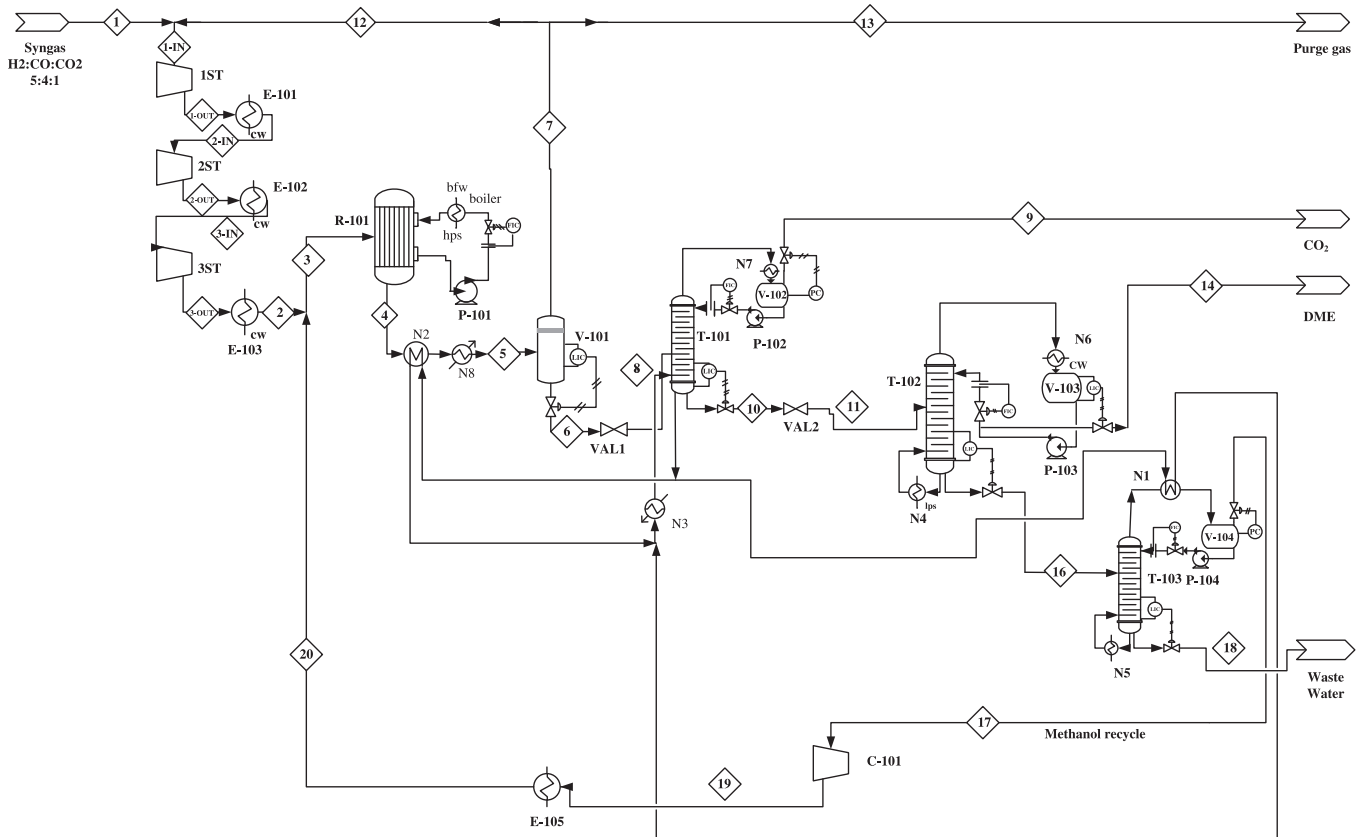


Fig. 10. Final process flow diagram for the production of DME with heat integration for $\Delta T_{\min} = 10\text{ }^{\circ}\text{C}$.

8. Conclusion

Thermodynamic approach provides an insight into a reactor performance. This information should aid in the detailed design of reactor kinetics. In sum, this paper describes an analysis, synthesis and design of a one-step DME production through a thermodynamic approach. Managing energy usage can dramatically reduce energy costs. Heat integration has been a very popular topic in the chemical process industries over the past decades, especially in regard to the issue of reducing the amount of energy usage. Therefore, we have also incorporated the pinch technology into the study in a consecutive four-step sequence. While it is true that every process is different, it is equally true that the methodology of visualizing, analyzing, and synthesizing are universal and transferable to other chemical processes.

Acknowledgments

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