Design of Components for Liquefaction of Nitrogen Using DWSIM

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The field of Cryogenics deals with low temperature refrigeration applications such as liquefaction of gases. The various cryogenic cycles such as the Linde cycle, Claude cycle, Kapitza cycle help in liquefaction of various gases such as nitrogen, helium etc. Of all the three cryogenic cycles, Linde cycle was chosen as it is the simplest cycle. Liquid nitrogen has wide applications in various industries like food processing and transportation, medicine, medical therapy, manufacturing of computers etc. Operating variables such as flow rates, temperature, pressure, energy in a thermal system operating in a steady state can be calculated by a process simulation. Since design of plants is a cost and time-consuming process in reality, chemical engineers use simulators to simulate design and operation of a chemical plant and its equipment which saves money and time. Today, many simulators are in use. DWSIM was our choice for the project as it was open software, easy to work on and gave accurate results.

Keywords: Cryogenics, Liquid Nitrogen, Linde cycle Simulation, Process design, Heat exchanger.

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1 Introduction

Conversion of any gas from vapor phase to liquid phase is called liquefaction. Liquefaction of gases is complex as it involves various processes and stages such as compressions, cooling, evaporation, condensation, and expansions. It is widely used in commercial, scientific, and industrial applications. Many gases can be put into a liquid state at normal atmospheric pressure by simple cooling.It is a conventional method of producing liquid from gases. It dates to the early 1820s when English scientist Michael Faraday liquified gases like chlorine, hydrogen sulphide, hydrogen bromide and carbon dioxide by the application of pressure for the first time. Yet, for many years later, the liquefaction of gases at critically low temperature remained unexplored. It wasdone only at the end of the 19th century. The first critically low temperature gases which were liquified were hydrogen (-399.5 F) and helium (-449.9 F). Liquefaction of gases in industries is accomplished by modifying the temperature, pressure and by using coolants [1].

Liquefied gases have numerous applications in various fields. They can be stored and transported easily as compared to others. Liquid nitrogen is used to preserve the freshness of packaged or bulk foods, cryogenic metal hardening, as fuel in space shuttles and in magnetic resonance characterization of materials etc. Their medical applications include preservation of vaccines, biomaterials, and cryosurgery. The specific applications of liquid nitrogen include freezing and storage of food products, cryopreservation of biological samples, cryotherapy and cryosurgery, inert atmosphere to shield from oxygen, cryogenic machining of materials etc [1]. Hence, the topic of liquefaction of nitrogen was chosen.

Liquid Nitrogen (N_2) is a gas under the category of critically low temperature gases. It becomes liquid at a very low temperature of -195.79 °C (77 K, -320 F). Liquid nitrogen retains the character of the N2 molecule even after liquefaction. The weak Van der Waals interaction between the N_2 molecules of liquid nitrogen results in its very low boiling point. It is colourless and has a density of 0.808 g/ml at its boiling point.Liquid nitrogen is widely used as coolant because of its low density and inert nature which prevent combustible reactions. It can cool in a comparatively lesser time than if it were to be done naturally. Liquid nitrogen may harm the human tissue, so utmost care should be taken in its production and storage.

Simulation is a handy tool to gain an in-depth understanding of the N_2 liquefaction process and the effects of various process parameters and unit operations. A simulation study can be utilized as a preliminary step to the carrying out of the physical process. Basically, simulation is an imitation of any process, that shows how operations are carried out in the process. It can be used to conduct scale-up or scale-down studies of processes. It is very helpful to predict the trajectory of any process under inoperable conditions.In chemical engineering, process design is the choice and sequencing of units for the desired physical and/or chemical transformation of materials. It is central to chemical engineering andis the summit of that field, bringing together all the field's components. It can be the design of new facilities, or it can be the modification or expansion of existing facilities. It is distinct from equipment design, which is closer in spirit to the design of unit operations.

There is many simulation software which can be used for simulating this process. DWSIM was chosen for this work as it is a free and an open-source software easily accessible for students of undergraduate courses. It can simulate steady state vapor liquid, liquid, solid liquid and aqueous electrolyte equilibrium processes. It gives better understanding of processes, as it has built-in thermodynamic models and unit operations as well as a large range of tools for managing reactions or creating components.The most common thermodynamic cycles used for liquefaction of nitrogen areLinde cycle (Linde Hampson cycle and precooled Linde Hampson cycle), Claude cycle and Kapitza cycle. Linde cycle being the simplest cycle of the three is used for our project of liquefying nitrogen[1].

2 Methods

2.1 Simulation of Liquefaction of nitrogen

Fig 2.1.1: Flowsheet of overall simulation of liquefaction of nitrogen

We have taken air (79% nitrogen ,21% oxygen) at a temperature of 298.15K, mass flow rate 5 kg/s and a pressure of 101325 pa (see Fig 2.1.2 and 2.1.3)[2].

The air mixture taken is sent into a mixer for homogeneous mixing which is then directed into the compressor where it compresses the gas adiabatically with 75% efficiency. There is no heat added or extracted from the air. The temperature of the air increases along with the increase in its pressure (seeFig 2.1.4).

Fig 2.1.4: Compressor conditions

The stream coming out of the compressor is still in the vapor phase but there is no change in composition of the feed (21% oxygen,79% nitrogen) (see Fig 2.1.5 and Fig 2.1.6).

Fig 2.1.5: Compressed air Properties. **Fig 2.1.6**: Compressed air Composition.

Next the stream enters the heat exchanger where water at 303 K, 101325 pa and 1.3kg/s mass flow rate is used as coolant. Water having low temperature exchanges heat with hot fluid flowing through the Heat exchanger. The heat exchanger conditions, coolant properties, and coolant composition (see Fig 2.1.7, Fig 2.1.8, and Fig 2.1.9 respectively).The coolant leaves the heat exchanger in liquid phase with temperature 353.275K. The heat exchanger inlet air properties and composition (see Fig 2.1.10 and Fig 2.1.11).

Calculation Parameters				Stream Conditions	Compound Amounts		
Calculation Type	Heat Transfer Efficiency		\checkmark	Flash Spec			
Flow Direction	Counter Current		\checkmark			Temperature and Pressure (TP)	
Cold Fluid Pressure Drop	$\mathbf{0}$	Pa	\checkmark	Temperature	303	к	
Hot Fluid Pressure Drop	$\mathbf{0}$	Pa	\checkmark	Pressure	101325	Pa	
Cold Fluid Outlet Temperature	353.275 K			Mass Flow	1.3	kg/s	
Hot Fluid Outlet Temperature	304.403 K			Molar Flow	72.161	mol/s	
Global Heat Transfer Coefficient	1000	W/[m2.K]	\checkmark				
Heat Exchange Area	50.296	m ₂		Volumetric Flow	0.00130661	m3/s	
Heat Exchanged	348.825 kW			Specific Enthalpy	-2605.15	kJ/kg	
Min Temperature Difference	$\overline{0}$	K.		Specific Entropy	-7.01192	kJ/kg.Kl	
Heat Loss		0 kW		Vapor Phase Mole Fraction			
Heat Transfer Efficiency	98 ₁	\mathcal{Z}	w				

Fig 2.1.7: Heat exchanger Conditions **Fig 2.1.8**: Coolant properties

Fig 2.1.9: Coolant composition

Fig 2.1.10: Heat exchanger inlet air properties **Fig 2.1.11**: Heat exchanger inlet air composition

The main outlet stream (considering the coolant outlet stream as another) from the heat exchanger enters the cooler with temperature 304.403K, the former mixture is in vapor phase[2].

Fig 2.1.12: Heat exchanger outlet/cooler inlet Fig 2.1.13: Heat exchanger outlet/ cooler inletair properties. air Composition.

In the cooler, the air temperature further decreases to 86.3005K and the composition remains the same (see Fig 2.1.14, Fig 2.1.15 and Fig 2.1.16).

Fig2.1.14: Cooler conditions **Fig2.1.15**: Cooler outlet properties.

Fig 2.1.16: Cooler outlet composition

The cool air stream then enters the throttling valve where itexpands, and its pressure gets further reduced by 86000 Pa. The stream leaving the throttling valve has a temperature of 80.6402 K and pressure of 100000 pa as shown below (see Fig 2.1.17, Fig 2.1.18 and Fig 2.1.19).

Fig 2.1.17: Throttling valve conditions

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Fig 2.1.18: Throttling valve outlet composition **Fig 2.1.19**: Throttling valve outlet properties

Then the stream is further directed to the vapor liquid separator, which separates the vapor phase of the stream from liquid phase. There are two streams coming out of the separator one being liquid (41.2% oxygen, 58.7%nitrogen) another being vapor (86.4% nitrogen, 13.5% oxygen). The liquid phase is the desired output (see Fig 2.1.20, Fig 2.1.21 and Fig 2.1.22).

vapour-liquid separator vapour-liquid separator

In case the desired liquid phase is not of satisfactory composition, then it is not withdrawn as a product, but is recycled back to the process with conditions same as that of the feed.

Mole Fractions Basis				Stream Conditions	Compound Amounts			
Solvent				Flash Spec		Pressure and Enthalpy (PH)		
Compound	Amount		Total: 1	Temperature		80.6402 K		\checkmark
Nitrogen		0.86442329	Normalize	Pressure		100000	Pa	\checkmark
Oxygen		0.13557671	Equalize	Mass Flow		3.61819	ka/s	\sim
Water			Clear	Molar Flow		126,715	mol/s	\sim
				Volumetric Flow		0.849553	m3/s	\sim
			Accept Changes	Specific Enthalpy		-224.62	kJ/ka	\checkmark
				Specific Entropy		-1.24453	kJ/kg.KJ	\checkmark
				Vapor Phase Mole Fraction				

Fig 2.1.23: Vapour stream composition vapour-liquid **Fig 2.1.24**: Liquid stream composition Separator vapour-liquid separator

The vapor stream at temperature 80.64 K must be liquified. It is sent to the cooler of 100% efficiency. where its temperature is further reduced and it gets liquified (see Fig 2.1.25).

Input Data Results		Annotations	Dynamics	Floating Tables		
Stream Conditions		Compound Amounts				
Flash Spec			Temperature and Pressure (TP)			\checkmark
Temperature				77	к	\checkmark
Pressure				100000	Pa	\checkmark
Mass Flow	3.61819				kg/s	\checkmark
	126.715 Molar Flow				mol/s	\checkmark
Volumetric Flow				0.00427289	m3/s	\checkmark
Specific Enthalpy				-431.044	kJ/ka	\checkmark
Specific Entropy				-3.86045	kJ/kg.Kl	
	Vapor Phase Mole Fraction			Ω		

Fig 2.1.25: Cooler outlet properties

The liquid stream containing 86.4% of nitrogen and 13.5% of oxygen must be separated to get exclusively liquid nitrogen. This is done using a component separator. The outlets of the component separator are nitrogen (liquid phase) at 77K and oxygen (vapor phase) (see Fig 2.1.26, Fig 1.1.27 and Fig 2.1.28).

\checkmark
\checkmark
\checkmark

Fig 2.1.26: Component separator conditions

Fig 2.1.27: Component Separator Outlet properties **Fig 2.1.28**: Component Separator Outlet

composition

2.2 Design of the Heat Exchanger from the process

2.2.1 Heat exchanger properties from simulation

Heat Exchangers like shell and tube counter current heat exchanger are devices which transfer heat from hot fluid stream to cold fluid stream. In heat exchanger hot fluid temperature decreases and there is increase in temperature of cold fluid. By losing heat hot fluid is prepared for the throttling process and similarly by gaining heat cold fluid heated up for the compression process.

HEAT EXCHANGER					
Object	HOT FLUID OUT	HOT FLUID IN	COLD FLUID OUT	COLD FLUID IN	
Temperature	304 403	373.015	353.275	303	К
Pressure	186000	186000	101325	101325	Pa
Mass Flow			1.3	1.3	kg/s
Volumetric Flow	2.35812	2.88963	0.00133822	0.00130551	m3/s
Mixture Density	2.12034	1.73032	971,437	994.942	kg/m3
Mixture Specific Enthalpy	5.93633	75.7013	-2336.83	-2605.15	kJ/kg
Mixture Molar Enthalpy	171.265	2184.01	-42098.6	-46932.6	kJ/kmol
Mixture Thermal Conductivity	0.0261055	0.030974	0.669873	0.617318	W/[m.K]
Molar Fraction (Mixture) / Nitrogen	0.79	0.79		0	
Molar Fraction (Mixture) / Oxygen	0.21	0.21	ñ	\circ	
Molar Fraction (Mixture) / Water	0	0			

Table 2.2.1: Heat Exchanger properties from the simulation

2.2.2 Energy balance

Energy balance calculation is done on a heat exchanger to determine operating parameters for hot and cold fluids such as inlet / outlet temperatures and flow rates [4,5,6].

$$
E_{in} = E_{out}
$$
\n(1)
\n
$$
E_{H_{in}} + C_{H_{in}} = E_{H_{out}} + C_{H_{out}}
$$
\n
$$
5 \times 75.7 + 1.3 \times 2605.15 = 5 \times 5.93 + 1.3 \times 2336.83
$$

$3765.195 \text{ kJ} \cong 3067.529 \text{ kJ}$

As the mass entering the heat exchanger is equal to the mass leaving it, it is considered that

$$
m_{H_{in}} = m_{H_{out}} = m_H
$$
 and
$$
m_{C_{in}} = m_{C_{out}} = m_C
$$

2.2.3 Manual Design [4,5,6]

Step 1: Temperature of hot fluid and cold fluid

Mass flow rate of $air = 5 kg/s$

 $T_{H1\,air} = 373\,k = 100\,°C$ $T_{H2\,air} = 304\,k = 31\,^{\circ}\text{C}$ $T_{C1 water} = 303 k = 30 °C$ $T_{c2 water} = 353 k = 80 °C$

Step 2: Average Temperature of hot and cold fluid

 $t_{\text{H air}} = 655.5 \text{ °C} = 338.5 \text{ k}$ (Molecular weight of air = 28.96)

 $t_{Cwater} = 55.5 °C = 328 k$ (Molecular weight of water = 18.05)

Step 3: Determination of the transport properties of hot and cold fluids at their average temperatures [5].

Step 4: Calculation of the mass flow rate of water from heat balance

$$
m_{H} \times C_{p} \times (T_{2} - T_{1}) = m_{C} \times C_{pC} \times (t_{2} - t_{1})
$$
 (2)
5 × 1.007 × (373 – 304) = $m_{C} \times 4.178 \times (353 - 303)$
 $m_{c} = 1.6 \text{ kg/s}$

Step 5: Calculation of inside and outside diameter of tubes. Since the tube sections are available at 14 BWG standard, the tube dimensions are:

 $14 BWG = 0.08" = 2.302 mm from Kerns Table(3)$

 $D_i = D_0 - 2 \times \text{thickness} = 19.05 - 2 \times 2.302 = 14.98 \text{ mm}$

 $D_{io} = 0.01906 \text{ m}; \quad D_{ii} = 0.01498 \text{ m}$

Assuming (15/16)" triangular pitch; 3/4th OD tubes with 14 BWG; Tube length of 2430 mm are laid in 15/16th triangular pitch; Velocity is 1 m/sec inside the tubesusing Table 10- 18 of Perry Handbook [1].

Step 6: Calculation of the LMTD for counter current flow

$$
LMTD = \frac{d(T1) - d(T2)}{\ln \frac{d(T1)}{d(T2)}} = \frac{(373 - 353) - (304 - 303)}{\Box \Box \frac{373 - 353}{(304 - 303)}} = 6.342 \text{ K}
$$
(4)

Calculation of the Corrected LMTD:

$$
R = \frac{373 - 304}{353 - 303} = 1.38
$$
and
$$
S = \frac{353 - 303}{373 - 303} = 0.714;
$$

$$
dT_{LMTD} = F_T \times LMTD \text{ where } F_T \text{ from graph is 0.95 (5)}
$$

Corrected LMTD = 0.95 × 6.342 = 6.02 K

Step 7: Calculation of the area required for heat transfer

 ℎ = = ()() (6) = *347*.*415*; = 334.24 A = 340.82 ∗ 10000 1000 ∗ 6.34 = 53.75 m²

Step 8: Mechanical design [5]

(Assumption: Using low carbon steel of 14 BWG thickness outer diameter=3/4th inch (19.05 mm); Tube length=2430 m)

Number of tubes = N_t =
$$
\frac{\text{Total surface area}}{\text{Individual heat transfer area}}
$$
 = $\frac{53.75}{0.1454}$ = 369.59 \approx 370 No. s (7)

Individual heat transfer surface area = $3.14 * 0.01905 * 2.43 = 0.1454 \text{ m}^2$ (8)

Each Pass =
$$
\frac{370}{2}
$$
 = 185 No.s

Shell diameter: The nearest tube sheet tube hole count for tema class L or M for a triangular pitch of 370 tubes the shell diameter is540mmreferred from Perry Handbook Table 11 [1].Permissible deviation=3mm.

Number of baffles: (Assumption: the shell contains 25% cut segmented baffles spaced 112mm apart)

$$
N_b = \frac{_{\text{Table length}}}{_{\text{Baffle spacing}}} - 1 = 21 \, \text{No.s} \tag{9}
$$

Pass partition plates=10mmselect pass partition plate thickness 10mm on channel head in tube side using IS Code Table 13.

Number and diameter of tie rods= 6 numbers of tie rods of 10mmusing IS Code Table 14.

Saddle support: The shell is mounted horizontally on 2 numbers of saddle support as shown in the drawing and provided with a banking saddle plate.

Tube sheet thickness: Minimum tube sheet thickness is 15mm; partition grove depth of 5 mm tube sheet thickness=20 mm (IS code Table 7)

Calculation of the shell thickness: Minimum shell thickness specified is 6.3 mm for safety and corrosion allowance select 10 mm. D_s = Shell OD = 540+(2*10) = 560mm using IS Code Table 14.

Step 9: Dimensions of nozzles

Shell side nozzle= 1 no inlet and 1 no outlet

$$
\frac{c}{s}
$$
 flow are of nozzle =
$$
\frac{\text{volume}}{\text{operating economic fluid velocity}}
$$
 (10)

For liquid OEFV = 1-2 m/s
\nFor gas OEFV = 1 m/s
\nCross flow area of Nozzle =
$$
\frac{5}{1.0435 \times 1}
$$
 = 4.79156 m2
\nInner diameter of the nozzle = DNT for air = $\sqrt{\frac{4 \times 4.79}{3.14}}$ = 2.469 m(11)
\nAlignment tolerance for standard nozzle using IS Code Table 16=1.5mm
\nDiameter including tolerance = 2471.5mm

Tube side:

Tube side nozzle= 1 no. inlet and 1 no. outlet

c $\frac{c}{s}$ flow area of the nozzle = $\frac{volume}{v}$ volumetric flow rate operating economic fluid velocity

For liquid OEFV = $1-2$ m/s

For gas OEFV =
$$
1 \text{ m/s}
$$

Cross flow area of Nozzle $\frac{1.6}{990.28*2}$ = 8.078*10⁻⁴ m²

Inner diameter of the nozzle = DNT for water = $\sqrt{\frac{4 * 8.078 * 0.0.0001}{3.14}}$ = 0.0320 mm

Alignment tolerance for standard nozzle using IS Code Table 16=1.5mm

Diameter including tolerance = 33.5 mm

Step 10: Calculation of the inside heat transfer coefficient (hio)

Dirt resistance: Assuming $R_d = 0.0005(m^2K)/W$; 14 BWG=0.08 inch = 2.032mm: Inner diameter= 14.98mm(di) using Perry Handbook table 11-3 [1].

Assuming velocity of 1 m/s

$$
N_{re} = \frac{d_i v_i}{k_i} \rho_i = 28782.3(12)
$$

$$
N_{pi} = \frac{c_{pi} \mu_i}{k_i} = 2.55
$$
 (13)

Sieder Tate Equation:

$$
\frac{h_i d_i}{k_i} = 0.023 \times N_{re}^{0.8} \times N_{pr}^{0.33} \times \varphi
$$
 (14)

$$
h_i = 6504.96 \frac{w}{m^2} \cdot k
$$

$$
h_{io} = h_i \times \left(\frac{d_i}{d_o}\right) = 5115.18 \frac{w}{m^2} \cdot k
$$

Step 11: Calculation of the outside heat transfer coefficient (h_o)

By Colburn Analogy of HTC

$$
\frac{h_0 d_{is}}{k_s} = 0.36 \times N_{re}^{0.55} \times N_{pr}^{0.33} \times \varphi^{0.14}
$$
(15)

$$
N_{re} = \frac{\text{DisGs}}{\mu s} Mass Velocity = G_s = \frac{m_s}{a_s}
$$

Shell side $\frac{c}{s}$ flow area = $a_s = \frac{ID * Baffle Spacing \cdot Clearance}{Pitch}$ (16)

$$
a_s = 0.0152 m^2
$$

$$
G_s = 330.68 \frac{kg}{m^2 \cdot s}
$$

For Triangular Pitch Dis = 0.0183 m

$$
N_{re} = 298100.68
$$

\n
$$
N_{pr} = .6.9
$$

\n
$$
\frac{h_o d_{is}}{k_s} = 0.36 \times N_{re}^{0.55} \times N_{pr}^{0.33} \times \varphi^{0.14}
$$

\n
$$
h_o = 11302.025 W/m^2 K
$$

\n
$$
h_o = 11302.025 W/m^2 K
$$

Calculation of the Overall heat transfer coefficient (U_D)

$$
\frac{1}{v_d \text{ calculated}} = \frac{1}{h_{io}} + \frac{1}{h_o} + R_d
$$
 (17)

$$
U_d \text{ calculated} = 1275.14 \frac{W}{m^2}.
$$

$$
U_{d\;Calculate\,d} > U_{d\;Assumed}
$$

- 1. Hence the heat exchanger design is valid.
- 2. Heat Exchanger is Overdesigned.
- 3. The assumption U_d = 1000 W/m²K

Neglecting R_d we get
$$
U_d
$$
 _{calculated} = 3521.421 $\frac{W}{m^2}$. K
\n $R_d = \frac{Ud \text{ calculated} - Ud \text{ assumed}}{Ud \text{ calculated} * Ud \text{ assumed}} = 7.160 \times 10^{-4} \text{ m}^2 \text{K/w}$

$$
R_d
$$
 provided $> R_d$ Required

Therefore, the calculated overall heat transfer coefficient is well within the design criteria.

2.2.4 Summary sheet of heat exchanger manual design

```
Length of the tube = 2430 mm
Triangular pitch = 254mm
```
Baffle space = 112 mm apart No of tubes $= 370$ No.s Total surface area = $53.75m²$ Shell diameter = 540 mm No of baffles = 21 No.s No of tie rods = 6 No.s Diameter of tie rod = 10mm Saddle support = 2 No.s Tube sheet thickness =20 mm OD of the shell = 560 mm Diameter of the nozzles Tube nozzle = 2 Nos; ID of Nozzle = 293.5 mm Shell Nozzle= 2 Nos; ID of Nozzle =33.5mm Shell Thickness = 10 mm Individual heat transfer coefficient: Tube side=5115.18 W/m2K Shell side=11302.025W/m2K Overall heat transfer coefficient Ud=1275.14 W/m2K Assumed U^d is 1000 W/m2K

3 Results and Conclusions

The process flow sheet for the liquefaction of nitrogen using the Linde cycle was developed using DWSIM simulation software. Manual design of the heat exchanger from the process was done. The calculated overall heat transfer coefficient of the heat exchanger was well within the design criteria.

This work gave us an insight into the industrial production of liquid nitrogen. Through the simulation, we have observed and learnt about the effect of various parameters like pressure, temperature, mass flow rate etc. on the yield of the final product. This simulation guided us to determine the optimum process conditions easily and to get the desired result efficiently.

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