

Production of Lithium and Sodium Lubricating Greases

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Abstract: *Improving grease lubricants quality plays a significant role in reducing a maintenance cost and saving the contacted surfaces. The purpose of Lithium and Sodium lubricating greases production process, which described in this paper, comes for designing economic process with quite high quality. Generally, Sodium and Lithium greases are largely required in market. The work start from laboratory then all data and results are transferred to the refinery plant. As the result, this process targets enhance the product quality to enlarge the interests.*

1. INTRODUCTION

In order to reduce the friction and wear between surfaces in high pressure and temperature engines and machines, low viscosity and dropping point lubricants are not preferable. Grease lubricants are higher viscosity and dropping point lubricants. Grease is one of the old petroleum lubricants over the world (Totten, et al. 2003). Grease can be described as a semi-fluid to solid, multi-color lubricant. The primary component of grease is the fluid lubricant that can be a petroleum oil, vegetable oil, and synthetic oil. Lubricant fluid identifies the lubrication quality of the grease. The other ingredients that uses in grease manufacturing are thickener and additives (Kholijah, et al. 2012).

Thickener is a salt of long chained fatty acid, such as Sodium Stearate, that produced through saponification reaction of metallic base like sodium hydroxide with long chained fatty acid like stearic acid. Thickener gives grease its properties such as water resistance, dropping point, etc. In addition, thickener in grease determines grease type, for example if the thickener is Lithium Stearate grease type will be Lithium grease (Manual Engineer, 1999). The third component of grease is additives. Additives improve performance of grease and protect the lubricated surface. Additives can be anti-oxidant, rust inhibitor and others, such as molybdenum disulphide or graphite (Rudnick, 2009).

Nowadays, a verity types of grease lubricant existing. In this paper, production of Lithium and Sodium Lubricating Greases will be described. Also, production procedure, equipment and operating conditions will be determined and calculated. The operating conditions and raw materials amounts which are used in Lithium and Sodium greases production, will be estimated experimentally, and then will be applied on real production plant.

2. SODIUM AND LITHIUM GREASES PROPERTIES

2.1 Sodium Grease

Sodium (Soda) grease is produced by blending of thickener (Soda soap) and additives into petroleum base oil stock. Sodium greases have a dropping point about 175 °C and it can be operated at 121°C. Therefore, Sodium greases are used in reasonably high temperature operations, such as electric motor bearings (Manual Engineer, 1999). Additionally, Sodium greases have an excellent protection against rust and it can be mixed with other greases to produce higher quality grease. However, sodium greases have a lower water resistance than other greases types (Totten, et al. 2003).

2.2 Lithium Grease

The first greases were used as multipurpose greases are Lithium greases. Lithium greases have a high water resistance and dropping point about 190 °C. It can be used at temperature up to 135 °C and extreme pressure and speed Machines (Totten, et al. 2003). Lithium greases have a smooth and buttery appearance with low fibrous content. Basically, Lithium greases can be manufactured by mixing lithium 12-hydroxystearate soap and additives into petroleum base oil. Rust inhibitor additive

is usually added in Lithium greases production, because of it misses for this feature (Manual Engineer, 1999). Complex Lithium greases are derivative greases from the original Lithium grease, with better characterizations. It is produced by adding complex soap which is called “complexing agent” (Manual Engineer, 1999).

3. LITHIUM AND SODIUM GREASES PRODUCTION PROCESS

In general, grease production process includes, batch and continuous process. Process type depends on the public demand for grease and raw materials availability. In this research, the production process will be batch process. Lithium and Sodium greases production process consists of:

3.1 Feed Stocks

a. Base Oil

Bright base oil (150 stock) is lubricant oil has been used as base oil for grease production in Iraqi refineries. It is paraffinic oil, produced from vacuum distillation residue after removal of asphalt. Table 1 shows some of base oil's properties:

Table1. Base oil physical properties (Lab. Data and (Green, 2007))

Density(Kg/m ³)	Viscosity(Pa.s)	V.I	Flash point(°C)	Pour point(°C)	Boiling point (°C)
910	0.03	90	300	0	650

b. Sodium Hydroxide (Naoh) And Lithium Hydroxide (Lioh)

Both of them are in solid form with good purity degree and suitable for industrial purpose.

c. Stearic Acid (C17H35COOH)

Stearic acid is a long chain fatty acid and it is used as solid.

d. Additives

20% Bismuth Ten-Cem® (rust inhibitor and corrosion inhibitor).

1- Dioctyldiphenylamine (Antioxidant).

According to the experimental production process in the laboratory, the composition of feed stock was as in below table:

Assumed batch: 5 ton

Table2. Feed composition and amounts

Materials	wt. %	Amount (Kg)
Base Oil	88%	4,400
NaOH/LiOH	5%	250
Stearic Acid	5%	250
20% Bismuth Ten-Cem®	1.4%	70
Dioctyldiphenylamine	0.6%	30

2- Saponification reaction and reactor

Grease forms after thickening of the base oil by thickener. The thickener in grease production is formed in situ by the saponification reaction. The saponification reaction involves, metallic base (NaOH) with fatty acid (Stearic acid) to produce the soap (thickener). The reaction is endothermic reaction and needs for heating. The reactor is mixed flow reactor with jacket for heating and capacity about 10 ton.

Reactions:



Table3. Reactor operating conditions

	Temperature (°C)	Residence time (hr.)	Pressure (atm.)	Agitator speed (rpm)
Saponification Reaction	160-200	2	5.8	85
Grease Manufacturing	200	1	5.8	85

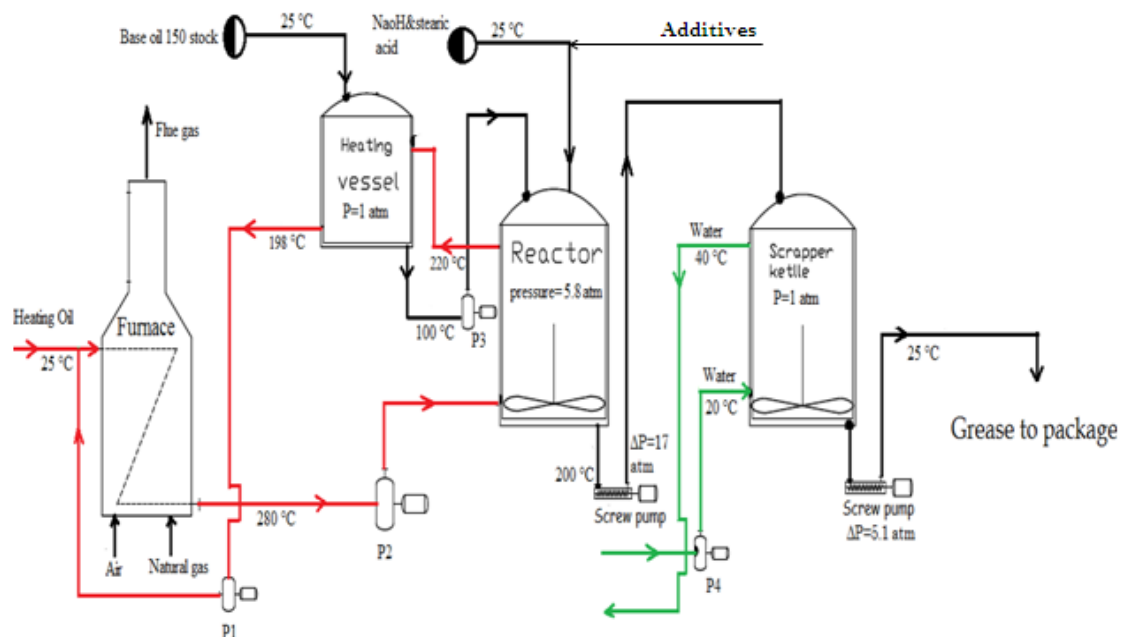


Fig1. Grease production process flow diagram (by Chem CAD)

3- Grease Plant Description: Grease plant consists of (see figure 1):

a- Base Oil Heating Vessel: Jacketed vessel is used to heating base oil feed stock from ambient temperature to 100 °C, before feeding to main reactor.

b- Mixed Flow Reactor (Contactor): High pressure, jacketed mixed flow reactor. Saponification reaction takes place in this reactor, then the base oil and additives are added to reaction products in the reactor.

c- Scraper Kettle (Finishing Vessel): Jacketed mixed vessel is used for cooling produced grease by cold water. Also, it offers a further mixing to homogenize the grease.

d- Furnace: Tubed burner uses a natural gas as fuel to heat heavy petroleum heating oil, which has a boiling point about (350 °C).

e- Pumps:

1. **Screw Pumps:** two pumps with high pressure drop, used to transfer the grease from the reactor to scraper kettle, then to packing stage.

2. **Centrifugal Pumps:**

- Two pumps are used to transfer the hot heating oil from the furnace to the reactor jacket and base oil vessel jacket.
- Third pump for pumping the hot base oil from heating vessel to the reactor.
- The last one for pumping cooling water of scraper kettle.

4. MATERIAL BALANCE

The products from this process are grease and small amount of water (15.9 Kg), which means all the reactants are totally converted to products. The water in grease is necessary to enhance grease ability of mineral oils absorption.

In of raw material = Out of products

So, produced grease = 5000 kg/ Batch

5. ENERGY BALANCE

Saponification Reactions:



E.B. on Reactor:

Table4. Physical properties (Green, 2007)

Material name	Specific gravity	Heat capacity (KJ/Kg.°F)	Weight(Kg)
Base Oil	0.91	1.207	8800
Grease	0.7	1.7	10000

Table5. Streams temperatures

Stream	Temp. inlet °C (°F)	Temp. outlet °C (°F)
Material stream	100 (212)	200 (392)
Heating Oil	280 (536)	220 (428)

Table6. Heats of formation (David and James, 2012)

Component	Heat of formation ,KJ/g mol
C ₁₇ H ₃₅ COOH	-964.3
NaoH	-426.6
C ₁₇ H ₃₅ COONa	-803.116
H ₂ O	-285.83

T_{ref}=25 °C=77 °F

$$\begin{aligned} \text{Heat}_{in} &= m \cdot C_p \cdot (T_{in} - T_{ref}) \\ &= 8800 \cdot 1.207 \cdot (212 - 77) \\ &= 1433916 \text{ KJ} \end{aligned}$$

$$\begin{aligned} \text{Heat}_{grease} &= 10000 \cdot 1.7 \cdot (392 - 77) \\ &= 5355000 \text{ KJ} \end{aligned}$$

$$\begin{aligned} \text{Heat of reaction} &= -803.116 - 285.83 - (-426.6 - 964.3) \\ &= 302 \text{ KJ} \end{aligned}$$

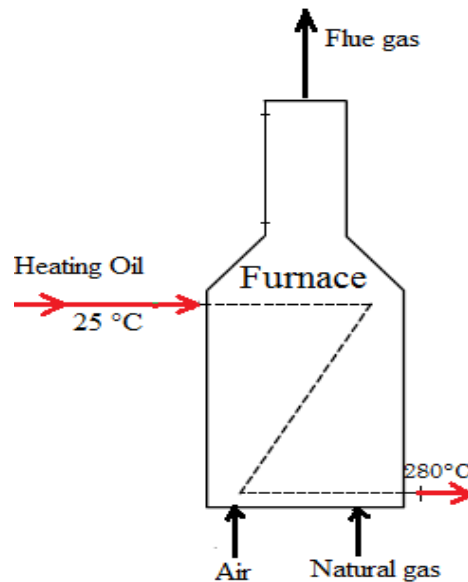
$$\begin{aligned} Q &= \text{Heat}_{grease} - \text{Heat}_{in} + \text{Heat of reaction} \\ &= 5355000 - 1433916 + 302 \\ &= 3921386 \text{ KJ} \end{aligned}$$

Heating Oil Quantity Need for Heating :

Time of process=3 hr.=10800 sec.

$$\begin{aligned} Q &= m \cdot C_p \cdot (T_{in} - T_{out}) \\ 3921386 &= m \cdot 1.207 \cdot (536 - 428) \\ m &= 30082 \text{ Kg} \end{aligned}$$

$$\begin{aligned} \text{Mass flow rate} &= 30082 / 10800 \\ &= 2.78 \text{ Kg/sec.} \end{aligned}$$



E.B. on Heating Vessel:

Table7. Streams temperatures

Streams	Temp. inlet °C (°F)	Temp. outlet °C (°F)
Material stream	25 (77)	100 (212)
Heating oil	220 (428)	198 (388.4)

T_{in}=T_{ref}=25°C → Heat_{in}=0

$$\begin{aligned} \text{So, } Q &= \text{Heat}_{out} = m \cdot C_p \cdot (T_{out} - T_{ref}) \\ &= 8800 \cdot 1.207 \cdot (212 - 77) \\ &= 1433916 \text{ KJ} \end{aligned}$$

Temp. Outlet of Heating Oil:

$$Q = m \cdot C_p \cdot (T_{in} - T_{out})$$

$$1433916 = 30082 \cdot 1.207 \cdot (428 - T_{out})$$

$$T_{out} = 388.4 \text{ } ^\circ\text{F} = 198 \text{ } ^\circ\text{C}$$

E.B. on Scraper Kettle:

Assume ΔT of Water = $20 \text{ } ^\circ\text{C}$

Table 8. Streams temperatures

Streams	Temp. inlet $^\circ\text{C}$ ($^\circ\text{F}$)	Temp. outlet $^\circ\text{C}$ ($^\circ\text{F}$)
Grease	200 (392)	25 (77)
Water	20 (68)	40 (104)

$$Q = \text{Heat}_{in} = m \cdot C_p \cdot (T_{in} - T_{ref})$$

$$= 10000 \cdot 1.7 \cdot (392 - 77) = 5355000 \text{ KJ}$$

The Cooling Water Mass Flow Rate:

$$m = Q / C_p \cdot (T_{out} - T_{in})$$

$$= 5355000 / 4.218 \cdot (40 - 20)$$

$$= 63478 \text{ Kg}$$

Heat of Furnace:

$$Q_f = m \cdot C_p \cdot (T_{out} - T_{in})$$

$$= 2.78 \cdot 1.207 \cdot (536 - 77)$$

$$= 16633686 \text{ KJ}$$

6. EQUIPMENT DESIGN AND CALCULATIONS:

a. Reactor Design According To (Sinnott, 2005):

Volume and Diameter of Reactor:

Grease Data:

$$\text{Density} = 700 \text{ Kg/m}^3$$

$$\text{Weight} = 10000 \text{ Kg}$$

$$\text{Volume} = \text{weight} / \text{density} = 10000 / 700 = 14.29 \text{ m}^3$$

$$\text{Total volume} = 14.29 + 0.1 \cdot 14.29 = 15.719 \text{ m}^3$$

$$V = (\pi/4) \cdot D^2 \cdot H$$

$$15.719 = (\pi/4) \cdot D^2 \cdot 5$$

$$D = \text{diameter of reactor} = 2 \text{ m}$$

Saponification Reactions Take Place in Reactor:



Stearic acid Sodium hydroxide Sodium Stearate water

The Reaction Rate of This Reaction:

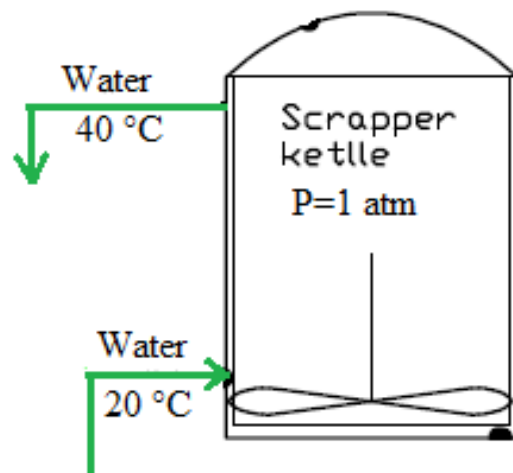
$$-r_A = K C_A^2$$

K = the reaction rate constant = 8.8 liters per mole. Minute

The conversion (X_A) = 90%

The reaction is second order

$$\text{Moles of NaOH} = W/M.Wt = 500/40 = 12.5 \text{ mole}$$



Moles of stearic acid = W/M . $W_t = 500/284 = 1.7$ mole

Stearic acid is the limiting material

Density of stearic acid = 1130 Kg/m³

V (volume of stearic acid) = weight/density = $500/1130 = 0.44$ m³

$V = 0.44 * 1000 = 440$ liters

$CA_0 = \text{moles of acid}/V = 1.7/440 = 3.9 * 10^{-3}$ mole/lit.

$-dCA/dt = KCA^2$

$$\frac{1}{CA} - \frac{1}{CA_0} = K * t$$

$CA = CA_0 * (1 - XA)$

$$(1/3.9 * 10^{-3} * (1 - 0.9)) - (1/3.9 * 10^{-3}) = 8.8 * t$$

$t = 262.2$ min.

Calculation the Thickness of Reactor Wall:

$$e = \frac{P_i * D_i}{2 * f - P_i}$$

e: thickness in (mm)

P_i: the pressure of the reactor (N/mm)

D_i: the diameter of reactor (m)

f : design stress for carbon steel

P_i = 5.9 bar

$$= 5.9 * 10^{-1} \text{ N/mm}$$

D_i = 2 * 10³ mm

f⁽¹⁾ = 105 N/mm

$$e = \frac{0.59 * 2 * 10^3}{2 * 105 - 0.59} = 5.63 \text{ mm}$$

$$e = 5.6 + 2(\text{corrosion}) = 7.63 \text{ mm}$$

Design the head of reactor:

Try an ellipsoidal head

$$e = \frac{P_i * D_i}{2 * f - 0.2 * P_i}$$

$$e = \frac{0.59 * 2 * 10^3}{2 * 105 - 0.2 * 0.59} = 5.62 \text{ mm}$$

$$e = 5.622 + 2(\text{corrosion allowance}) = 7.62 \text{ mm}$$

b. Furnace Design:

Symbols:

R: Fraction of heat absorbed by radiation surface.

α_{Acp} : Effective surface.

P_L : Partial pressure of (CO₂+H₂O) multiple length of radiation Beam.

P_f : Flame Emissivity.

Ø : Exchange factor.

T_g: Flue gas temperature.

$(\frac{RQ}{\alpha_{Acp} \phi})$: Rate of Heat Absorption Factor

Natural gas is fuel that burned to heat the heating oil in the furnace, M.wt. = 17.9

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Table9. Natural Gas composition (Dora refinery data)

Component of NG	Percent,%
CH ₄	90.7
C ₂ H ₆	6.2
C ₃ H ₈	2.1
C ₄ H ₁₀	1.0

Net heat=49524 KJ/Kg , Thermal efficiency=0.65, Heat required=16633686 KJ

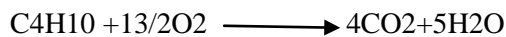
$$\text{Heat of Combustion} = \frac{16633686}{0.65} = 25590286 \text{ KJ}$$

$$\text{Amount of fuel used} = \frac{25590286}{49524} = 516.7 \text{ Kg}$$

Table10. Natural gas composition (Nelson, 1958)

Component	Percent,%	Weight, Kg	M.wt	Moles
CH ₄	90.7	468.6	16	29.28
C ₂ H ₆	6.2	32.03	30	1.06
C ₃ H ₈	2.1	10.8	44	0.245
C ₄ H ₁₀	1.0	5.16	58	0.09

Reactions of Fuel:



Flame Temperature Calculation:

$$Q = \Delta H_p - \Delta H_r + \Delta H_{rxn}$$

$$Q = 0, \text{ adiabatic system}$$

$$\Delta H_r = 0, \text{ fuel entering at } 298\text{K}$$

$$\text{So } 0 = \Delta H_p + \Delta H_{rxn}$$

Table11. Heats of formation table (David and James, 2012)

Component	$\Delta H_f(\text{KJ/gmole})$
H ₂ O	-241.82
CO ₂	-393.5
CH ₄	-74.84
C ₂ H ₆	-84.67
C ₃ H ₈	-103.83
C ₄ H ₁₀	-124.7

Table 12. Constants table (David and James, 2012)

Component	a	b	c	D
CO ₂	36.11	4.233e-2	-2.887e-5	7.464e-9
H ₂ O	33.46	0.688e-2	0.76e-5	-3.593e-9
N ₂	29	0.2199e-2	0.5723e-5	-2.871e-9
O ₂	29.1	1.158e-2	-0.607e-5	1.311e-9

$$\text{So } \Delta H_{rxn} = -6932.46 \text{ KJ}, \Delta H_p = m \int_{298}^{Tf} C_p' dT$$

Where, C_p' is average heat capacity of gases produce from combustion.

Finally the non-linear equation...

$$10.27T + 1.162 * 10^{-3} T^2 + 3.08 * 10^{-7} T^3 - 1.176 * 10^{-10} T^4 - 10104.8 = 0$$

By Solver:

So, the flame temperature is 309.37°C at 60% excess air

Furnace Design:

Part 1: Radiation Section

$Q_t = 25590286 \text{ KJ}$, total heat of furnace

Let $R = 0.7$

$Q_{rad.} = R * Q_t = 17913200.31 \text{ KJ}$

$Q_{rad. Rate} = \frac{Q_{rad.}}{3} = 5971066.77 \text{ KJ/hr}$, where time of process is 3 hour.

$Q_{proj.} = 533715.52 \text{ KJ/m}^2 \cdot \text{hr}$, (Nelson, 1958)

$A_{proj.} = \frac{Q_{rad. Rate}}{Q_{proj.}} = 11.19 \text{ m}^2 = 120.36 \text{ ft}^2$

Heating oil rate = 2.78 Kg/sec

Velocity = 1.50 m/sec , assumed

Average oil density = 830 kg/m³

Area = 0.0022 m²

$D_i = 0.0533 \text{ m}$

From pipe standards, (Ludwig, 1965).

$D_i = 0.054 \text{ m}$

$D_o = 0.06 \text{ m}$

Length tube = 7 ft. = 2.13 m , assumed

$No. of tubes = \frac{A_{proj.}}{D_o * L} = 87$

Center to center distance = 5 in. = 0.13 m, assumed

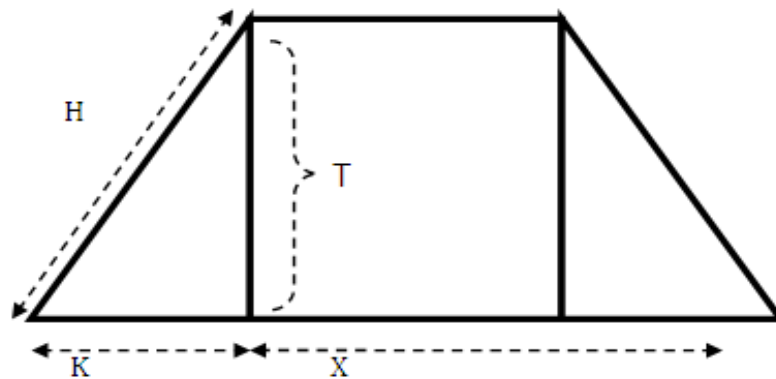
$A_{cp} = C - C * No. of tubes * L = 23.55 \text{ m}^2 = 253.39 \text{ ft}^2$

$\alpha = 0.87$, (David and James, 2012)

(CO₂+H₂O) partial pr. = 0.187 atm, (Nelson, 1958)

$P_L = 0.94 \text{ atm.ft.}$

Areas of Radiation Section



$H = 4.46 \text{ m}$

$T = 4.305 \text{ m}$

$K = 1.15 \text{ m}$

$X = 1.81 \text{ m}$

Rectangle volume=16.62 m²

Triangle volume=4.97 m²

Mean beam length = $\sqrt{\text{Rectangle volume} + \text{Triangle volume}} = 1.86 \text{ m}$

A_{total}= 74.46 m²

Pf= 0.41, (Nelson, 1958)

Ø= 0.68, (Kern, 1950)

T_g= 871°C

} (Nelson, 1958)

Pseudo-flame Temp.(Tf)= 1482 °C

$$\frac{RQ}{\alpha_{Acp} \varnothing} = \frac{A_{proj} \cdot Q_{proj}}{\alpha_{Acp} \varnothing} = 37736.65$$

$$\frac{0.98Q}{\alpha_{Acp} \varnothing} = 91000,$$

Q_{new}= 14685614.27 KJ/hr

R=0.41

Comment: The design procedure will repeated dependence upon new R value until the values of R converge

Part 2: Convection Section

Table 13.Base oil physical properties (Lab. Data and (Green, 2007))

Component	Viscosity(pa.s)	K(w/m.k)	M.wt.	Cp (J/Kg.K)	Density(Kg/m ³)
Base oil	0.0275	0.122	109	1304.2	830

• **Calculate Heat Transfer Coefficient of Base Oil Flow in Convection Section Pipes (Hi):**

We will use a traditional expression for calculation of heat transfer in fully developed turbulent flow is that recommended by Dittus and Boelter:

$$Nu = 0.023Re^{0.8} Pr^{0.4}, \text{ (Holman, 2002)}$$

$$Nu = \frac{hidi}{Kf}$$

$$Re = \frac{\rho u di}{\mu} = 2377, \text{ Velocity and inside diameter as in radiation section}$$

$$Pr = \frac{\mu cp}{Kf} = 294$$

So, hi=260.6 w/m².k

• **Calculate Pure Convection Film-Transfer Rate for Flue Gas Flowing at Tubes (Hc):**

The empirical equation by Monrad is the only comprehensive formulation of convection transfer rates

$$hc = \frac{1.6G^{0.667} T^{0.3}}{D^{0.33}} \quad \text{(Nelson, 1958)}$$

Table 14.Flue gas physical properties

Component	Velocity (m/s)	Density(Kg/m ³)	Temperature(°C)	G (Kg/m ² .sec)
Flue gases	6	0.3	871	1.79066

hc =64.1361 w/m². °C

$$Q = UA(Tg - To)$$

$$\frac{1}{U} = \left(\frac{1}{hc} + \frac{1}{hi} \right) \frac{di}{do}$$

U = 59.1 W/m². °C

$$Q=2559028.6 \text{ KJ/hr}$$

$$\text{So } A = 60.2 \text{ m}^2$$

$$\text{Length of tubes (L)} = 7\text{ft} = 2.13 \text{ m}$$

$$\text{No. of tubes} = \frac{A}{Ld\pi} = 149$$

7. CONCLUSION

One grease production unit is quite paramount to meet refinery and public demand. Many applications in the industry depend on lubricating greases to reduce the wear and friction between surfaces. Lubricating greases have an ability to endure a high pressure, temperature and loadings.

In overall, this paper has focused on raw materials quality and availability, the economy of equipment design, and reducing the manufacturing time. Lithium and Sodium lubricating grease has been adopted in this research in order to higher need of these categories. The work on this research lasted for 6 month, 3 month spent in Chemical Engineering Laboratory. Then, 2 month of the work period spent in refinery in Iraq, that to apply all laboratory work.

Finally, this paper is not fully determines all aspect of Lithium and Sodium greases production process. Additionally, the appropriate design for the heating vessel and scrapper kettle can save more money and time. High quality base oil and suitable additives should be selected for grease performance-enhancing and surface-protecting.

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Amthal Zaidan Algailani, Chemical engineer graduated from Chemical engineering department/ University of Baghdad at 2012 with a rank of 1st out of 54 students. (2012 – 2014) working as production engineer in a pharmaceutical industries Co. in Iraq. At 2015, I started my master study at Chemical and Process engineering school/University of Leeds/UK.