A study on design of Batch reactor and Horizontal condenser in the production of Citronellol

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Abstract: Citronellol, or dihydrogeraniol is a natural acyclic monoterpenoid, which is found in citronella oils, oils of rose (18-55%). Citronellol is used in perfumes and insect repellents, and as a mite attractant. In this study a batch reactor and horizontal condenser were designed for achievement of maximum yield. Material and energy balance calculations were performed. Design calculations were performed on batch reactor and horizontal condenser and the results were noted. The volume of the reactor required was around 2.58 m^3 .The height of the reactor was 3.3m and the diameter of the reactor was 2.2 m. The heat transfer area required in the horizontal condenser was found to be 10.56 m^2.number of tubes required to pass the fluid was found to be 28.

Keywords: batch reactor; horizontal condenser; material balance; Citronellol; energy balance.

1.INTRODUCTION

Citral a natural compound is found in plant essential oils [1]. Application of citral is abundant in cosmetics and also used in insecticides [2, 3]. On hydrogenation reaction citral gets converted to geraniol and citronellol [4]. Various catalysts like Pd, Ni, Pt, Rh were used in the hydrogenation of citral [5, 6]. Citral hydrogenation can be performed in batch and continuous reactors. Investigation on citral hydrogenation was carried out in continuous mode in trickle bed reactor [7]. In our research work batch reactor was used to investigate the hydrogenation of citral. Material balance and energy balance were performed for the unit operations involved in the process. A study on design of batch reactor and horizontal condenser was extensively done to understand the design configuration and to improve the rate of production by providing optimal conditions for the reaction in the reactor.

2. EXPERIMENTAL

2.1Hydrogenation of Citral

The Catalytic Citral hydrogenation is carried out in the semi batch reactor with agitation has the capacity of 1000 liters. The reactor has oil sealed arrangement and rupture disc to prevent system from over pressurization. The oil seal pressure of reactor is maintained at AP of 2kg/cm greater than operating pressure. The feed consisting of 1178.4 kilograms of Citral with 20 Kilograms of Raney Nickel catalyst in form of slurry with 4.08 kg of de mineralized water is added to reactor through pump initially. Three hundred and thirteen (1.2) kilograms of KOH flakes (caustic flakes), in order to maintain pH, is diluted in de mineralized water and then it is flushed in charging line. After which Citral is flushed to ensure maximum usage of KOH. After the feed is charged into reactor, it is agitated at 500rpm with Hydrogen being fed at 8kg/cm. The temperature is maintained at 100°C for fresh catalyst since the reaction fairly exothermic, the steam is cut off after the required temperature is reached and then cooling is passed in the coils by which reaction temperature is controlled. After 3hours of Hydrogen consumption, the sample is sent for GC (gas chromatography). The product is discharged when Citronellol concentration reaches required value. Then the reactor is cooled to 40°C to 45°C. The Citral by catalytic hydrogenation gets converted to Citronellol. The conversion of Citral to Citronellol is around 85%. The reactor is depressurized and then the product is drained in the vessel. The drained solution is then filtered for catalyst recovery and then distillate to remove impurities.

Material balance and energy balance
Material Balance for Reactor:

INLET:

Citral –1833.73 kg/hr.

Hydrogen – 24.09 kg/hr.

Raney nickel catalyst – 20 kg

KOH flakes – 1.2 kg

Demineralized water – 4.8 kg

Total feed = Citral+ Hydrogen

= 1833.73+24.09

= 1857.82 kg/hr.

For further reaction will occurs the product formed,

Citronellol +hydrogen =1624.07 kg/hr.

Total amount of Geraniol present = total feed - (Citronellol + hydrogen)

= 1857.82- 1624.07

= 233.74 kg/hr.

Total Amount of Citronellol in outlet = 1599.98 kg/hr.

Total Amount of Hydrogen in outlet =1.52 kg/hr.

Table 1.1

Compound	**Input (kg/hr.)**	**Output (kg/hr.)**
Citral	1833.73	-
Hydrogen	24.09	1.52
Geranoil	-	233.74
Citronellol	-	1599.98

Energy balance for reactor

Inlet temperature = 47 °C

Outlet temperature = 100 °C

Reference temperature =25 °C

ΔT 320K-298K =22 K

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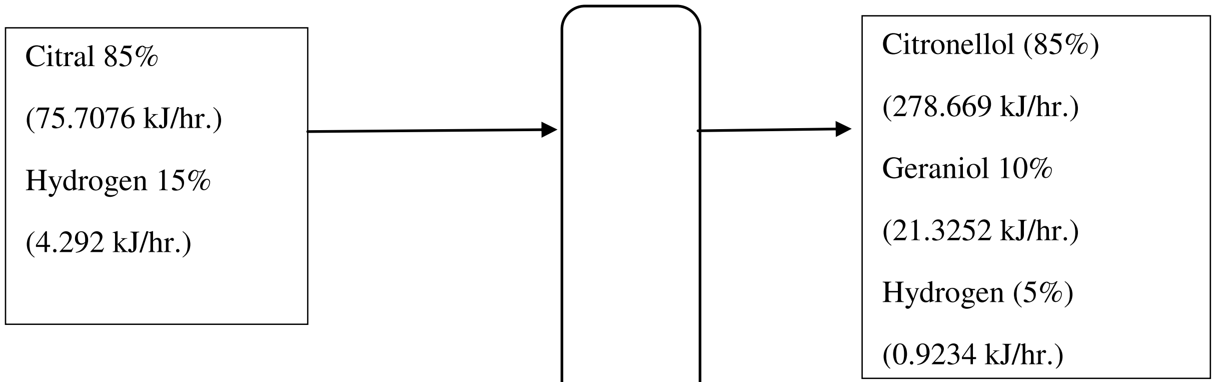
m = 12.045 kmol/ hr.

INPUT:

REACTOR



Input = Q= m Cp ΔT

Q= 12.045*0.2857*(22)

Q= 75.7076 kJ/hr.

Table 1.2

Compound	m kmol/hr.	C_p kJ/ kmol K	ΔT K	Q kJ/hr.
Citral	12.045	0.2857	22	75.7076
Hydrogen	12.045	0.0162	22	4.2928

Q= 80.004 kJ/h

OUTPUT:

ΔT =373K-298K =75 K

Output Q= m C_p ΔT

Q= 10.2386*0.3629*75

Q= 278.669 kJ/h

Table 1.3

Compound	m kmol/hr.	C_p kJ/ kmol K	ΔT K	Q kJ/hr.
Citronellol	10.2386	0.3629	75	278.669
Hydrogen	0.76	0.0162	75	0.923
Geraniol	1.04	0.2734	75	0.923

Q= 300.917 kJ/hr.

Heat of formation (Citronellol) = -187.1

Heat of formation (Citral) = -120.45

ΔH reaction= ΔH product - ΔH reactant

ΔH product=(10.2386 * - 187.1) +(10.23 *0)

= -1915.52 kJ/hr.

ΔH reactant=(12.045-120.45) + (12.045*0)

= -1450.82 kJ/hr.

ΔH_R^0 = -1915.52 + 1450.82

= -464.7 kJ/hr.

Value is negative. So Reaction is exothermic reaction

Qin + $\Delta H_R^0 = Q_{out} + Q_{released}$

80.004-464.7 -300.91 = $Q_{released}$

$Q_{released}$=-685.61 kJ/hr.

Energy released during the reaction Q= -685.61 kJ/hr.

3.RESULTS AND DISCUSSION

Design of Batch Reactor

Assumptions are made in designing the reactor is as follows

- The reaction that takes place is exothermic
- Steam is used as the heating medium initially and then cut off when required temperature is reached the cooling water is used to control the reaction temperature
- The density and the specific heat of mixture remain constant
- Isobaric process is carried out

Design of reactor Calculations

Weight of Citral =1833.73kg

Weight of water = 24.09 k

Weight of Citronellol = 1600 kg

Raney Nickel catalyst = 20 kg

Let the weight of mixtures W= Weight of Citral + Weight of Catalyst

= 1833.73+20

= 1877.82 kg

Density of Water = 997 kg/m^3

Density of Citral = 893 kg/m^3

Density of Raney Nickel catalyst = 1200 kg/m'

Viscosity of fluid = 1.11*10^{-3} Pas

Weight Fraction

Weight fraction of Citral = Wt. of Citral/Wt. of mixtures

= 1833.73/1877.82

= 0.976

Weight fraction of catalyst = Wt. of catalyst/ Wt. of mixtures

= 20/1877.82

= 0.010

Total Density

Density of Citral * Wt. fraction of Citral = 893 *0.976

= 871.56

Density of Catalyst * Wt. fraction of Catalyst = 1200 * 0.010

= 12

Total density = 871.56 +12

= 883.56

Viscosity of solution = 1.11*10^{-3}Pa. s

Volume of Reactor

Volume of Citral = mass/density

= 1833.73/893 = 2.05 m^3

Volume of Catalyst = mass/density

= 20/1200

= 0.0166 m

Volume of Reactor = 2.0696 m

= 2069.6 lit

Vapour Space

Vapour space required = 25% of total volume

= 2069.6* 0.25

= 517321.3

= 517.32 lit

Total volume required for reactor = 2069.6 +517.32

= 2586.32 lit

Assuming reactor to be cylindrical,

Volume of reactor = (T* D * Height)/ 4

Where, D = Diameter

And also assume H/D ratio is 1.5

Diameter of Reactor

Volume of reactor = (7* D^2* Height)/ 4

D^2= (vol * 4)/ (7*1.5D)

D^3 = vol *4/3.14 * 1.5

D^3=2586.32*4/3.14*1.5

D^3=2196.45

Dia= 2.2 m

Height of Reactor

Height = 1.5*D

= 1.5*2.2

H= 3.3 m

Diameter of reactor = 2.2 m

Height of Reactor = 3.3 m

Impeller Diameter

Impeller diameter = Reactor dia/3

= 2.2/3

D = 0.73m

Impeller Reynolds no= (speed of impeller*(dia of agitator)*density) / viscosity of solution

Speed of impeller = 472 Rpm

Dia of Agitator = 0.688 m

Density = 859.72

Viscosity of solution = 1.71*10 pa s

Impeller Reynolds Number

Impeller Reynolds no = (472/60)*(0.68) *859.935/1.71*10

= 7.86*0.44*859.935/1.71*103

= 3127.271/1.71*10

= 1828813.81

Power number Np = 6

Power Consumption

Power consumption = Np * density * $(impeller\ speed\ /60)^3$* (Dia^5)

= 6*859.72* $(\frac{472}{60})^3 * 2.2^5$

= 6 * 859.72 * (486.82) * (51.53)

= 4353306.7

= 4353.30 kW

Batch time = 15 hr.

= 4353.3/15

= 290.22 kW/h.

Design Summary

1) Diameter of reactor = 2.2 m

2) Height of reactor = 3.3m

3) Impeller diameter = 0.73m

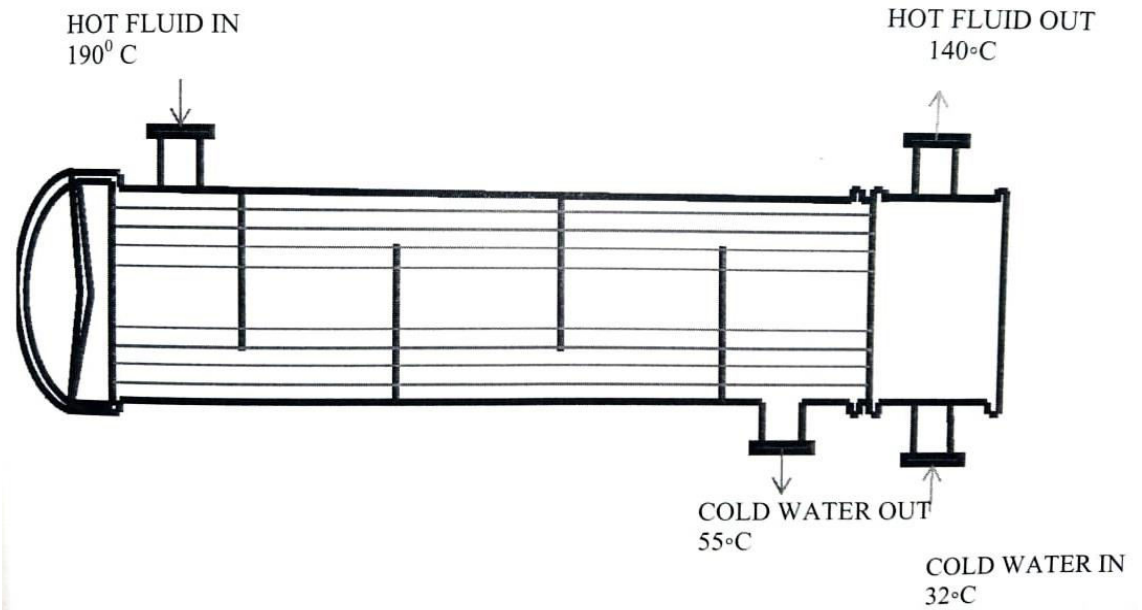
4) Reactor volume= 2.0696 m^3

5) Total volume for reactor = 2.58 m^3

6) Impeller Reynolds no = 1828.81

7) Power consumed = 4353.30 kW

Design of Horizontal Condenser



1. Define the duty: heat transfer rate, fluid flow-rates and temperatures.

2 Collect together the fluid physical properties required: density, viscosity and thermal conductivity

3. Decide on the type of exchanger to be used.

4. Select a trial value for the overall coefficient, U.

S. Calculate the mean temperature difference, ΔT_t

6. Calculate the Heat transfer area required

7. Decide the exchanger layout.

8. Calculate the overall coefficient and compare with the trial value.

9. Calculate the pressure drop for the shell side and tube side

Properties of feed

Properties	Shell Side	Tube Side
Fluid	Light Hydrocarbon	Cooling Water
Inlet temperature	$190^o\ C$	$32^o\ C$
Outlet temperature	$140^o\ C$	$55^o\ C$
No: of passes	1	2
Pressure drop allowance	23 kpa	46 kpa
Fluid flow rate	187.135 kg/hr.	-

Condenser Heat Load (Q)

Q= mλ

Where, Q= heat duty.

m = mass flow rate

λ = latent heat

Step 1: Heat Duty

$$\boldsymbol{Q_h = mCp\Delta T + m\,\lambda_c}$$

= 187.13 * 0.2734 (190- 140) + (187.13 * 65)

Q_h =14726.19 kJ/hr.

Q_h = 40.9 kJ/s

Mass Flow Rate of Chilled Water

$Q_h = Q_c$

$Q_h = mCp\Delta T$

Q/$Cp\Delta T$=m

m =14726.19/4.187 * (55 -32)

m = 152.919 kg/hr.

m = 22.46 kg/s

Driving Force for Heat Transfer

LMTD Calculation

T_1 = 190 → T_2 = 140

$t_2 = 55$ ← $t_1 = 32$

$\Delta T_1 = 135$ ΔT_2 = 108

Logarithmic Mean Temperature Difference

LMTD = (ΔT_1-ΔT_2)/ ln (ΔT_1/ΔT_2)

= (135-108)/ln (135/108)

= 121.07 °C

LMTD Correction Factor, $\mathbf{F_T}$

P = t_2-t_1/T_1-t_1 = 0.145

R=T_1-T_2/$t_1 - t_2$ = 2.173

Fouling Factor, Ft = 0.98

ΔTm = Ft * LMTD

= 0.98*121.07

=118.64 C

Estimated Area for Heat Transfer

Heat Transfer Area

Let us assume Uo= 450 W/ m^2 $^\circ C$

Heat transfer area required

A =Q/ (U * ΔTm)

A = 40.90 * 1000/ (450 * 118.64)

A=10.56 m^2

Determination of Number of Tubes

Tube dimensions 20 mm OD X 16 mm ID X 4.2 m long

Pitch = 25 mm

Square pitch

Number of Tubes

Number of tubes = Trial area/ surface area per tube

= 10.56/ (π * 0.016 * 4.2)

= 28.24 tubes = 28 tubes

From counter table

Shell ID = 237 mm = 0.237 m

Number of tubes = 28 tubes

Number of passes = 2

The condenser unit selected will be 237 mm shell ID, 28 no: of tubes 20 mm OD, 16 mm ID, 4.2 m long tube placed on 25 mm Square pitch.

Film Co-efficient Tube Side

Whereas the flow is laminar, shall we take, Seider Tate equation

$h_i d_i$ / K= 0.023 * $(N_{Re})^{0.8} * (P_r)^{0.33}$ * $(\mu / \mu_w)^{0.14}$

Flow area pass

$A_t = \left(\frac{\pi}{4}\right) * D_{i^2} * (N_T / N_P)$

= $\left(\frac{\pi}{4}\right) * 0.02^2$(28/2)

= 0.0043 m^2

G_t = m/At = 0.051/0.0043

= 12.08 kg/m^2 s

Reynolds number

$N_{Re} = G_t D_i / \mu$ {given: μ =0.7 Cp, k = 0.63W/m "c}

=0.012 * 12.08/ (0.7*10)

= 276.11

P_r= Cp * μ /K

= 4.187 * 103* 0.7* 10*/0.63 = 4.65

Whereas the flow is laminar, shall (Nre< 2100) we take, Seider Tate equation

$h_i d_i$ / K= 0.023 * $(N_{Re})^{0.8} * (P_r)^{0.33} *$ (μ / $\mu_w)^{0.14}$

(μ / μ_w)=1

h_i= 0.023 (276.11)(4.65)*0.63/ (0.016)

= 134.91 W/m^2 °C

h_{iO} = 134.91 * (0.016/0.02)

= 107.92 W/m^2 °C

Film Co-Efficient Shell Side

Mean temperature:

Shell side (T_s) = ($T_{H\ out} + T_{H\ in}$)/2

= (190+140)/2

= 165 °C

Tube side (T_W) = ($T_{C\ out} + T_{C\ in}$)/2

= (55+32) /2

= 43.5 °C

Mean temperature of condensate = (Ts +Tw) / 2

= (165+43.5) /2

= 104.25 °C

Tube loading = m/ (L * $Nt^{2/3}$)

= 0.42/ (4.2 *$28^{2/3}$)

= 0.038 kg/ms

$N_T = C\ (Lb/\ Pt)^2$

28 = 0.78 (Lb. / 0.025$)^2$

Lb. = 149.21 mm

Lb. = 0.149 m

N_r = 2/3 (0.149 / P_t)

= 2/3 (0.149 / 0.025)

= 3.97

Condensing Coefficient

$h_c = 728.7\ W/\ m^2$ ^{o}C

Tube wall Resistance

$R_w = D_O \ln(D_O / D_i) / 2Kw$

= 0.02 ln (0.02/ 0.016) / (2*50)

= 4.46* $10^{-3} m^2$ ^{o} C/ W

$R_{do} = 2.5 * 10^{-4} m^{2\ o}\ C/W$

$R_{di} = 2.5 * 10^{-4} m^{2\ o}\ C/W$

$R_{do} = 2.5 * 10^{-4} * (0.02/\ 0.016)$

$= 3.125 * 10^{-4} m^{2\ o}\ C/W$

Overall Film Co- Efficient

$U_O = [\,1/\ h_O\ + Rd_O + Rd_{di0} + Rw + 1/\ h_{iO}]^{-1}$

= 0.0106 + 0.1650 * 10^{-3}

U_O= 527.4 W/ m^2 ^{0}C

Required Area

Ar. =Q/$U_O \Delta T_m$

= 40.1 / (527.4 * 118.64)

= 6.4 m^2

Available Area

Aa = $\pi * OD * L * N_t$

= 3.14 * 0.02 * 4.2 *28

= 7.2 m^2

Determination of Percent Excess Area

% Excess Area = 7.2 – 6.4 / 6.4

= 12.5%

Pressure Drop Tube Side

ΔP_t= {(f*$G^2{}_t$ * L*N_p) / (2*$10^6 * S * \Phi_t * D_i$)} + {(2.5 * Np V^2 S)}

f =0.7 * $(N_{Re})^{-0.33}$

= 0.7* $(276.14)^{-0.33}$

= 0.109

ΔP_t = {(0.10*18.08^2 * 4.2 * 2) / (2 * 10^6 * 0.016 * 1) + (2.5* 2*12.08^2 * 1)}

= 45.59 kPa

Pressure drop is less than 70 kPa so design is safe.

Pressure Drop Shell Side

$$\Delta P_S = 0.5\{f * G^2{}_S * D_s * (N_b + 1)\}/\ (2 * 10^6 * S * \Phi_t * D_e$$

$$D_e = \{\ 4 * \left(P^2{}_t - \left(\frac{\pi}{4}\right) * D^2{}_O\right\}/\ \{\pi * D_e\}$$

= 0.0198 m

Baffle spacing = 0.5m

Clearance (c') = 0.002 m

Shell dia = 0.390 m

Area = Ds * c * Bs / Pt.

= 0.237 * 0.002 * 0.59/0.025

A = 0.0948 m^2

$G_S = 22.46\ /\ 0.094$

= 249.55 kg/m^2 s

$\mu/\ \mu_W$ = 1

N_B =Effective length / baffle spacing

= 4.16/ 0.23

= 20 baffles

f = 1.87 * $(N_{Re})^{-0.4}$

f = 4.6

ΔP_S = (0.3903 *249.55^2* 4.2 * 28) / (2 *$10^6 * 0.019 * 0.79$)

$\Delta P_S = 22\ kPa$

Result

Pressure drop is less than 35 kPa so design is safe.

Design of Summary

1) Heat duty of heat exchanger = 40.09 kW

2) Area required for heat transfer = 6.4

3) Area available for heat transfer = 7.2

4) Number tubes = 28

5) % Excess area == 12.5 %

6) Number of passes on tube side = 2

7) Number of passes on shell side = 1

8) Shell ID = 390 mm

9) Film heat transfer coefficient tube side = 134.91 W/ m^2°C

10) Film heat transfer coefficient shell side = 728.7 W/m^2°C

11) Pressure drop tube side ΔPT = 45.59 kPa

12) Pressure drop shell side ΔPs = 22.86 kPa

4.CONCLUSIONS

The demand for Citronellol is always higher due to their strong aromatic strength. The current batch reactor has the capacity 1000kg per batch, the theoretical results showed extremely significant results and data's. The vapor space required in the reactor was found to be 517.32 liters. The diameter of the reactor was found to be 2.2m.The required height of the reactor was around 3.3 m. proper mixing in the reactor is performed by impellers and its diameter was found to be 0.73 m. In the horizontal condenser the area of heat transfer was found to be 10.56 m^2 and the no of tubes required for better heat transfer was found to be 28.The design study on these equipments revealed the importance of various parameters needed to maintain optimum condition for the better yield of Citronellol.

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