Design of pilot plant for the production of essential oil from *Eucalyptus* leaves

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*Received 05 December 2005; revised 12 April 2006; accepted 05 July 2006*

Study presents design, construction and test run of a pilot plant for the production of essential oils (0.864 l/h) from *Eucalyptus* leaves. Analysis shows that the rate at which steam passes through the leaf bed may deviate from linear relationship to curve depending on the loading capacity. An oil/leave production rate of \(3.0 \times 10^{-2}\) ml/g was obtained. In designing pilot plant, tank still has a dimension of 0.45 m diam and length of 1.65 m. Packed bed height was 0.565 m with an expected pressure head loss of 0.013 m. Overall heat transfer coefficient was calculated as 176.12 W/m\(^2\)°C. The condenser has a heat load of 4970 kJ/s requiring a cooling water flow rate of 0.047 kg/sec.

**Keywords:** Essential oils, *Eucalyptus* leaves, Pilot plant

**IPC Code:** C11B1/04

**Introduction**

In this study, essential oil was extracted from *Eucalyptus* leaves. Comprehensive experiments on characterization of the technological parameters and crude obtained were performed in laboratory condition.

**Process Design of Pilot Plant**

In this project, steam distillation technique consists of the tank still or reactor, condenser and separator (Fig. 1). Steam required for the process was obtained from a steam generator or water distillation equipment\(^1,2\).

**Design Conditions**

The technological conditions for designing the process were: Optimum steam: oil ratio (per gram leaves), \(9.33 \times 10^{-4}\): \(9.42 \times 10^{-6}\) ml/sec; Optimum steam supply rate (per gram leaves), \(9.33 \times 10^{-4}\) ml/sec; Residence time after first water oil mixture drops, 50-60 min.; Density of essential oil, 853.8 kg/m\(^3\); Density of steam oil mixture, 853.96 kg/m\(^3\); Viscosity of steam oil mixture (\(\mu\)), \(6.92 \times 10^{-4}\) Ns/m\(^2\); Heat capacity of mixture (\(c\)), 4.0 kJ/kg°C; Thermal conductivity of mixture (\(k_0\)), 0.69 W/m°C; Condensate temperature, 40°C; Operating pressure, atmospheric; Density of leaves, 1800 kg/m\(^3\); and Operating bulk density, 310 kg/m\(^3\).

**Material Balance**

In the packed bed process, material is heated to the steam temperature (unsteady state). The moving steam transfers the deposited oil on the leaves surface to the condenser (steady state). Maximum volume of oil removed from the leaves was \(3 \times 10^{-2}\) ml/sec/kg. Therefore, at a production rate of \(2.4 \times 10^{-4}\) l/s or 0.864 l/h per batch cycle, maximum quantity of leaves required was 28.8 kg/h (Table 1). Quantity of steam required = mass flow rate of steam x induction time. Experimentally, maximum induction period is 21 min, however 25 min was considered for this project, i.e. 0.0205x25x60 = 30.75 kg. Theoretically, the condensate flow rate is the same as the steam. Allowing for film condensate on the leaves surfaces of 10%, the condensate flow rate is assumed as 0.01845 kg/sec.

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![Fig. 1—Pilot plant for production of essential oil](image-url)
Energy Balance

At unsteady state, packed leaves bed could be likened to plug flow system in which the fluid (steam) at 100°C loses the heat to the bed (Fig. 2). Heat loss by the steam will continue until a steady state is reached at which the temperature of the bed is equal to that of the steam.

Condensate, 0.00205 kg/sec
Oil, 2.05x10^-4 kg/sec (2.4x10^-7 m^3/sec)

At steady steam supply rate in a given reactor of height H, induction period is dependent on the steam-leaves ratio or packing density. Heat supplied to the system $Q_{\text{supplied}}$ is calculated as

\[ Q_{\text{in}} = m_s L \]

where, \( m_s \) = mass flow rate of steam, 0.0205 kg/s; \( L = \) latent heat of vaporization of water.

\[ Q_{\text{supplied}} = 0.0205 \times 22600 = 463.3 \text{ kJ/sec} \]

At a steam transfer efficiency (90%), it is expected that additional heat (46.33 kJ/s) will be required from the steam generator to the tank still. Therefore,

Required process heat = 463.30 + 46.33 = 509.63 kJ/s

Heat Received by Tank Still and Contents

Heat adsorbed by the system \( (Q_{\text{recieved}}) \) = heat content of the oil \( (Q_{\text{oil}}) \), spent leaves \( (Q_{\text{sl}}) \), condensate removed from the reactor with marginal fall in temperature \( (Q_{\text{c}}) \), condensate on leaves surface \( (Q_{\text{cls}}) \), heat of vaporization of condensate \( (Q_{\text{cv}}) \) and construction material \( (Q_{\text{cm}}) \),

\[ Q_{\text{recieved}} = Q_{\text{oil}} + Q_{\text{sl}} + Q_{\text{c}} + Q_{\text{cls}} + Q_{\text{cv}} + Q_{\text{cm}} = 0.004 + 0.565 + 309.96 + 34.44 + 46.33 + 20.85 = 412.15 \text{ kJ/s} \]

Design of the Tank Still

Calculation of the Tank Still Operation Volume

Volume of the packed bed using bulk density, V = 28.8/310 =0.093 m^3. For a cylindrical shaped vessel, V = 0.093 = 0.785 D^2H. For this design, a height to diam ratio H:D=1.3. Therefore, V=0.093 m^3, D = 0.45 m, H = 0.585 m. At equilibrium

\[ Q_{\text{supplied}} = Q_{\text{recieved}} + KF \Delta T_{\text{cm}} \]

\[ kF\Delta T_{\text{cm}} = 463.3 - 412.17 = 51.13 \text{ kJ/s} \]

\[ Q_{\text{cm}} = KF\Delta T_{\text{cm}} = 51.13 \text{ kJ/s} = \text{heat lost through the tank still wall} \]

K = 1/(\( \delta/\lambda + 1/\alpha_i \)) = 0.275W/mK

where, \( \delta = \) insulator thickness, 20 mm; \( \lambda = \) heat transfer coefficient of insulator (fiber with binder and baked), 5.5 kW/mK; \( \alpha = \) heat transfer coefficient of construction material (aluminum), 0.2059 kW/mK.

\[ \Delta T_{\text{cm}} = 100 - 25 = 75\text{°C}. \]

Therefore, \( F = 51.13/(0.275 \times 75) = 2.48 \text{ m}^2 = \pi DH \]
where, \( D = \text{diam of the tank still; this will be taken as 0.45 m as earlier calculated from the material balance; } \) 
\( H = \text{the overall height of the tank, m.} \)

An inner holding casing shall be provided to ensure easy transfer of heat and removal of spent material. This shall be constructed from the same material as the outer conducting aluminum alloy casing. An allowance of 0.005 m between the two casings shall be provided. Total area of the inner holding casing = 0.01x15.1 = 0.151m². Therefore,

\[
L = \frac{(2.48 - 0.151)}{(3.142x0.45)} = 1.65 \text{ m}
\]

**Pressure Drop Across The Packed Bed**

Pressure drop across the packed bed is dependent on the packing density of the leaves. As observed experimentally, an optimum steam leaves ratio is necessary for the process. Pressure drop across a packed bed of a single incompressible fluid through an incompressible bed of solid particles could be correlated by the formula. The bed was considered as fairly incompressible based on experimental observations. However, corrective coefficient will be introduced to account for possible compression of the leaves bed.

\[
\Delta P = \frac{[2f_mG^2L(1 - \varepsilon)^3\rho_e\phi^3\varepsilon^3]}{[D_2g_e^2\rho_e^2(1 - \varepsilon)^3]} = \frac{[2x23.48x0.0205^2x0.585(1-0.17)^2]}{[0.142xg_e^2x995x0.9375^2x0.17^3]} = 0.01309/g_e
\]

The observed insignificant pressure drop is expected considering that the process takes place at atmospheric condition. Using the velocity-head concept approx velocity drop can be obtained as

\[
\Delta P = \Delta h = 50(v^2/2g_e) = 0.01309g_e, \text{ therefore, } \frac{v}{0.02288kg/s} - \text{ mass velocity}
\]

This implies that the actual mass velocity of steam from the generator to the tank still should be maximum at 0.0205 + 0.02288 = 0.04338kg/sec.

**Condenser Design**

Heat capacity of the steam-essential oil mixture = 4000.0 kJ/kg°C; Heat load, \( Q = m_wc\Delta T = 0.020705x4000x(100-40) = 4970 \text{ kJ/sec}; \) Heat capacity of water = 4.2 kJ/kg°C; and Cooling water mass flow rate, \( G_w = Q/c\Delta T = 4970/(4200x(50-25)) = 0.047 \text{ kg/sec}. \) The mean log temperature change

\[
\Delta T = [(100-40)-(50-25)]/ln[(100-40)/(50-25)] = 40°C.
\]

To calculate the dimensionless temperature ratio for a one pass and two tubes passes (horizontal type),

\[
R=(100–40)/(50–25)=2.4 \text{ and } S=(50–25)/(100–40)=0.33
\]

From the temperature correlation factor graph, \( F_i = 0.85. \) Therefore,

\[
\Delta T_m = 0.85 \times 40 = 34°C
\]

From the correlation Table of hot fluid versus cold fluid, the overall heat transfer coefficient \( U \) was taken as 300 W/m²°C. Therefore,

Provisional area, \( A_p = 4970/(300x34) = 0.49 m^2 \)

For Condenser design, a 16 mm internal diam (\( d_i \)), 20 mm outer diam (\( d_o \)) and 0.55 m tube length (\( L \)) made from aluminum was chosen. For welding, \( L = 20 \text{ mm} \) outer diam (\( d \)) and \( 0.55 \text{ m} \) tube length (\( L \)) was made from aluminum was chosen.

**Shell Side Coefficient (Coolant)**

Different parameters are as follows: Mean water temperature, \( t=(50+25)/2=38°C; \) Tube cross sectional area = 0.785x16² = 201 mm²; Tubes per pass=15/2=8; Total flow area, \( A_{Tw} = 8x201x10^{-6} = 1.6x10^{-3} \text{m}^2; \) Water mass velocity, \( G_w = G_e/A_{Tw} = 0.047/1.6x10^{-3} = 29.22 \text{ kg/s.m}^2; \) Water linear velocity, \( u = 29.22/995 = 0.029 \text{ m/sec}; \) Heat transfer coefficient, \( h_t = (4200(1.35 + 0.02t) x u^{0.5})/d_i^{0.2} = 299.85 \text{ W/m}^2\text{ oC}; \) and \( \text{Re} = G_xd_i/\mu = 29.22x16x10^{-3}/8x10^{-4} = 584.4. \)

**Shell Side Coefficient**

Different parameters are\(^{37} \) as follows: Choice of baffle spacing, \( I_B = d/5 = 150/5 = 30 \text{ mm}; \) Square tube pitch, \( P_i = 1.25d_o = 1.25x20 = 25 \text{ mm}; \) Cross flow area, \( A_i = [(P_i - d_o)/P_i]d_iI_B = [(25-20)/25] x 150x30x10^{-6} = 9.0x10^{-4} \text{ m}^2; \) Equivalent diam, \( d_e = 1.27/d_i(P_{i}^2 - 0.785d_i^2) = 1.27x20x25^2 - 0.785x20^2 = 19.75 \text{ mm}; \) Linear velocity, \( u_i = G_{x_i}/\rho = 23/853.8 = \)
Therefore, that collection will take place every hour per batch. Mean shell temperature = (100 + 50)/2 = 75°C; Re = 23x19.75x10³/0.69 = 658.33; Pr = cₚµ/kₒ = 4x10³x 6.92x10⁻⁴/0.69 = 4.01; and from the chart at a 15% baffle cut, jₚ = 2.5x10⁻². Overall heat transfer coefficient is calculated as,

\[
\frac{1}{U} = \frac{1}{h_s} + \frac{1}{h_{ad}} + \frac{[d_s In(d_s/d_i)]/2k_w}{+ (d_o/d_i)x1/h_{id}} + (d_i/d_o)/1/h_f
\]

where, hₚ and hₚ are the inside and outside fluid film coefficient, = 6000 W/m² °C; kₒ = thermal conductivity of the tube wall material (aluminum alloy) = 205.9 W/m² °C. Thus,

\[
\begin{align*}
1/U &= \frac{1}{189.41+1/6000} + \frac{[20x10³ In(20/16)]}{2x205.9} + 20/16 x 1/6000 + 20/16 x 1/299.85 = 0.001123 \\
+ 0.0001667 + 0.00001084 + 0.0002083 \\
+ 0.004169 &= 0.0056778.
\end{align*}
\]

\[
U = 176.12 \text{ W/m}² \text{ °C}
\]

This is below the assumed value of 300 W/m² °C. It shows the low heat load of the condenser and consequently the potential for process expansion.

At Tube side, from the chart at Re = 584.3, jₚ = 0.2x10⁻¹

\[
\begin{align*}
\Delta P &= |j_p(L/d_o)(\mu/\mu_o)^{0.14} + 2.5|\mu_u^2/2 (8x0.02) \\
x(0.55/0.016) &x 0.98 + 2.5\times 995 \times 0.029²/2 = 2.2N/m²
\end{align*}
\]

At shell side, from the chart at Re = 658.33, jₚ = 0.2x10⁻¹

\[
\begin{align*}
\Delta P &= |j_p(d_o/d_i)(L/I_h)\mu_u²/2 8x0.02x (150/19.75) \\
x (0.51/30x10⁻³) \times 853.8x0.027²/2 = 6.43N/m².
\end{align*}
\]

The obtained pressure drops in the condenser show that the baffle pitch is adequate.

Separator Unit

In the separator, oil and water layer are allowed to accumulate to be drawn off periodically. The collection vessel is made from glass with separate outlet valve for the two components. It is expected that collection will take place every hour per batch. Therefore,

Volume of the vessel = 2.424x10⁻⁵ x 3600 = 0.08726 m³

Allowing 20% extra volume for safety, the actual operating volume = 0.1047 m³. The vessel height h = 0.65 m. The oil is about 1% of the total volume of 0.00087 m³. Expected oil height h at a base diam of 0.4 m;

\[
h = 0.00087/(0.785x0.4²) = 0.0087/0.1256 = 0.0069 \text{ m or 6.9 cm}
\]

Conclusions

Design parameters for the fabrication of an essential oil pilot plant (8.64 l/h) have been obtained. Tank still is 0.45 m wide and 1.65 m long. Packed bed height was 0.565 m with an expected pressure head loss of 0.013 m. Overall heat transfer coefficient was calculated as 176.12 W/m² °C. The condenser has a heat load of 4970kJ/s requiring a cooling water flow rate of 0.047kg/sec. Financial analysis showed that the project has a pay back period of 2½ years and percentage profit turnover (PPT) of 61%.

Acknowledgement

Authors thank the Raw Material Research and Development Council, Abuja for the moral and financial contributions toward this project.

References