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

E M Okonkwo[1], J O Odigure[2], J O Ugwu[1,*], K Mu'azu[1], I S Williams[1], B E Nwobi[1], F K Okorie[1] and V N Oriah[1]

[1]National Research Institute for Chemical Technology, Zaria, Nigeria

[2]Federal University of Technology, Minna, Nigeria

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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 3.0×10^{-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 176.12W/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

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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), 9.33×10^{-4}: 9.42×10^{-6} ml/sec; Optimum steam supply rate (per gram leaves), 9.33×10^{-4} ml/sec; Residence time after first water oil mixture drops, 50-60 min.; Density of essential oil, 853.8kg/m^3; Density of steam oil mixture, 853.96 kg/m^3; Viscosity of steam oil mixture (μ), 6.92×10^{-4} Ns/m^2; Heat capacity of mixture (c), 4.0 kJ/kg°C; Thermal conductivity of mixture (k_f), 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.

*Author for correspondence
E-mail: ugwu_jo@yahoo.com

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 3×10^{-2} ml/sec/kg. Therefore, at a production rate of 2.4×10^{-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.

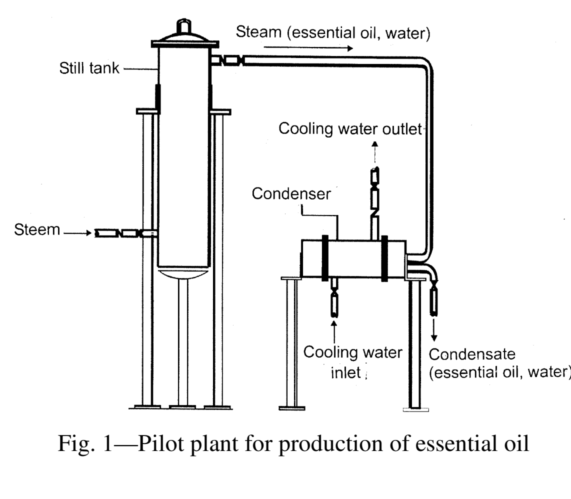

Fig. 1—Pilot plant for production of essential oil

Table 1 — Summary of material balance

	In		Out	
	Kg/h	kg%	kg/h	kg%
		Tank still (water, nil)		
Leaves	28.80	27.87	28.787	27.86
Steam	73.8	71.42	73.8	71.43
Oil	0.738	0.71	0.738	0.71
Total	103.338	100	103.325	100
		Condenser (Leaves, nil)		
Steam	73.8	26.38	-	-
Water	-	-	73.8	26.38
Oil	0.738	0.26	0.738	0.26
Coolant	205.2	73.35	205.2	73.35
Total	279.738	100	279.738	100
		Separator (Leaves and steam, nil)		
Water	73.8	99.01	73.8	99.01
Oil	0.738	0.99	0.738	0.99
Total	74.538	100	74.538	100

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[3-6].

Condensate, 0.00205 kg/sec

Oil, 2.05×10^{-4} kg/sec ($2.4 \times 10^{-7} m^3$/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 $Q_{supplied}$ is calculated as

$$Q_{in} = m_s L$$

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

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

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

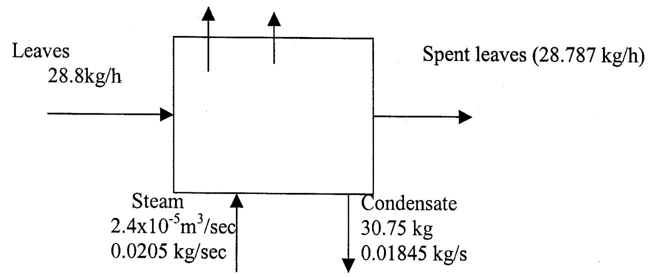

Fig. 2 — Heat balance at unsteady state

Heat Received by Tank Still and Contents

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

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

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

$$Q_{supplied} = Q_{recieved} + KF\Delta T_{cm}$$

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

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

$$K = 1/(\delta/\lambda + 1/\alpha_1) = 0.275 \text{W/mK}$$

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

$$\Delta T_{cm} = 100 - 25 = 75°C.$$

Therefore, $F = 51.13/(0.275 \times 75) = 2.48 \text{ m}^2 = \pi DH$

where, D = diam of the tank still; this will be taken as 0.45 m as earlier calculated from the material balance; H = 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 = 0.151m^2. Therefore,

$$L = (2.48 - 0.151)/(3.142x0.45) = 1.65 \text{ m}$$

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[1]. 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.

$$\Delta P = [2f_m G^2 L(1 - \varepsilon)^{3-n}]/[D_p g_c \rho \phi_s^{3-n} \varepsilon^3]$$
$$= [2x23.48x0.0205^2 x0.585(1-0.17)^2]/$$
$$[0.142xg_c x995x0.9375^2 x0.17^3] = 0.01309/g_c$$

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

$\Delta P = \Delta h \approx 50(v^2/2g_c) = 0.01309g_c$, therefore,
v = 0.02288kg/s - 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, $Q = m_m c\Delta T$ = 0.020705x 4000x(100-40) = 4970 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 kg/sec. The mean log temperature change

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

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

R=(100–40)/(50–25)=2.4 and S=(50–25)/(100–40)=0.33

From the temperature correlation factor graph, F_t = 0.85. Therefore,

$$\Delta T_m = 0.85 \text{ x } 40 = 34°C$$

From the correlation Table of hot fluid versus cold fluid, the overall heat transfer coefficient U was taken as 300 W/m^2 °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 was taken as 0.51 m. Area of 1 tube, A = circumference x L = $\pi 2.0x10^{-2}$ x 0.51 = 0.032 m^2. Number of tubes $N_t = A_p/A$ = 0.49/0.032 = 15.29.

Estimated shell diam
$D_h = d_o(N_t/k_1)^{0.453} = 0.02(15/0.215)^{0.453}$ = 137 mm

where, k_1 is constant = 0.215. A bundle clearance of 13 mm will be allowed. Therefore, shell diam, d_s = 137 + 13 = 150 mm.

Tube Side Coefficient (Coolant)

Different parameters are as follows: Mean water temperature, t=(50+25)/2=38°C; Tube cross sectional area = $0.785x16^2$ = 201 mm^2; Tubes per pass=15/2=8; Total flow area, A_{Tw} = $8x201x10^{-6}$ = $1.6x10^{-3} m^2$; Water mass velocity, $G_s = G_w/A_{Tw}$ = 0.047/ $1.6x10^{-3}$ = 29.22 kg/s.m^2; Water linear velocity, u = 29.22/995 = 0.029 m/sec; Heat transfer coefficient, h_i = $\{4200(1.35 + 0.02t) \text{ x } u^{0.8}\}/d_i^{0.2}$ = 299.85 W/m^2 °C; and Re = $G_s x d_e/\mu$ = $29.22x16x10^{-3}/8x10^{-4}$ = 584.4.

Shell Side Coefficient

Different parameters are[3,7] as follows: Choice of baffle spacing, $I_B = d_s/5$ = 150/5 = 30 mm; Square tube pitch, $P_t = 1.25d_o$ = 1.25x20 = 25 mm; Cross flow area, $A_s = [(P_t - d_o)/P_t]d_s I_B]$ = [(25-20)/25] x $150x30x10^{-6}$ = $9.0x10^{-4}$ m^2; Equivalent diam, d_e = $1.27/d_o (P_t^2 - 0.785d_o^2)$ = $1.27/20(25^2 - 0.785x20^2)$ = 19.75 mm; Linear velocity, $u_1 = G_{s1}/\rho$ = 23/853.8 =

0.027 m/s, where, ρ-density of steam/oil mixture; Mean shell temperature= (100 + 50)/2 = 75°C; Re = $23x19.75x10^{-3}/0.69$ = 658.33; Pr = $c_p\mu/k_f$ = $4x10^{-3}x\ 6.92x10^{-4}/0.69$ = 4.01; and from the chart at a 15% baffle cut, $j_h = 2.5x10^{-2}$. Overall heat transfer coefficient[8,9] is calculated as,

$$1/U = 1/h_s + 1/h_{od} + [d_o In(d_o/d_i)]/2k_w + (do/d_i)x1/h_{id} + (d_o/d_i)/1/h_I$$

where, h_{id} and h_{od} are the inside and outside fluid film coefficient, = 6000 W/m^2 °C; k_w = thermal conductivity of the tube wall material (aluminum alloy) = 205.9W/m^2 °C. Thus,

$$1/U = 1/890.41+1/6000 + [20x10^{-3} In(20/16)]/2x205.9 + 20/16 \times 1/6000 + 20/16 \times 1/299.85 = 0.001123 + 0.0001667 + 0.00001084 + 0.0002083 + 0.004169 = 0.0056778.$$

$$U = 176.12\ W/m^2\ °C$$

This is below the assumed value of 300 W/m^2 °C. It shows the low heat load of the condenser and consequently the potential for process expansion[1,5].

At Tube side, from the chart at Re = 584.4, $j_f = 0.2x10^{-1}$

$$\Delta P = [8j_f(L/d_i)(\mu/\mu_w)^{0.14} + 2.5]\rho u_t^2/2\ (8x0.02 x(0.55/0.016) \times 0.98 + 2.5)995 \times 0.029^2/2 = 2.2N/m^2$$

At shell side, from the chart at Re = 658.33, $j_f = 0.2x10^{-1}$

$$\Delta P = [8j_f(d_s/d_e)(L/I_B)\rho u_s^2/2\ 8x0.02x\ (150/19.75) \times (0.51/30x10^{-3}) \times 853.8x0.027^2/2 = 6.43N/m^2.$$

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 = $2.424x10^{-5} \times 3600 = 0.08726\ m^3$

Allowing 20% extra volume for safety, the actual operating volume = 0.1047 m^3; $V = 0.785\ D^2xh$.

Taking the internal vessel diam as 0.4 m. The vessel height h = 0.65 m. The oil is about 1% of the total volume of 0.00087 m^3. Expected oil height h at a base diam of 0.4 m;

$$h = 0.00087/(0.785x0.4^2) = 0.00087/0.1256 = 0.0069\ m$$ 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/m^2 °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%.

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