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DESIGN AND CONSTRUCTION OF PILOT SCALE PROCESS SOLVENT EXTRACTION PLANT FOR NEEM SEED OIL

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Abstract
A pilot Neem oil solvent extraction plant of 9.65kg/day Neem seed kernel capacity was designed and fabricated. Grade 304 stainless steel was used for construction of the extractor, evaporator, condensate receiver and the flat blade turbine impeller. The concept of overall heat transfer coefficient was adopted for calculating the heat transfer areas of the extractor and evaporator and their sizing. The extraction was carried out at 50°C extraction temperature and particle size of 0.425 – 0.710mm at an efficiency level of 81.91%. The percentage yield obtained was 36.86% when flat blade turbine impeller was operated at 84 rpm for 40 minutes contact time. The GCMS result shows the composition of the extracted Neem oil to be oleic acid, 40.41%; stearic acid, 27.65%; palmitic acid, 25.36%; octanal, 3.90%; elaidic acid, 1.23%; lactone, 0.97%; and methyl stearate, 0.48%. This percent composition compares favourably with literature values. The properties of the Neem oil were found to be: specific gravity, 0.9111; pH, 6.5; refractive index, 1.4668; iodine value, 70.21g/g; acid value, 34.33mgKOH/g and Saponification value, 180.95 mgKOH/g.

Keywords: Neem oil, Design, Construction, Pilot Scale Process and Extraction.

1. Introduction
Neem tree, which is also known as *Azadrichta indica*, is one of the best known trees in India, which is known for its medicinal properties. The main reason behind the popularity of the Neem oil is that it is used to treat some of the most common ailments that the people face [1].
Neem oil is a vegetable oil pressed from the fruits and seeds of the neem (*Azadirachta indica*), an evergreen tree which is endemic to the Indian continent and has been introduced to many other areas in the tropics such as Nigeria. Neem oil varies in colour; it can be golden yellow, yellowish brown, dark brown, greenish brown or bright red. It has a rather strong odour that is said to combine the odopurs of peanut and garlic. It is composed mainly of triglycerides and contains many titerpenoid compounds which are responsible for the bitter taste. It is hydrophobic in nature; inorder to emulsify it in water for application purposes it must be formulated with appropriate surfactants. Azadirachtin is the most well known and studied titerpenoid in the neem oil. The azadirachtin content of neem oil varies from 300ppm to over 2500ppm depending on the extraction technology and quality of the neem seeds crushed. Neem oil also contains steroids (campesterol, bête- sitosterol, stigmasterol). The solvent extraction of Neem oil at laboratory scale has low production rate. The need to have higher production rate using solvent cannot be over emphasized, hence the need to design and fabricate an agitated pilot solvent extraction plant and use flat blade turbine impeller as a step forward toward industrial production of Neem oil using solvent.

2. Literature review
There are several methods to obtain Neem oil from the seeds like mechanical pressing, supercritical fluid extraction, and solvent extraction. Mechanical extraction is the most widely used method to extract Neem oil from Neem seed. However, the oil produced with this method usually has a low price, since it is turbid and contains a significant amount of water. Extraction using supercritical fluid, the oil produced has very high purity; however the operating and investment cost is high. Extraction using solvent has several advantages. It gives higher yield and less turbid oil than mechanical extraction, and relative low operating cost compared with supercritical fluid extraction [2].
From past works, solvent extraction of Neem oil from Neem seed using agitated vessel extraction at bench scale gives a yield of 33.19% using ethanol as solvent, 50°C extraction temperature and 0.425 – 0.710 mm particle size for 3 hours [1]. Yield of

41.11% was reported when Soxhlet apparatus was used at an extraction temperature of 50°C, ethanol as solvent and 0.425 – 0.710 mm particle size for 3 hours [2]. Agitation and mixing increase the mass and heat transfer in the extractor, thereby enhancing the leaching of oil from the Neem seed kernel.

Neem oil extract, which is the fatty acid-extract of Neem tree seeds, is the most widely used product of the Neem tree. Neem seeds are about 25 - 45% oil and provide the major source of Neem chemicals [3]. The average composition of Neem oil is shown in Table 1. The standard properties of Neem oil are shown in Table 2.

Table 1: Average Composition of Neem Oil

Formula	Fatty acid	Composition range
Linoleic acid	$C_{18}H_{32}O_2$	6-16%
Oleic acid	$C_{18}H_{34}O_2$	25-54%
Palmittic acid	$C_{16}H_{32}O_2$	16-33%
Stearic acid	$C_{18}H_{36}O_2$	9-24%
Linolenic	$C_{18}H_{30}O_2$	ND*
Palmitoleic acid	$C_{16}H_{30}O_2$	ND*

Source: [3]. ND = Not Determined*

Table 2: Standard Properties of Neem Oil

Property	Literature Value	Unit
Odour	Garlic	-
Specific gravity at 30°C	0.908-0.934	-
Refractive index at 30°C	1.4615-1.4705	-
pH	5.7 – 6.5	-
Iodine value	65 – 80	g/g
Acid Value	40	mg KOH/g
Saponification value	175-205	mg KOH/g

Source: [1, 4, and 5].

The percentage yield of oil is given as:

$$\% \text{ yield} = ((W_1 - W_2)/W_1) \times 100 \quad (1)$$

where W_1 is the eight of seed particle before extraction, W_2 is the Weight of seed particle (Cake) after extraction. [1]

3. Material and methodology

The block diagram and flow sheet for the extraction process of oil from Neem seed were developed and shown in Figures 1 and 2 respectively.

3.1 Design consideration

3.1.1 Design of Extractor

Capacity of the extractor $= \frac{0.3348}{0.0347} = 9.65$ kg/day of Neem Seed Kernel

3.1.1.1 Energy Balance over the Extractor

From Figure 3, For a batch process without chemical reaction, the energy balance is given as

$$AH_a + BH_b + CH_c + H_g = DH_d + EH_e + FH_f \quad (2)$$

Where A, B…F are the mass flow rates of components and H_a…H_f are the respective enthalpies of the components ,while H_g is the heat energy absorbed from the heating coil by the materials.

Enthalpy of a substance [6] at constant pressure = $Cp(T_2 - T_1)$ where Cp is the specific heat capacity of the component, J/kg °C. Using (2) it can be shown that: $366.4052 + H_g = DH_g + 547.7169$.

Let the heat energy of cake, DH_g be the heat energy of NSK minus the heat energy of Neem oil, so that DH_g now becomes 1.8909 J/s from which H_g which is also the heat absorbed by the material H_f is now 183.203 J/s.

3.1.1.2 Calculation of the Area of Extractor

The following data is used:

(a) Film coefficient of organic solvent = 340 – 2800 W/m²°C
(b) Film coefficient of oil = 50 – 1500 W/m²°C
(c) Film coefficient of air = 5 – 25 W/m²°C
(d) Heat energy supplied by the heating coil Q = 183.203 J/s
(e) Range of value for the ethanol/oil mixture is 151.41 – 2346.56 W/m²°C

The assumed overall heat transfer coefficient, U [7] is given as:

$$U = \frac{0.7 \times h_i \times h_o}{h_i + h_o} \quad (3)$$

where h_i is the inside film coefficient of ethanol/oil mixture and h_o is the outside film coefficient of air. Film coefficient of the mixture lower limit is calculated using (3) to be 151.41 W/m²°C, while the lower limit was found to be 2346.56 W/m²°C.

Using (3), the heat transfer coefficient U was calculated as 8.12 W/m² °C

Assuming no heat loss, $Q = UA\Delta T$ where Q is the quantity of heat, J/s, A is the area, m² and ΔT is the temperature difference. From the forgoing, area of the extractor, A can be found to be 0.7521 m²

Let $H = 2.5Dt$, where H is the Height and Dt is diameter of extractor, both in meters, then area of the extractor A is given by:

$$A = \pi DtH + \pi Dt^2/4 \quad (4)$$

Total volume charged into the extractor vessel = volume of solvent for extraction + volume of NSK = 0.02163 m³. The volume of extractor V is given by:

$$V = (\pi Dt^2 H)/4 \quad (5)$$

When we substitute $Dt = 0.295 \cong 0.30$m and $V = 0.02163$, we can show from (5) that height of mixture in the extractor, $H = 0.3060$m.

Let the actual height of the extractor, H be 27.5% above the calculated value of 0.306m for safety purpose so that $H_{actual} = 0.306 + 0.275 \times (0.306) = 0.39$m. Since $0.75\text{m} > 0.39\text{m}$, it is economical to construct the extractor using 0.39m as the height.

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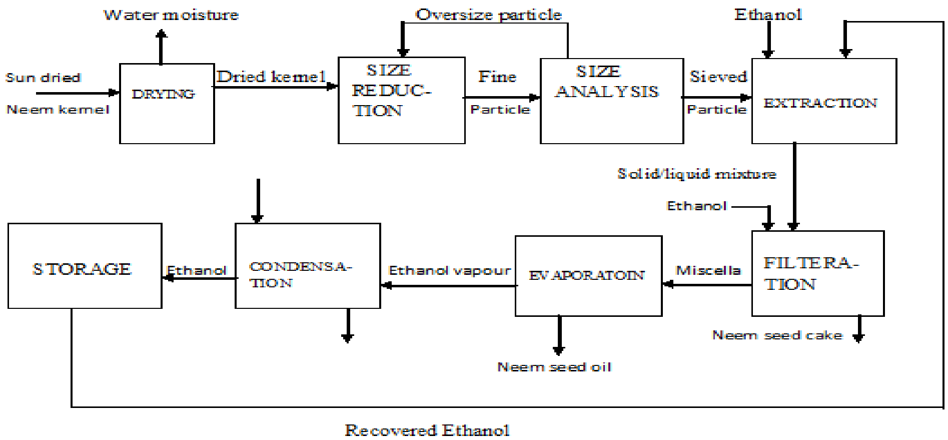
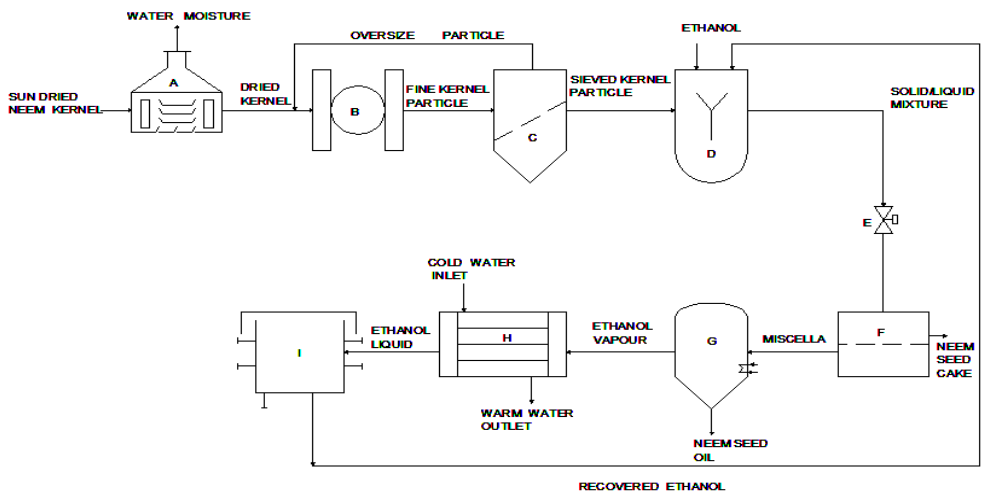


Figure 1 : Block diagram for the Extraction of oil from Neem seed



KEY: A – DRYING OVEN B – GRINDER C – LABORATORY TEST SIEVE D – MIXER EXTRACTOR E – CONTROL VALVE F – FILTER G – EVAPORATOR H – CONDENSER I – STORAGE TANK

Figure 2: Flow sheet for the Extraction of Oil from Neem Seed

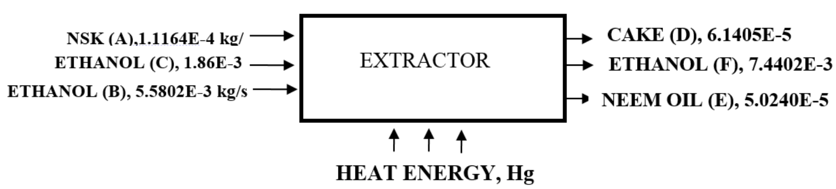


Figure 3: Simplified diagram for the extraction of oil from NSK

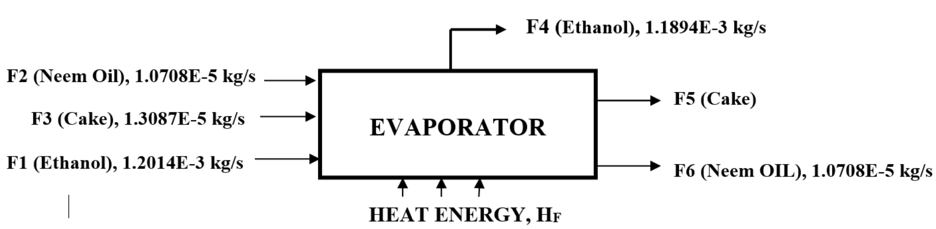


Figure 4: Simplified diagram for the recovery of Neem oil

The assumed overall heat transfer coefficient (U= 8.12 W/m^2^{o}C) is validated if the calculated overall heat transfer coefficient based on calculated film coefficient is equal to the assumed overall heat transfer coefficient value

3.1.1.2 Calculation of Film Coefficient

For heating liquid in a baffled cylindrical tank equipped with a coil and a turbine impeller, the individual heat transfer coefficient [7] is given as:

$$h = 0.17\left(\frac{k}{D_c}\right)\left(\frac{D_a^2 \times N \times \rho}{\mu}\right)^{0.67}\left(\frac{C_p \times \mu}{k}\right)^{0.37}\left(\frac{D_a}{D_t}\right)^{0.1}\left(\frac{D_c}{D_t}\right)^{0.5}\left(\frac{\mu}{\mu_w}\right) \quad (6)$$

where, h is the individual heat transfer coefficient between coil surface and liquid mixture, W/m^{2o}C, Dc is the outside diameter of heating coil = 0.005m, k is the thermal conductivity of liquid at average temperature of 35^{o}C ($k_{ethanol}$ = 0.176 W/m^{o}C , k_{oil} = 0.180 W/m^{o}C), μ is the absolute viscosity of liquid at 35^{o}C ($\mu_{ethanol}$ = 0.000882Pa.s and μ_{oil} = 4.375E-4 Pa.s), μ_w is the wall viscosity, Da is the diameter of impeller = 0.15m and Dt is the extractor diameter = 0.3m, N is the impeller speed = 1.40 rps (84rpm), ρ is the desnsity of liquid at 35^{o}C ($\rho_{ethanol}$ = 788.36 kg/m^3 and ρ_{oil} = 905 kg/m^3) and Cp is the specific heat capacity of liquid ($Cp_{ethanol}$ = 2440 J/kg^{o}C and Cp_{oil} = 2053 J/kg^{o}C).

Assuming $\mu_w = \mu$, film coefficient due to ethanol $h_{ethanonl}$ using (6) is calculated to be: 1743.17 W/ m$^{2\ 0}$C. Similarly, the film coefficient of oil, h_{oil} = 2245.10 W/m^{2o}C

Film coefficient of the mixture then becomes 2025.72 W/m^{2o}C

Film coefficient of air

For a vertical cylinder with a constant flux, the Nusselt number [8] is given as

$$h = 0.6\left(\frac{k}{D_t}\right)\left(Ra_D \times \frac{D_t}{H}\right)^{0.25} \quad (7)$$

for Ra_D D/L≥ 10^4

$$Ra_D = \frac{g\beta(T_w - T_a)D_t^{\,3}}{(v \times \alpha)} \quad (8)$$

where, β = 1/T_a , T_a is the ambient temperature = 20^{o}C , T_w is the wall temperature = 40^{o}C, Dt is the extractor diameter = 0.3m , H is the height of extractor = 0.39m, Ra_D is the dimensionless Rayleigh number, k is the thermal conductivity of air at average temperature of 30^{o}C = 0.02647 W/m^{o}C, β is the coefficient of thermal expansion = 1/20 = 0.05/^{o}C, v is the kinematic viscosity at 30^{o}C = 15.9848E-6 m^2/s, α is the thermal diffusivity at 30oC = 2.262E-5 m^2/s. From (8), Ra_D is calculated to be 7.325E+8 which then yields that h_{air} is 8.16 W/m^{2o}C. The overall heat transfer coefficient is given by: U = 1/(1/$h_{mixture}$ + x/k + 1/h_{air}) where x is the thickness of extractor material = 0.001m, k is the thermal conductivity of stainless steel at 30^{o}C = 16.15 W/m^{o}C. So U is then calculated to be 8.12 W/m^{2o}C

Since $U_{calculated}$ = $U_{assumed}$, the area of the extractor is correct.

3.1.2 Design of Evaporator

Residence time for evaporation = 14070.25s

The volume of ethanol to be evaporated is 1% less the total volume for extraction = 0.02144m^3 Total volume into evaporator = volume of cake + volume of ethanol + volume of extracted oil = 0.02261 m^3. Mass flow rate of ethanol out of the evaporator is less 1% = 1.2014E-3 – (1E-2 × 1.2014E-3) = 1.1894E-3 kg/s

For a batch process without chemical reaction, the mass balance is given as:

F1 + F2 + F3 = F4 + F5 + F6 (9)

From (9), F5 =2.5087E-5 kg/s

3.1.2.1 Energy Balance over the Evaporator

From Figure 4, for a batch process without chemical reaction, the energy balance is given as

$F1H_1 + F2H_2 + F3H_3 + HF = F4H_4 + F5H_5 + F6H_6$ (10)

where F1, F2 …………F6 are the flow rate materials as shown in Figure 4, H_1, H_2……H_6 are enthalpy of the corresponding material while H_F is the heat energy from the heating coil and hot plate. Substituting values into (10), we have:

$$89.05 + H_F = F4H_4 + F5H_5 + 0.6155$$

One hundred percent recovery of ethanol from the cake is not feasible and 1% of ethanol is assumed to be retained by the cake and heat content of the 1% ethanol is equally assumed to be 1% of the total heat content of the ethanol so: $F5H_5$ = 2.7473 J/s

$F4H_4$ = sensible heat + latent heat energy of ethanol = 1087.51J/s and H_F = 1001.82J/s.

3.1.2.2 Calculation of Area of Evaporator

The following assumptions will be used for the calculation of the area of the evaporator:

(a) A shell and tube arrangement, with air being in an arbitrary shell round the evaporator
(b) Heat is transfered by convection from the heating coil to the bulk of the liquid
(c) Thermal resistance of the evaporator material is negligible, therefore heat is transfered by convection from the hot plate to the bulk of the liquid.
(d) Nucleate boiling due to bubble formation is occurring because the excess temperature (78^{o}C – 50^{o}C = 28^{o}C) is within the range 9^{o}C – 50^{o}C.

Table 3: Data for Mass Balance over the Evaporator

S/No	Component	Mass (kg)	Flow rate (kg/s)	Volume (m^3)
1	Ethanol	16.904	1.2014E-3	2.1654E-2
2	Neem oil	0.15066	1.0708E-5	1.6648E-4
3	Cake	0.18414	1.3087E-5	7.8928E-4

The assumed film coefficient of the ethanol/oil mixture = 151.41 – 2346.56 W/m^{2o}C
Assumed film coefficient of air = 5 – 25 W/m^{2o}C. The assumed overall heat transfer coefficient U will be found to be 6.788 W/m^{2o}C. Assuming no heat lost, then heat generated will equal heat duty of the evaporator = 1001.82 J/s so that $A_{assumed} = Q/(U\times \Delta T) = 5.271\ m^2$
Area of evaporator, A is given by:
A = πDtH +πDt^2/4
Assuming, ratio of evaporator diameter to evaporator height, H/Dt = 30.23
Diameter of evaporator, Dt = 0.2346m and H = 7.09m. Total volume into the evaporator = 0.02261m^3. Volume of extractor, V = πDt^{2}H/4. Height of liquid in the evaporator, H = 0.523m

3.1.2.3 Calculation of the Total Height of Evaporator based on the Liquid Height, Support for Perforated Stainless Steel, Condenser and Discharge Point for Seed Cake.

Thickness of support for the perforated stainless steel = diameter of bolt = 0.003m, Length of support for the perforated stainless steel = length of bolt = 0.005m, Diameter of the perforated stainless steel = diameter of evaporator = 0.2346m, Thickness of perforated stainless steel = thickness of construction material = 0.001m
Let height of discharge outlet for solid be 20% above the height of cake receiver = 0.066 + 0.2 × 0.066 = 0.0792m, Circumference of evaporator = π×Dt = 3.142 × 0.2346 = 0.7370m, Arc length of the discharge outlet for solid cake = 1/5 of evaporator circumference = 0.7370/5 = 0.1474m, Diameter of stopper to be used is 0.05m and diameter of condenser inlet = 0.02m, Let the clearance distance between the top of the mixture in the evaporator and the base of the stopper be 3.48% above the height of liquid = 0.0348 × 0.523 = 0.0182m, Height of evaporator = heigth of miscella in evaporator + clearance above the liquid + stopper thickness + condenser diameter + stopper thickness + thickness of support for perforated steel + thickness of perforated stainless steel + heigth of cake discharge outlet = 0.523 + 0.0182 + 0.015 + 0.02 + 0.015 + 0.003 + 0.001 + 0.0792 = 0.6744m, Let the Neem oil discharge pipe diameter be 1/44.96 of the actual height of evaporator = 1/44.96 × 0.6744 = 0.015m, Thickness of flange material = 1.5mm (0.0015m). Width of flange attached to the evaporator = 1/7 of evaporator diameter = 1/7 × 0.2346 = 0.0335m. Since 0.6744m < 7.09m, the evaporator can be economically constructed using 0.6744m as height. The assumed overall heat transfer coefficient (6.79 W/m^{2o}C) is validated if the calculated overall heat transfer coefficient based on calculated film coefficient is equal to the assumed overall heat transfer coefficient value.

3.1.2.3 Calculation of Film Coefficient

For nucleate boiling, the heat transfer coefficient is calculated using Labantsov correlation [9]

$$h = 0.075\left(1 + 10\left(\frac{\rho_v}{\rho l - \rho v}\right)^{0.67}\right)\left(\frac{k_l^2}{\nu\alpha(\mathrm{Ts} + 273.15)}\right)^{0.33} q^{0.67} \quad (11)$$

where ρ_l is density of ethanol at 50^{o}C = 0.7753 kg/m^3, ρ_v is the density of ethanol vapour at 78^{o}C = 0.7510 kg/m^3, k_l is the thermal conductivity of ethanol at 50^{o}C = 0.000594 W/m^{o}C, ν is the kinematic viscosity of ethanol at 50^{o}C = 4.62E-6 m^2/s, T_s = 50^{o}C and q = 1001.82J/s. From these data, $h_{ethanol}$ is calculated using (9) to become 180.14 W/m^{2o}C
Assuming the oil did not evaporate, $\rho_{l,oil} = \rho_{v,oil}$ then h_{oil} can also be evaluated with (11) to become 59.93W/m^{2o}C.
Heat transfer coefficient of the ethanol/oil mixture is found to be 150.13 W/m^{2o}C using equation (XXX).

3.1.2.4 Film Coefficient of Air

Taking T_a = 20^{o}C and T_w = 40^{o}C and using (8), Ra_D is found to be 3.503E+8. Using (7), h_{air} is found to be 7.11 W/m^{2o}C. Thermal conductivity of stainless steel at 59^{o}C = 16.36 W/m^{o}C, $h_{mixture}$ = 150.13W/m^{2o}C , h_{air} = 7.11 W/m^{2o}C, so U can be evaluated to be 679W/m^{2o}C.
Since $U_{calculated} = U_{assumed}$, the area of the evaporator is correct.
Every component of the pilot solvent extraction plant was coupled together as shown in Plate 1 for the extraction of Neem oil from Neem seed.

Table 4: Summary of Extractor Vessel Specification

Parameter	Value
Capacity	9.65kg/day of Neem Seed Kernel
Area	0.7521m^2
Diameter	0.300m
Thickness	0.001m
Actual height	0.390m
Outlet discharge diameter	0.015m
Outlet length	0.04m
Diameter of flexible reinforce rubber tube	0.02m
Length of flexible reinforce rubber tube	0.60m

3.2 Solvent Extraction

The extraction of oil was done using food grade ethanol as solvent in a pilot solvent extraction plant. The pilot plant is mainly made up of extractor, evaporator and condensate receiver and flat blade turbine impeller was used for agitation in the extractor. The pilot plant was adequately checked and appropriate one way, stainless steel and ½ inch ball valves; V_1,V_2 and V_3 were closed. The electrical fittings were equally checked and ascertained to be in good conditions. The chiller was switched on and set to 0°C and allowed to work for 30 minutes to attain stability and cool the condenser; this was done to aid easy condensation of the food grade ethanol vapour to liquid.

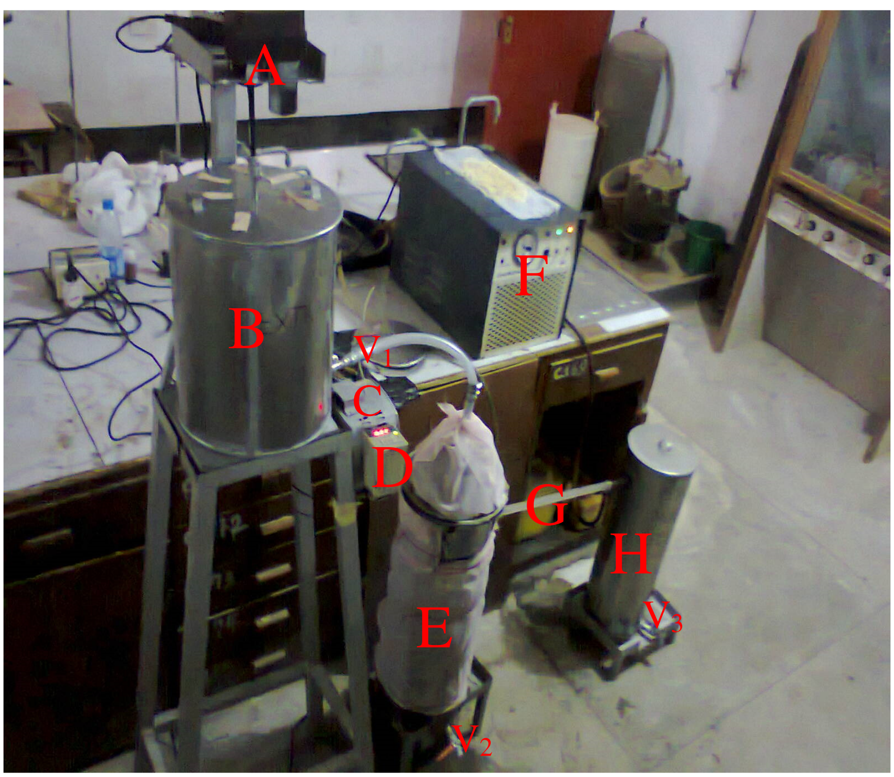
Now, 21.23 litres of food grade ethanol and 0.3348kg (W_1) of ground Neem seed kernel of particle sized 0.425 – 0.710mm were charged into the extractor. The main switch and 50°C switch were put on. The electric heater for the extractor was switched-on and the temperature controller set to 50°C for a period of time to stabilize the system at 50°C. The stability was noticed by the aid of a temperature sensor placed in the extractor and a click short sharp sound that was heard and the temperature controller light changed from green to red which indicates that the system is stabilized at 50°C.

Once the stability was attained, the electric motor was switched-on and regulated at 37 rpm with the aid of a speed control unit using flat blade turbine impeller which was already mounted on the shaft; mixing and agitation commenced immediately for a period of 20 minutes and 40 minutes. After extraction, the electric heater and electric motor were switched-off and the control valve, V_1 was fully opened. The mixture flow through the reinforce rubber tube and inverted funnel for filtration to take place with the aid of a stainless steel filter mesh of size 0.00001m (0.01mm) attached to the cake receiver. The impeller shaft was disconnected from the electric motor and top cover of the extractor was opened and 0.424 litre of ethanol was introduced for washing to take place through percolation.

After washing, the cake receiver was collected via the cake discharge outlet and placed in an oven. The weight of the cake was taken after every one hour until constant weight was achieved (W_2). The control valves V_1, V_2 and V_3 were shut and the temperature sensor was transferred to the evaporator.

Table 5: Summary of Evaporator and its Accessories Specification

Parameter	Value
Capacity	2.629E-2 kg/hr of Neem oil
Area	5.721m^2
Diameter	0.235m
Height of liquid	0.523
Height of evaporator	0.6744m
Discharge outlet diameter	0.015m
Height of discharge outlet for solid cake	0.0792m
Arc length of discharge outlet for solid cake	0.1474m
Diameter of stopper	0.05m
Diameter of condenser inlet	0.02m
Width of flange	0.0335m



KEY

A = ELECTRIC MOTOR

B = EXTRACTOR

C = SPEED CONTROLLER

D = TEMPERATURE CONTROLLER

E = EVAPORATOR

F = CHILLER

G = CONDENSER

H = CONDENSATE RECEIVER

V_1 = CONTROL VALVE FOR MIXTURE

V_2 = CONTROL VALVE FOR COLLECTING NEEM OIL

V_3 = CONTROL VALVE FOR COLLECTING CONDENSED ETHANOL

Plate 1: Pilot Solvent Extraction Plant for Extracting Neem Oil from Neem Seed

The 78°C switch was switched-on and the temperature controller set to 78°C. The heating was maintained at 78°C so that evaporation of the food grade ethanol can take place. The vapour ethanol passed through the condenser and was collected in the ethanol condensate receiver as liquid ethanol. The above procedure was repeated at 84 rpm for extraction time of 20 minutes and 40minutes.

4. Results and Discussion

4.1 Results

At 37rpm for 20 and 40 minutes contact time, the yield were 24.28 and 28.62% respectively as seen from Table 6; while at 84rpm for 20 and 40 minutes the yield were 29.32 and 36.86% respectively as seen from Table 7. The percentage yield of oil increases with increase of contact time for both mixing intensity of 37 and 84 rpm within the experimental region. Increase in time allows more leaching to occur leading to higher percentage yield. Agitation of the mixing medium increases turbulence and causes the particles to come in contact with fresh solvent within the mixing vessel and led to higher extraction rate and better yield. This was clearly seen when the mixing intensity changes from 37rpm to 84rpm for both contact time.

4.1.1 Extraction Rate

The reported extraction rate of Neem oil from an agitated round bottom flask with stirrer at 50°C extraction temperature, 3 hours contact time, ethanol as solvent and 0.425 – 0.710mm particle size was 19.9g/3hours (6.633g/hour) with 33.2% yield (Workneh, 2011) as seen in Table 8, while the maximum extraction rate from the designed and constructed pilot solvent extraction plant was 116.20g/40 minutes (174.3g/hr) at 50°C extraction temperature, food grade ethanol as solvent and 0.425 – 0.710mm particle size as seen from Table 9. Based on this, the extraction rate of Neem oil using solvent had improved by 26 times (6.633 : 174.3) per hour when using the pilot solvent extraction plant and compared to the earlier laboratory scale experiment.

4.1.2 GCMS Result

The GCMS analysis identified the presence of seven components in the oil as shown by the peaks on the chromatogram (Figure 5). The spectrum of the identified individual component was compared with the spectrum of a known component as shown in Figures 6 and 7 for the first two components. From Figure 6, the identified spectrum was line #1 and Hit #1 spectrum has 87% fitness when compared to line #1. The identified component was $C_8H_{16}O$ (Octanal). Figure 7 shows spectrum line#2 and Hit #1 spectrum has 86% fitness when compared to spectrum line #2. The compound identified was $C_6H_{10}O_2$ (Lactone). Similarly, the spectrum of all the components were studied and the identified components and their uses are shown in Table 11.

The characterized properties of the extracted Neem Oil as seen in Table 10 fall within the literature values, except the acid values. This difference may be due to locations where the Neem seeds were obtained and time of harvesting.

Table 6: Percentage Yield of Oil at 37 rpm for 20 minutes and 40 minutes Contact Time.

S/NO	Times (minutes)	Initial Mass of Neem Seed Particle (g)	Final Mass of Neem Seed Particle (g)	Percentage Yield (%)
1	20	334.8	253.51	24.28
2	40	334.8	238.98	28.62

Table 7: Percentage Yield of Oil at 84 rpm for 20 minutes and 40 minutes Contact Time.

S/NO	Times (minutes)	Initial Mass of Neem Seed Particle (g)	Final Mass of Neem Seed Particle (g)	Percentage Yield (%)
1	20	334.8	236.64	29.32
2	40	334.8	211.40	36.86

Table 8: Extraction Rate of Neem Oil using Ethanol as Solvent

Author	Initial mass of Neem seed particle (g)	Final mass of Neem seed particle (g)	Mass of oil extracted (g)	Time of extraction (hr)	Production rate (g/hr)
Workneh (2011)	60	40.1	19.9	3	6.633

Table 9: Extraction Rate of Neem Oil using Ethanol as Solvent

Initial mass of Neem seed particle (g)	Final mass of Neem seed particle (g)	Mass of oil extracted (g)	Time of extraction (hr)	Production rate (g/hr)
334.8	211.40	116.20	0.667	174.3

Table 10: Characterized Properties of the Extracted Neem Oil

S/No	Properties of Extracted Neem Oil	Literature Values
Specific gravity	0.9111	0.9080 - 0.9340
Refractive index	1.4668	1.4615-1.4705
pH	6.2	5.7 – 6.5
Iodine value	70.21g/g	65 – 80 g/g
Acid value	34.33 mgKOH/g	40 mg KOH/g
Saponification value	180.95 mg KOH/g	175 – 205 mg KOH/g

Table 11: Composition of Extracted Neem Oil

S/No	Common Name	Composition of Extracted oil (%)	Literature Value (%)	Use(s) of Identified Neem Oil Components
1	Octanal	3.90	-	Skin Regeneration and Wound Healing
2	Lactone	0.97	-	Manufacturing of Plastic (% is not significant)
3	Palmitic acid	25.36	16 – 33	Manufacturing of soap
4	Elaidic acid	1.23	-	Pharmaceutical Solvent
5	Methyl stearate	0.48	-	Skin Conditioner
6	Oleic acid	40.41	25 – 54	Production of Detergent, Soap and Cosmetics
7	Stearic acid	27.65	9 – 24	Manufacturing of cosmetic and pharmaceuticals

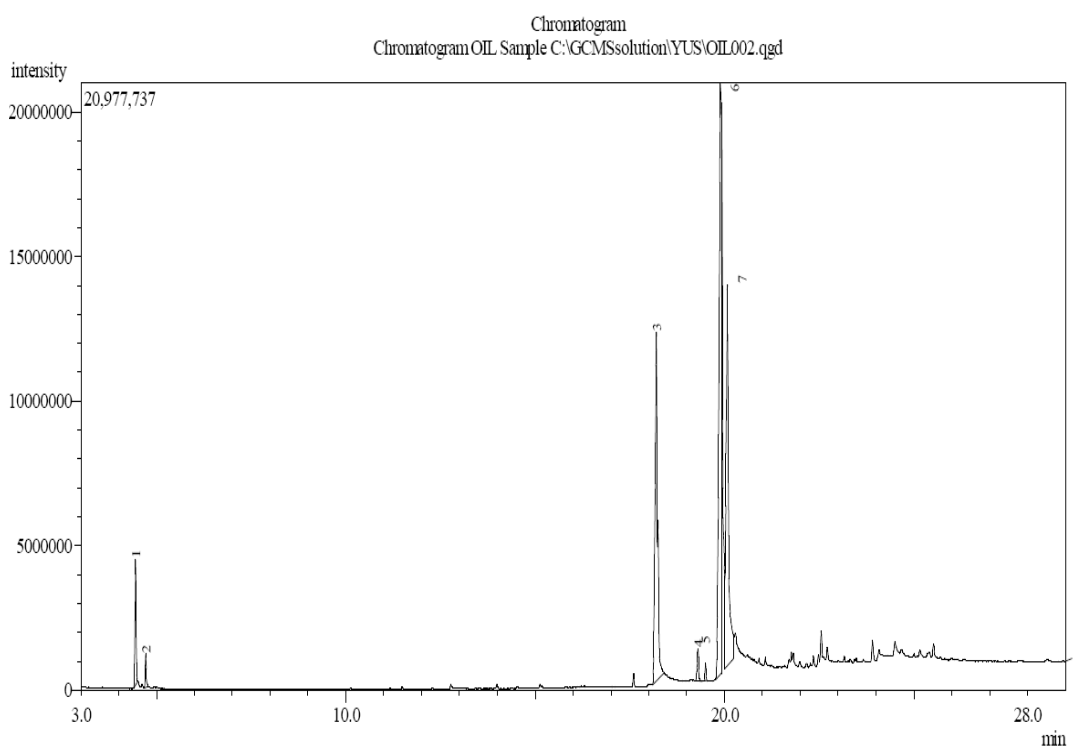


Figure 5: Chromatogram of the Extracted Oil

5. Conclusion and recommendation

5.1 Conclusion

Percentage yield of Neem oil increases with increase in contact time and mixing intensity within the experimental limit. The maximum yield of 36.86% was obtained when the pilot solvent extraction plant was operated at 84 rpm for a contact time of 40 minutes. The extraction rate of Neem oil using solvent had improved by 26 times (6.633 : 174.3) per hour when compared to the earlier laboratory scale experiment, which employed agitated vessel. The Neem oil can be used in soap, cosmetic and pharmaceutical industries. The physical and chemical properties of the extracted Neem oil were: specific gravity, 0.9111; pH, 6.5; refractive index, 1.4668; iodine value, 70.21g/g; acid value, 34.33mgKOH/g and saponification value, 180.95 mgKOH/g.

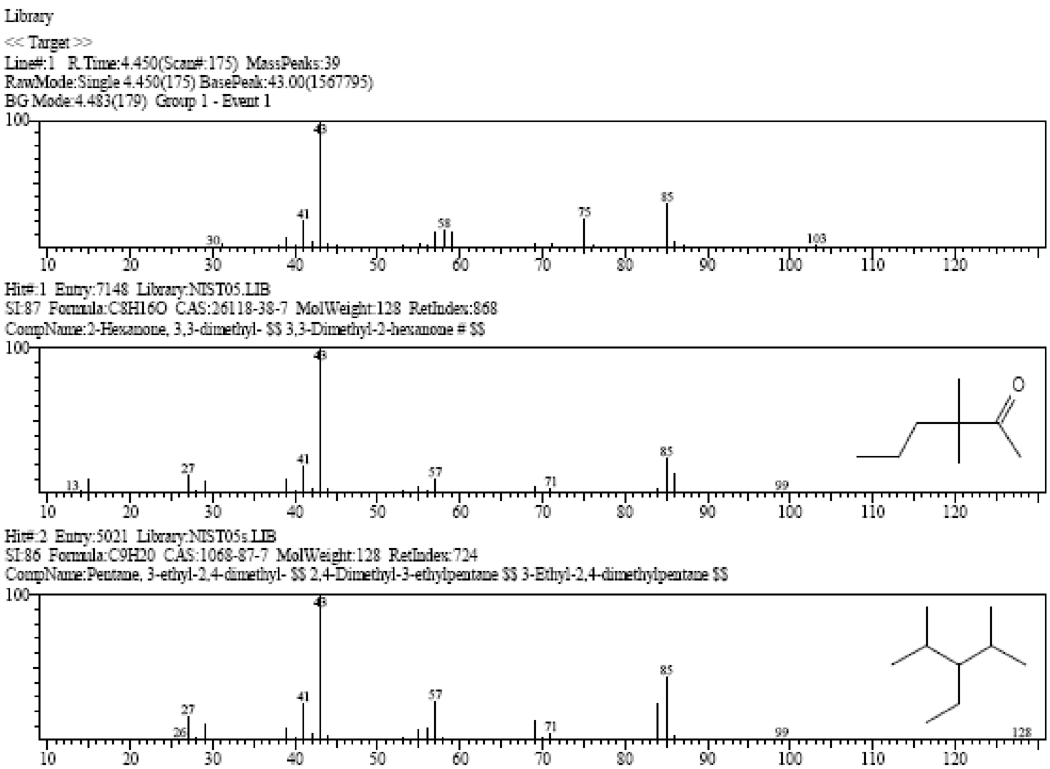


Figure 6: The Comparison for Chromatogram line#1

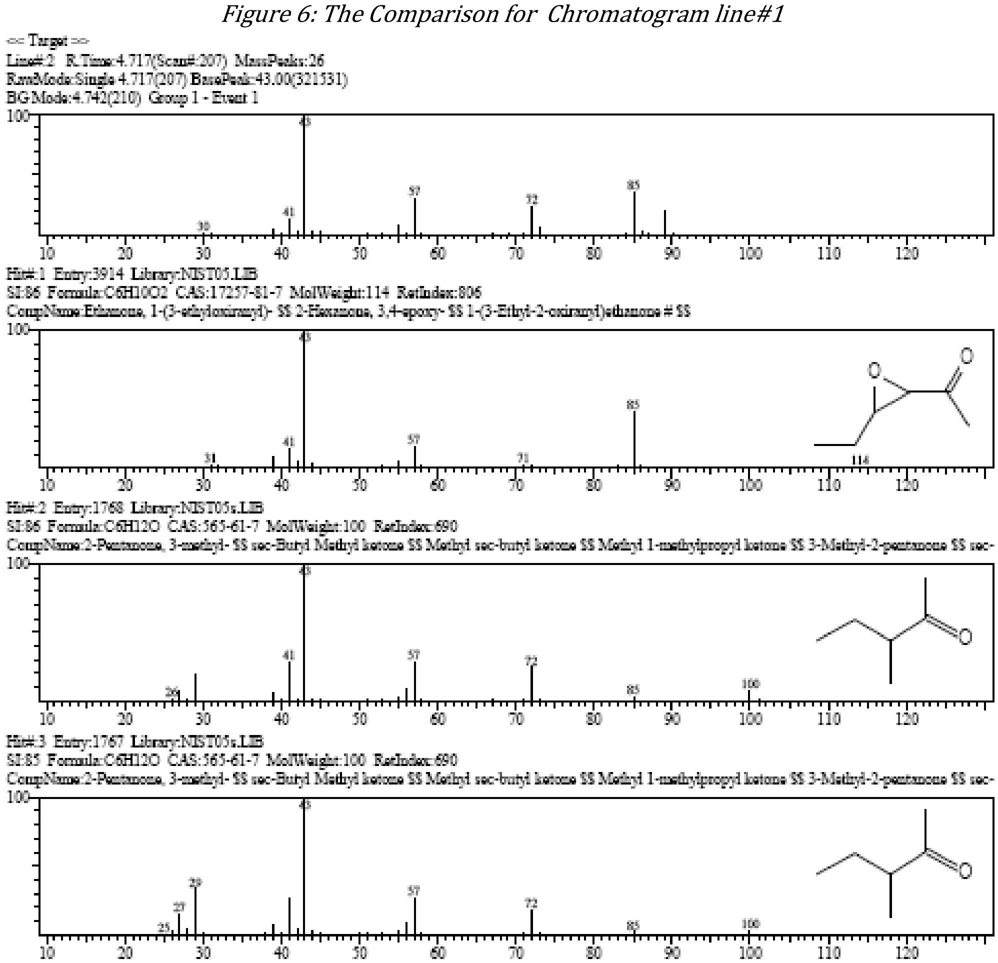


Figure 7: The Comparison for Chromatogram line#2

5.2 Recommendation

(a) The effect of particle size, temperature and solvent – solute ratio should be investigated using the same pilot solvent extraction plant.

(b) The thermodynamic and kinetic studies of the leaching process should be studied using the same pilot solvent extraction plant.

References

1. Workneh, Wondesen. "*Extraction and Characterization of Essential oil from Morgosa seed"* www.libsearch.com/search/soxhlet%25. Accessed on January 2 , 2012.
2. Liauw,M.Y. Natan, F.A. Widiyanti, P. Ikasari,D. Indraswati, N. and Soetaredjo F.E.. (2008). *"Extraction of Neem oil (Azadirachta indica A. Juss) using N-Hexane and Ethanol: Studies of oil Quality, Kinetic and Thermodynamic"*, Asia Research publishing Network *Journal of Engineering and Applied Sciences*, Vol.3,No.3, 2008, Pages 49-50.
3. Anya,U. A. Chioma, N. N. and Obinna, O. (2012). Optimized Reduction Of Free Fatty Acid Content On Neem Seed Oil, For Biodiesel Production. *Journal of Basic and Applied Chemistry*, vol.2, Number 4, 2012, pages 21-28.
4. *Neem uses*. Retrieved on January 7, 2012 from http://www. Neem uses.com/neem_oil.php.
5. Kovo, A. S "Application of full 4^2 Factorial Design for the Development and Characterization of Insecticidal Soap from Neem Oil "http://www. Lejpt. academicdirect.org/A08/29_40.htm. Accessed on January, 2012.
6. Himmelblau, D.M. *Basic Principles and Calculations in Chemical Engineering,* 6th Edition, Prentice-Hall of India, Private Limited New Delhi, 2005.
7. Backhurst, J.R. & Harker, J.H. *Process Plant Design*. Heinemann Chemical Engineering Series, London, 1983.
8. Janna, W.S. *Engineering Heat Transfer*. 1st Edition, PWS Publishers, United States of America, 1986.
9. S. A Alavi Fazel and S. Roumana. "Pool Boiling Heat Transfer to Pure Liquid" http://www. wseas.us/e-library/conferences/2010/Cambridge/CFH/CFH-31. Accessed on March 14, 2012.