

Investigation into the Usage of Solvent for Extracting Neem Oil from Neem Seed for Industrial Application

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Doi:10.5901/ajis.2014.v3n5p39

Abstract

Neem oil was extracted using food grade ethanol in an agitated pilot scale solvent extractor. The maximum percentage yield was 36.86% and was obtained when flat blade turbine impeller was operated at 84 rpm for 40 minutes contact time at 50°C extraction temperature and particle size of 0.425 – 0.710mm. The Gas Chromatography and Mass Spectrometer result shows the composition of the extracted neem oil to be 40.41% oleic acid, 27.65% stearic acid, 25.36% palmitic acid, 3.90% octanal, 1.23% elaidic acid, 0.97% lactone and 0.48% methyl stearate,. This percent composition compares favourably with standard values. The composition of the neem oil suggests its application for industrial usage. The properties of the neem oil extracted 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. These values compare favourably with standard values.

Keywords: *Neem oil, Extraction, Mixing, Agitation and GCMS.*

1. Introduction

Neem tree, which is also known as *Azadirachta indica*, is one of the best known trees in India, which is known for its medicinal properties (Kovo, 2008).

There are several methods for obtaining neem oil from the seeds. These include: mechanical pressing, supercritical fluid extraction, and solvent extraction. Mechanical extraction is the most widely used method to extract oil from neem seed. However, the oil produced with this method usually has a low price, because it is turbid. Extraction using supercritical fluid, produces oil with very high purity; however the operating and investment cost is high. Extraction using solvent has several advantages. It gives higher yield and it is less turbid than oil obtained from mechanical extraction, it also has low operating cost compared with oil from supercritical fluid extraction (Liauw, 2008).

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 contain about 25 - 45% oil and provide the major source of neem chemicals (Anyia, 2012). The average composition of Neem oil is shown in Table 1.

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%
Palmitic 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: (Anya, 2012). ND* = Not Determined.

The standard properties of Neem oil are shown in Table 2.

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: Neemuses.com, Workneh, 2011, Kovo, 2008

Agitation refers to the induced motion of a material in a specified way, usually in a circulatory pattern inside some sort of container. Mixing is the random distribution, into and through one another, of two or more initially separated phases. Mixing is applied to processes to reduce the degree of non-uniformity, or gradient of a property in a system such as concentration, viscosity, temperature and so on. Mixing is achieved by moving material from one region to another to enhance mass and heat transfers (McCabe, 1993).

The GCMS instrument is made up of two parts. The gas chromatography (GC) portion separates the chemical mixture into pulses of pure chemicals and the mass spectrometer (MS) identifies and quantifies the chemicals. The GC separates the chemical based on their volatility, or ease with which they evaporate into a gas. The MS is used to identify chemical based on their structure (GCMS, n.d).

2. Methodology

2.1 Design of Experiment (DOE)

A 2² factorial design was adopted with two-variables two-level DOE using Minitab 14 computer software. The run-by-run experimental design is shown in Table 3. The runs were replicated twice giving a total of 8 runs (4 x 2) to minimize error.

Table 3: DOE for the Extraction of Oil from Neem Seed Kernel Using Flat Blade Turbine Impeller.

Run Order	Impeller Speed (rpm)	Contact Time (min)
1	84	20
2	37	20
3	37	20
4	37	40
5	37	40
6	84	40
7	84	40
8	84	20

2.2 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. Impeller was used for agitation in the extractor.

The pilot plant was adequately checked and appropriate 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 allow 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. 21.23 litres of food grade ethanol and 0.3348kg (334.8g) of ground Neem seed kernel of particle sized 0.425 – 0.71mm 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 XMTD electronic temperature controller manufactured by XY Instrument Ltd, China was 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 manufactured by Brook Crompton Doncaster, England was switched-on and regulated at 84 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. The above procedure was repeated based on the guide obtained from Minitab 14 computer software design of experiment (DOE). The DOE is shown in Table 3, while the impeller and the pilot solvent extraction plant are shown in Plates 1-2 respectively.

Plate 1: Flat Blade Turbine Impeller



Plate 2: Pilot Solvent Extraction Plant for Extracting Neem Oil from Neem See



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

After extraction, the electric heater and electric motor were switched-off and the control valve, V₁ was fully opened. The mixture flow through the reinforce rubber tube and through the 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 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 is achieved.

The control valves V₁, V₂ and V₃ were shut and the temperature sensor was transferred to the evaporator. 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 already cooled condenser and was collected in the ethanol condensate receiver as liquid ethanol. After 4hr 25mins of evaporation, a sample of oil was collected via V₂ and analyzed. The collected Neem oil was dried in an oven for 10 minutes to dried-off any residual food grade ethanol. The main switch was switched-off and V₃ opened to collect the recovered solvent for recycling.

3. Result and Discussion

The optimum percentage yield of oil from the neem seed was 36.86% obtained when operating Flat Blade Turbine Impeller at 84 rpm for 40 minutes contact time. The results show that increase in mixing intensity and contact time increases the yield of oil from the neem seed particles. This is because, the higher the agitation of the medium, the faster the rate of oil transfer from the neem seed to the solvent medium and the longer the contact time, the higher the quantity of oil extracted.

The results obtained from the experiment is shown in Table 4.

Table 4: Percentage Yield of Oil from Mixer - Extractor for using Flat Blade Turbine Impeller

Run order	Impeller speed (rpm)	Contact time (min)	Cake weight (g)	YIELD (%)
1	84	20	236.64	29.32
2	37	20	253.51	24.28
3	37	20	249.04	25.62
4	37	40	242.70	27.51
5	37	40	238.98	28.62
6	84	40	211.40	36.86
7	84	40	215.68	35.58
8	84	20	234.66	29.91

The GCMS analysis identified the presence of seven components in the oil as shown by the peaks on the chromatogram (Figure 1). The spectrum of the identified individual component was compared with the spectrum of a known component as shown in Figures 2 and 3 for the first two components. From Figure 2, 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 3 shows spectrum line#2 and Hit #1 spectrum has 86% fitness when compared to spectrum line #2. The compound identified was $C_9H_{10}O_2$ (Lactone). Similarly, the spectrum of all the components were studied and the identified components and their uses are shown in Table 5.

Figure 1: Chromatogram of the Extracted Oil

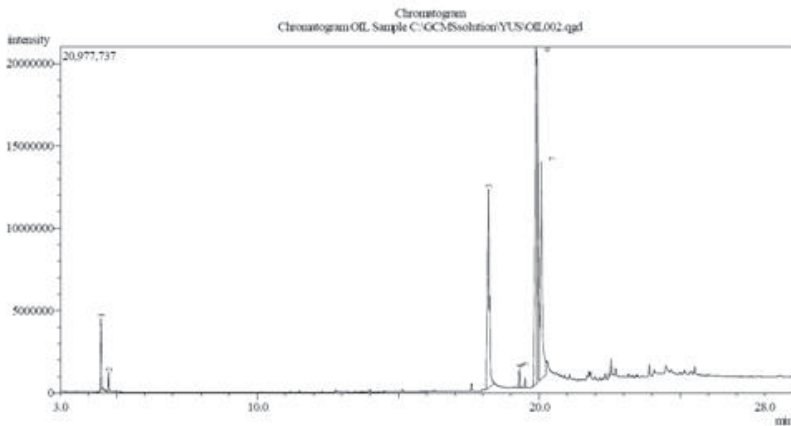


Figure 2: The Comparison for Chromatogram line#1

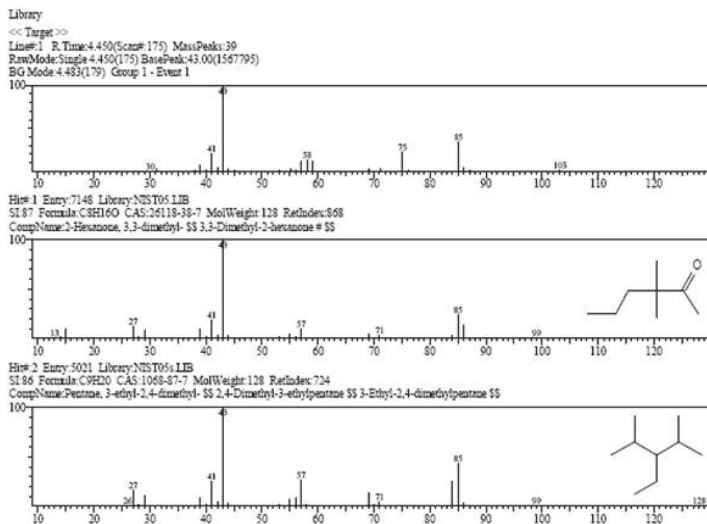


Figure 3: The Comparison for Chromatogram line#2

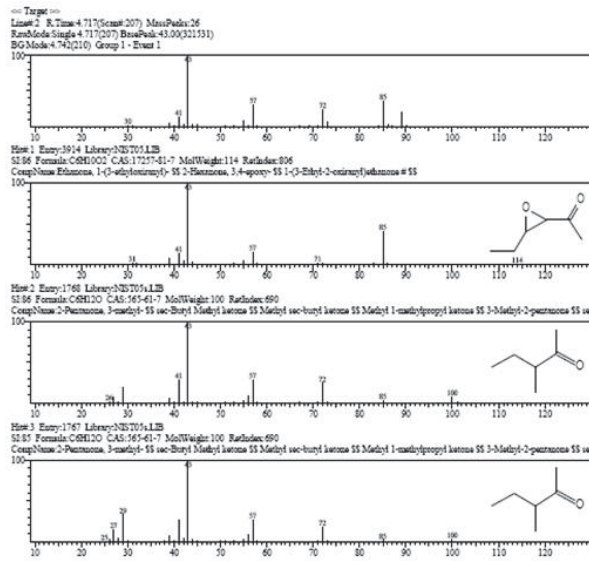


Table 5: Composition of Extracted Neem Oil

S/No	Common Name	Composition of Extracted oil (%)	Literature Use(s) of Value (%) 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

4. Conclusion

Neem oil was extracted using food grade ethanol as solvent in an agitated pilot solvent extraction plant using the DOE as guide and the highest percentage yield was 36.89% within the experimental limit when flat blade turbine impeller was operated at 84 rpm for a contact time of 40 minutes.

The GCMS analysis of the extracted neem seed oil composition shows that it contains: octanal (3.90%), lactone (0.97%), palmitic acid (25.36%), elaidic acid (1.23%), methyl stearate (0.48%), oleic acid (40.41%) and stearic acid (27.65%). Based on this composition, the neem oil can be used for industrial applications. The characterized 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.

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