Pilot Scale Extraction of NEEM Oil Using Ethanol as Solvent

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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^{o}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 find applications in domestic and 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.

1.0 INTRODUCTION

Neem oil is not used for cooking purposes. In India, it is used for preparing cosmetics(soap, hair products, body hygiene creams, hand creams and Ayurvedic. Traditional Ayurvedic uses of neem include the treatment of acne, fever, leprosy, malaria, ophtalmia ans tuberculosis. It has been used in traditional medicine for the treatment of tetanus, urticaria, eczema, sarofula and

erysipelas. Puri [1] has given an account of traditional uses and therapeutic indications and pharmacological studies of this oil in his book on neem. 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. Extraction of oil has been of great interest worldwide and this has been as a result of the constant increase in the world population. The Neem oil produced cannot cater for all need of the population which includes domestics and industrial uses [2].

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 and contains a significant amount of water and metals. 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 [7].

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 [3]. 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%
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.

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^{o}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:[2,4 and 5]

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 [6].

In this study, food grade ethanol was used for the extraction of oil from the neem seed using agitated pilot solvent extraction plant. The effect of turbine impeller speed (mixing intensity) and contact time on percentage yield of oil from the neem seed was investigated for 2 different impeller types. Minitab 14 software was used to get the Design of Experiment (DOE) and GCMS analysis was carried out to find the suitability of the extracted oil for domestic and industrial usage.

METHODOLOGY

Design of Experiment (DOE)

A 2^2 factorial design was adopted with two-variables two-level DOE using Minitab 14 computer soft ware. The run-by-run experimental design were shown in Tables 3 and 4 for impellers A1

and A2 respectively. The runs were replicated twice giving a total of 8 runs (4 x 2) to minimize error for each impeller type. The two factors and their levels considered are:

(b) Turbine impeller speed : 37 and 84 rpm

(c) Contact time : 20 and 40 minutes.

Table 3: DOE for the Extraction of Oil from Neem Seed Kernel for Impeller A1(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

Table 4: DOE for the Extraction of Oil from Neem Seed Kernel for Impeller A2 (Rushton Turbine Impeller) .

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

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^oC 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^oC 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^oC for a period of time to stabilize the system at 50^oC. 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^oC. 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 (A1) 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 are shown in Tables 3 and 4 for impellers A1 and A2 respectively, while impellers A1 and A2 Plates 1and 2 respectively.

Plate 1: Impeller A1

Plate 2: Impeller A2

Plate 3: Pilot Solvent Extraction Plant for Extracting Neem Oil from Neem Seed

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 into 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 V_1, V_2 and V_3 were shut and the temperature sensor was transferred to the evaporator. The 78^{o}C switch was switched-on and the temperature controller set to 78^{o}C. The heating was maintained at 78^{o}C so that evaporation of the food grade ethanol can take place.

The vapour ethanol is passed through the condenser and was collected in the ethanol condensate receiver as liquid ethanol. After 4hr 25mins of evaporation, a sample of oil was collected via V_2 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 V_3 opened to collect the recovered solvent for recycling.

RESULT AND DISCUSSION

The percentage yield of oil from the Neem seed obtained was 36.86% d when operating impeller A1 (Flat Blade Turbine Impeller) at 84 rpm for 40 minutes contact time; while for impeller A2 (Rushton Turbine Impeller) under similar operating conditions have the best percentage yield of 31.25%. The difference in percentage yield can be associated with the presence of a disc on Rushton turbine impeller which hindered the upward flow of the mixture there by reducing the rate of leaching of the oil from the neem seed around that region. The results show that increase in mixing intensity and contact time increases the yield for individual type of the impellers. 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 were shown in Table 5 and 6.

Table 5: Percentage Yield of Oil from Mixer - Extractor for Impeller Types A1 using Food Grade Ethanol as Solvent.

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

Table 6: Percentage Yield of Oil from Mixer - Extractor for Impeller Types A2 using Food Grade Ethanol as Solvent.

Run order	Impeller speed (min)	Contact time (min)	Cake weight (g)	YIELD (%)
1	84	20	255.59	23.66
2	84	40	232.60	30.53
3	37	40	254.62	23.95
4	84	20	251.07	25.00
5	37	20	266.68	20.35
6	37	40	256.68	23.33
7	37	20	264.65	20.95
8	84	40	230.19	31.25

The Neem oil obtained from the optimum operating conditions was used for the GCMS analysis. The GCMS analysis identified the presence of seven components in the oil as shown by the peaks on the chromatogram (Figure 1) and the spectra in figures 2-8. The components' peaks identified as 1-7 from left to right of the chromatograph correspond to the respective names and the percentage composition of the components from serial number 1-7 as shown in Table 7.

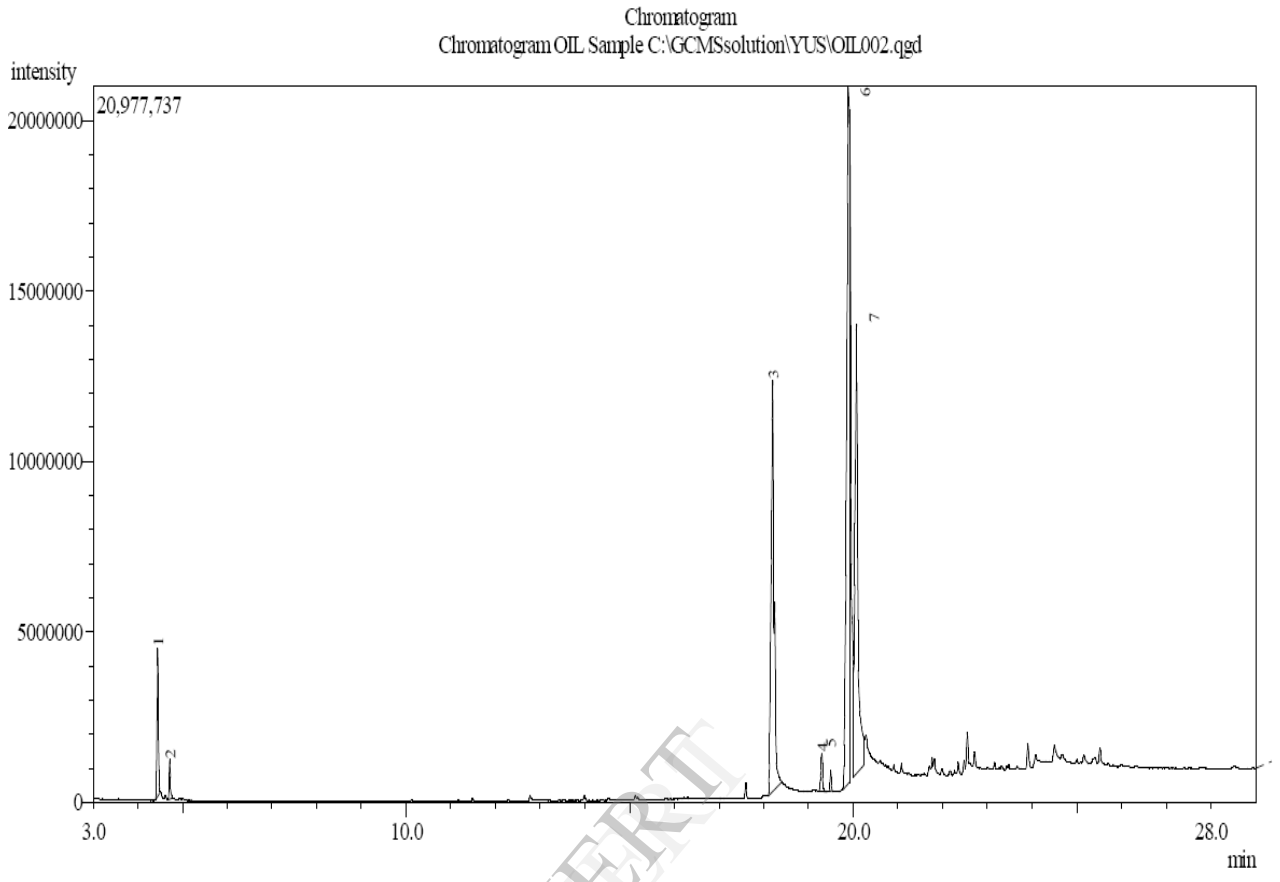

Figure 1: Chromatogram of the Extracted Oil

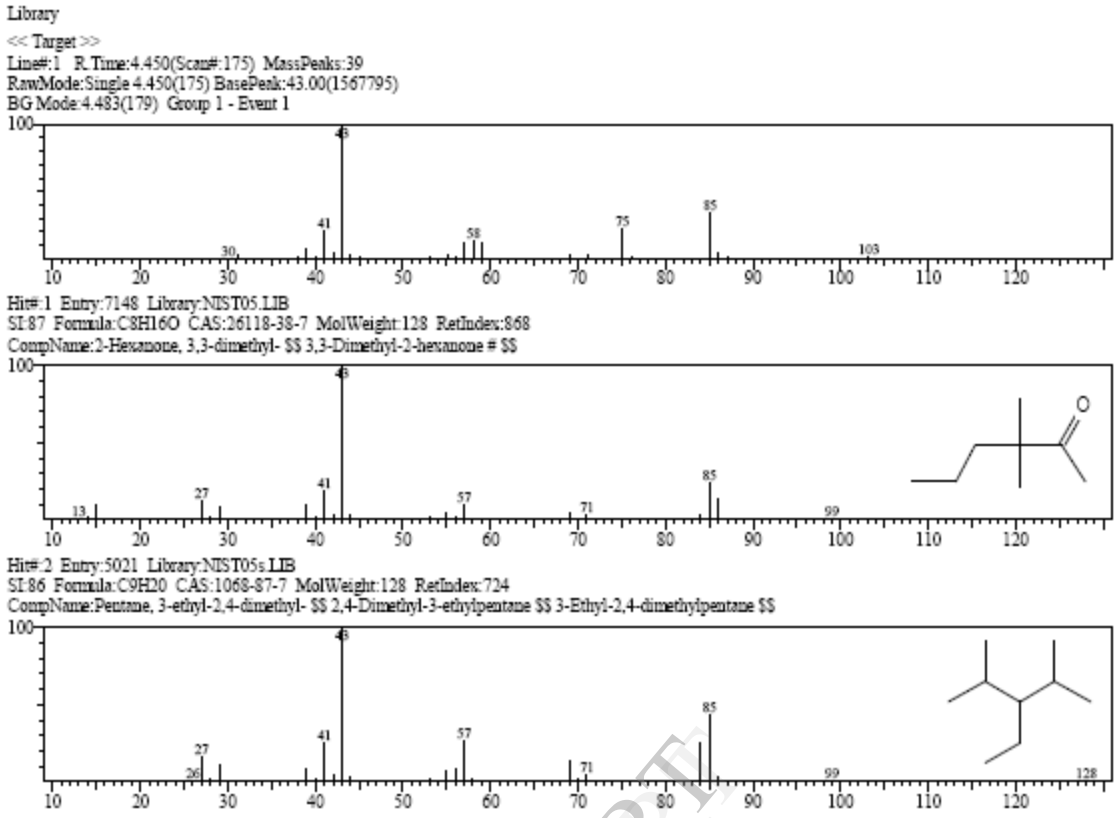

Figure 2: The Comparison for Chromatogram Line#1

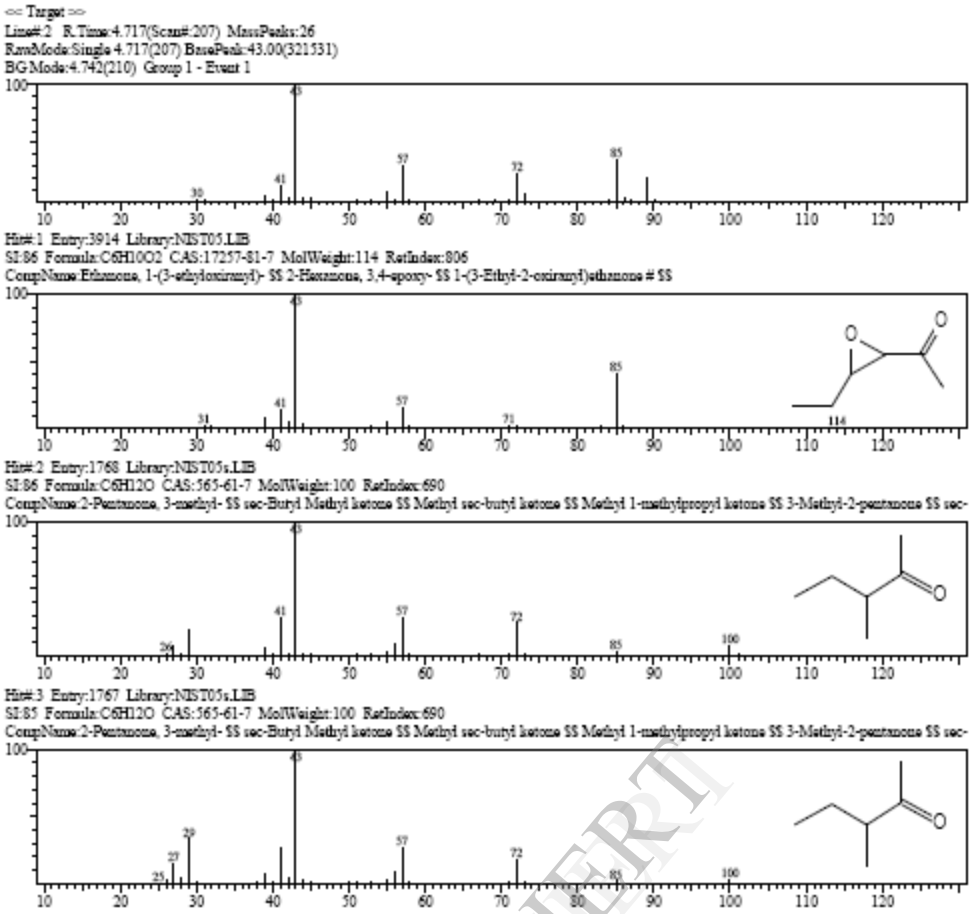

Figure 3: The Comparison for Chromatogram Line#2

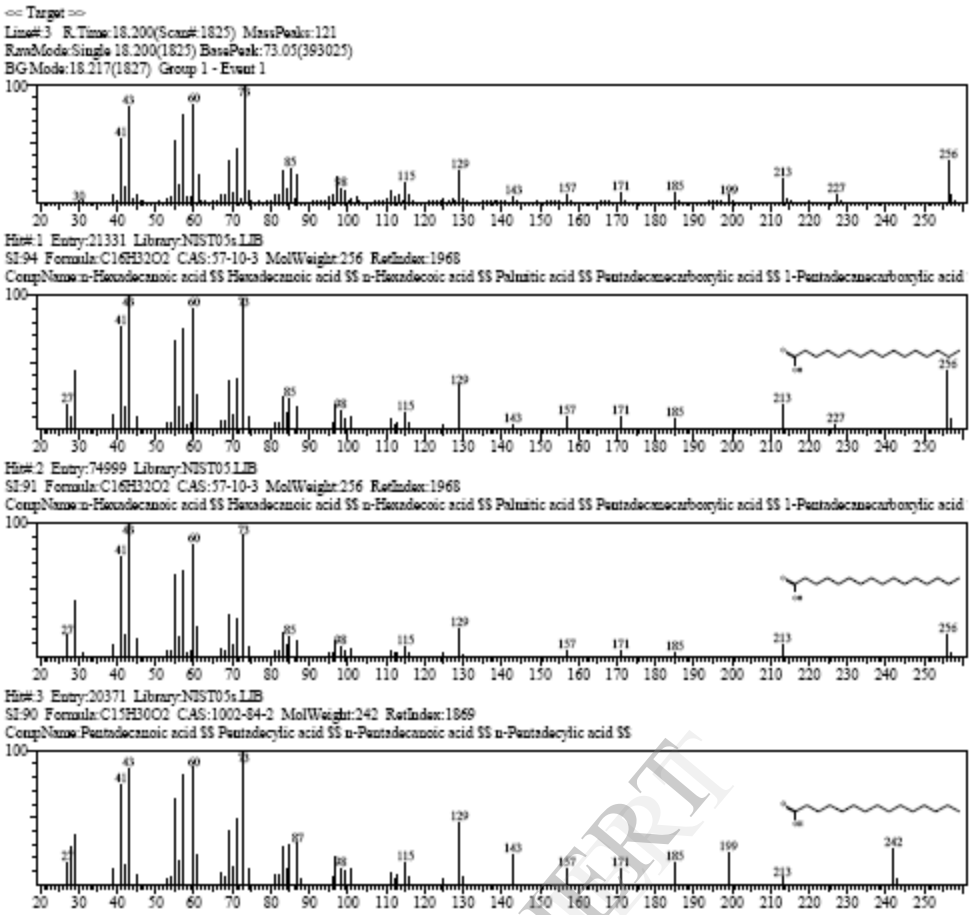

Figure 4: The Comparison for Chromatogram Line#3

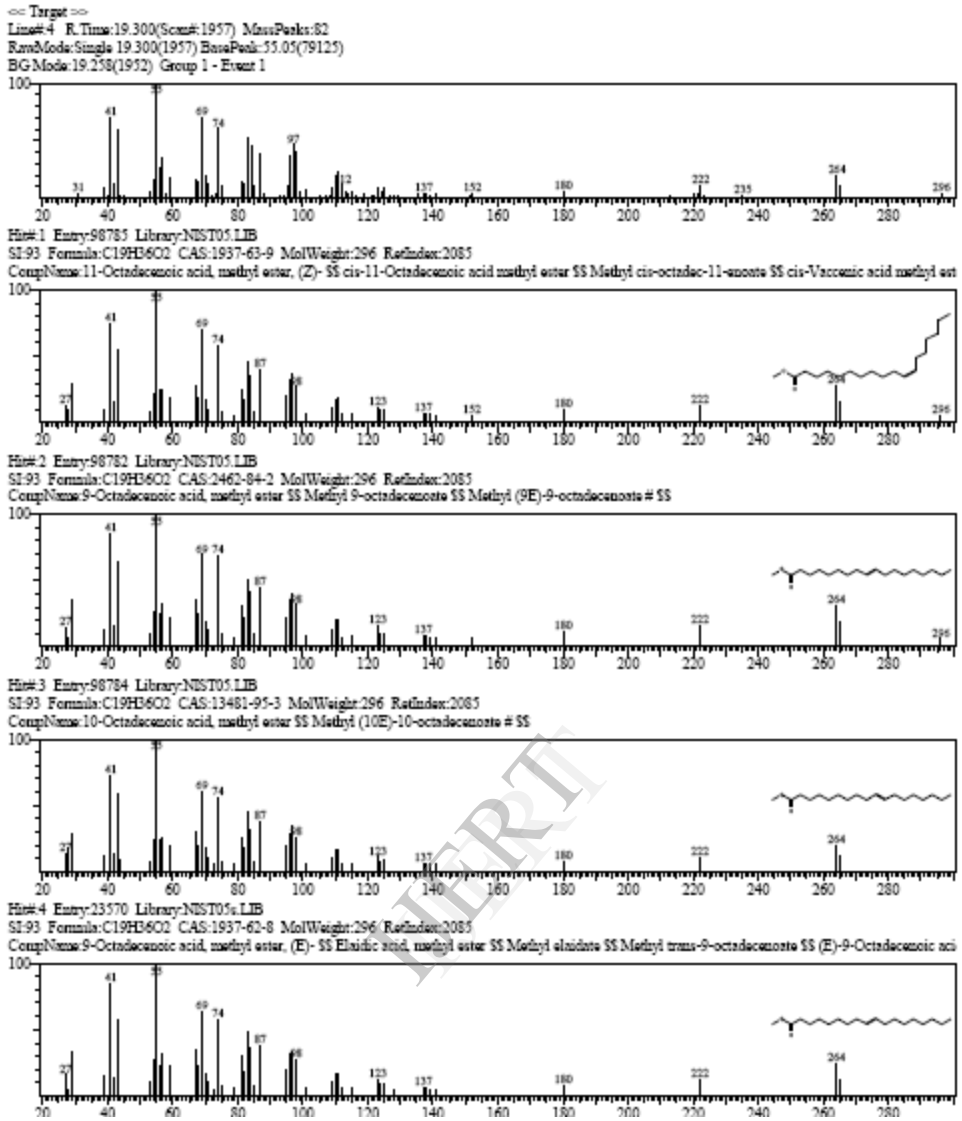

Figure 5: The Comparison for Chromatogram Line#4

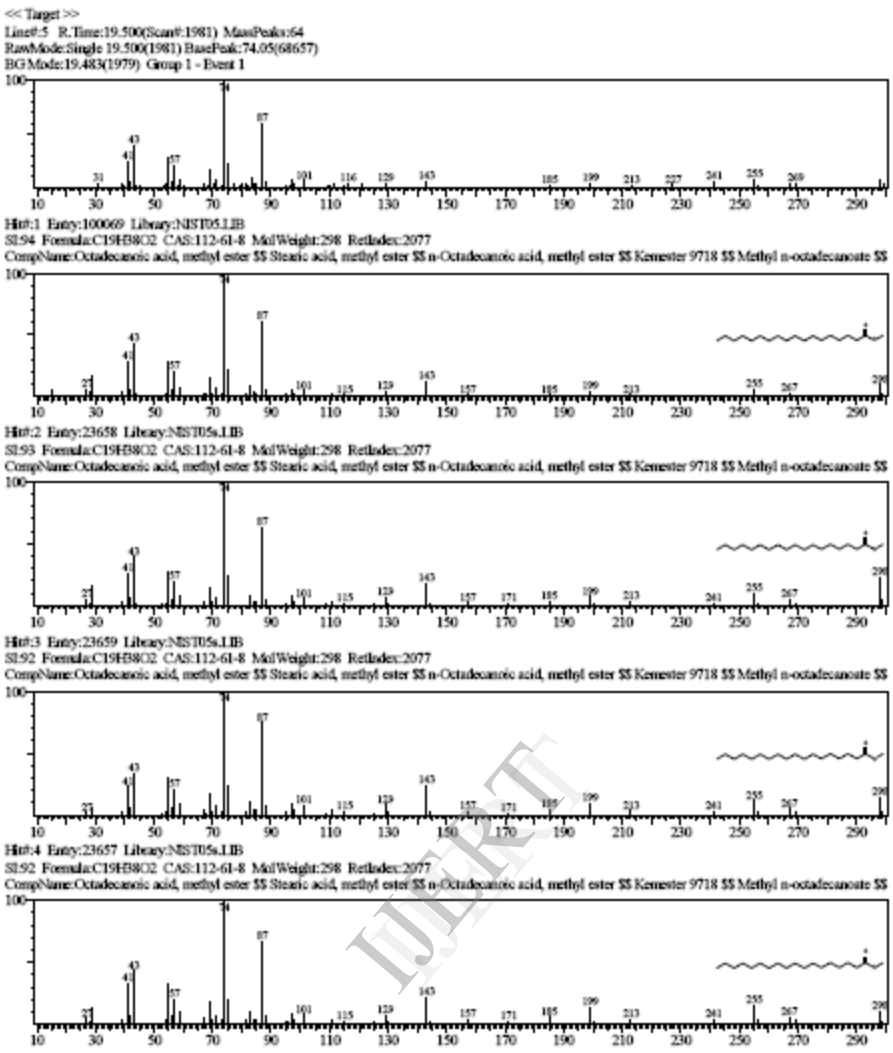

Figure 6: The Comparison for Chromatogram Line#5

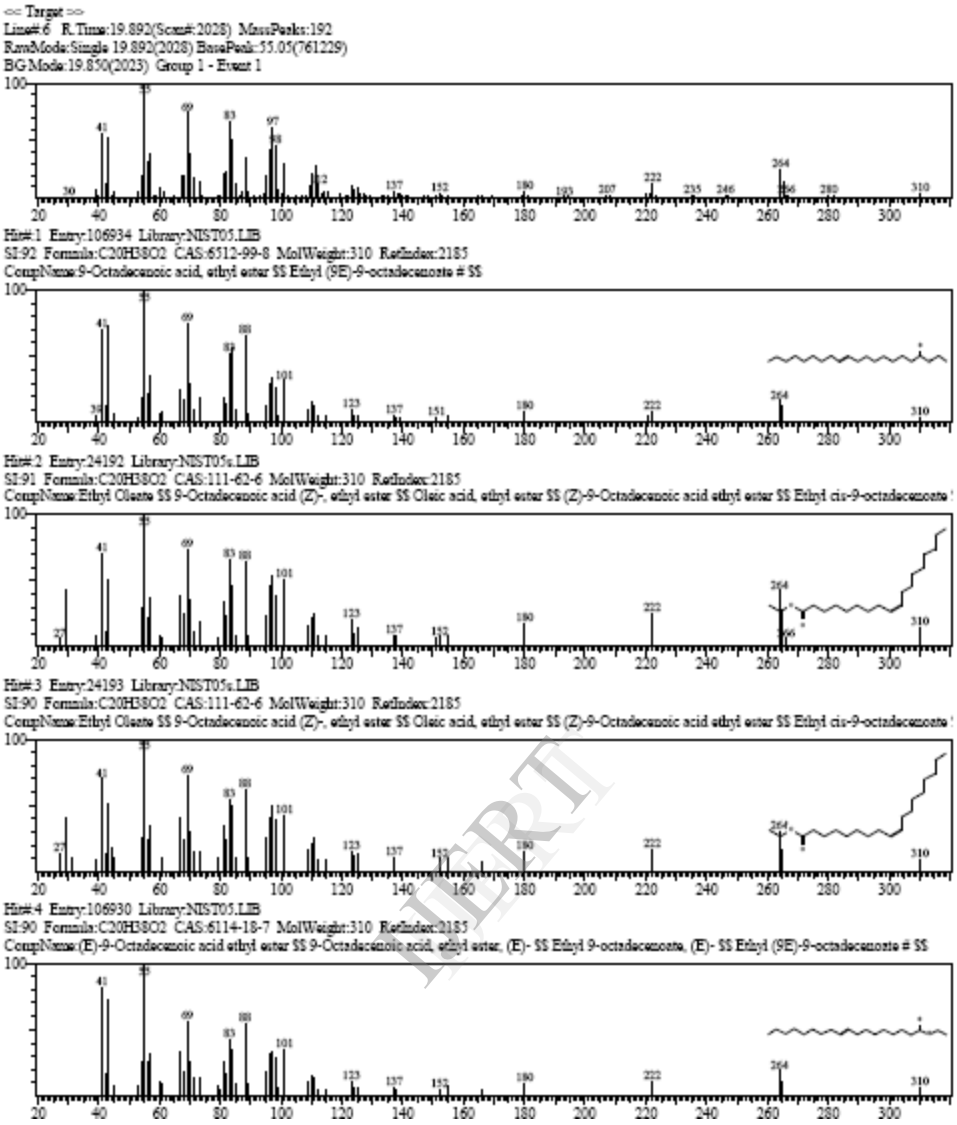

Figure 7: The Comparison for Chromatogram Line #6

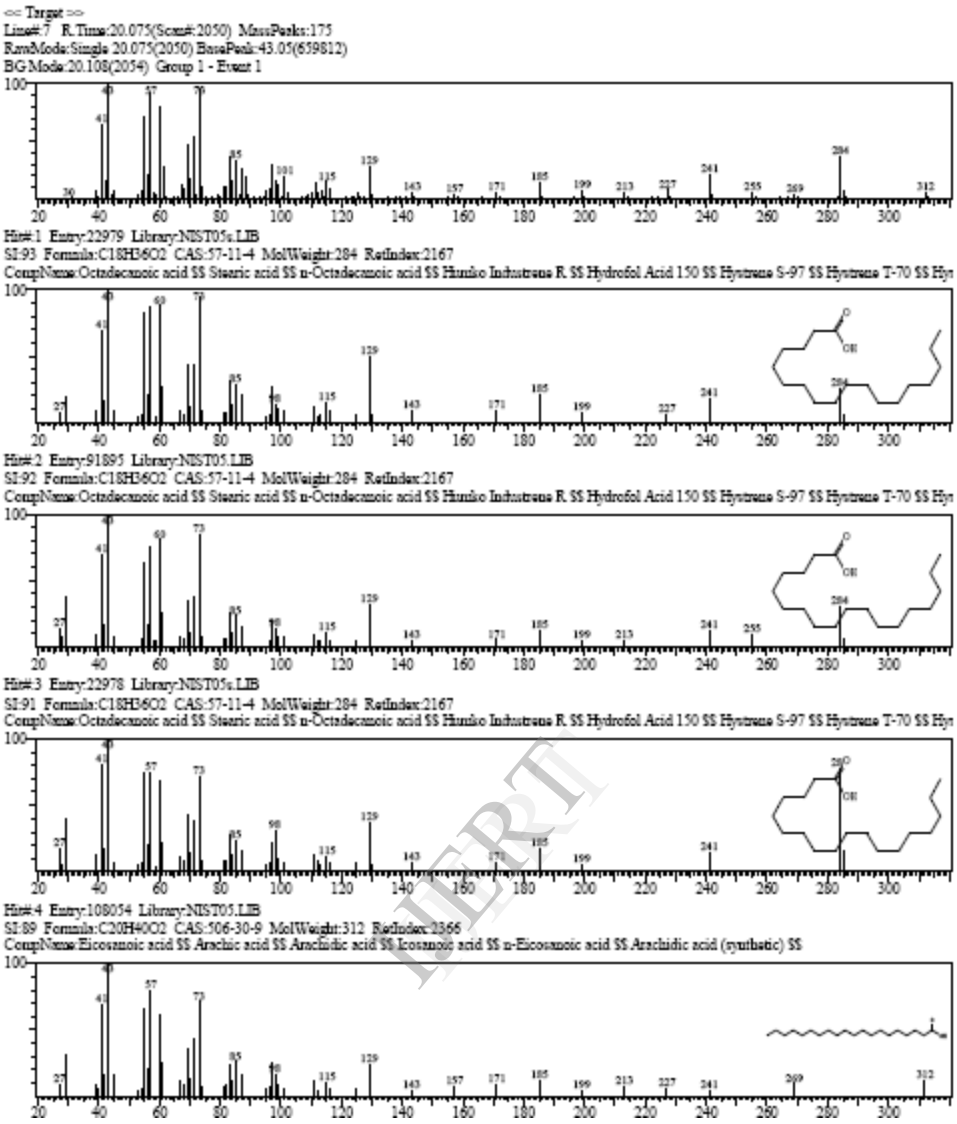

Figure 8: The Comparison for Chromatogram Line #7

Table 7: 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

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 impeller type A1 was used 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 domestic and 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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