ABUAD Journal of Engineering and Applied Sciences (AJEAS)
ISSN: 2992-5584
Volume 2, Issue 1, 28-35

Application of Aspen Plus to Modelling and Simulation of Neem Seed Oil Extraction

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Date Submitted: 18/01/2024
Date Accepted: 29/03/2024
Date Published: 30/06/2024

Abstract: *The demand for neem oil keeps on increasing due to its wide applications in pharmaceutical, agro–allied industries, cosmetics and soap production. To avoid wastage of material and safety of operation and personnel, the designed plant must be simulated to predict its performance before actual construction. The property method based on the* non–random two–liquid *(NRTL) model of Aspen Plus V12 was used to simulate the designed plant and its predicted performances were 87.13% and 99.60% for quantity of neem oil to be obtained from the evaporator and quantity of solvent (ethanol) recovery from the condenser as against 85.93% and 94.93% of the practical result with 1.38% and 4.69% errors respectively. The extracted oil has iodine, acid and saponification values of 60.10 g I_2/100 g, 4.2 mg KOH/g and 210.0 mg KOH/g respectively. The fatty acid compositions of oil are oleic acid, 46.61%; stearic acid, 11.83%; palmitic acid, 16.54%; 11 – octadecenoic acid, 3.58%; cis–vaccenic acid, 5.90%; cyclopropaneoctanal, 11.19%; squalene, 0.21% and trimethylsilyl–di(timethylsiloxy)-silane, 4.14%. Based on the quality and fatty acid composition of the extracted neem oil, it is highly recommended for cosmetics and soap production.*

Keywords: Neem tree, simulation, scale-up, Aspen Plus V12, property method.

1. INTRODUCTION

Neem tree is highly medicinal and cultivated in different parts of the world such as Asia (India and Indonesia), Africa, America and Europe [1, 2, 3]. Neem tree was introduced in Nigeria from Ghana by the colonial authorities in the year 1928 [4, 5]. Neem trees are readily available in the Northern part of Nigeria. Over four million (> 4000000) neem trees are in Borno, Kano, Katsina and Sokoto; and 90% of the Savanna Zone forestry of Nigeria is covered by neem tree under the afforestation programme [6]. Neem Oil Market was valued at USD 1.82 Bn. in 2022 and is expected to grow significantly from 2023 to 2030. The global neem market is growing with a compound annual growth rate (CAGR) of 13.4% from 2023 to 2030 and is expected to reach USD 3.8 Bn by 2030 [7]. Neem oil is a vegetable oil that is currently on high demand from emerging economics such as India and China [8]. [9] states that scale–up is simply the method used in design and construction of a large-scale system by using experimental results obtained from the small scale or pilot equipment. It is done in chemical industry to produce goods on a commercial scale.

Simulation is simply the operation of a model equation representing a system. The behaviour or performance of the actual system can be inferred based on the operation of the model during simulation. Simulation is very important tool in evaluating the performance of a system before building it so as to reduce chances of failure [10]. The Aspen Plus V12 was used based on its inbuilt interconnected components that are solved either under steady–state or dynamic mode or batch wise. The process modelling involved choosing the property method before creating the process flow sheet. The property method of Aspen Plus simulator was adopted due to its high accuracy for determining the physical/thermodynamics properties and phase behaviour of the components along with a detailed model equation for each unit operation from its library. This method is mostly chosen because it depends on process parameters such as temperature, pressure, flow rate and fractional composition of the components [11].

Neem oil was extracted from neem seed in an agitated pilot plant (9.61 kg/day) and yield of 36.86% was reported [12]. The challenge associated with the pilot plant is low production capacity and no reported studies have been found in current extant literature aimed at addressing this challenge. To boost the local extraction capacity of neem seed oil, there is need to scale up the pilot plant in other to increase its production capacity. The non–random two–liquid (NRTL) of the Aspen Plus model was adopted for the modelling. The NRTL model was considered because it fit best for equilibrium concerning polar components and the vapor phase behaviour can be compared to an ideal gas due to the operating pressure of less than 2 atm. For a successful simulation, the sequential procedure included selection of simulator, opening of new case, inputting components and fractional composition of the components, selection of property method and database, design of process flow diagram and inputting of operating conditions, running the simulation and obtaining of results. The results obtained from the procedure, will be compared with the actual performance of the scaled-up plant. The need for large scale production of neem oil to meet increasing demand has been emphasized by previous researchers such as [13, 14, 15, 5, 16 and 17].

2. MATERIALS AND METHODS

2.1 Flow Sheet for the Extraction of Oil from Neem Seed

The flow sheet for the solvent extraction of neem seed oil is shown in Figure 1.

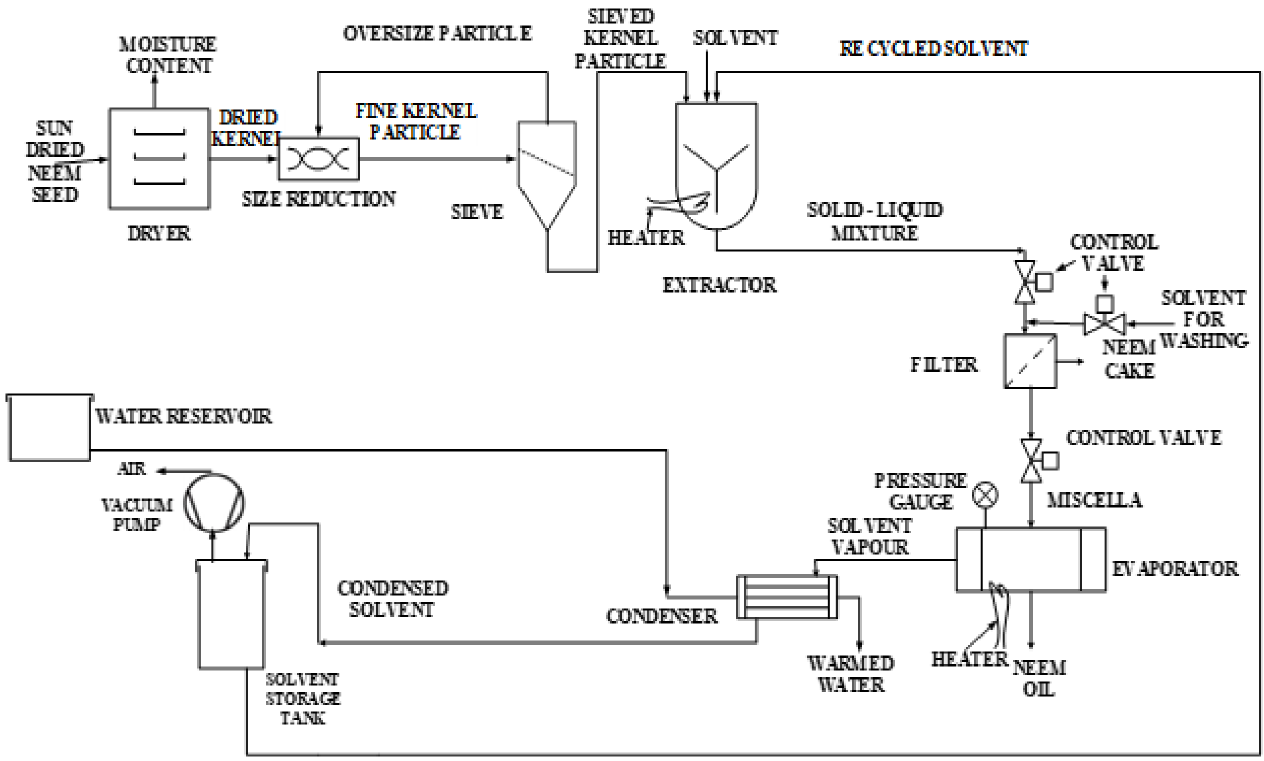

Figure 1: Flowsheet for the solvent extraction of neem seed oil

2.2 Data Calculation for Non–conventional Materials

The Aspen Plus V12 has inbuilt properties for conventional materials (ethanol, water, oil and air). The properties of the non-conventional materials (neem seed kernel and neem seed cake) must be calculated and inputted.

Calculation of Molecular Weight of Neem Seed Kernel (NSK)

The major components of neem seed kernel are oil, protein, carbohydrate and moisture. Protein is majorly made up of amino acid. The major amino acids in neem seed kernel are glutamic acid and arginine acid [18]. The glutamic acid ($C_5H_9NO_4$) has molecular mass of 147.13 [19]. The arginine has a molecular mass of 174.2 [20]. Carbohydrate is stored in nuts and seeds in form of starch [21]. Starch can either be amylose or amyloptin. The amylose ($C_{14}H_{26}H_{11}$) is found in seed, nut and legume [22] and it has a molecular mass of 370.35 [23]. Table 1 shows the molecular mass of the neem seed kernel.

Assumption

i. Major components of neem seed kernel are considered in calculating molecular mass of neem seed kernel.

ii. Moisture is completely removed during drying.

Table 1: Molecular mass of neem seed kernel

Component		Molecular Mass (kg/kmol)
Neem oil		884
Protein	Glutamic acid	147.13
	Arginine acid	174.2
Carbohydrate		307.35
Neem seed kernel		1 512.68

Calculation of Specific Heat Capacity of Neem Cake

The specific heat capacity of neem oil = 2.0827 J/g K [24].
The molecular mass of neem oil = 884

The Specific heat capacity of neem oil in J/kmol K is calculated as follows:

2.0827 J	1000 g	884 kg
g K	1 kg	kmol

= 1, 841,107 J/kmol K

Specific heat capacity of neem kernel = 1, 488.87 J/kg K = 2,252,184 J/kmol K [25].

The neem cake specific heat capacity is determined based on the percentage composition of neem oil being 45% and the neem cake being 55%, while the neem kernel is 100%.

$1 \times 2{,}252{,}184 = 0.45 \times 1{,}841{,}107 + 0.55X$
$X = 2{,}588{,}519$ J/kmol K.

Calculation of Density of Neem Cake

i. Mass of neem kernel = 1.56 kg
ii. Mass of oil (45%) = 0.702 kg
iii. Mass of neem cake = 0.858 kg
iv. Volume of neem kernel = 1.872×10^{-3} m^3
v. Volume of neem oil = 7.7569×10^{-4} m^3

Volume of neem cake = $1.872 \times 10^{-3} - 7.7569 \times 10^{-4} = 1.0963 \times 10^{-3}$ m^3

Density of neem cake = $\frac{0.858}{1.0963 \times 10^{-3}}$ = 782.63 kg/m^3

Calculation of mass flow rates of neem seed kernel into the dryer, neem seed kernel into the extractor and mass flow rate of ethanol for washing into the filtration unit

i. Total mass of neem seed kernel = 1.8224 kg (This the mass of fresh neem seed kernel before drying).
ii. Assumed processing time = 45 minutes
iii. Mass flow rate of neem kernel into dryer = $\frac{1.8224}{45 \times 60}$ = 6.7497×10^{-4} kg/s
iv. Mass of neem seed into the extractor = 1.56 kg
v. Mass flow rate of neem seed into extractor = $\frac{15.6}{45 \times 60}$ = 0.005778 kg/sec
vi. Mass of ethanol for washing = 0.312 kg
vii. Assumed time to charge ethanol into the filter for washing = 2 minutes

Mass flow rate ethanol into the filter $\frac{0.312}{2 \times 60}$ = 0.0026 kg/sec

2.3 Required Data for Simulating the Aspen Plus Model

Table 2: Data used for simulating the Aspen Plus model

Component	Molecular Weight	Specific heat capacity (J/kmol K)	Density (kg/m^3)
Ethanol	-	-	-
Water	-	-	-
Air	-	-	-
Neem oil (Triolein, Tripalmitin and Tristearin)	-	-	-
Neem Kernel	1512.68	2,252,184	833.2
Neem Cake	628.68	2,588,519	782.63

The additional required data for simulating the Aspen Plus model for each unit based on the non–random two–liquid model is shown in Table 3.

Table 3: Additional data for simulating the Aspen Plus V12

Dryer		**Crusher**	
Property	Value	Property	Value
Fractional composition	Neem Kernel: 0.856 and water: 0.144	Distribution function	RRSB (Robin Rammler Sperling Benneth)
Flow rate of neem kernel	0.00067497 kg/sec	Select parameter	D50
Valid phase	Vapour – Liquid	Pressure	1 atm
Pressure	1 atm	Stream class	MIXCIPSD
Temperature	50 °C	D50	0.00042 m
Stream class	MIXCIPSD	Valid phase	Solid
Type	Shortcut	Communition law	Bond
Critical moisture content	4.4% (0.044)	**Screen**	
Shape factor	0.63 – 0.99 (0.99 was used)	Property	Value
Air composition	1	Valid phase	Solid
Mass flow rate of Air	0.29 kg/sec [26]	Selection function	Ideal
Extractor		Cut size	0.0068
Property	Value	Pressure	1 atm
Temperature	50 °C	Stream type	MIXCIPSD
Pressure	1 atm	**Filter**	
Valid phase	Vapour – liquid	Property	Value
Ethanol into extractor		Screen size	100 μm (0.1 mm)
Temperature	20 °C	**Ethanol into filter for washing**	
Pressure	1 atm	Property	Value
Flow rate	0.005778 kg/sec	Temperature	20 °C
Mass fraction	Ethanol: 0.96; Water: 0.04	Pressure	1 atm
Stream class	MIXCIPSD	Flow rate	0.0026 kg/sec
Evaporator		Mass fraction	Ethanol: 0.96; Water: 0.04

Property	Value	**Condenser**	
Temperature	50 °C	Property	Value
Pressure	207.02 mmHg	Type	Shortcut
Valid phase	Vapour – liquid	Minimum cold stream outlet temp.	15 °C
		Minimum temp. approach	1 °C
		Minimum correction factor	0.99

The flow sheet generated in the ASPEN PLUS V12 is shown in Figure 2 and definition of terms on the flow sheet is as follows: S1 = Fresh neem seed kernel into the dryer, S2 = Air, S3 = Air moisture out of the dryer, S4 = Dried neem seed kernel, S5 = Reduced neem seed kernel particles from the crusher, S6 = Specific particle size of neem seed kernel into the extractor, S7 = Overflow of neem seed kernel, S8 = Mixture of neem kernel, neem oil and ethanol solvent, S9 = Cake of neem seed kernel, S10 = Miscella (Mixture of neem oil and ethanol), S11 = Ethanol, S12 = Neem oil and S13 = Cooling water.

The predicted mass fraction of components from the Extractor (S8), Filter (S9 and S10), Evaporator (S11 and S12) and Condenser (S13) are shown in Table 4.

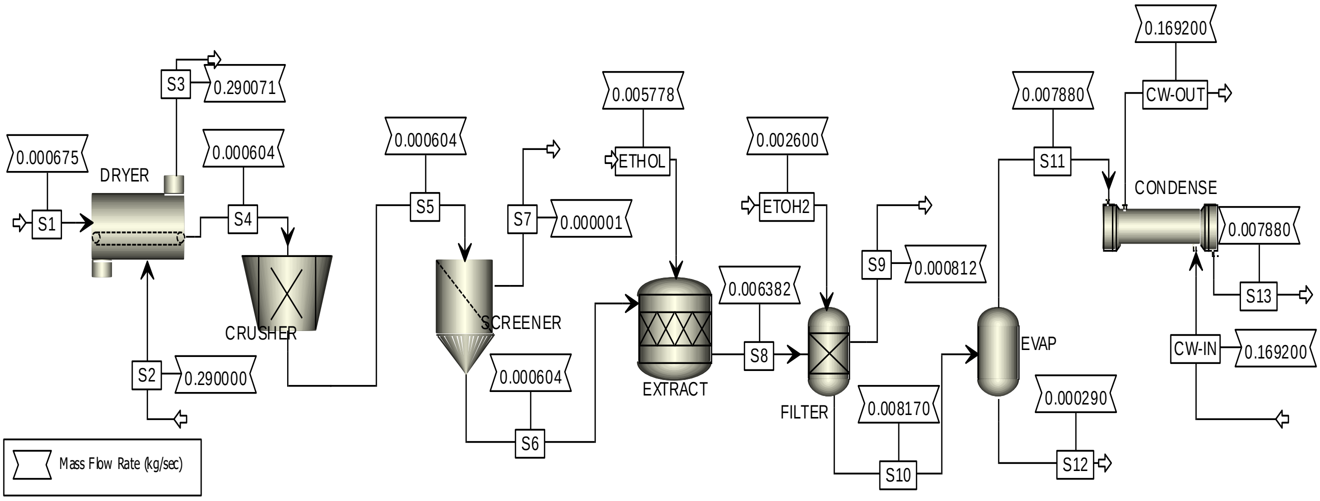

Figure 2: Aspen Plus flow sheet for the solvent extraction of neem seed oil

2.4 Extraction of Neem Oil

The neem oil was extracted using the scale–up constructed plant shown in Figure 3.

KEY
A = CONTROL PANEL
B = MIXER (EXTRACTOR)
C = ELECTRIC MOTOR
D = VESSEL FOR WASHING SOLVENT
E = FILTRATION UNIT
F = EVAPORATOR
G = CONDENSER
H = CONDENSATE RECEIVER
I = COOLING WATER RESERVOIR
J = STEP – DOWN TRANSFORMER
K = VACUUM PUMP
L = STABILIZER
M = PRESSURE GAUGE

Figure 3: Scaled-up solvent extraction plant

The extraction of neem seed oil was done using technical grade ethanol as solvents (ISO 9001.ISO 14001 certified and manufactured by Mansoura for Resins and Chemical Industries CO.SAE (MRI), Egypt). The plant was adequately checked to be sure all components of the plant were intact and well secured. The electric motor **C** for stirring the impeller was mounted on the cover of the mixer and the shaft carrying the flat blade turbine impeller connected to it. Valves V1, V2, V3, V4, V5, V6 and V7 were closed. All electrical fittings and connections were equally checked and ascertained to be in good conditions.

16.55 kg of ice block was added to 49.83 litre of water in the reservoir tank **I** and mercury–in–glass thermometer was used to monitor the temperature at 15 °C. Valve V5 was opened to allow the cooling water pass through 1 – 2 shell–and–tube heat exchanger (condenser) **G** so as to attain cooling stability. This was done to aid condensation of the ethanol vapour to liquid. The main switch was put on to monitor voltage input, which was between 220 – 240 Volts using an independent power source (Petrol Generator) to avoid power interruption.

1.56 kg of neem seed kernel particle of size 0.42 mm and 19.9 litre of ethanol were charged into the mixer **B** through

V1 and 0.4 litre of ethanol was charged into the washing solvent vessel **D**. The knobs of the mixer/evaporator heater control and the mixer/timer control were set to positions '1' and '1' respectively on the control panel **A** to switch on the electric heater (1000 W) for the mixer. The temperature controller was set to the required temperature of 60 ºC and the timer was set to the required contact time of 30 minutes. The set contact time was not activated until the required extraction temperature was attained; the activation of the timing was achieved when the mixer/time control knob was changed to position '2'.

A period of time was allowed to enable the system get stabilized. The stability of the system was confirmed with the aid of a temperature sensor placed in the mixer and a click short sharp sound that would be heard and the appearing of green light on the temperature controller. Once the stability was attained, the electric motor **C** was switched on and regulated at 77 rpm with the aid of a stirrer speed controller using flat blade turbine impeller which was already connected to the speed control unit and the mixer/timer control was quickly changed to position'2' to start timing and back to position '1' to continue heating. Mixing and agitation commenced immediately for a period of 30 minutes. Once the set time elapsed, the heating automatically stopped and stirring continued to avoid settling of particles. V2 and V4 were opened to allow the mixture of neem cake, neem oil and ethanol to flow into the filtration unit **E** through the inverted funnel and the stirrer speed controller was turned to zero point. V3 was opened for the washing solvent to flow into the filtration unit for washing the neem cake.

Filtration took place in the filtration unit with the aid of a stainless-steel filter mesh of size of 100 μm attached to the cake receiver. The miscella or mixture of neem oil and ethanol flowed through the already opened V4 into the evaporator **F**. After the cake washing, V4 was closed and the filtration unit was opened to collect the cake receiver and the mixer was opened to collect any cake left over in the mixer. The total cake in the receiver was placed in an oven for drying the cake. The drying was done using oven manufactured by SANFA Limited, England; Model NO: DHG – 9030 with temperature range of 50 ºC to 200 ºC at the boiling temperature of the solvent, 78 ºC. The weight of the cake was taken after every 20 minutes until constant weight was obtained. Valves V1, V2, V3 and V4 were shut and the knob of the mixer/evaporator heater control was set to position '2' to switch on the evaporator heater (1000 W).

The evaporator temperature controller was set to 78 ºC, the recovery temperature of ethanol solvent. The ethanol vapour passed through the already cooled 1 – 2 shell–and–tube heat exchanger (condenser) and was collected in the condensate receiver **H** as liquid solvent. After 3 hours 30 minutes of evaporation, a sample of oil was collected via V6 and analyzed using pH meter. Based on the viscous physical appearance of the product (neem oil) and the pH value, the evaporator heater was switched off by turning the knob of the mixer/evaporator heater control to position zero, the main switch was put off and V6 was closed. The collected neem oil at 78 ºC was dried in an oven at 78 ºC for 20 minutes to remove any residual ethanol. V7 was opened to collect the recovered solvent for recycling.

3. RESULTS AND DISCUSSIONS

Theoretical predictions and practical results

The fractional mass composition of each component from extractor, filter, evaporator and condenser units are shown in Table 4.

Table 4: Fractional composition of components from selected units

Components	Unit							
	Extractor			Filter		Evaporator		Condenser
	Input Ethanol	Input Neem seed	Output	Input (cake)	Output (Filtrate)	Output (Vapour)	Output (Product)	Output
Ethanol	0.96	0	0.869185	0.197428	0.96511	0.99596	0.122923	0.995959
Water	0.04	0.044	0.040378	0.40278	0.00410	0.00404	0.005807	0.004041
Neem kernel	0	0.956	0	0	0	0	0	0
Neem cake	0	0	0.050644	0.396676	0	0	0	0
Triolein	0	0	0.017279	0.001353	0.01337	0	0.378337	0
Tripalmitin	0	0	0.01069	0.000837	0.00827	0	0.23406	0
Tristearin	0	0	0.011823	0.000926	0.00915	0	0.258872	0

The simulation of the neem seed extraction process was based on the mass balance. The scaled-up plant design was modelled with Aspen Plus V12, a simulation tool which can evaluate material flow. The neem seed kernel and neem cake were specified as non-conventional components.

Based on the result generated by the Aspen Plus model as shown in Figure 2, the mass flow rate of neem seed kernel (NSK) S6 into the extractor was 0.000604 kg/s (52.19 kg/day) as against the actual 50 kg/day. The Aspen Plus predicted from the evaporator a mass fractional composition of 0.1229, 0.0058 and 0.8713 for the ethanol, water and neem oil (the sum of triolein, tripalmitin and tristearin which are the major components in neem oil) respectively as shown in Table 4; as against the assumption of 0, 0 and 1 respectively. The predictive value in terms of mass fractional composition of the major component (neem oil) from the evaporator shows an efficiency of 87.13% from the Aspen Plus model. Table 5 shows the comparison between the performance predicted by the simulator and the actual result obtained together with the percentage error from the extractor, evaporator and condenser.

Table 5: Comparison of Aspen Plus predicted (theoretical) results and practical results

Equipment	Component	Theoretical	Practical	Percentage error (%)
Extractor	Neem seed kernel	52.19 kg/day	50 kg/day	4.38
Evaporator	Neem oil	87.13%	85.93%	1.38
	Ethanol	12.29%	1.26%	1.26
	Water	0.58%	0	0.58%
1 – 2 condenser heat exchangers	Ethanol	99.60%	94.93%	4.69
	Water	0.40%	0	0.40
	Neem oil	0	0	0

Based on the equipment (dryer, crusher and screener) before the extractor, the efficiency of the operation was 95.62% as 52.19 kg/day was the input to the extractor instead of 50 kg/day; this showed a percentage error of 4.38%. The maximum yield of 38.67% was obtained as against the 45% considered for the design, which translated to percentage yield of 85.93% as shown in Table 5. The simulator predicted percentage yield of 87.13% from the evaporator as against the 85.93% actual; this translated to a percentage error of 1.38%.

The actual or practical recovery of solvent was 94.93% as against the 99.60% predicted by the Aspen Plus model. This showed a percentage error of 4.69%. All the percentage errors observed in Table 5 are within the experimental limit of maximum of 5% [27]. The neem oil obtained from the evaporator was dried in an oven at 78 ºC to further reduce the ethanol associated with the neem oil. The quantity of ethanol associated with the neem oil and removed in the oven was 1.26% as against the 12.29% predicted by the Aspen Plus model as shown in Table 5. There is 0% of neem oil associated with the recovered ethanol from the 1 – 2 heat exchangers as predicted by the simulator and equally shown practically in Tables 4 and 5.

Physiochemical properties of extracted oil

The physiochemical properties of the ethanol extracted oil are shown in Table 6.

Table 6: Physicochemical properties of the extracted neem oil

S/N	Parameters	Standard value	Ethanol extracted oil
1	pH	5.7 – 6.5	3.65
2	Specific gravity	0.908 – 0.934	0.905
3	Refractive index	1.4615 – 1.4705	1.519
4	Iodine value (g I_2/100 g)	65 – 80	60.10
5	Acid value (mg KOH/g)	0.4 – 4	4.20
6	Saponification value (mg KOH/g)	175 – 205	210.0

The physiochemical properties of the extracted neem oil did not fall within the specified range of value as shown in Table 6. The standard refractive index for vegetable oil is 1.3 – 1.6 [28]. The 1.519 is outside the standard range for the neem oil, but fall within the range of vegetable oil and closed to the 1.469 value reported by [28]. The difference is insignificant and within acceptable experimental error. The acid value is as low as 4.20 mg KOH/g which is close to 4 mg KOH/g reported by [29] and closed to the ASTM D6751 standard value range of 0.4 – 4 mg KOH/g. The iodine values obtained was 60.10 g I_2/100 g which is lower than the standard values, but close to 58 g I_2/100 g and 63.8 g I_2/100 g reported by [28] and [29] respectively. Previous researchers, such as [30, 1, 31] obtained 75, 122.5 and 89.35 g I_2/100 g as iodine values outside the standard range respectively. The 210.0 mg KOH/g saponification value is outside the standard range value of 175 – 205 mg KOH/g. The obtained values are higher than the 185.05 mg KOH/g and 184.78 mg KOH/g reported by [28] and [30] respectively. The variation of the obtained values from the standard range can be attributed to time of harvesting the seed, geographical location, soil composition and weather condition of the environment.

Fatty acid composition of extracted oil

The major fatty acid composition of the neem oil was determined using GC–MS and the respective percentage composition is: oleic acid, 46.61%; stearic acid, 11.83%; palmitic acid, 16.54% and cyclopropaneoctanal, 11.19%. The major fatty acid composition of the neem oil obtained is similar to the oil composition obtained by other researcher from avocado oil (60.7% oleic acid, 10.8% palmitic acid and 1% stearic acid); canola oil (65.7% oleic acid, 4.5% palmitic acid and 3.3% stearic acid); olive oil (75.7% oleic acid, 11.1% palmitic acid and 4.3% stearic acid); palm oil (42.9% oleic acid, 39.6% palmitic acid and 4.3% stearic acid) [32]. The percentage of oleic acid obtained in this work falls within the range of values obtained by [1] (60.92%) and [12] (40.41%) from neem oil. The 27.65% stearic acid composition obtained by [33] is higher than the value obtained in the oil with 11.83%. The fatty acids composition detected in the neem oil were in agreement with those reported by [34] where they found the percentage composition of neem oil to be 49.9% oleic acid, 17.9% palmitic acid and 19.4% stearic acid. According to [32] oleic acid has the highest percentage composition as fatty acid in oil; this is clearly seen in avocado oil (60.7%), canola oil (65.7%), olive oil (75.5%) and palm oil (42.9%). These findings agreed with the findings in this work where oleic acid has the highest fatty acid percentage composition of 46.61%. The differences in the percentage composition can be attributed to variation on time of harvest, climatic condition, soil composition on which the neem trees grow and processing method.

4. CONCLUSION

Aspen Plus V12 was used to predict the performance of the scaled-up plant before construction. The actual constructed plant shows a performance of 85.93% as against

87.13% predicted in term of the quantity of neem oil obtained from the evaporator with a percentage error of 1.38%. The actual or practical recovery of solvent was 94.93% as against the 99.60% predicted by the Aspen Plus model. This shows a percentage error of 4.69%. The iodine, acid and saponification values of the extracted neem oil were 60.10 g I_2/100 g, 4.2 mg KOH/g and 210.0 mg KOH/g respectively. These values fall favourably within the standard values and are similar to the ones obtained by previous researchers. The fatty acid compositions of oil are oleic acid, 46.61%; stearic acid, 11.83%; palmitic acid, 16.54%; 11–octadecenoic acid, 3.58%; cis–vaccenic acid, 5.90%; cyclopropaneoctanal, 11.19%; squalene, 0.21% and trimethylsilyl–di(timethylsiloxy)-silane, 4.14%. Based on the quality and fatty acid composition of the extracted neem oil, it is highly recommended for cosmetics and soap production.

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