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EXTRACTION AND COMPARISON OF ESSENTIAL OIL FROM NEEM SEED BY USING SOXHLET EXTRACTION AND SIMPLE DISTILLATION METHODS

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Abstract:
Extraction of essential neem oil from neem seed were carried by soxhelt extraction and simple distillation method using different types of solvents and parameters. Physico-chemical characteristics of the extracted oil were also determined by using classical wet chemical method. Results were generated in both techniques. Soxhlet extraction using hexane has 40.35%, using ethanol-hexane mixture of 60:40% volume proportions have 43.71%, using ethanol 42.65% and using methanol 42.89%. In simple distillation method results reveal that using hexane 42.35%, in methanol 39.5%, using ethanol 40.72% and using ethanol-hexane mixture of 60:40% has 41.26 %. For all solvent type particle size has 355µm, extraction time 1hr up to 3hrs and applied constant and varies temperatures. At smaller extraction time, hexane produced oil yield greater than from ethanol and methanol. In soxhlet extraction, ethanol not produced oil at one-hour extraction time. Thus, by effective determination of factors like particle sizes, solvent type, temperature, and time it is possible to investigate the result on the quality and quantity of neem oil.

Keywords: Neem Oil; Particle Size; Soxhlet Extraction; Simple Distillation; Solvent Type.

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1. Introduction

Neem (*Azadirachta indica A. Juss*) is one of the very few trees known in the Indian sub-continent. This tree is belonged to *Meliceae* family and grows rapidly in the tropic and semi-tropic climate. It is also observed that this tree could survive in very dry and arid conditions. Neem seed is a part of neem tree which has high concentration of oil. Neem oil is widely used as insecticides, lubricant, drugs for variety of diseases such as diabetes and tuberculosis. This oil could also prolong leather goods when it is applied on them. There are several methods to obtain neem oil from the seeds like mechanical pressing, supercritical fluid extraction, and solvent extraction (Puri, 1999).

The Neem oil is not used for cooking purposes, it is used for preparing cosmetic (soap, hair products, body hygiene creams, hand creams) and in Ayurvedic unani and folklore traditional

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medicine in the treatment of wide range of affiliations. The most frequently reported indications in ancient Ayurvedic writing are skin diseases. Inflammations and fevers are more recently rheumatic disorders, insect repellent and insecticide effects. Formulations made of neem oil also find wide usage as a bio-pesticide for organic farming, as it repels a wide variety of pests including the mealy bug, beet armyworm, aphids, the cabbage worm, thrips, whiteflies, mites, fungus gnats, beetles, moth larvae, mushroom flies, leaf miners, caterpillars, locust, nematodes and the Japanese beetle (Natarajan et.al, 2003).

Neem oil is not known to be harmful to mammals, birds, earthworms or some beneficial insects such as butter flies, honey bees and lady bugs if it is not concentrated directly into their area of habitat or on their food source. It can be used as a house hold pesticide for ant, bedbug, cockroach, housefly, sand fly, snail, termite and mosquitoes both as repellent and larvicide. Neem seed oil has also been used as a renewable source for the preparation of polymeric coatings. It has been converted into various polymeric resins, including polyester amides and polyether amides. These resins may be utilized further for preparation of polyurethane coatings (Natarajan et.al, 2003).

The use of solvents and extraction process for the separation of neem oil were generally preferred choice. This is due to very high oil yield and less turbid oil obtained than from mechanical pressing. Due to the relatively low operating cost compared to supercritical extraction. The solvent hexane is the most commonly used, as well as preferred choice in extraction of oils from seed, even in neem oil solvent extraction. This is largely due to its availability at a reasonable cost and its suitable functional characteristics for oil extraction. Amongst such characteristics is its high solvent power for triglycerides at fairly low temperatures, non-reactivity with oil and oil miscella, as well as with equipment (Liaum et al., 2008).

The objectives of present study were compared soxhlet extraction and simple distillation method using some selected organic solvent to take out essential oil from neem seed. The effects of parameter such as temperature, time and particle size were studied in this research work. As the solvents, we used ethanol, hexane, methanol and ethanol-hexane mixtures.

2. Materials and Methods

2.1. Materials and Equipments

Neem seed used for the study was obtained from Dire Dawa city. Dry oven was used for drying neem seed. Analytical balance was used for weighed neem powder sample. Sieve was used for separate fine particles from neem powder. Filter paper was used for filtration process. Heater Mantle was used for heating the solution during soxhlet extraction time. Soxhlet chamber was used for extraction process. Distillation column was used for distillation process. Conical flasks and 100 mL volumetric flasks were used for measuring and preparing sample solution respectively. Burette, beakers and pipette were used for carried titration process for measured acid value and saopnifcation value.

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2.2. Chemicals (Reagent)

N-hexane, Methanol, Toluene, Ethanol and mixtures of ethanol - hexane were used for extraction of oil from need powdered. Alcoholic potassium hydroxide (KOH), hydrochloric acid (HCl) and phenolphthalein indicator were used for determination of saponification value.

2.3. Size Reduction and Sieve Analysis of Neem Seeds

Neem kernel was crushed in KIKA-WERKE GMBH mill with sieve size 2mm. The sample was sieved using vibrated shaker with set of sieves sizes arranged in descending order 1mm, 710µm and 355µm. This is because to investigate the effect of particles size on yield and quantity of the oil. Therefore, using 355µm sieve is more effective and gives greater amount of oil than the else.

2.4. Preparation of Neem Seed Powder

Neem seed used for the study was obtained from Dire Dawa city. Prior to use, the neem seeds were repeatedly washed to remove dirt and other impurities material, and subsequently dried in oven at 50°C until it attained constant moisture content. Then, neem seeds were pulverized to get the particle sizes of 355µm and prepared for extraction process (Maria et al. 2008)**.**

2.5. Method for Extraction of Neem Oil

2.5.1. Soxhlet Extraction Method

100 g of neem powder was placed into the thimble and placed in the soxhlet chamber .500 ml of selected solvents were placed in a round bottom flask and assembled for soxhlet extractor then the distillation process was begun. After completed the extraction process, the solvent and extractor were placed on water bath to evaporate the solvent. Then extracted neem oil was weighed by using the following equation:

$$\% \text{ oil yield} = \frac{W1-W2}{W1} X100 \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad (1)$$

Where: W_1=Sample weight initially placed in the thimble and W_2= sample weight after dried in the oven (Natarajan et al. 2003).

2.5.2. Method for Simple Distillation

100g of neem powder sample was Weigh and placed in conical flaks and measured 500ml of organic solvents (200ml of n- hexane and 300ml of ethanol/Methanol). Mix and effectively shake the solution for about 15 minutes. Then Put the solution in the stable place for about 3 hours to enhance sedimentation process. Filter the solution with the aid of vacuum filtration mechanism. Distillation of liquid mixture (ethanol, hexane, oil, water).

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Method for Acid Value and Saponification Value

Acid value and saponification value were determined according to the methods prescribed by AOAC (1984). Specific gravity and pH were determined according to the method of AOAC (1990).

3. Result and Discussion

Extraction of essential neem oil from neem seed were carried by soxhlet extraction and simple distillation method using some selected organic solvents and parameters. The effects of parameter such as temperature, time and particle size were studied. Results were tabulated in the following tables. Three replicate measurements were done for each parameters (N=3). The first six tables were represented the result of soxhlet extraction method.

Table 1: show result of Soxhlet extraction techniques using hexane with particle size 355µm and constant temperature

Trial	Temperature (^{0}c)	Time (min)	Oil yield (%)
1	70	60	37.02
2	70	120	39.43
3	70	180	40.35

Table 2: show results of Soxhlet extraction using hexane with constant particle size (355µm) and varies temperature.

Trials	Temperature (0c)	Oil yield (%)
1	70	41.08
2	78	41.89
3	86	42.41

Table 3: show results of Soxhlet extraction using Ethanol-Hexane mixtures of 60:40% of volume proportion at constant temperature

Trials	Temperature(^{0}C)	Time (min)	Oil yield (%)
1	70	60	25.06
2	70	120	37.87
3	70	180	43.71

Table 4: show results of Soxhlet extraction using Methanol with particle size 355µm and at constant temperature

Trials	Temperature(0c)	Time(min)	Oil yield (%)
1	70	60	25.38
2	70	120	39.05
3	70	180	42.89

Table 5: show results of Soxhlet extraction using Ethanol with particle size 355µm and at constant temperature

Trials	Temperature (0c)	Time (min)	Oil yield (%)
1	70	60	-
2	70	120	40.54

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3	70	180	42.65

Table 6: show result of Soxhlet extraction using ethanol- hexane mixture 40:60% of volume proportion with particle size 355 µm and constant temperature

Trials	Temperature (0c)	Time(min)	Oil yield (%)
1	70	60	26.23
2	70	120	36.42
3	70	180	40.21

Results of Simple Distillation Methods

The following tables shown the result of simple distillation method for each parameter replicate analysis were done (N=3).

Table 7: show results of simple distillation method using hexane with constant particle size (355µm) and temperature at varies time interval

Trials	Temperature	Time	Oil yield
1	70	60	29.6
2	70	120	34.85
3	70	180	42.35

Table 8: show results of simple distillation method using hexane with constant particle size (355µm) and varies temperature.

Trials	Temperature (0c)	Oil yield (%)
1	70	42.78
2	78	43.24
3	86	43.84

Table 9: show results of simple distillation method using methanol with constant particle size (355µm) and temperature at varies time interval

Run number	Temperature(0C)	Time	Oil yield (%)
1	70	60	20.63
2	70	120	39.34
3	70	180	43.23

Table 10: show results of simple distillation method using ethanol with constant particle size (355µm) and temperature at varies time interval

Trials	Temperature (^{0}C)	Time	Oil yield (%)
1	70	60	25.38
2	70	120	38. 43
3	70	180	40.72

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Table 11: show results of simple distillation method using ethanol-hexane mixture 60:40% with constant particle size (355µm) and temperature at varies time interval

Run number	Temperature(0C)	Time	Oil yield (%)
1	70	60	26.23
2	70	120	36. 42
3	70	180	41.26

3.1. Physico-Chemical Characteristics of Extracted Neem Oil

In this research work the physico-chemical properties of the extracted essential neem oil was investigated by using classical wet chemical method (volumetric method). Results were presented below in table-12.

Solvents	Acid value (mg KOH/g)	Sap.Value (mg KOH/g)	Specific gravity	P^H
Ethanol	17.35	195.56	0.85	5.38
Hexane	18.05	202.04	0.90	6.04
60:40% hex/eth	14.46	194.48	0.90	4.86
40:60% hex/eth	17.11	198.36	0.89	4.98

3.2. Effect of Temperature

Results obtained from the analysis showed that an increase in temperature generally favors an increase in the oil yield in both techniques. This phenomenon is due to the fact that oils are generally more soluble at elevated temperatures (Sen et al. 1992). At higher temperatures, the viscosity of the solvent is reduced while the diffusivity, as well as evaporation rate is increased. This increases the contact time between the solvent and the oil bearing material (Ayoola et al., 2014). From the above result, soxhlet extraction using hexane at constant temperature (70^0C) has 40.35% and a temperature at 86^0C has 42.41%. At same temperature, the results showed that ethanol -hexane mixture of 60:40 % volume proportions were better extraction liquid for higher yields of oil attained. In simple distillation method, using hexane at varies temperature recorded 43.84% and varies time 42.35%. Using methanol at constant temperature and varies time has 43.23%.

3.3. Effect of Time

Oil yield obtained (expressed in percent) was extraction time reliant. In general, the oil yield increased with increase in extraction time and there was no considerable increase after 3hour (Ayoola et al., 2014). Results shown from table-1, soxhlet extraction using hexane the time changed from 1hr to 3hrs the oil yield rose 37.02% to 40.35%. In case of ethanol- hexane mixture of 60:40% and 40:60% volume proportions the oil yield rapidly rose 25.06% to 43.71% and 26.23% to 40.21% respectively. The same condition was appeared in methanol. Ethanol produced no oil at one hour of extraction time. As the extraction time increased, mixtures of 60% ethanol 40% hexane gave the maximum oil yield. These show that the combination of solvents favors higher oil yield compare to using these solvents separately.

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Results of physco-chemical properties of extracted neem oil point out that, extraction using hexane solvent reported high acid value (18mgKOH/g) and saponification value (202.04 mgKOH/g) than the other solvents. Acid value indicates the amount of free fatty acids presents in an oil. Acid value is good indicator of oil degradation caused by hydrolysis. Also, higher extraction temperature increased the acid value because the extraction temperature influenced the hydrolysis of neem oil (Adeeko & Ajibola, 1990). Saponification value indicates the average molecular weight of triglycerides in the oil (Fasina &Ajibola, 1989, Ayoola et al., 2014). And also an increase in temperature increased the saponification value because higher temperature caused lipid to breakdown therefore reduced the average molecular weight of the oil (Fasina &Ajibola, 1989, Adeeko & Ajibola, 1990).

4. Conclusion

This research work was proposed to examine the influence of different features such as Particle sizes, solvent type, temperature, and time on the extent of neem oil. Unpredictability of these operating conditions is the pre-dominant factors for the quantity of the oil.

There are different techniques for essential oil extraction from neem seed. In the current study, soxhlet extraction and simple distillation method were nominated. Using soxhlet extraction method the maximum oil yield obtained 43.71% in ethanol-hexane 60:40% volume of solvents with particle size of 355μm and for three hours extraction time. Using simple distillation method, maximum oil yield obtained 43.23% in methanol. The quantitative difference in the amount of the oil was due to particle size and extraction time.

Determination of the appropriate size of the particles and optimal time for the recommended particle size needs to have a consideration to get the maximum amount of the required product. Volume proportions of ethanol-hexane mixtures (60:40 and 40:60% respectively) served as efficient solvent alternatives to the use of hexane in essential neem oil extraction.

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Conflict of Interest

The author(s) declare(s) that there is no conflict of interest regarding the publication of this paper."

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