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A Search for Alternative Solvent To Hexane During Neem Oil Extraction

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ABSTRACT

The growing environmental concerns on the toxicity and flammability of hexane motivate the need to obtain a more environmentally friendly solvent capable of extracting as much oil as hexane or even more, while giving acceptable oil quality. This study therefore investigated the possibility of replacing Hexane with either Ethanol or the mixtures of both in the extraction of Neem Oil from Neem seed. It was observed that as the extraction time increased, oil yield increased, though there was little or no increase in oil yield at 5hours and 6 hours of operations. At different extraction time, Hexane produced oil yields greater than that obtained from Ethanol. Hence, Ethanol did not prove to be a good replacement for Hexane, even at higher temperature of 75^{0}C for Ethanol. Optimum temperature of extraction was found to be 55^{0}C. Surprisingly, mixtures of Ethanol and Hexane gave commendable results. Particularly, Ethanol-Hexane mixtures of 60/40, 50/50, and 40/60 % (volume proportions) gave better oil yields of 44%, 43% and 41.2% respectively than that of Hexane (40.25%) at the 6 hours of operation, thereby making the combinations good substitutes for Hexane.

Keywords: *Ethanol, Hexane, Neem Oil, Soxhlet Extraction.*

I. INTRODUCTION

Neem Oil is a vegetable oil gotten from the fruits, leaves and seeds of neem tree. The tree is a member of the Mahogany family, with generic name Azadirachta Indica A. Juss, it is an evergreen tree which is endemic to the Indian subcontinent and has been introduced to many other areas in the tropics. It has also been found to thrive in the semi tropics, arid and semi arid climates in some countries including Nigeria where it is known as Dongoyaro, meaning “tall boy”. [1,2,3]

Neem oil is generally light to dark brown in colour. It has a bitter taste and an offensive odour similar to the combined odours of garlic and peanut. It comprises mainly of triglycerides (esters formed from a molecule of glycerol and three molecules of fatty acids), and is very rich in azadirachtin- the key component acting as insect repellent, anti-feedant, anti-fungal and anti-viral, among others, it is perhaps the most important commercial product of neem for organic farming and medicines.[4,5,6] Also, it is being increasingly used in manufacturing a large number of skin products: body soaps, body lotions, and beauty care facial packs in combination with other natural ingredients.[7]

Research reveals three ways in which neem oil may be extracted from its seeds and they are: mechanical pressing, supercritical fluid extraction and solvent extraction. Mechanical pressing involved crushing of neem seed to extract oil. Though oil obtained by cold pressing preserves the key components of the oil (particularly azadirachtin), but reports however show that such oil has low yield and poor market value (quality) as the oil is turbid and has high water and metal contents.[1,2] Supercritical fluid extraction process utilizes carbon dioxide at critical temperatures and pressures to extract the active ingredients of the neem leaves or seeds. It produces high quality oil but it is very expensive to operate and involves the release of CO_2 to the atmosphere.[8]

The use of solvent extraction for the extraction of neem oil is generally the preferred choice. This is due to very high oil yield and less turbid oil obtained than that from mechanical pressing. And also due to the relatively low operating cost compared to supercritical extraction.[2]

The vast usefulness and applications of the neem oil progressively places an increasing demand for the availability of the extracted and processed oil.[9,10,11,12,13,14,15] The pharmaceutical industry and, particularly, the agro-allied industry, where neem oil is rapidly finding relevance in plant protection make the need for the extracted oil be on the increase.[9,16]

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.[7]

However, the interest in alternate solvents to hexane has continued and is motivated by one of a combination of factors: the desire for a non flammable solvent, a more efficient and less hazardous and environmentally friendly solvent. An average of one major accident associated with the use of hexane occurs per year.[7] In addition, n-Hexane, the main component of

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commercial hexane, is one of 189 Hazardous Air pollutants listed in the Clean Air Act, and hexane has hence being regarded as both a Criteria Pollutant and a Hazardous Air Pollutant. This has thus generated the need to find alternate solvents to hexane, in a bid to join hands with other efforts round the world to cumber air pollution and toxicity.[7]

In an oil extraction process investigation, ethanol, a supposedly good substitute for hexane in neem oil extraction is found to give an oil yield of 41.11% at 50^0C as against 42.29% when hexane was used at the same temperature.[7] While this is encouraging, a study prove that oil solubility in ethanol is higher at high temperatures, as is the case with soybean oil. Hence, there is a possibility that more oil could still be extracted from the neem seed using ethanol at temperatures higher than 50^0C, and possibly more oil than that extracted using hexane at 50^0C, which is about the possible temperature-limit of hexane due to its high risk of flammability.

Also, reports show that ethanol-hexane mixtures have potential for oilseed extraction.[17] Cottonseed and soy oils, extraction rate and total extractible increased with the use of hexane-alcohol mixtures in different proportions.[17] These suggest the need to inquire and research whether ethanol-hexane mixtures or ethanol alone (as extraction solvent) will be able to yield similar or better results using hexane as solvent, during oil extraction from the neem seeds.

This study is therefore an attempt to seek a way of solvent extracting as much oil from the neem seed as possible using a less toxic solvent than hexane. And it is believe a success of this study would culminate in more neem oil for use in industries and may also lead to a decrease in cost of purchase of solvents.

II. METHOD

Having obtained the seeds, the seeds were stored in such a manner that allowed passage of air for further drying and prevention of the fungi growth.

Depulping and Dehulling: Depulping, the separation of seeds from fruits, was followed by dehulling, the process of removing the outer seed coat. Dehulling was done to ensure high extraction efficiency as seed coats contain little or no oil. To achieve this, seed pulps were soaked in warm water to soften the outer seed coat and the seed coats and fruit were then peeled off by hand.

Seed Cleaning: This was done to remove foreign materials such as sticks, stems, leaves, bad seeds, sand and dirt, to ensure that the oil produced is not contaminated and of high quality.

Drying: This was done to remove the moisture content of the seeds so as to ensure high extraction efficiency. The drying of the seeds was done using electric dryer operated at a temperature of 55^0C for about 18hours.

Grinding: This was done to obtain a very large surface area for extraction to be completed.

Heating: This was the final stage of the seeds preparation steps. It involves heating the ground seeds just before extraction to remove moisture content completely. This was done for 10mins at a temperature of 55^0C.

Extraction Procedure: After the completion of the seed preparation steps, extraction of oil from grinded neem seeds was done. Extraction experiments involved the use of: soxhlet extractor (coupled with heating mantle) and extraction solvents (at different combinations), varied extraction time and varied temperature.

Experiments were carried out to achieve the set objectives of the determination of the maximum oil yield obtainable with: hexane, ethanol and hexane-ethanol mixtures.

Distillation: Distillation of oil-solvent mixture obtained was carried out at 80^0C to obtain pure neem oil.

III. DISCUSSION OF RESULTS

Effect of Temperature on Oil Yield

Results obtained, when hexane and ethanol were used separately (as solvent), showed that an increase in temperature generally favours an increase in oil yield. This phenomenon is due to the fact that oils are generally more soluble at elevated temperatures [4].

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. The net effect of this is that more oil is dissolved in the solvent per time, thereby increasing oil yield.

At same temperature, the results showed that hexane is a better extraction liquid, for higher yields were obtained. Even when ethanol is used at higher temperatures (temperatures above boiling point of hexane), oil yields obtained were still very low (**Figure 1**). And in each case, the optimum extraction temperature is 55^0C.

These shows that pure ethanol cannot be a good substitute for hexane, as extraction liquid. Further investigated were carried out to see whether combination of ethanol and hexane would produce higher oil yield than pure hexane at the optimum temperature of 55^0C

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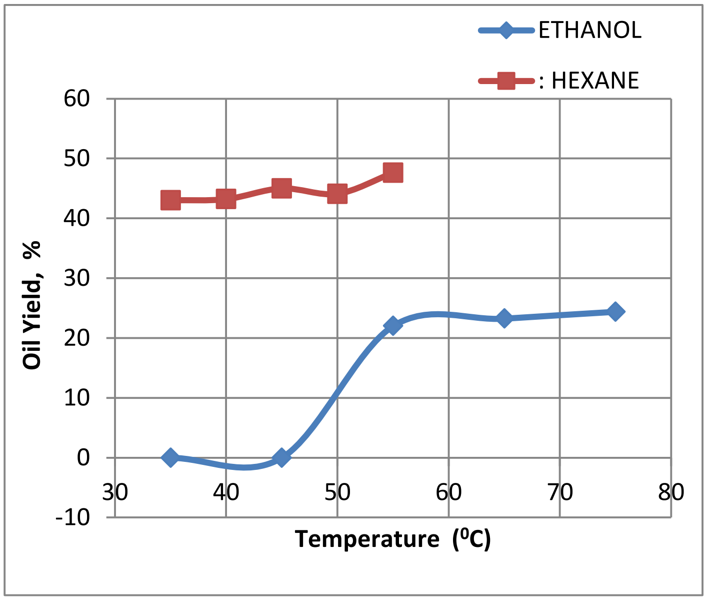

Figure 1: Oil Yield obtained during Neem Oil Extraction at different Temperature using Ethanol and Hexane

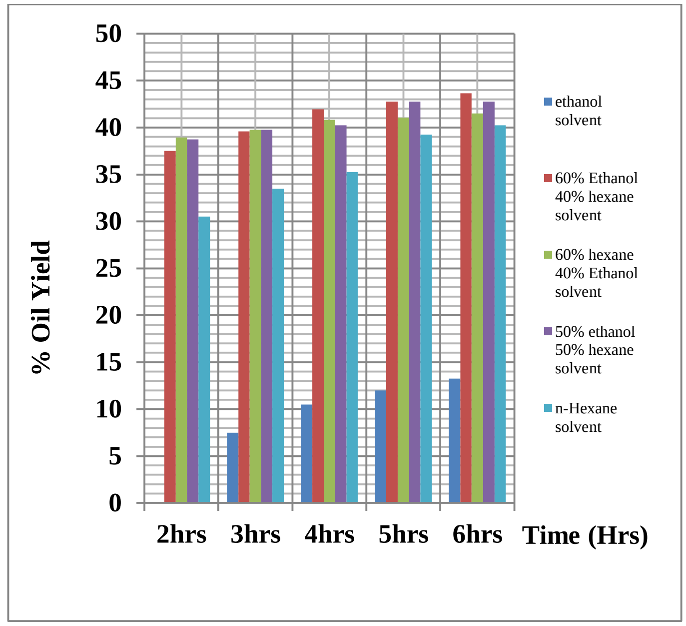

Figure 2: Oil Yield obtained during Neem Oil Extraction at different Time using Ethanol, Hexane and their combinations

Effect of Time on Oil Yield

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. The net effect of this is that more oil is dissolved in the solvent per time, thereby increasing oil yield.

Oil yield obtained (expressed in percent) was extraction time dependent. In general, the oil yield increased with increase in extraction time and there was no significant increase after 5hours **(Figure 2)**. When pure ethanol and hexane were used separately (as extraction solvents), higher oil yields were obtained with hexane. Infact, ethanol produced no oil at 2 hours of extraction time. As the extraction time increased, 60% ethanol/40% hexane gave the maximum oil yield, followed by 50% ethanol/50% hexane. While at 2 and 3 hours of extraction, 40% ethanol/60% hexane produced maximum oil yield, followed by 50% ethanol/50% hexane. These show that the combination of solvents favours higher oil yield compare to using these solvents separately. And that Hexane performs better as extraction solvent at short period of operation while at long period of extraction process, ethanol is preferred. And also, 60% ethanol/40% hexane gave the maximum oil yield.

Justification of Ethanol-Hexane mixture

The ability of the ethanol-hexane mixture to perform better than ethanol or hexane is due to the fact that the mixture is able to reduce the flammability challenges usually associated with pure hexane solvent. The effect of being able to operate at a temperature higher than the boiling point temperature is due to an increased rate of: diffusion, evaporation and cooling (or condensation). This invariably causes increased contact time between the solvent mixture and the oil bearing material which thus increases solubility and oil yield.

Oil Analysis

The effect of using different solvents (at different combinations) in the extraction of neem oil was considered by analysing the quality of the oil produced. The results of analysis showed that oil extracted was neem oil and that it certified its quality, for the values obtained fall within the standard values.

Table 1: Properties of Oil Extracted

Solvent	Acid Value	Sap. Value	Specific Gravity	pH
Ethanol	19.35	198.45	0.87	5.7
Hexane	20.05	201.25	0.90	6.1
50/50Hex./Eth.	18.79	197.75	0.91	5.9
60/40Hex./Eth.	12.90	199.99	0.92	5.8
40/60Hex./Eth.	17.11	198.39	0.90	5.8

Std Values		**175-205**	**0.9 - 0.93**	**5.5 - 6.0**

IV. CONCLUSION

Ethanol cannot serve as a good substitute to hexane as a solvent, in the solvent extraction of neem oil from neem seeds. This is due to the observed low extraction efficiency (in terms of maximum oil obtainable) of ethanol (even at higher temperatures than that of hexane).

The proportions of ethanol-hexane mixtures (60/40, 50/50, and 40/60% respectively) served as efficient solvent alternatives to the use of hexane only, in neem oil solvent extraction. These mixtures are more environmentally friendly, have a reduced risk of flammability, less toxic and give higher oil yields than both hexane and ethanol. And can be operated at higher temperature.

This should encourage the use of ethanol-hexane mixtures due to the increased extraction efficiency. Another advantage is also the reduced cost associated with the use of hexane only, as a solvent. Further experiments need to be carried out for the implementation on a large scale. To be established by considering more parameters like seed particle size, seed quality, seed and oil compositions.

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