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A brief review on the comparative study of essential oil compounds extracted from lemongrass (Cymbopogon citratus) using Microwave assisted Distillation and Solvent Free Microwave Extraction.

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Abstract— This is a review of a comparative study for the performance of three different essential oil extraction techniques on the grounds of the yield and time of extraction of lemongrass oil (Cymbopogon Citratus) from its leaves using the Microwave assisted Hydro-distillation (MAHD), and Solvent free microwave extraction. Isolation of lemongrass oil with Solvent Free microwave extraction offers a slight advantage over MAHD when collated on the basis extraction yield. Solvent free microwave extraction was found to be superior and produced better oil quality. Solvent free microwave extraction came out as a green technology causing least adverse effects on the environment and yielding top grade oil.

Keywords— Solvent free microwave extraction; essential oil; Microwave assisted Hydro distillation; Lemongrass

Introduction

There are various herbs and plants that are used for various purposes by primitive culture over many years. These herbs and plants are vital resources of aroma, flavours, medicines, essential oils, food additives, and others for daily purpose. Essential oil is a significant product obtained from plant material. They are extracted from roots, leaves, flowers, herbs, barks, etc. The oil is obtained either from extraction or distillation.

Essential oils are concentrated and volatile floral extracts of aromatically potential plants, out of the total species of flora discovered till now only 10% of them bear aromatic potential in different parts such as seed, flower, stem, leaves, roots etc. The amount of volatile oil present varies for different parts of the plant from 0.1 % to 10 %.

Lemongrass is a grass type aromatic plant, it possesses its aromatic valuables in the stems and leaves, having a lemon alike odor. Lemongrass has been exploited as a medicinal plant for more than 2000 years because of the high citral content delivering antimicrobial properties [1]. The oil obtained from lemongrass is one of the most popular sources for natural citral and because of high quantity of citral present it is considered as a great raw material for the pharmaceutical, fragrance, flavor and cosmetic industries. There are more than 200 species of lemongrass examined till now one of which is the Cymbopogon Citratus, it is a specie of lemongrass raised from the subtropical parts of Africa, Asia and South America [2]. Belonging to the family of Poaceae or the grass family with Cymbopogon is the genus. The oil obtained from this specie of lemongrass is called the West India oil [2].

Distillation first came into consideration back in the 9th century AD when the Arabs discovered the process, then in the 13th century utilized by the pharmacies for the production of remedy oils [3]. Till today's time numerous distillation and oil extraction techniques have been invented and innovated in search of an efficient engineering model for the extraction of essential oils

like the traditional method of Hydro distillation, Steam Distillation, Hydro Steam distillation, Microwave assisted Hydro-Distillation, Solvent Free microwave extraction and many more. The amount of time expended, the oil grade acquired, energy invested and cost of operation are the grounds which determines the efficiency of a particular process, if it is worthy enough to fill in for the foregoing method and can be established on Industrial scale. Despite being popular on industrial scale and producing fairly good quality oil conventional methods like hydro distillation have several snags like the phenomena of isomerization of the compounds in the oil due to long distillation cycles destroying compounds sentient to heat, and copious extraction time. Several advance methods have been introduced which prevail over the drawbacks of the traditional methods producing exemplary oil quality in a short period of time, which leads to lower energy consumption and less operating cost. The factor of time has a great dependence on the type of energy source used for the extraction process. Microwave heating equipment's have taken over, providing effective and efficient heating, faster mass and heat transfer, reduced induction time, rapid extraction rate etc. This review is based on the comparison of Microwave assisted hydro distillation and solvent free microwave extraction which utilize microwave as a heating source for the abstraction of Lemongrass (Cymbopogon Citratus) oil in view with the time taken for extraction, the total yield obtained and the cost of extraction.

Over 200 distinct essential oils are utilized globally. The maximum consumed essential oils are of orange, lemon oil, jasmine, eucalyptus, lavender, peppermint, rose, and clove. South Asian countries like India, China are leading producers of essential oils. India is major exporter of lemongrass and sandalwood. 3In European regions like France, Indonesia, Israel, Greece are leading exporters of basil oil.

MATERIAL AND METHODS

Raw Material

Cymbopogon Citratus or lemongrass where Cymbopogon is the combination of "kymbe" (boat) and "pogon" (beard) in Greek referring towards the flower spike arrangement [4]. It belongs to the Poaceae family or the grass family and Cymbopogon is the genus. It is found in the sub-tropical continents of Asia, America and parts of Africa has tall grass like leaves, with tufted roots. They possess volatile oil in their short stems and long leaves, which contain nearly 12% (w/w) of essential oil [3]. Long strap like fragrant leaves of lemongrass is 1.3 to 2.5 cm wide and about 3 feet in length, with dropping tips. They are bright green in colour and release fresh lemony aroma when crumpled. This is a non-flowering plant. Ketones, hydrocarbon terpenes, esters, alcohols and aldehydes are the major constituents found in Cymbopogon citratus, the concentration of these constituents may vary geographically [5]. The Citral concentration decide the quality of the oil, it is a combination of two aldehyde compounds Neral (Citral-a) and Geranial (citral-b) which are stereoisomeric monoterpenes, where Neral is the cis-isomer and Geranial is the trans-isomer [6]. Citral-a and citral-b show antibacterial properties on gram-negative and gram-positive bacteria. The other major isolate of lemongrass oil is myrcene, which did not display any antibacterial properties. Previous researches also confirm antidiarrheal [7], antifungal [8], anti-inflammatory [9], antimalarial [10] and anti-amebic properties of lemongrass essential oil. Lemongrass oil also has numerous health benefits and helps improving menstruation, nausea and digestion. It also had positive results curing muscle cramps, headaches, rheumatism and spasms. The raw material should not be harvested very long the extraction as it will degrade the quality of raw material, dead leaves and parts of stem should be eliminated from the raw material before subjecting it to extraction. The plant material goes through various pre-treatment processes, leading to high yield and quality of oil. A longer time gap between harvesting and extraction may lead to the production of top-grade oil with high citral content but may reduce the quantity of isolates from the oil. Lemongrass leaves are chopped into considerably small parts and are then dumped into the flat bottom flask for better extraction results.

The composition of essential oil majorly depends on the extraction process and the length of time for which the process is going on. One more aspect that influence the

yield of essential oil is the pre-treatment process. Studies were conducted using Cymbopogon Citratus leaves treated with different types of drying methods like Sun-drying for 24 hours, Oven drying for 7 hours, and shed drying for 48 hours.

Researchers concluded that oven drying pretreatment process was most efficient as it produced the maximum yield (2.45 %) followed by shed drying (2.12 %) and then through sun drying (2.10 %) [11]. All the samples were extracted through hydro-distillation method using Clevenger type apparatus.

Varying the size of the raw material fed to the extractor vessel also alters the overall yield percentage of the oil.

Plant material to solvent (water) ratio is also one of the process parameters measured to describe the variation in the yield and how the amount of solvent fed affects the quality and quantity of the oil. The maximum amount of yield recovered was when the plant material to water ratio was 1:8. Further increasing the ratio to 1:10 the yield % declined because of the use excessive water, which resulted in generation of thermal stress in the mixture [12]. Also, when the amount of water used was reduced, a gradual decrease in the yield of the oil was observed by the researchers. These results were carried out using microwave assisted hydro-distillation method.

Microwave assisted Hydro-distillation

Microwave assisted hydro distillation is an evolving and ingenious technique that is used for the extraction of essential oils from plant. The procedure of microwave is related to the irradiation of microwave energy that converts into heat energy. The heat energy is used to heat the solvent and plant material of extraction process thus stimulating the kinetics.

The merits of MAHD process comprise of lesser extraction time, higher quality products, less consumption of solvent, cheaper, and eco-friendly. The utilization of microwave in this technique to extract essential oil started around the year 1980s. With the advancements in the technology of microwave, it has recently appeared as one of the excellent extraction methods that is economically feasible.

The working principle of microwave-assisted hydro-distillation (MAHD) is notably a modified version of the conventional HD. MAHD employs microwaves to heat up the solvent. The boiling flask containing the solvent usually water and the plant material are introduced to a controllable microwave oven commonly operated at 2.45 GHz [13] [14]. The oven cavity contains the raw plant material in the flask, the middle nozzle of the flask is connected to the condenser placed outside the oven cavity. The utilization of microwaves power speeds up the essential oil extraction, which can be accomplished in a few minutes and leads to shorten the time required to achieve the same amount of extract. This method is very interesting for both laboratory and industrial scale applications due to its efficient heating, rapid energy and mass transfer rate, and being environmentally benign. The short time of extraction due to the presence of the microwave as the heating source gives it a huge edge over the conventional methods.

Factors affecting the yield and performance of Microwave assisted hydro-distillation include the nature of the solvent used, raw material to solvent ratio and microwave power.

One of the major disadvantages accounted while using a non-polar solvent in MAHD process as they are poor absorbers of microwave radiations and hence do not provide effective and homogeneous heating to the feed. To overcome this issue modifiers are added to the non-polar solvents improving the absorption capacity of the solvent for microwave heating. Pretreating the raw material with non-polar solvent results in positive outcomes [1]. Surround heating is observed when appropriate amount of matrix to solvent ratio is applied in the extraction process. Water being a good absorber of microwave radiation when used in excess amounts may absorb heat radiations from the microwave and result in unnecessary consumption of additional electrical energy, affecting the economy of the process.

Size of the extracting vessel also affects the pace of extraction, smaller vessels produce high amount of pressure within them, which accelerates the process of extraction, reducing the time of extraction.

Temperature of the extraction process is interrelated to the microwave power offered by the user. Greater the microwave power higher the extraction temperature, but increase in temperature becomes irrelevant after a certain point of time as excessive temperature increase could cause thermal degradation of the heat sensitive constituents found in the lemongrass oil [15].

The mechanism that differentiates conventional hydro-distillation process from microwave assisted hydro-distillation are mass transfer and heat transfer. The extraction in MAHD occurs because of the alteration in cell assembly, due to the action of electromagnetic waves. Direction of heat and mass transfer process is identical which fast-tracks the process of extraction.

Heat dissipation in MAHD occurs volumetrically and not from outside surrounding to the raw material as in Hydro-distillation.

Extraction of Lemongrass (Cymbopogon Citratus) essential oil was carried out using Microwave assisted hydro-distillation technique. Harvested lemongrass plant was subjected to shed drying for 48 hours at normal temperature and pressure conditions. 100 grams of lemongrass material was fed to a two-liter capacity round bottom flask with 200 ml of water. Microwave was operated at a constant power supply of 850 W for 45 minutes. A yield of 1.85 % was obtained with 89.33 % citral content [16]. The research proved the efficiency of MAHD over conventional hydro-distillation.

Solvent free Microwave Extraction

Solvent free microwave extraction is a fundamental integration of microwave heating and dry distillation. This is performed without using any solvent at atmospheric pressure. The separation and consolidation of volatile substances are accomplished in a single stage. The impact of extraction period and microwave radiation on the output and formation of material is examined.

Solvent free microwave extraction as the name suggests is a no solvent extraction process. It operates with no amount of solvent in the processing of essential oils makes this process idiosyncratic from other microwave assisted techniques. This process is an amalgamation of the microwave heating and dry distillation at atmospheric pressure [14]. The raw material is pre-treated with water at normal room temperature before the extraction to moisten the outer surface of the material generally for an hour. Water is considered as the agent of pre-treatment for solvent free microwave extraction. This is done for faster heat transfer from the microwave to the inner glands of the raw material. The flask contains the chopped lemongrass material with no solvent involved and is placed inside the microwave oven cavity, wrapping the flask with PTFE or Teflon to bring down the situations of leakage [13]. This process deals with transferring of heat from microwave to the in-situ water of the plant material which swells up the plant body, making release of the oil easy from the glands. The in-situ water contains oil constituents, which is released through azeotropic distillation process [17]. The condenser is structured at the top of the flask outside the oven where the vapours are condensed and received in the form of a heterogeneous mixture of water and oil, which is then separated by simple decantation and then the oil can be treated with suitable drying agents to remove water traces from the oil [18].

A solvent free microwave extraction process was carried out using a microwave with maximum operating power of 500 W. 150 grams of lemongrass was fed to the PTFE cavity of the microwave, offering homogeneous heating. No solvent or water was added with the raw material. Percentage of citral content was found 72.60%. Total time for the extraction was noted 40 minutes [19]. The use of microwave provides with contact less heating and negligible thermal gradients. Involving no traces of solvent makes this process what we call a “Green Technology” which will leads extraction engineering for a sustainable future. Negligible solvent usage (water) or solvent free, efficient heating, rapid extraction rates, and low investment of energy proves solvent free microwave extraction an ideal extraction technique [1].

COMPARING PERFORMANCE OF MAHD AND SOLVENT FREE MICROWAVE EXTRACTION

The performance of Microwave assisted hydro-distillation and Solvent free microwave extraction were reviewed using the isolation of lemongrass volatile oil. Both the processes are microwave powered, both of the

processes were found to be superior when compared with the traditional methods like hydro-distillation and steam distillation [1]. Both the microwave assisted techniques delivered products in short time and least induction time.

Use of solvent, time of extraction, operating conditions, solvent to feed ratio, operating power of microwave, nature of the lemongrass raw material are the subsets of an effective and efficient extraction technique of Microwave powered extraction units [1]. Microwave assisted hydro-distillation and Solvent free microwave extraction when analyzed, head-to-head multiple observation were made and the processes were collated on the basis of Extraction Yield, extraction Time and the overall cost of extraction.

Comparison on the extraction Yield

The quantity of yield is one of the major aspects that determines the eminence of the process through which the abstraction of the essential oil has been carried out. The yield of the oil is determined by,

$$\frac{\text{Amount of essential oil obtained}}{\text{Amount of raw material used}} \times 100 = \%\ \text{yield of essential oil}$$

The yield of the essential oil produced is conditional on several aspects like the use of solvent, the solvent to raw material ratio, the operating power of the microwave and time. MAHD used water as a solvent with high dielectric properties, also water is a great absorber of microwave radiation [1]. Several organic solvents such as ethanol, methanol and acetone are also used for the extraction purpose. Whereas no solvent was utilized for the extraction of lemongrass essential oil, lemongrass leaves were soaked into water for an hour before the raw material was dumped into the oven cavity. The performance of Microwave operated extraction techniques depend on the power at which the process is taking place. Increase in the power at which the microwave is operating and transmitting heat energy to the feed increases the yield of the system up to a level, further increase in the microwave heating has negligible effects on the yield. It was observed that SFME produced a high yield at low microwave power, but there was no increment in the yield after a certain amount of time. MAHD produced high yield than SFME that is 1.7% in 90 to 120 minutes while SFME produced 1.6% of yield. The amount of citral (geranial and neral) also known as citral-a and citral-b, which determines the purity of the oil was found higher in SFME. Observations were made by previous research work and were concluded in Table 1.

Technique	Solvent to raw material ratio	Microwave power (Watt)	Yield in %	Reference
Microwave assisted hydro distillation	8:1	250 W	1.46	[20] [21]
	5:1	288 W	0.55	
	5:1	464 W	1.5	
	5:1	640 W	1.7	
Solvent free microwave extraction	-	288 W	0.7	[20]
	-	464 W	1.6	
	-	640 W	1.6	
	-	500 W	72.6 % Citral (a+b)	[19]
		850 W		

Table-1: Comparison in Yield

Time of extraction

It was observed that the time of extraction is proportional to the amount of yield produced [22]. Time taken for extraction Solvent free Microwave extraction using microwave as a heating source, ensure rapid extraction, it was observed that most of the lemongrass essential oil was extracted in a short span of 20 minutes, which proves the rapid rate of extraction from SFME [23]. But the yield was nearly constant for the remaining time. MAHD produced

higher yields because of the presence of water as a solvent which ensures effective heating, which raptures the oil glands of the plant material releasing oil. At the extraction time span of 90 minutes highest yield by MAHD was observed [23].

The variation in time of MAHD and SFME process mainly depends on the use of heating media or water during the extraction process. Microwave Hydro-distillation involves the usage of water. Being polar in nature it is a decent absorber of microwave radiations. Water heats up, absorbing the radiations and hence supply heat to the fed lemongrass material, which initiates the course of extraction. Whereas, in Solvent free microwave extraction the plant material is treated with water to increase the moisture content in the plant material and then it is fed to the oven cavity. The in-situ water present in the plant material absorbs the radiations and release the oil present in the feed. The amount of water used in MAHD is more as compared to SFME and hence effective heating is observed in MAHD, promoting less time of extraction.

SFME on the other hand due to internal heating of the water in the plant material, a rapid temperature rise is observed which destroys the plant material and release the oil through the water present in the material itself.

Cost of Extraction

Economics of a process is determined and characterized by several factors like the cost of the raw material, equipment cost, electric power consumed, time of extraction etc. As the heating equipment is same in both the processes, there is no comparison possible on the equipment used. The only factor that differentiates the two processes is the factor of Time taken for the extraction. 20 min of extraction from SFME produced considerably high yield, but MAHD took around 90 to 120 minutes for a yield slightly higher than what achieved by SFME [23] [20].

The cost of production and running of the equipment was found higher in MAHD because of high electricity consumption due to a longer extraction time.

Conclusion

The review paper explains the different essential oil extraction methods and their merits. There is a comparison is shown between MAHD and SFME techniques for lemongrass. MAHD technique is potentially better as compared to some traditional methods as it exhibits an excellent heating effect that stimulates the rate of production of essential oil whereas Solvent-free microwave distillation provides more merits like it is faster, eco-friendly, and more efficient. SFME is more effective than MAHD. Negligible inclusion of organic solvents, rapid extraction rate and fairly high yield of lemongrass (Cymbopogon Citratus) proves SFME as a superior extraction method. Low electricity consumption was observed due to shorter extraction time of SFME, which will prove this method cost effective for large scale production of lemongrass essential oil. Also, because of no solvent involved in the SFME process it was proved to be a Green Technology, causing least adverse effects to the environment.

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