Extraction and Formulation of Perfume from Locally Available Lemon Grass Leaves

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ABSTRACT

The availability of wild plants and their use as economically efficient sources of food for man and animals is obvious. In most parts of Nigeria, plant materials whose edible use is not established are regarded as waste materials. Available materials like lemon grass leaves that contain essential oils can be utilized in the production of perfumes that can mask body odours. Lemon grass extracts obtained using soxhlet extraction and solvent extraction (maceration) were utilized in the formulation of perfume using methanol and ethanol as solvent media. An oil yield of 4.5% and 3.8% were obtained for solvent extraction and soxhlet extraction methods respectively. Physicochemical properties of the two formulations revealed that the essential oil has saponification value of 21.04mgKOH/g and the densities of the two formulations in methanol and ethanol were 0.768gcm^{-3} and 0.82gcm^{-3} at 60°c while the boiling point for both formulations was 85°. The essential oil can be profitably used for cosmetic grade and perfume formulations.

Keywords: Essential oil, Extraction, Perfume, Physicochemical Parameters

INTRODUCTION

Since the beginning of recorded history, many natural and man-made materials have been used in attempt to mask (or enhance) body odor called perfumes. Perfume is a fragrant liquid made from extracts that have been distilled in alcohol and water (Venkar *et al.,*2015). No perfume will scent exactly the same on any two people, because of differences in their body odors, temperature etc (Ranitha *et al.*, 2014).

Venkar *et al*. (2015), defined perfume as extracts or essence that contains a percentage of oil distilled in alcohol. Many ancient perfumes were made by extracting natural oils from plants through pressing and steaming.

Oloyede *(*2009), is of the view that perfumery (the art of making perfumes) began in ancient Mesopotamia and Egypt and was further refined by the Romans and Persians. Traditionally, perfumes were made from plant and animal substances and prepared in the form of water, oils, unguents, powders, and incenses. Most modern perfumes are alcohol-based and contain synthetic scents.

Modern perfumery began in the late 19th century with the commercial synthesis of aroma compounds such as vanillin, which allowed for the composition of perfumes with smells previously unattainable solely from natural aromatics alone (Tajidin *et al.,* 2012).

According to Venkar et al. (2015), perfume usually refers to all forms of fragrances (in general, containing over 15% of fragrance oils) in alcohol.

Techniques involved in perfume extraction from plants include; solvent extraction, distillation and effleurage methods among others. These methods to a certain extent, distort the odor of the aromatic compounds that are obtained from the raw materials (Mancada *et al.*, 2014; Suryawanshi *et al.,* 2016).

Key ingredients required to produce perfume are; essential oils (which is extracted from various organic or non-organic sources), alcohols which dissolves the essential oil and fixatives which combine to give the scent of the perfume.

Essential oils are fragrant materials that have been extracted from a source material directly through distillation or expression; usually obtained in the form of an oily liquid. (Cassel and Vargas, 2006).

Fixatives are any natural substances that help a fragrance last longer on the skin by lowering the evaporation rate of the alcohol as alcohol-based scents are the most fleeting. So a substance is required to add for "anchor" the scent. *Hartman and Ross (2010), outlined that* Natural fixatives are resinoids (benzoin, labdanum, myrrh, olibanum, storax, tolu balsam) and animal products (ambergris, castoreum, musk and civet) while Synthetic fixatives includes substances of low volatility (cyclopentadecanolide, ambroxide, benzyl salicylate) and virtually odorless solvents with

very low vapor pressures (benzyl benzoate, diethylphthalate, triethylcitrate)

According to Chagonda *et al.* (2000), the primary purpose of alcohol in perfume is to cause the perfume oils to evaporate faster than they ordinary would. This gives the perfume a considerably stronger impression than it actually has and also explains why their scent fades dramatically within one or two hours.

Ranitha *et al.* (2014), reported that the lemongrass genus has about 55 species, most of which are native to South Asia, Southeast Asia and Australia. Two major types have considerable relevance for commercial use: East Indian lemongrass *(Cymbopogon citratus)* which is native to India, Sri Lanka, Burma and Thailand, and West Indian lemongrass *(Cymbopogon citratus)* assumed to originate from Malaysia. The plants grow in dense clumps up to 2 meters in diameter and have leaves up to 1 meter long

The reported life zone for lemongrass is 18 to 29 degrees centigrade with an annual precipitation of 0.7 to 4.1 meters with a soil pH of 5.0 to 5.8 (East Indian) or 4.3 to 8.4 (West Indian). The plant grows best in a warm, humid climate in full sun or in a sandy soils with adequate drainage.

Citral, (the aldehyde responsible for the lemon odor) generally determines the quality of lemongrass oil. Some other constituents of the essential oils are terpineol, myrcene, citronellol, methyl heptenone, dipentene, geraniol, limonene, nerol, and farnesol as reported by Ranitha *et al.* (2014).

Amenaghawon *et al.* (2014), on carrying out steam distillation, modeling and kinetic studies (which are not instantaneous), observed that the oil yield was improved by using loose packing of the plant material within the steam distillation equipment.

Maturity stages of lemongrass on essential oil, chemical composition and citral contents influence the essential oil yield, according to Tajidin *et al.* (2012). The effects of appropriate level of maturity determine the quality of essential oil in lemongrass.

Essential oils are different from fatty oils because they evaporate on contact with the air and they possess a pleasant taste and strong aromatic odor. They are readily removed from plant tissues without any change in composition. In addition, they have a complex chemical nature. Cassel and Vargas (2006) outlined the main groups as the hydrocarbon terpenes and the oxygenated and sculptured oils.

Pure essential oils are mixtures of more than 200 components, normally mixtures of terpenes or phenylpropanic derivatives, in which the chemical and structural differences between compounds are minimal (Jack and Raymond, 2010). They can be essentially classified into two groups viz; volatile and non-volatile fractions.

METHODS
Sample Procurement and Treatment
Fresh sample of lemongrass leaves was collected from a garden located at Gusau Road in Sokoto State, Nigeria. The sample was dried, powdered and used for oil extraction.

Oil Extraction
A medium size soxhlet apparatus was set-up with 300g of the powdered sample in a thimble and 600ml of n-hexane solvent in a round bottom flask. The solvent was heated at 65°c gently with the aid of a heating mantle. The vapour passing through the vapour tube was condensed by the condenser, and the condensed hot solvent dropped into the thimble containing the sample. As it reaches the top of the tube, it siphoned over into the flask. The process continued until complete extract was obtained. In order to remove the excess solvent from the natural essential oil, the extract was heated on water bath at 60^0 for 2 hours.

Solvent Extraction (Maceration) Method
Powdered dry lemongrass leaves (300g) was added to 600ml of the solvent (n-hexane) in a 1000ml flask and stopped. In order to completely extract all the oil in the lemongrass, the content was allowed to stand for 38hrs; after which the extract was decanted into a 500ml beaker. Ethanol (200ml) was added to dissolve the extract. The mixture was transferred to 1000ml separating funnel and allowed to come to equilibrium, which separated into two layers. The lower ethanol extract and the upper hexane layer were carefully separated, collected into two separate 250ml beakers and placed in a water bath at 60°. This was done to remove the excess ethanol leaving only the natural essential oil.

Determination of Percentage Oil Yield
The yield of oil was determined by weighing the extract on an electronic weighing balance. The difference between the final weight (weight of the beaker with extract) and the initial weight (weight of the empty beaker) gave the weight of essential oil. The percentage oil yield was calculated using the method of Sanger *et al.* (2011), based on equation 1 below.

$$\% \ oil \ yield = \frac{M.E}{M.L} \times 100 \ -------------------1$$

Where M.E = Weight of Essential Oil, M.L = Weight of Lemongrass Sample

Determination of Saponification Value

Saponification, referred to as sap, is one of the important physicochemical parameters which estimates the number of milligrams of potassium hydroxide KOH, required to saponify 1gram of fat and/or biodiesel.

Twenty five (25ml) of ethanoic potassium hydroxide (0.1M) was pipetted and mixed with 2g of essential oil sample; the mixture was then heated gently to emulsify the oil sample in the ethanoic potassium hydroxide. Few drops of phenolphthalein (1% w/v) was added, and the mixture was titrated against HCl (0.1M) with constant shaking until the disappearance of purple color.

Blank titration was also conducted in the same way, but without adding the oil sample such that the saponification value could be computed. The saponication value was computed using the method of Elinge *et al.* (2013), based on equation 2.

$$Saponification\ Value = \frac{56.1 \times M(VA - VB)}{W} - - - - - - - - - - - -2$$

Where

M = concentration of acid used; V_B = volume of acid during blank titration;
V_A = volume of acid when essential oil titrated against 0.1M HCl; W= weight of sample.

PERFUME FORMULATION

Using Methanol

Lemongrass essential oil extract (15ml) was measured in a beaker containing 5ml of methanol. To improve the longevity of the perfume, benzyl salicylate (5ml) was added to the mixture. The solution was shaken and poured into a 50ml bottle.

Using Ethanol

Lemongrass essential oil extract (15ml) was measured and placed in a beaker containing 5ml of ethanol. Benzyl salicylate (5ml) was added to the mixture to improve the longevity of the perfume. The solution was shaken and poured into a 50ml bottle.

Determination of Perfume Density

The density of each formulation was carried out based on the principle of Elinge *et al.* (2013), which was calculated according to equation 3.

$$Density = \frac{mass}{volume} = \frac{W_1 - W_0}{V} - - - - - - - - - - - - - - - - - 3$$

Where

W_1 = Weight of Beaker and Perfume; W_0 = Weight of Empty Beaker; V = Volume of Perfume.

Determination of Perfume Boiling Point

Fraction of the two perfumes formulations (30ml each) was measured and transferred into a beaker placed on a heating mantle at 60^{0}C. The boiling points of the two formulations was recorded using a thermometer.

Determination of Boiling Point

The temperature at which the vapor pressure of the liquid equals the external pressure surrounding or the temperature at which it changes state from a liquid to a gas throughout the bulk of the liquid the method of Edward (2007) was adopted in determining the boiling point.

RESULTS

The result shows weight of the lemongrass extract obtained by solvent extraction method with time. The weight of oil (g) obtained from the lemongrass extract with time is presented in Table 1.

Table 1: Weight of Oil Recovered Using Soxhlet Extraction and Solvent Extraction (maceration) with Respect to Time

Weight of oil Recovered (g)		Time (minutes)
Soxhlet Extraction	**Solvent Extraction**	
3.12	3.50	180
2.90	2.86	360
2.53	2.35	540
1.21	1.90	720
1.15	1.65	900
0.95	1.24	1200

Table 2: Percentage Oil Yield Recovered from Solvent and Soxhlet Extraction Methods.

Method of Extraction	Oil Yield (%)
Solvent Extraction (maceration)	4.5
Soxhlet Extraction	3.8

Table 3: Physicochemical Parameters of Oil Extract and Perfume Formulations.

Parameter(s)	Value	Methanol	Ethanol
*Density g/cm^3		0.768	0.82
*Boiling point (°C)		85	85
**Saponification Value (mgKOH/g)	21.04		

Values(s) with asterisk (*) were determined after perfume formulation, double asterisk (**) were determined before perfume formulation.

DISCUSSION

Table 1 shows the weight of oil obtained from solvent extraction with time. It can be seen that the weight of extract decreases with time, showing that the solvent (hexane) evaporated with time resulting in weight loss of the content and increase in oil content with time. The oil yield obtained (13.5g essential oil per 300g of dry lemongrass leaves) with 4.5% is higher than (2.9 g of essential oil per 100g of dry lemongrass leaves) and (3.1g of essential oil per 140g of dry lemongrass leaves) reported by Suryawanshi *et al.* (2016) and Ronak *et al.* (2017) respectively. Differences in the oil yield could be attributed to variation in methods used, specie of the lemon grass sample, temperature of extraction, geographical location etc. The weight of extract was measured at every 3hr interval to determine the oil yield at varying time, as the time increases the Ethanol solvent reduces thereby leaving the essential oil in the mixture.

The result of essential oil yield produced by soxhlet and solvent extraction methods was presented in Table 2. Exactly 3.8% oil yield per 300g of dry lemongrass leaves at 60^{0}C, obtained for soxhlet extraction appeared higher than 3.1g of essential oil per 140g reported by Ronak *et al.* (2017) for dry lemongrass leaves. This could be due to either method adopted, sample quality or instrumental error etc. To determine the oil yield at varying time, the weight of extract was measured at every 3hr interval. As the time increases the Ethanol solvent reduces thereby leaving the essential oil in the mixture. Solvent extraction method produced an oil yield of 4.5% which is 0.7% more than 3.8% obtained for soxhlet extraction. The higher oil yield obtained could be due to exposure of the sample to the bulk of the solvent or due to high contact time as the powdered sample soaked in an air tight container for 38hrs after which the mixture was separated. It could be presumed that the best method in the extraction is solvent extraction method because it gave more oil yield than soxhlet extraction method. This conforms to works done by Suryawanshi *et al.* (2016).

Table 3 shows the result of some physico chemical parameters determined for both the oil extract and perfume formulations using ethanol and methanol. s 21.04mgKOH/g this value shows that it has a lower molecular weight which is due to lower density. It is a measure of average molecular weight or chain length of all the fatty acid present. The long chain fatty acids found in fats have a low saponification value because they have a relatively fewer number of carboxylic functional group per unit mass of the fat as compound to short chain fatty acid.

Recent report by Mukhtar and Dabai (2016), captured a density of 0.94g/cm^3 for soybean oil which is higher than 0.768g/cm3 and 0.82g/cm3 obtained for perfume formulations using methanol and ethanol solvents respectively.Furthermore, the boiling point of the two perfume formulations (85^{0}C), is the same with 85^{0}C reported by David (2004), and less than 100^{0}C reported by John and Julian (2001), for water.

CONCLUSION

The lemongrass essential oil was successfully extracted from dry lemongrass leaves using solvent extraction and soxhlet extraction methods, although solvent extraction method gave higher essential oil than the soxhlet extraction method, both methods prove effective for essential oil extraction. The essential oil was used successfully in perfume formulation by using fixatives and different solvents (alcohols) as ingredients.

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