Screening and Extraction of Essential Oils from Different Indigenous Non-food Plants for Detergent Production

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Abstract: In this study, an alternative plant oil is investigated for substitution of pine oil, which is the most expensive ingredient in pine gel detergent. Firstly a headspace sampling technique known as BID-SDME was optimized for the initial screening for presence of essential oils in the plants as to compare their profiles with that of the commercial pine oil. The parameters optimized were as follows: use of 1 µL heptadecane with 0.5 µL of an air-bubble as an extraction solvent, 55 °C sampling temperature, 15 minutes sampling time and 1500 µL sample volume. Following the screening of the potential plants, oil was extracted from different locally growing non-food plants using a modified steam distillation for comparison of oil content. Of the screened plants, *Tagetes minuta* known locally as either *monkhane* or *lechuchutha* resulted in the highest yield of 0.83 -1.35% while sewage algae and pine leaves resulted in yields of 0.28 - 0.44 and 0.54 - 0.97% (v/m) respectively. *Tagetes minuta* oil was then used for detergent preparation. However, the detergent could not set the same way as the pine gel counterpart and displayed higher CMC (0.002219 mol/L) compared to that of the pine gel 0.001691 mol/L demonstrating a somewhat lower detergency. Despite these challenges, *T. minuta* still demonstrated a potential that will lead to improved profit margin should the optimization be carried out further.

Keywords: Pine gel detergent, pine oil, *Tagetes minuta* oil, Critical Micelle Concentration, fluorescence.

1. Introduction

As a means of counteracting unemployment in developing countries like Lesotho, individuals tend to go in the commercial sector and become entrepreneurs. However capital investment still remains a daunting challenge. Detergent production on a small scale is one of the processes that do not require complex capital investments as it generally involves simple mixing of reagents. Some of the reagents are industrially produced and are cheaper while others are obtained directly from nature and get to be very expensive. For instance, pine oil is an ingredient of Pine gel detergent and it is produced as a by-product in wood production and this makes it very expensive thus lowering profit margins in the detergent production.

This was a problem experienced by one of the local detergent manufactures in Leribe and the basis of the study was to help find a solution to the problem. Pine gel is an effective, fast working, environmentally friendly and most versatile all-purpose disinfectant, detergent and sanitizer that is made from pure and natural pine-oil that when applied leaves behind a long lasting fresh pine forest fragrance due to the presence of β-Phellandrine and β-Pinene monoterpenes that result in a pleasing aroma [1]. Pine gel is usually formulated by mixing controlled amounts of surfactant e.g linear alkyl benzene sulphonic acid then neutralizing with caustic soda then adding pine oil. Mixing linear alkyl benzene sulfonic acid with caustic soda solution neutralizes the acid hydrogen of the sulfonic acid with the hydroxide ion in the caustic soda solution resulting in linear alkyl benzene sulfonate salt. This compound has two parts attached chemically; one is the sulfonate which is hydrophilic and attaches to water molecules and the other is the linear alkyl benzene group that attracts the pine oil. This ability to attract both water and the pine oil is what results in a gel.

The aim of the study was to find a cheaper and easily accessible alternative for pine oil (the most expensive reagent in the pine gel detergent) from locally available non-food plants while still maintaining similar properties in the detergent. The plant *Tagetes minuta* locally known as *lechuchutha* or *Tagetes minuta*, blackjack in English, belongs to the Asteracae family. The species is an invasive weed and aggressively colonizes disturbed areas and fallow farmlands [2]. The plant in Lesotho is reported to be used as an insecticide and applied to crops mixed with other local plants. The plant also possesses anti-microbial properties which are similar to those that pine oil possesses and this plant oil may serve as alternative to pine oil [2].

Since there is a direct interaction between the oil and the surfactant molecules, it is crucial to determine the efficiency of the newly produced detergent in comparison to that of pine gel. This is usually determined by the critical micelle concentration. Micelle are spontaneously formed aggregated units with the hydrophobic ends forming the core while the hydrophilic ends form a surface hydrated by water molecules [3]. It is the presence of micelle that results in the detergency. Critical concentration is defined as the minimum concentration of molecules at which the intermolecular hydrogen bonding, micelles, or other aggregates start forming. The value of the CMC can be determined by the change in the physicochemical properties of the surfactant solution as the surfactant concentration increases. Ideally low CMC values of surfactants are favorable [4].

The 1:3 ratio is one of the commonly used procedures for the CMC determination, it was initially used for determination of pure micellar solutions but has also found application in mixed surfactant systems. This method makes use of the dependence of the fluorescence vibrational structure of pyrene on solvent polarity. This method makes use of the intensity of the ratio of the 1st and 3rd vibrational peaks hence the name (1:3 ratio). Below the CMC the pyrene 1:3 ratio value corresponds to a polar environment of the water molecules; as the surfactant concentration increases the pyrene 1:3 ratio decreases rapidly, indicating that the pyrene is sensing a more hydrophobic environment since it is will be enclosed into the core of the micelle. Above the CMC, the pyrene 1:3 ratio reaches a roughly constant value because of the incorporation of the probe into the hydrophobic region of the micelles [5].

2. Materials and Methods

Plant harvesting and screening of plant volatiles:

Plant samples were harvested by plugging off the plant leaves the same day of the extraction to prevent loss of plant volatiles that may occur during the storage of the plant and were washed thoroughly with water. A weighed mass of plants and a measured amount of oil was placed in a vial and tightly closed to prevent any plant loss during the extraction. The extraction of plant volatiles was achieved through the BID-SDME method initially described by Williams et al [6] using 1 µL heptadecane with 0.5 µL of an air-bubble as an extraction solvent, 55 °C sampling temperature, 15 minutes sampling time and 1500 µL sample volume.

Essential oil extraction:

Plant samples were harvested by plugging off the plant leaves the same day of the extraction to prevent loss of plant volatiles that may occur during the storage of the plant and were washed thoroughly with water. Plant leaves were weighed and ground using a blender and added to a distilling flask then water was added for extraction and placed on a heat source. After a 3 hour extraction time elapsed, diethyl ether was added into the receiving flask to allow further separation of the oil from the water. The ether was then evaporated and oil was obtained.

GC-MS Analysis:

A Shimadzu QP 2010 GC-MS fitted with a Zebron ZB-5MS column (30 m x 0.25 µm ID x 0.25 mm film thickness) was used for the analysis. One µL of each oil sample was directly injected into the GC at a split ratio of 300:1 at an inlet temperature of 250 °C. Similar oven conditions as those of GC were applied with few

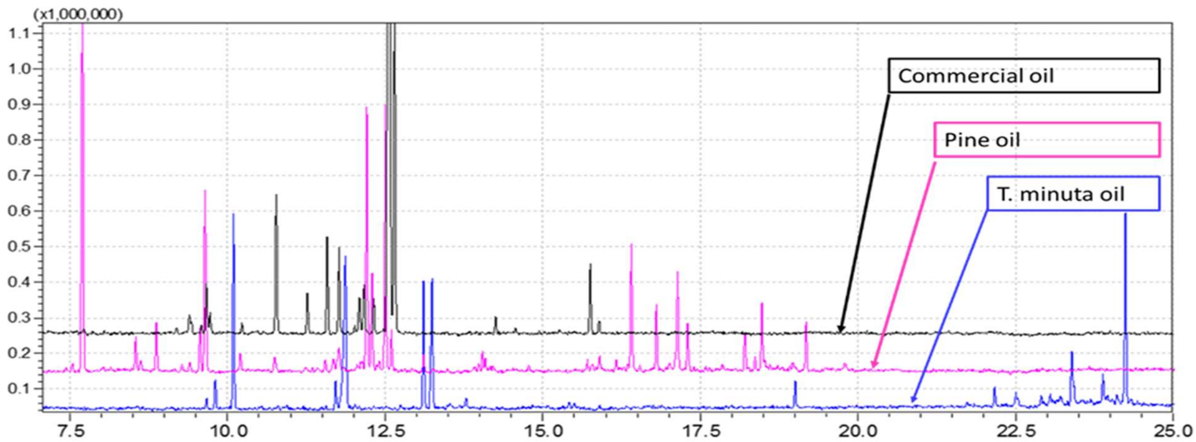

Figure 1: Total ion GC-MS Chromatogram of extracted oils (a) *Tagetes minuta* (b) pine oil.

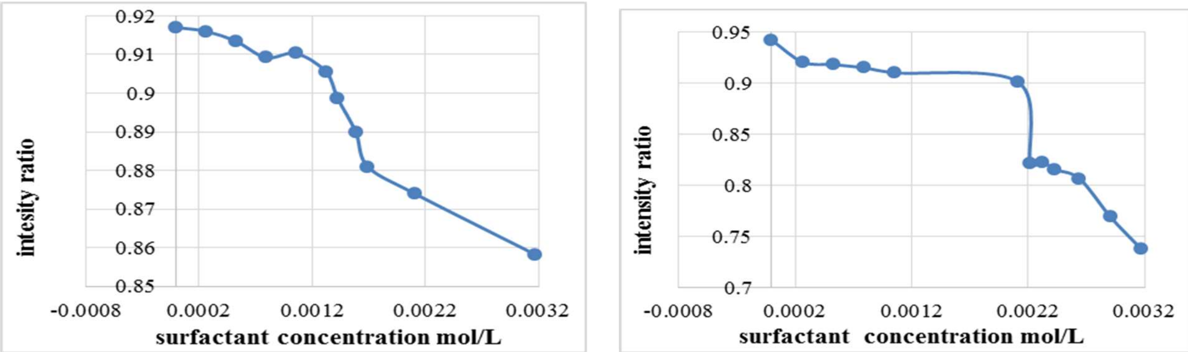

Figure 2: Graphs of fluorescence intensity versus surfactant concentration for the determination of critical micelle concentration. (A) Pine Gel, and (B) *Tagetes minuta*..

variations as follows. Helium was used as a carrier gas at a flow rate of 1 ml/min. For MS detection, ionization was achieved by electron impact at 70 eV and the scan range was from 50 – 350 m/z. The compounds were identified using the NIST library.

Fluorescence analysis:

Fluorescence measurements were performed on a Shimadzu RF-1501 Spectrofluorophotometer. Fluorescence emission spectra of a number of surfactant solutions containing 50 µL of 0.025 M of pyrene were recorded using an excitation wavelength of 335 nm, and the intensities I1 and I3 were measured at the wavelengths corresponding to the first and third vibronic bands located near 373 and 384 nm. The ratio I is the so-called pyrene 1:3 ratio. All fluorescence measurements were carried out at 25.0 ± 0.1 °C. The excitation and emission slit width were adjusted to 10 nm and the scan speed was fast with high detection sensitivity.

3. Results and Discussion

GC-MS analysis:

Figure 1 shows only seven major compounds in Tagetes minuta and it is in accordance with the one in literature. Pine oil chromatogram shows more peaks when compared to that of *Tagetes minuta*, indicating that pine oil contains more compounds than Tagetes minuta despite the latter's oil being coloured while that of pine tree is colourless.

Properties of the prepared detergent:

Tagetes minuta gel is slightly thinner than pine gel and this is may be due to presence of fewer essential oils in *Tagetes minuta* while pine oil has more and this was seen from the chromatograms. It has yellow in color while pine gel colorless without any dye used post processing. It takes a longer time to set however upon addition of a few drops of pine oil the setting process takes a shorter time. It has also a very strong scent that is characteristic smell of the *Tagetes minuta*

Detergent efficiency determination by CMC method:

The plots in figure 2 illustrate the efficiency determination by fluorescence of both detergents from the inflection points. Pine gel had a lower CMC value of 0.001691 mol/L while *Tagetes minuta* had a CMC value of 0.002219 mol/L. This is a reflection that pine gel has a better detergency than *Tagetes minuta* gel, however this has not compromised detergency of the product.

4. Conclusions

Plant oils from algae, pine and *Tagetes minuta* were extracted; *Tagetes minuta* oil (with higher oil yield) was used in the detergent preparation. The efficiency of the new detergent was determined in comparison to that of the pine gel and was found to be lower than that of pine gel.

5. References

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