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Theme:

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EXTRACTION AND PURIFICATION OF OIL FROM GARLIC BULB

by

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ABSTRACT

The aim of this research work is to extract and refine essential oil from garlic seed. Soxhlet extractor was used for the extraction purpose with hexane as the solvent. The extraction yielded 2.04% of oil from the garlic seed. The oil extracted was subjected to refining process by degumming, neutralization and bleaching using inactivated clay as adsorbent. Different physico-chemical parameters for both the crude and refined extracts were determined. The pH, specific gravity, refractive index, Saponification value, peroxide value and iodine value were recorded as 5.62, 0.9038, 1.463, 208.97mgKOH/g, 0.5meq/kg and 79.31gI$_2$/g, respectively for the crude garlic oil and 5.83, 0.9027, 1.459, 204.76mg KOH/g, 0.1meq/kg and 76.77 gI$_2$/g respectively for refined garlic oil. The characterization analysis revealed that tested parameters, were within the standard values except for the saponification and specific gravity of crude garlic oil.

KEYWORDS: Garlic Seed, Solvent Extraction, Essential Oil

INTRODUCTION

Garlic resembles onion except that it has flattered solid leaf blades and produces a composite bulb, consisting of several small densely crowded, angular, bulbets or cloves enclosed within the white, pink or purplish skin of the parent bulb. Each clove is derived from the auxiliary bulb of the younger foliage leaf and consists of a protective cylindrical sheath a single thickened storage leaf and a small central bud. Essential oils are commonly known as natural ingredients for cosmetics and pharmaceuticals. In the past they were regularly used in ancient Rome, Greece, Egypt and through out the middle and Far East for their essence as perfumes, food flavours, deodorants, pharmaceuticals and embalming antiseptics. Essential oils are concentrated aromatic plant extracts from certain species of flowers, grasses, fruits, leaves, roots and trees. Examples of essential oils are oil extracted from garlic, ginger, lemon grass, Eucalyptus Oil, black pepper and clove oil etc.(Gernot, K, 2005, Iwalokun, et-al, 2004)

The oils are collected mainly through a process of steam distillation, concentrated and used as perfume and food flavouring scents. They have a long tradition of providing a variety of therapeutic benefits and many of the traditionally known benefits have been confirmed through modern scientific research. It has been established that the use of essential oils in a cosmetic has an antiseptic and antimicrobial action as well as healing and soothing effect on the skin. The oils are called essential oils because they are essential to life and essence to the plant. The first isolation of a component compound of garlic took place in 1844 when German chemist identified diallyl disulfide in distilled garlic oil. One hundred years later, an American research team extracted a macerate of the frozen plant with acetone and found an odourless residue free of organosulfides, which when water was added produce the typical garlic odour. Later a Swiss team identified a precursor to the odiferous principal in garlic and the mechanism leading to its release. (Block, 1985, Ransom et-al, 1992, Balch, 2000)

The aims of this research work include extraction of essential oil from garlic seed through solvent extraction process; purification and characterization of the crude and refined garlic oil for easy identification, and also to assess its quality. Due to the increasing cost of drugs and also to the continuous spread of multi drug resistant organisms that causes diseases which has become a serious threat to public health, there is need to look for an alternative and cheaper source of treatment or drug that is readily available and can also perform equal or more task than synthesized drugs. The extent to which oil will be removed from a fixed mass of solid (garlic) is dependent on Solvent flow rate, particle size, solvent chosen, temperature and agitation of the fluid.

METHODOLOGY

EQUIPMENTS AND MATERIALS

TABLE 1: LIST OF INSTRUMENTS AND EQUIPMENTS

INSTRUMENTS/EQUIPMENT	*MANUFACTURES*
Soxhlet extraction apparatus	QUICK FIT, ENGLAND
Mortar and pestle	Locally Made (Nigeria)
Oven	GALLENKAMP. GERMANY
Digital Weighing balance	BRIAN-WEIGH, ENGLAND
Heating Mantle	ENGLAND
Thermometer	ZEAL ENGLAND
Stop watch	SMITHS, GERMANY
PH meter	CORNING LTD, USA
Visco tester	GLADON, JAPAN
Refractometer	BELLINGHAM STANLEY
Density bottle	PYREX, ENGLAND
Beaker, Measuring cylinder	PYREX, ENGLAND
Conical, Round bottom, Flat bottom flask	PYREX, ENGLAND
Burette/ Pipette/ stand/ filter paper/ funnel	PYREX, ENGLAND

TABLE 2: LIST OF CHEMICAL AND MATERIALS

CHEMICALS/MATERIALS	*MANUFACTURES*
Garlic seeds	Central Market, Minna
Distilled water	Fisheries Lab, FUT Minna
Hexane	May and Baker Limited, England
Potassium Hydroxide (KOH)	May and Baker Limited, England
Sodium Hydroxide (NaOH)	May and Baker Limited, England
Phenolphthalein	May and Baker Limited, England
Ethanol	May and Baker Limited, England
Hydrochloric Acid (HCl)	May and Baker Limited, England

EXPERIMENTAL PROCEDURE

The process for the extraction and purification of oil from garlic seed is divided into three stages; these are preparatory stage (pre-treatment), extraction stage and the purification stage. Decorticating the seed was the first step of pre-treatment and this involves the removal of the outer layer or skin from the garlic bulb. Then the other impurities were separated. The clean seed was then dried in an oven at temperature of 100°C for a period of 3 hours to remove the moisture content of the bulb, the bulb was weighed before it was placed in the oven and thereafter the weight was re-taken at an interval of 1 hour. This procedure was repeated until constant weight was obtained (Amoo et-al, 2004)

Extraction of oil using soxhlet apparatus with hexane as solvent

The dried garlic bulb/seed was crushed in a mortar. This is to offer a greater surface area, so as to increase the yield of oil. 20g of dried garlic sample was weighed and transferred into the thimble after which the weight of sample and the thimble was noted. The thimble containing the sample was covered with a cotton wool before it was inserted into the extractor. A known volume of the solvent hexane (250ml) was poured into the round bottomed flask. The extractor was then connected to a flask at its bottom and to a condenser above it, which was already clamped to a retort stand. In addition, the condenser was already connected to 2 pipes. One pipe connects it to a water supply and the other removes water from the condenser. The soxhlet apparatus was heated by a heating mantle set at 40°C. The heating made the hexane to evaporate and condense by a condenser into the extractor, of which a golden yellow colour of solution was observed in the extractor.

This coloured solution rose up to the level of extractor capillary tube and then flows down through the capillary tube into the flask. The coloured solution observed to be a mixture of the oil and solvent remains in the flask. Still, the solvent evaporates from the flask leaving the oil with some of the solvent. This process continued for five hours (5 hrs), until it was observed that the solvent condensing in the extractor formed no coloured solution. The process was stopped and the apparatus was disconnected, thimble containing the sample was removed from the extractor. The apparatus was connected back without the thimble, for recovery of the solvent and to obtain the oil. The flask was heated at 40°C for the solvent to evaporate from the flask and condense into the extractor, but this time the condensed solvent in the extractor was not allowed to reach the level of the capillary tube of the extractor, because the solvent will reflux back into the flask to begin the process of solvent recovery all over. So when it reached two-third (?) of the capillary tube height the extractor was removed and the solvent inside it was poured into the measuring cylinder and the volume was noted and recorded, then the solvent was poured into the recovery bottle. This process was continued until the solvent was recovered and only the oil was left in the flask with very minute amount of solvent.

The flask containing the oil was removed and dried on a hot plate set at 100°C for 30 minutes to remove some of the solvent the oil retained. Then the weight of oil together with flask and the oil were noted and recorded respectively. Each time the extractor was disconnected in the process, the temperature of the heating mantle was reduced to 0°C, which minimized the escape of the solvent. At the end of the recovery, the thimble was filled with fresh sample and the thimble then placed back into the extractor. The process was repeated for 5 (five) fresh samples. (Lew, 1990 Amoo et-al, 2004,)

Purification of garlic seed oil

Degumming

Distilled water was heated in a beaker and allowed to boil for 20 minutes. The garlic oil was degummed by the addition of hot distilled water. The mixture was stirred for 2 minutes and allowed to stand in the separating funnel. Thereafter, the aqueous layer was removed. The procedure was repeated to ensure removal of the residual gums. (Block, 1985)

Neutralization

In the neutralization process, the degummed oil was poured into a beaker and heated to about 80°C, after which 2ml of 0.1M NaOH was added and stirred to a uniform solution. Sodium chloride about 10% of the weight of the oil was added to help settle out the soap formed. This was transferred into a

separating funnel and allowed to stand for 20 minutes; the soap formed settles at the bottom of the beaker and the oil was decanted leaving the soap. Hot distilled water was added continuously to the oil solution until the soap in solution was completely removed. The neutralized oil was then drawn off into beaker. (Lew, 1999 and Amoo et-al, 2000)

Bleaching

20g of neutralized oil was poured into a beaker and 3g of activated charcoal, about 15% by weight of oil was added. The mixture was stirred, and heated to 90°C. The temperature was allowed to rise to 110°C for another 30 minutes. The content was filtered hot with a filter paper at about 70°C.This process was repeated twice to clearly remove the dark brown colour of the crude garlic oil. (Amoo et-al, 2000)

Characterisation of garlic oil

Specific Gravity determination

An empty beaker was weighed. A specific volume of distilled water was added to the beaker and weighed. The beaker was emptied and dried. An equal quantity of oil was put in the beaker and weighed. The weight of the oil divided by the weight of equal volume of the distilled water gives the specific gravity. (Block, 1985)

Refractive index determination

The refractive index was determined using a refractometer. This was done by placing a few drops of the oil sample on the face of the refractometer. The refractive index was read after a few minutes. (Amoo et-al, 2000)

Iodine value determination

The method specified by International Standard Organisation (ISO) 3961 (1989) was employed. 0.2g of garlic oil sample was weighed into a conical flask, 20ml of carbon tetrachloride and 25ml of DAM's reagent were added to the flask. A stopper was fixed and the content of the flask was vigorously swirled. The flask was then placed in the dark for 1 hour 30 minutes. At the end of the time, 20ml of potassium iodide solution and 150ml of water were added. The contents of the flask was titrated with 0.1mol sodium thiosulphate solution until the yellow colour due to iodine has almost disappeared .Few drops of starch solution were then added and titration continued until the blue colour disappeared after vigorous shaking. The same procedure was repeated for the refined garlic oil and blank test. (Block, 1985)

Saponification value determination

The method specified by International Standard Organisation (ISO) 3657, (1988) was employed. 2g of the garlic oil was weighed into a conical flask; 25ml of ethanolic potassium hydroxide was then added with the aid of a pipette. The flask was attached to a reflux condenser and placed on an electric heater. The contents of the flask were allowed to boil gently for 60 minutes with shaking from time to time. 1ml of phenolphthalein indicator was then added to the flask and the content of the flask titrated with 0.5M Hydrochloric acid until the pink colour of the indicator just disappeared. The procedure was repeated for the refined garlic oil sample and for blank test. Two determinations were carried out on each test sample. (Amoo et-al, 2000)

Peroxide value determination

The method specified by international standard organization (ISO) 3960 (1975) was employed. The experiment was carried out in diffused daylight. 2g of sample was weighed into a 500ml conical flask, 10ml of chloroform was added to dissolve the sample quickly by stirring, then 15ml of acetic acid was added and 1ml of freshly prepared saturated potassium iodide solution was added. The flask was then closed immediately, stirred for 1 minute and kept for exactly 5 minutes away from light at room temperature. 75ml of water was added as indicator. The liberated iodine was then titrated against 0.01M sodium thiosulphate solution. The same procedure was carried out for other samples and the blank test was carried out by the same procedure by omitting test sample.(Block, 1985)

RESULTS

The data obtained from the experiments were tabulated in Table 4. While in Table 5 and 6, comparison is made between the properties of crude and refined Garlic with the standard values for oil.

TABLE 4: THE EXPERIMENTAL RESULT

Percentage yield of oil	2.04%
Solvent recovery	73.6%
Percentage Moisture content	62.9%

TABLE 5: PHYSICAL PROPERTIES OF CRUDE AND REFINED GARLIC OIL

Property	*Crude garlic oil*	*Refined garlic oil*
Colour	Dark Brown	Pale yellow
Odour	Unpleasant smell of garlic	Weak smell of garlic

TABLE 6: COMPARISON OF THE PROPERTIES OF THE CRUDE AND REFINED GARLIC OIL WITH STANDARD

Property	*Standard Value*	*Crude Garlic oil*	*Refined Garlic oil*
Specific Gravity	0.901 – 0.9034	0.9038	0.9027
Refractive index	1. 452 – 1.470	1.463	1.459
PH	5.3 – 6.4	5.62	5.83
Saponification Value (mgKOH/g)	185 – 206	208.97	204.76
Iodine value (g I_2 /100g of Oil)	70 – 90	79.31	76.77
Peroxide value	< 10	0.5	0.1

DISCUSSION

The garlic oil was extracted using the soxhlet extraction equipment with hexane as solvent which gives an average yield of 2.04% of oil. Six extraction runs were conducted and total quantity of oil extracted was 2.452g with 73.6% of the total solvent recovered. From Table 5 which shows the physical properties of crude and refined garlic oil, the colour of the crude oil was observed to be dark brown, while the refined oil is pale yellow; this is as a result of bleaching which removes the colouring materials present. The values of specific gravity for both crude and refined oil were found to be 0.9038 and 0.9027 respectively as shown in Table 6. The value for refined oil was found to be in range of the standard, while the value of the crude oil was found to be outside the recommended standard range of 0.901 - 0.9034, this deviation in value of the specific gravity of the crude garlic oil is due to the impurities present which adds to the weight of the crude sample. The pH value of the crude oil, 5.62 shows that the oil is more acidic when compared to 5.83 obtained for the refined oil. This may be as a result of the purification process. The refractive index which is used in detecting adulteration in fats and oil shows there is a variation in the value of the crude and refined oil, the refractive index of the crude garlic oil 1.463 and that of the refined oil, 1.459.

The chemical analysis carried out includes the saponification value which is also used to check adulteration and to indicate fats and oil that can be used for industrial purposes. Fats and oil with low saponification value may not be useful in the industry (Amoo *et al.*, 2004). The value of the crude and the refined is 208.97 mg KOH/g and 204.76 mg KOH/g respectively. It was found that the saponification value of crude garlic oil is not in line with the standard value. This shows that, for the crude oil, more alkaline would be required to enable it neutralize the available free fatty acid librated by the oil, when compared with the refined oil.

The peroxide value is used as an indicator of deterioration of oils. Fresh oils have values less than 10 meq/Kg. Values

the crude oil is 0.5 while that of the refined garlic oil is 0.1, the variation in peroxide value is attributed to the alkaline treatment of the crude oil.

The iodine value is also an index for assessing the ability of oil to go rancid [1, 3]. This is the measure of the proportion of unsaturated acid present. The iodine values for the crude and refined are 79.31 and 76.77. The crude and refined oil could be classified as a non-drying oils, since their iodine values are lower than 100 [7]. However there is no iodine present in the oil but test measure the amount of iodine, which can be absorbed by unsaturated acid.

The above results obtained are subjected to experimental error during the course of the analysis.

CONCLUSION

The extraction and refining of oil from garlic seeds was successfully carried out using soxhlet apparatus with hexane as the solvent which gives an average yield of 2.04% and 73.9% solvent recovery. The garlic extract was purified by degumming, neutralizing and bleaching in order to remove impurities and undesired substances present in the oil. The physicochemical analysis showed specific gravity of 0.9038, PH of 5.62, Refractive index of 1.463, saponification value of 208.97mgKOH/g, peroxide value of 0.5, Iodine value of 79.31 for crude garlic oil and 0.9027, 5.83, 1.459, 204.76mgKOH/g, 0.1, 76.77 are the values of specific gravity, PH, Refractive index, saponification value, peroxide value, Iodine value for refined garlic oil respectively. Many of physicochemical properties of both the crude and the refined oil compared favourably with standard values except for the saponification and specific gravity of crude garlic oil.

The seed oils therefore have potential for development for use as domestic and industrial oils.

RECOMMENDATION

1. Garlic oil compared to other oil bearing seeds contains very little percentage of oil. Therefore it is recommended that further research should be carried out on improving the yield of the oil.
2. The use of adsorbent other than clay that can be sourced locally can also be used for further research work to assess the bleaching performance
3. The use of other non-toxic solvent other than Hexane e.g petroleum ether, Diethyl ether, and methanol chloroform should be investigated.
4. The deodorization of garlic oil should also be investigated.

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