



OCCASION

This publication has been made available to the public on the occasion of the 50th anniversary of the United Nations Industrial Development Organisation.

TOGETHER

for a sustainable future

DISCLAIMER

This document has been produced without formal United Nations editing. The designations employed and the presentation of the material in this document do not imply the expression of any opinion whatsoever on the part of the Secretariat of the United Nations Industrial Development Organization (UNIDO) concerning the legal status of any country, territory, city or area or of its authorities, or concerning the delimitation of its frontiers or boundaries, or its economic system or degree of development. Designations such as "developed", "industrialized" and "developing" are intended for statistical convenience and do not necessarily express a judgment about the stage reached by a particular country or area in the development process. Mention of firm names or commercial products does not constitute an endorsement by UNIDO.

FAIR USE POLICY

Any part of this publication may be quoted and referenced for educational and research purposes without additional permission from UNIDO. However, those who make use of quoting and referencing this publication are requested to follow the Fair Use Policy of giving due credit to UNIDO.

CONTACT

Please contact <u>publications@unido.org</u> for further information concerning UNIDO publications.

For more information about UNIDO, please visit us at www.unido.org

21777

Distr. RESTRICTED

ISED/R.82 25 February 1997

UNITED NATIONS INDUSTRIAL DEVELOPMENT ORGANIZATION

ORIGINAL: ENGLISH

TECHNO-ECONOMIC DEVELOPMENT OF MEDICINAL AND AROMATIC PLANTS FOR INDUSTRIAL UTILIZATION IN NIGERIA

DG/NIR/92/015/11-52

NIGERIA

Technical report: work performed and recommendations *

Prepared for the Government of Nigeria by the United Nations Industrial Development Organization

Based on the work of M. K. Raina, essential oil technologist

Project Manager: T. De Silva. Chemical Industries Branch

^{*} This document has not been edited.

TABLE OF CONTENTS

Page No:

.

•

.

.

Abstract i			
1.	Introduction 1		
11.	Activities		
111.	Conclusions		
IV.	Recommendations 1	2	
V .	Acknowledgements 1	3	
ANNEXES.			
Annex	1. Job description 1	4	
Annex	2. List of potential plants 1	5	

ABSTRACT

The consultant reached the project site in Abuja on the 17th January, 1997 and started work at the National Institute for Pharmaceutical Research and Development (NIPRD) on the 18th January. He had a detailed discussion with the Director, Prof. C. Wambebe and his colleagues in the Institute and went round the various laboratories of the Institute and met the scientific staff who appraised him of their project work.

Thereafter the consultant performed the duties as detailed in the job description (Annex 1) until 11th February 1997. The consultant was debriefed by the Project Manager at the headquarters in Vienna on the 12th February 1997.

There is an ever increasing demand for natural essential oils for use in food products, pharmaceuticals, natural insecticides and pesticides besides the flavour and fragrance industries. Nigeria has a very conducive climate for growing several aromatic plants which could be distilled for their essential oils for domestic use and export.

As a part of its main objectives, the National Institute for Pharmaceutical Research and Development (NIPRD) of the Federal Ministry of Science and Technology Nigeria has been engaged in identifying appropriate aromatic plants for their essential oils which could be used both in Nigeria and exported. A well designed multipurpose pilot plant has been set up with the assistance of UNIDO and is being commissioned in a few months. Objective of the consultant's mission was to help NIPRD in identifying aromatic plants which can grow well in Nigeria and train the counterpart staff on methods of essential oil production using the Pilot plant, process parameters and Quality control methods. Also I was assigned the task of demonstrating use of fractionation column for production of isolates and enrichment of oils. Since the pilot plant was not yet commissioned, I had to demonstrate the methodology on laboratory scale and described/discussed all the parameters of essential oil production and their quality control aspects. Essential oils of <u>Eucalyptus citroidora</u>, <u>Cymbopogon citratus</u> and <u>Ocimum basilicum</u> were distilled from fresh plants growing in the farms of NIPRD and the oils analysed using GC-MS. Several batches of each were distilled to validate process parameters.

The methods commonly employed for fractional distillation of essential oils into various isolates and for enrichment were discussed in detail. The importance of adhering to quality control parameters to achieve consistent quality of essential oil was stressed.

A list of essential oil bearing plants(Annex 2) and a number of aroma chemicals (page) which will have international markets have been provided . An appropriate set up for fractionation of oils has been recommended to be installed in Pilot plant. Systematic agro-techniques for growing these aromatic plants must be undertaken before mass cultivating through outgrowers and for this an area of 10 hectares in the farm land be properly fenced and irrigation facilities provided.

Additional training facilities will be required by counterpart staff for both pilot scale trials and on use of sophisticated instruments. However, this should be done only after sufficient quantities of aromatic plants are available for processing in the pilot plant.

A seminar was delivered on the following subjects which was attended by the all the scientific staff of the Institute.

- Production and Quality Control parameters of Essentials oils
- Criteria for Evaluation of Herbal Medicines and their Standardization.

I. INTRODUCTION

Nigeria is one of the most populated countries in Africa estimated at about 110 million, occupies a vast geographical area with varying climatic conditions. The economy of the country is mostly agriculture especially in the rural sector. The varied climatic conditions in different regions of the country help in wild growth of a large number of medicinal and aromatic plants which are used as Traditional medicines by majority of the population. The forest regions of the country covers about 849, 496 km² which is about 86.4% of the total land area. The Nigerian Savanna region has been subdivided into five ecological zones and a large variety of trees are growing in these forests for use as fire-wood, timber, poles and shelterbelts. A number of aromatic plants into essential oils could be a good source of foreign exchange earner, through exports to developed countries especially for fragrance and flavour industries. This could well fit in as a diversified export commodity as a part of Federal Government's policy of encouragement of non-oil exports (Petroleum based crude oil).

As a part of health-care facilities available in the country, the Federal Government has felt a need to strengthen this area of vast importance affecting and total population of the country. The need to establish many industries manufacturing medicines both modern and traditional was felt and in 1987, an institute was established which would systematically investigate and organise scientific studies in the health care needs of the country. Thus the National Institute for Pharmaceutical Research and Development (NIPRD) was established and its operations started in 1989. The Institute has the following clear objectives laid down by the Ministry of Science and Technology.

- i) undertake research and development work into drug and pharmaceutical raw materials from indigenous natural resources and by synthesis.
- ii) conduct appropriate investigations and consequent applications vis-a-vis the evaluation, preservation, purification, standardisation, safety and rational utilization of traditional medicines.
- iii) develop methodologies for quality assessment of both orthodox and herbal medicines including their raw materials.
- iv) serve as reference centre for research work on the biopharmaceutics, pharmacokinetics, storage and stability of imported and locally manufactured drugs.
- conduct research and development work into pharmaceutical biotechnology, nutrition, cosmetics and environmental sciences for improved quality of life and the conservation of medicinal and aromatic plants.
- vi) establish and operate a quality assurance laboratory for pharmaceutical raw materials and products.
- vii) promote and sponsor staff development through training courses, workshops, fellowships within and outside Nigeria.
- viii promote and sponsor the local development and production of drugs, pharmaceutical raw materials, cosmetics, food supplements, pharmaceutical machinery, devices and accessories.
- ix) promote the pilot production unit of the Institute into a limited liability business venture.
- x) transfer pharmaceutical products and machinery technologies to private sector industries and render consultancy and extension services to such and other organisations.

- xi) establish and maintain relevant laboratories, clinics, medicinal plant gardens and other institutions in strategic ecological zones of Nigeria as may be necessary for the performance of the functions.
- xii) compile and publish relevant data resulting from the performance of the functions of the Institute.
- xiii sponsor such national and international conferences, workshops, symposia etc as it may consider appropriate.
- xiv) patent and register new products and processes with appropriate national bodies, international organizations and selected countries.
- xv) enter into commercial and other appropriate agreements with relevant national and multinational corporations regarding the marketing and utilization of the Institute's products and services.
- xvi) liaise with higher institutions, Government agencies, corporate bodies, international organisations, multinational bodies and other relevant establishments within and outside Nigeria in the pursuance of the mandates of the Institute and
- xvii undertake such other activities as are necessary or expedient for the performance of its functions.

NIPRD is a well organised Institute in Nigeria with proper organisational set up of various laboratories like:

- i) Department of Medicinal Plant Research and Traditional Medicines.
- ii) Department of Pharmacology and Toxicology.
- iii) Department of Medicinal Chemistry and Quality Control.
- iv) Department of Pharmaceutical Technology.
- v) Department of Pharmaceutical Microbiology and Biotechnology.
- vi) Library and Information Services.
- vii) Animal House.

With the assistance of UNIDO/UNDP, the Institute has progressed well in the light of the objectives laid down. Most of the laboratories have been set up and are fully operational and a well designed pilot plant is being commissioned within a few months. The building is complete and the equipments are ready for installation. Final touches are being given to electrical wiring and steam lines and within 2-3 months the pilot plant could be fully operational.

The Institute has sufficient land adjoining the laboratory (about 52 hectares) where systematic cultivation practices of selected medicinal and aromatic plants will be taken up. A great deal of development work is required for the proper cultivation practices of selected aromatic plants, standardization of their optimum concentration of essential oil, the quality parameters and the economics so that the oils thus produced could be internationally accepted. Agro-technology of several aromatic plants have been worked out in many developing countries and assistance could be sought from these practices in initiating the trial

cultivation using similar parameters for planting, fertilizer inputs, harvesting time etc. Modification could be done wherever necessary depending on the requirements of the soil/climate.

A small nursery for propagation of selected medicinal and aromatic plants is also located in the Institute premises. This needs to be strengthened to enable proper nursery raising of the selected medicinal and aromatic plants for cultivation trials and development of agro-techniques in a more organised way. Efforts should also be made to instal a green house which can supplement the rational use of nursery raising of some of the essential oil bearing plants which need specific climatic conditions for favourable growth. The Institute has plans for setting up of Tissue Culture Laboratories wherein this Green House will be very useful in transplanting the tissue culture raised plants to soil conditions.

There is not much data available on the use of aromatic plants or their essential oils in Nigeria. However scientists at the Department of Chemistry, University of Ibadan have been undertaking some preliminary studies of some of the aromatic plants growing wild in Nigeria. Some of these essential oil bearing plants could be systematically cultivated and their oil utilised for domestic consumption and for export. Thus the species would be preserved and the country can earn valuable foreign exchange by exporting the essential oils. Besides this, the employment opportunities in cultivation and distillation processes will increase especially in the rural sector where farmland could be utilised property. The involvement of women in these cultivation practices will increase, generating more income for poor families.

II. ACTIVITIES

My assignment involved working closely with Prof. J. Okogun, Head, Department of Medicinal Plant Research and Traditional Medicine and Research Scientists Mrs. N. M. Enwerem and Mrs. Y. Kunle. Details of the activities are listed below under the same headings as given in Job Description (Annex 1)

1. <u>Study the available data on indigenous aromatic plants and select a priority list of plants</u> which have a potential for industrial utilization

There is no single publication giving details of various essential oil bearing plants which have been studied for their commercial utilization. However, a number of aromatic plants growing in Nigeria have been studied for their essential oil content and their analysis undertaken on GC/GC-MS at the Department of Chemistry, University of Ibadan, Nigeria. Most of these aromatic plants have only academic value since the concentrations of essential oils are very small and no efforts have been made to undertake systematic cultivation of any of these for proper commercial utilization. I have gone through the reprints of these publications from various journals and salient features of these are listed below:

i) Ocimum viride Leaves

Steam distillation - yield 0.06-0.20% GC/GC-MS analysis showed 28 components. Thymol was major constituent (47.98%) (Planta Medica 1986, 52, 200-202)

ii) Annona senegalensis (Leaves and Fruits)

Steam distillations - Leaves yielded 0.02% oil, Fruits yielded 0.06% oil. GC/GC-MS analysis of the oils showed 19 mono-and sesquiterpenoids, the major constituents being car-3-ene in fruit oil and linalool in the leaf oil. (Planta Medica, 1986, 52, 202-209).

iii) <u>Clausena anisata</u> (Leaves)

Steam distillation, yield - 0.58%

GC/GC-MS analysis - 19 components identified. Phenylpropanoids constituted major concentration (96%) of the oil. Methyl chavicol was found in concentration of 92%. *This oil has a potential use as substitute for anise oil in pharmaceutical preparations (Planta

Medica, 1986,52,505-506)

iv) Zanthoxylum leprieurii (Leaves)

Steam distillation, yield - 0.035% GC - GC-MS analysis - Sesquiterpenoids constituted bulk of essential oil (50.8%) B-pinene, caryophyllene and caryophyllelol were major components. (Fitoterapia, 1986, 57,267-270)

v) Ageratum convzoides (Leaves)

Steam distillation, yield - not specified Oil separated into non-polar and polar fractions by elution with n-pentane and ethylacetate respectively.

GC-MS analysis showed that in addition to two chromenes, precocene I and II, six more chromenes were detected using this method. These chromenes have well known insect anti-juvenile hormones which also induce sterility. These compounds also possess phototoxic activities against micro-organisms. (Journal of Chromatography, 1987, 403,358-362).

vi) <u>Piper guineense</u> (Fruits)

Steam distillation, yield - 0.35% GC/GC-MS analysis showed myristicin, sarisan, safrole and elimicin as major components. (Journal of Agricultural and Food Chemistry, 1988, 36, 880-882).

vii) Zingiber officinale (Rhizomes)

Hydro-distillation, yield - 1.02-1.8% column chromatography/ GC/GC-MS analysis showed major components as geraniol, neral, 1,8-cineole, zingiberene, B-bisabolene, B-sesquiphellandrene. (Flavour and Fragrance Journal, 1988, 3,85-90).

viii) Ocimum canum (Leaves)

Hydro-distillation, yield - 0.02% GC/GC-MS analysis showed Eugenol as major constituent in addition to 28 other compounds. (Flavour and Fragrance Journal, 1989, 4, 17-18).

ix) Laggera alata (Leaves)

Hydro-distillation, yield - 0.7% - 0.8% GC/GC-MS analysis showed 34 components. There was considerable variation in concentration of the constituents in plant samples collected from Ife and Ibadan. (Planta Medica, 1989, 55, 573-574).

x) <u>Vitex agnus</u> - castus (Leaves)

Hydrodistillation, yield - 0.023%

GC/GC-MS analysis showed presence of 31 compounds of which major components were 1,8cineole (50.9%),sabinene (10.8%), -pinene(9.0%) (Journal of Essential oil Research, 1990,2,115-119).

xi) <u>Cyperus tuberosus</u> (Rhizomes)

Hydrodistillation, yield - 0.65% GC/GC-MS analysis showed presence of 43 compounds of which humulene, B-caryophyllene and four of their isomeric epoxides were major ones accounting for 70% of the oil. Essential oil from <u>Cyperus rotundus</u> showed significant compositional differences which may be chemotaxonomically significant. (Flavour and Fragrance Journal, 1991, 6, 261-264).

xii) <u>Psidium guajava</u> (Fruits)

Hydro-distillation, yield - 0.009%.

GC/GC-MS analysis showed presence of 25 compounds accounting for 80% of the oil. Free fatty acids were abundant (34%). Large amount of B-caryophyllene and oxygen-containing sesquiterpenes (25%) were present. (Flavour and Fragrance Journal 1991, 6, 233-36).

xiii) Mitracarpus scaber (Aerial Parts)

Hydrodistillation, yield - 0.04% GC/GC-MS analysis showed presence of 26 compounds, 11 were free fatty acids constituting major components. Hexa-decanoic acid was the most abundant component (51.2%) (Flavour and Fragrance Journal, 1993, 8, 269-71)

xiv) Ocimum basilicum (Leaves)

Hydro-distillation, yield - 0.22-0.25% GC/GC-MS analysis of oil from plant from Ife contained

predominantly methyl chavicol (84%) while Ibadan sample contained linalool (30.7%) and eugenol (16.9%) as major constituents.

(Acta Pharmaceutica Fennica 1987, 96, 101-106)

xv) <u>Cleistopholis patens</u> (Leaves, Fruits, Stem Bark)

Hydrodistillation, yield leaves - 0.05%; fruit - 0.04%, stem bark - 0.024% GC/GC-MS analysis showed only 30 of the 61 components present in all three oils. E-B -ocimene was the main constituent of leaf oil while as in fruit oil, linalool, Z- and E - linalool oxides were found up to 58%. Mono and sesquiterpenene hydrocarbons made up 68% of the stem bark oil. (Planta Medica, 1988, 54, 338-340).

Root oil contained bornyl acetate and cadinol as major constituents. (Planta Medica, 1987,53, 228-229).

xvi) Pinus species (Needles)

A review of the various species of Pinus needle oils comprising about 52 lower terpenoids has been reported. The needle oil composition is generally characterized by considerable qualitative and quantitative variations which are valuable for chemotaxonomic studies of Pinus genus. (Flavour and Fragrance Journal, 1988, 3, 1 -11).

A well organized scientific study on the essential oil content of the leaves of three Eucalplus species viz <u>E. citroidora, E. camaldulensis</u> and <u>E. tereticornis</u> has been done by the Forestry Research Institute of Nigeria at Zaria in 1980's.

Several thousands of hectares of forests were covered with these three species in late 50's for firewood, poles, timber and shelter belts. The leaves of especially <u>Eucalyptus citroidora</u> which contains higher per cent of essential oil (4%) as compared to other species could be distilled for industrial utilization.

Two Pinus species (<u>P. caribaea</u>, <u>P. oocarpa</u>) are growing in the forest regions of Nigeria which could be successfully tapped for its resin content and the needles used for distillation of essential oil.

The following aromatic plants are growing well in the nursery/farms of NIPRD.

i	Ocimum gratissimum
ii	Cymbopogon citratus
iii	Eucalyptus citriodora
iv	Ocimum basilicum

A list of aromatic plants which has potential for industrial utilization both for domestic consumption and for export is given in Annex 2.

2. Train the counterpart staff on methods of essential oil production using the pilot plant.

Since the Pilot plant is not commissioned yet, experiments were done in laboratory using Clevenger apparatus for distillation of some essential oils from the leaves of <u>Eucalyptus citroidora</u>, <u>Cymbopogon citratus</u> and <u>Ocimum basilicum</u> growing in the farms of the Institute. The distillation of oils were repeated to assess the oil distilled within the specified time. i.e., at the end of I hour, 2 hours, 3 hours and 4 hours. It was observed that most of the oil was distilled within 2-3 hours, thus one could standardize the time needed for optimum yield of the oil. These samples of oils were analysed by GC-MS.

The Scientific staff were given short notes on various methods of Essential oil production and explained the procedures, advantages and disadvantages of various processes, controls on temp, cooling procedures etc. These were also explained on the drawing of the Pilot Plant. Some of the salient points made are given below:

Parts of plant material used

- * Oil glands mints, grasses
- * Breakdown of epidermal cells Pines, Eucalyptus
- * Schizogenous cavities/Lysigenous cavities
 - Umbelli ferous Fruits
 - Rutaceae Plants
- * Resin canals/ Gum -Resins

Methods of essential oil production:

* Hydrodistillation or Cold Press or Solvent Extraction

- * Water Distillation
- * Steam and water Distillation
- * Steam Distillation
- Cold Press Expression

Return of distillate water back to still for reboiling to minimize loss of oxygenated components like phenols which dissolve to some extent in distillate.

ADVANTAGES:

Hydrodistillation

- * Higher Oil yield
- * Oil Components less susceptible to hydrolysis and polymerization
- * Minimum loss of polar compounds
- * Quality of oil superior and reproducible
- * Relatively Faster process than only distillation

Steam distillation

Through boiler. Plant material on perforated grid above steam inlet

- control steam inlet
- Heat contact controlled not above 100°C
- Less thermal degradation
- Most widely accepted process

DISADVANTAGES OF STEAM DISTILLATION

- * Higher Capital Expenses
- * A mix-up of high value essential oils and low valued oils necessary to balance the cost of production.
- * May not be feasible to process in fields Mobile distillation

Expression - Cold Press

- * For citrus oils
 - Essential oil glands in peels are crushed/broken to release oil
- 3. Assist the counterpart staff in process parameter development and the preparation of standard operating procedures.

The details of various critical factors which could influence the quality of essential oil during distillation were discussed. The need for strict adherence to temperature, time of distillation and effective condensation procedures were emphasized. Since the essential oils were mainly used in fragrance and flavour industry, a standardized note of the oil has to be maintained at all time so that the ultimate perfume/ flavour does not differ. It is also very important that the aromatic plants cultivated have standardized agrotechniques followed to achieve consistent quality essential oil. If there are any plant improvements in breeding and selection of hybrid varieties of planting materials, those need to be standardized prior to mass cultivation.

The need for preparation of standard operation procedures were emphasized to the counterpart staff. These would include the assessment of quality parameters of aromatic plants received for processing

in the pilot plant. The moisture level of plants depending upon their nature (whether fresh or dried materials), the loading of plant material in the kettle, time given for distillation, temperature at which the distillation is done and the time required for complete distillation. All these parameters need to be followed strictly to achieve consistent quality of the essential oil.

4. Develop methods for the quality control of processes and products.

The various steps to be taken during processing of aromatic plants in pilot plant to achieve a consistent quality essential oil were described to the counterpart staff of the Institute. Emphasis was made on these parameters in view of the complex nature of volatile substances biosynthesized by living organisms and any changes in process parameters could adversely affect the quality of the oil produced. International Standards have been prescribed for several commercially known essential oils by Standards Organizations like International Standards Organization, British Standards, Essential oil Association of USA, Indian Standards, German DIN Standards, French AFNOR Standards and some Pharmacopeias of the world.

The various Quality Control parameters include Sensory evaluation, Physical tests, Chemical tests and Instrumental analysis techniques. These are enumerated below:

Constituents of essential oils :

Hydrocarbons and their oxygenated derivatives comprising Alcohols, Acids, Esters, Aldehydes, Ketones, Amines, etc.Mono, Sesqui and some Diterpenes

Quality assessment parameters:

- * Sensory Evaluation Expert noses
- * Physical Moisture, Sp. gr. Opt. Rot, Ref. Index, Freezing Pt., Solubility in Alcohol
- * Chemical Acid value, Ester value, Phenol content, carbonyl value.
- Instrumental Chromatographic and Spectroscopic Techniques.

GC - most important and widely used

- * Fused-silica capillary column very versatile for separation, identification and quantification
- * Mobile Phase N₂ or Helium
- Separation Due to differences in partition coefficients of constituents.
- * 1uL of 10% solution oil in N-Hexane or Diethyl ether.

GC/MS - Detection of compounds separated

- Library search system
- Very Versatile and Efficient.

Enrichment

For acceptable Quality as per requirements of Fragrance and Flavour Industry. Deterpenation usually done.

Storage

In inert material containers, most commonly Aluminium containers

Total Quality Management

- ISO 9000 Series Standards
 - Several International Association laid Specifications for common Essential oils used.

Process parameters and SOP'S

- * Quality of Aromatic Plants
- Extraction Parameters Standardized
- * Quality Control and Analysis Techniques Standardized.

5. <u>Demonstrate the use of the fractionation column in the production of isolates and in enrichment and rectification of oils.</u>

The methods employed for fractional distillation of essential oil were described and discussed since there were no fractionation columns available for conducting laboratory experiments nor was any essential oil available in sufficient quantity for undertaking trials.

High vacuum fractional distillation, being the most common method used, details of its operation were described to counterpart staff. Emphasis was made on optimization of vapour/liquid ratio in perfect equilibrium to enable separation of compounds from the mixture. The vapour/liquid mixture in the column constantly change during distillation and thus it is important that proper equilibrium is maintained. Based on this data, column dimension, type of packed columns etc., are designed and used for production of isolates and in enrichment and rectification of oils.

For deterpenation, batch fractional distillation is preferred to continuous distillation as a number of different fractionations can be performed on same equipment. In vacuum fractional distillation, the vacuum determines the boiling point of the mixture, the higher the vacuum, the lower the boiling point. Also, as the pot temperature is lowered, there is a corresponding decrease in off-notes or burnt notes of the distillate.

Most commonly used column for deterpenation of oils is Koch-Sulzer which has a cylindrical segmented sections of exact column dimensions made of parallel corrugated strips of stainless steel woven wire fabric. The packings are generally about 6.7 inches thick and are stacked on top of each other to fill the column. The fractional distillation is useful to reduce the level of monoterpene hydrocarbons in an essential oil. Other method is by Rotating or Static film evaporators which also effectively reduce the monoterpene hydrocarbon content of essential oil. Thin film evaporators have advantage of minimizing any thermal degradation by subjecting the oil to a low residence time into heating zone.

The consultant also discussed about other commonly used/extracted aromatic materials. Common examples of compounds like B-caryophyllene and eugenol isolated from clove leaf oil for further processing into other compounds requiring similar base skeletons were cited. These are caryophyllene oxide and caryophyllene alcohol produced from B-caryophyllene, while eugenol acetate, methyl eugenol and other ethers as well as isoeugenol and its esters/ethers are produced from eugenol that has been converted into isoeugenol. Other most commonly components from essential oils by fractional distillation are linalool, geranial, methyl chavicol, anethole, etc. From turpentine oil a wide variety of compounds are obtained by fractional distillation for use as starting materials of various synthesis products. Menthol is a very important flavour additive isolated from mentha oil by freeze crystallization.

Biotechnological approach is used by using fermentation and enzymatic reactions of natural substrates from essential oils to meet the specific demand of some flavour industry in particular. Another area being pursued is tissue culture technology to produce a component of the oil or full oil itself in a sterile

10

environment. But so far this method has not been commercially undertaken.

Fractional Distillation under vacuum involving single solvent, two-phase solvent system and supercritical CO_2 were also briefly discussed. It was emphasized that mono and sesqui-terpenes are much less soluble in alcohol than are the oxygenated constituents. As a result of this, alcohol washing can be used to remove the oxygenated compounds of an oil. The traces of alcohol remaining need to be washed with brine and by fractional distillation. The oil thus obtained would be free from both mono and sesquiterpene hydrocarbons.

The two phase solvent system has been perfected commercially on citrus oils. Solvents with different polarities are used in counter-current fashion at low temperature. To prevent oxidation during extraction, oxygen and air must be completely eliminated.

Other forms of aromatic material commonly used were also described:

Other aromatic materials

CONCRETE

Extract of fresh plant parts by Hydrocarbon solvent yields waxy semi-solid dark coloured material.

ABSOLUTE

Concentrated Alcoholic Extract of concrete contains only alcohol - soluble materials.

POMADE

Extracts of fresh flowers by fat solvent

RESINOID

Solvent extract of Gum-resin or Oleo-gum-resin with hydrocarbon solvent. Viscous to semi-solid mixtures.

OLEO-RESIN

Solvent ext. of dry spice/herb - free from extracting solvent.

BALSAM

Natural exudate rich in benzoic and Cinnamic acids and their esters.

6. <u>Recommend the Aroma Chemicals that could be produced using locally produced oils and the equipment and training requirements to initiate their production.</u>

Since there have been no efforts made to cultivate any of the aromatic plants so far in Nigeria, the exact aroma chemicals which could be extracted from the essential oils would depend on the nature of the aromatic plant which can grow well in this climate.

However based on certain assumptions of regular extraction of oils from selected aromatic plants, the following aroma chemicals could be extracted for export to Flavour/Fragrance industry. Preliminary cultivation trials of these aromatic plants have already been done and Nigeria climate is suitable for their

favourable growth. Only the agro-techniques on commercial scale have to be done so that the oils produced and methods of extraction of aroma chemicals are cost effective.

Aroma chemicals	Source of Plant
Citral	Cymbopogon citratus
Citronellal	Eucalyptus citroidora
Menthol	Mentha arvensis
Linalool	Ocimum basilicum
Eugenol	Ocimum gratissimum
Methyl chavicol	Ocimum basilicum Clausena anisata
Geranial	Zingiber officinale

<u>Miscellaneous</u>

A Seminar was organised by the Director of NIPRD wherein I gave a lecture to the scientific staff of the Institute on the following subjects.

- Production and Quality control parameters of Essential oils.
- Criteria for Evaluation of Herbal Medicines and their Standardization.

The Seminar was well attended and the discussions that followed were very useful and lively.

In order to obtain data on import of any essential oil/aroma chemical, the consultant met the concerned officials of Customs Department, Nigerian Export Promotion Council and the Ministry of Commerce. These seems to be a sizeable import of some aroma chemicals and data would be given to NIPRD.

Such aromatic plants could be taken up on a priority basis by NIPRD to enable the country to save foreign exchange for import of these essential oils/aroma chemicals. Since the whole data is classified into Drugs and Pharmacuticals and the individual subject data would be taken out in due course. Similarly, Export Promotion Council would assist NIPRD in identifying proper marketing outlets for essential oils/aroma chemicals in the world market.

III. CONCLUSIONS

1. The climate and soil of Nigeria is quite favourable for systematic cultivation of a number of aromatic plants which can be processed for their essential oil for use in domestic industry and for export to flavour and fragrance Industries. However, agro-techniques for commercial cultivation need to be developed for these aromatic plants and their essential oils have to be distilled and evaluated as per international standards.

2. The National Institute for Pharmaceutical Research and Development (NIPRD) under the Federal

ministry of Science and Technology, Abuja has the necessary expertise and infrastructure to organise systematic cultivation trials and distillation of essential oils from these aromatic plants. The quality parameters and analysis of these oils can be conveniently taken up at NIPRD since the requisite sophisticated instruments like Gas chromatograph/Gas chromatograph with Mass Spectrometry are available.

3. A well designed multipurpose Pilot Plant having a capacity of processing 100 - 250 kg of plant material (depending on nature of plants - fresh or dried) will be commissioned within 2-3 months to enable standardize process parameters for distillation of essential oils conforming to international standards.

4. Aromatic chemicals can be extracted/distilled from these essential oils by using the fractional distillation unit in the pilot plant as value-added products having a demand in Flavour/Fragrance Industries and thus the country could earn valuable foreign exchange.

5. Based on research work done at University of Ibadan, some new aromatic plants specific to Nigeria could be grown in trial plots and their essential oils evaluated for possible introduction in the international market as new oils or substitutes for existing oils/aroma chemicals.

6. The scientific staff need to be given specific training in Agro-technology, Distillation/Extraction and fractionation of oils with proper Good Laboratory Practices; analytical techniques and work experience of handling sophisticated instruments like GC and GC-MS.

7. A close liaison is needed between NIPRD and Nigerian Export Promotion Council, Ministry of Commerce and Import/Export control of Customs for proper identification of products needed as import substitutes and creating a marketing network for export. This has emerged based on consultant's personal interaction with officials of these departments of the Federal Government.

8. Forest regions of Nigeria are growing <u>Eucalyputs citroidora</u> in a vast area mainly for its timber. The leaves are not used and these could be processed in the pilot plant and oil evaluated for possible export or its citronellal fraction could be distilled by using fractional distillation.

IV. RECOMMENDATIONS

1. NIPRD has sufficient land available (52 hectares) within the campus. It is highly recommended that at least 10 hectares of the land be properly fenced so that cattle do not enter the area and irrigation facilities be provided for proper cultivation trials to be conducted under controlled conditions. Based on the laboratory data of essential oils distilled from these experimental plots, systematic cultivation should be extended to produce raw materials for processing in the pilot plant. A list of potential aromatic plants has been given in Annex 2 based on agro-climatic conditions and their international demand.

2. Agro-techniques for selected essential oil bearing plants have to be initiated at the earliest, to enable NIPRD to involve growers for mass cultivation of these aromatic plants. Presently, there is no expertise available for the development of agro-techniques/cultivation practices for aromatic plants at NIPRD. Therefore an experienced agronomist should be recruited to undertake these activities. This should result in the development of high yielding planting material and good cultivation practices that the oils produced not only will meet the international standards of quality but also are cost effective in the international market.

3. Monographs on such aromatic plants must be prepared giving details of soil conditions, time of planting/sowing seeds, irrigation requirements, use of fertilizers, harvesting time etc. It is important to lay down methods of post-harvest technology which has to be disseminated to farmers in rural areas of Nigeria.

4. The essential oils distilled from these aromatic plants have to be evaluated and their composition

ascertained. Parameters for the distillation process which could give optimum yields and quality for each oil and standards for the control of their quality have to be finalised.

5. Fractional Distillation of essential oils have to be undertaken to produce specific aroma chemicals for export as value added commodities. The protocols for fractional distillation of each essential oil giving details of process parameters and controls for various isolates/aromachemicals/concretes etc. which could finally meet specifications of flavour and fragrance industries, will have to be drawn up by NIPRD with the assistance of experienced consultants.

6. The scientists especially working on essential oils need to be given adequate training in analysis of oils on sophisticated instruments like GC and GC-MS for effective interpretation of data and in process and product development. It is recommended that industrial consultants should be fielded for this purpose.

7. There may be a need for the installation of field distillation units in rural areas for such crops which cannot be transported quickly for distillation of their oil. The field operations would also help in employment of women and thus help in poverty alleviation. The quality control/fractionation processes have to be supervised by the NIPRD

8. A few Nigerian aromatic plants growing wild like Hyptis suaveolens, Annona senegalensis are well known for their insect control properties. Systematic cultivation trials should be undertaken and the action of their essential oils should be evaluated scientifically. There is a great demand for such natural oils in the world market for their eco-friendly and low toxicity levels. The export of the oil would earn valuable foreign exchange for the country.

V. ACKNOWLEDGEMENTS

The consultant would like to express sincere thanks and gratitude to Prof. C. O. N. Wambebe, Director/Chief Executive, National Institute for Pharmaceutical Research and Development, Abuja, Nigeria for his help and courtesy extended throughout his stay in Abuja.

He is thankful to Mrs. N. M. Enwerem and Mrs. Y. Kunle, Research Scientists, NIPRD for their active co-operation in performing his work assignments.

The consultant is grateful to Mr. Alhaji M. D. Usman, Director, Federal Ministry of Commerce and Tourism, Mr. Badmus and Mr. M. R. Agbogun from Nigerian Export Promotion Council and Mrs. O. O. Ahmadu, comptroller of Customs, Import and Export Control, Nigeria for their help and cooperation extended during his meeting with them, at short notice.

Thanks are also due to all Heads of Departments of NIPRD and their staff for active co-operation and courtesy.

His sincere thanks to Mr. Tunde Olorode for typing the manuscript and to Mrs. Musa for hospitality.



14

Annex 1

UNITED NATIONS INDUSTRIAL DEVELOPMENT ORGANIZATION

JOB DESCRIPTION DG/NIR/92/015/11-52/0730AO

- Post title Essential oil technologist
- Duration 1 m/m

Date required ASAP

- Duty station Abuja, Nigeria
- Purpose of project The project intends to provide institutional facilities which will strengthen the capacity and technical capability of the National Institute for Pharmaceutical Research and Development (NIPRD), to enable it operate a multi-disciplinary research on the medicinal and essential oil containing plants which are locally available vis-a-vis isolation, identification, production and pharmacological evaluation of such products.
- Duties The consultant working in collaboration with NIPRD/UNDP and under the general supervision of the National Project Director will be specifically expected to carry out the following duties:
 - Study the available data on indigenous aromatic plants and select a priority list of plants which have a potential for industrial utilization.
 - Train the counterpart staff on methods of essential oil production using the pilot plant.
 - Assist the counterpart staff in process parameter development and the preparation of standard operation procedures.
 - Develop methods for the quality control of processes and products.
 - Demonstrate the use of the fractionation column in the production of isolates and in enrichment and rectification of oils.
 - Recommend the aroma chemicals that could be produced using locally produced oils and the equipment and training requirements to initiate their production.

The consultant should prepare a comprehensive report (on Word Perfect 5.2 to submit to UNIDO on a diskette and on a hard copy) incorporating the findings and recommendations.

Qualifications A chemist or a technologist with over 10 years of experience in the industrial production of essential oils and aroma chemicals.

English

Annex 2

15

List of Aromatic Plants which have a potential for industrial utilization

- 1) Cymbopogon citratus
- 2) Eucalyptus citroidora
- 3) Ocimum basilicum
- 4) Ocimum gratissimum
- 5) Mentha arvensis
- 6) Mentha piperata
- 7) Mentha spicata
- 8) Zingiber officinale
- 9) Citrus sps.
- 10) Hyptis suaveolens
- 11) Clausena anisata