# QUALITY CHARACTERISTICS OF CLOVE AND NUTMEG AT DIFFERENT STAGES OF MATURITY

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#### THESIS

submitted in partial fulfilment of the requirement for the Degree MASTER OF SCIENCE IN HORTICULTURE Faculty of Agriculture

Kerala Agricultural University

Department of Horticulture COLLEGE OF AGRICULTURE VELLAYANI, THIRUVANANTHAPURAM

#### DECLARATION

I hereby declare that this thesis entitled "Quality characteristics of clove and nutmeg at different stages of maturity" is a bonafide record of research work done by me during the course of research and that the thesis has not previously formed the basis for the award to me of any degree, diploma, associateship, fellowship or other similar title of any other University or Society.

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#### CERTIFICATE

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<u>CONTENTS</u>

	· ·	PAGE
INTRODUCTION	•••	1 - 3
REVIEW OF LITERATURE	•• •	4 - 20
MATERIALS AND METHODS	• • •	21 <b>-</b> 31
RESULTS	<b>0 • •</b> .	32 - 84
DISCUSSION	0 0 0	85 <b>-</b> 100
SUMMARY	• • •	101 -, 104
REFERENCES	0 • 0	i - vi
APPENDICES		

ABSTRACT

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### viii

LIST OF TABLES

TABLE	TITLE	PAGE
1	Growth characteristics of clove at different maturity stages	33
2	Moisture content of clove flower buds at different maturity stages	36
3	NVEE of clove at different maturity stages	37
4	Direct and indirect effects of growth parameters on NVEE content of clove	39
5	Volatile oil content of clove at different maturity stages	42
6	Direct and indirect effects of growth parameters on clove oil	<sup>,</sup> 43
7	Physical properties of clove oil at different maturity stages	46
8	Flavour characteristics of clove oil at different maturity stages	48
. 9	Growth characteristics of nutmeg fruits at different maturity stages	52
10	Growth characteristics of nutmeg kernel at different maturity stages	55
11	Moisture content of rind and kernel of nutmeg fruit at different maturity stages	59

(contd.)

LIST OF TABLES (Contd.)

TABLE	TITLE	PAGE
. 12	NVEE in nutmeg fruit at different maturity stages	61
13	Direct and indirect effects of growth parameters on NVEE in nutmeg	64
14	Volatile oil in nutmeg at different maturity stages	66
15	Direct and indirect effects of growth parameters on nutmeg oil	68
16	Flavour characteristics of nutmeg oil at different maturity stages	71
17	Flavour characteristics of mace oil at different maturity stages	, , 79
18	Components in nutmeg oil	93
19	Components in mace oil	97

ix

# LIST OF FIGURES (contd.)

Figure	Title	After page
14	Gas chromatographic pattern of nutmeg oil at the second month of sampling	77
15	Gas chromatographic pattern of nutmeg oil at the third month of sampling	77
16	Gas chromatographic pattern of nutmeg oil at the fourth month of sampling	77
17	Gas chromatographic pattern of nutney Gas chromatographic pattern of sampling	77
18	Gas chromatographic pattern of Hudmas is at the sixth month of sampling	77
19	Gas chromatographic pattern of nucleos das chromatographic pattern of sampling	77
20	Gas chromatographic pattern of made is at the fifth month of sampling	84
21	Gas chromatographic pattern of maching	84
22	Gas chromatographic patternof mace oil at the seventh month of sampling	84
23	Variation in major components of	93
24.1	nutmeg of aromatic phenol and	95
to 24.6 25.1 to	Constituents of aromatic phenol and	99
25.		

ì

LIST OF TABLES (Contd.)

TABLE	TITLE	PAGE
12	NVEE in nutmeg fruit at different maturity stages	61
13	Direct and indirect effects of growth parameters on NVEE in nutmeg	64
14	Volatile oil in nutmeg at different maturity stages	66
15	Direct and indirect effects of growth parameters on nutmeg oil	68
16	Flavour characteristics of nutmeg oil at different maturity stages	71
17	Flavour characteristics of mace oil at different maturity stages	, <b>79</b>
18	Components in nutmeg oil	93
19	Components in mace oil	97

LIST OF FIGURES

<u>Figure</u>	Title	<u>After page</u>
. 1	Growth curves of clove flower bud	33
2.1	Changes in volatile oil and NVEE in flower buds, flowers and fruits of clove	36
2.2	Changes in moisture (%) in flower buds flowers and fruits of clove	36
3	Variation in major components of clove oil at different maturity stages	49
4	Gas chromatographic pattern of clove oil at the first month of sampling	50
5	Gas chromatographic pattern of clove oil at the second month of sampling	50
6	Gas chromatographic pattern of clove oil at the third month of sampling	50
7	Gas chromatographic pattern of clove oil at the fourth month of sampling	50
8	Gas chromatographic pattern of clove oil at the fifth month of sampling	50
9	Gas chromatographic pattern of clove oil at the sixth month of sampling	50
10	Gas chromatographic pattern of clove oil at the seventh month of sampling	50
11.1	Growth curves of nutmeg fruit	53
<b>11.2</b>	Growth curves of nutmeg kernel	53
12.1	Changes in moisture (%) in rind and kernel of nutmeg	58
12.2	Changes in NVEE(%) in rind and kernel of nutmeg	58
13	Volatile oil content of nutmeg and mace	67

(contd.)

# LIST OF FIGURES (contd.)

Figure	Title	<u>After page</u>
14	Gas chromatographic pattern of nutmeg oil at the second month of sampling	77
15	Gas chromatographic pattern of nutmeg oil at the third month of sampling	77
16	Gas chromatographic pattern of nutmeg oil at the fourth month of sampling	77
17	Gas chromatographic pattern of nutmeg oil at the fifth month of sampling	77
18	Gas chromatographic pattern of nutmeg oil at the sixth month of sampling	77
19	Gas chromatographic pattern of nutmeg oil at the seventh month of sampling	77
20	Gas chromatographic pattern of mace oil at the fifth month of sampling	84
21	Gas chromatographic pattern of mace oil at the sixth month of sampling	84
22	Gas chromatographic patternof mace oil at the seventh month of sampling	84
23	Variation in major components of nutmeg oil	93
24.1 to 24.6	Constituents of aromatic phenol and phenol ethers in nutmeg oil	95
25.1 to 25.3	Constituents of aromatic phenol and phenol ethers in mace oil	99

## LIST OF PLATES

<u>Plate</u>	Title	<u>After page</u>
1	Growth and development of clove at different maturity stages	33
2	Clove bud prior to anthesis	33
3	Clove bud at the anthesis stage	33
4	Growth stages from post-anthesis to "mother-of-clove"	33
5	"Mother-of-clove"	33
6	Nutmeg fruits at different growth stages	52
7	Nutmeg kernels at different growth stages	52
8	Nutmeg kernel with mace	56

## xiii

### LIST OF APPENDICES

<u>Appendix</u>	<u>Title</u>
I.A	Test of significance of fitted regression of growth parameters on sampling periods of clove.
I.B	Test of significance of regression coefficient of growth parameters on sampling periods of clove.
II.A	Test of significance of fitted regression of growth parameters on sampling periods of nutmeg fruit.
II.B	Test of significance of regression co-efficient of growth parameters on sampling periods of nutmeg fruit.
III.A	Test of significance of fitted regression of growth parameters on sampling periods of nutmeg kernel.
III.B	Test of significance of regression coefficient of growth parameters on sampling periods of nutmeg kernel.
IV.A	Test of significance of regression of NVEE and oil content on moisture in clove.
IV.B	Test of significance of regression coefficient of NVEE and oil content on moisture in clove.
V.A	Test of significance of regression of NVEE and oil content on moisture in nutmeg kernel.
V.B	Test of significance of regression coefficient of NVEE and oil content on moisture in nutmeg kernel.

# INTRODUCTION

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#### INTRODUCTION

Clove and nutmeg are the two important tree spices of the tropics., The commercial product of clove (<u>Eugenia caryophyllata</u> Thunb.) is the dried unopened flower bud. The nutmeg tree (<u>Myristica fragrans</u> Houtt.) is unique among spice crops as it provides two separate products, namely, the nutmeg which is the dried kernel of the seed and the mace which is the dried aril surrounding the seeds.

Clove and nutmeg are evergreen trees attaining a height of 10 to 12 meters. The clove tree flowers twice in a year, the first during July to September and the second during November to January. The fruit on ripening attains a purplish-red colour and is called 'mother-of-clove'. The nutmeg tree is dioecious in habit. Though the fruiting is seen throughout the year, some peaks are observed.

Clove and nutmeg are natives of Moluccas Islands. In India, clove and nutmeg are mainly grown in the Southern States of Kerala, Tamil Nadu and Karnataka. In Kerala, clove is chiefly grown in Kottayam, Ernakulam and Trivandrum districts, while nutmeg is mainly cultivated in Kottayam, Ernakulam and Trichur districts.

The spices obtained from clove and nutmeg are used for flavouring purpose and in pharamceutical preparations. Clove oil is used for flavouring food products and in perfumery industry. Nutmeg and mace oils are used for flavouring food products, liqueurs, in perfumery and for scenting soaps.

The use of these spices in various food products has been a traditional practice. The modern food ' processing industry aims at diversification of products from these spices which may have great demand and acceptability in domestic and foreign markets. The dried as well as ground spices have inherent disadvantages like microbial contamination and variability in flavour, strength and quality which are not preferred by the food technologists at home and abroad. The extracted spice oils are hygienic products which are of superior quality, free from microbial contamination and quite acceptable to the food industry.

The aroma of a spice oil is dependent on its composition (Lewis, 1984). Spice oils extracted from clove and nutmeg at different maturity stages differ in their flavour characteristics primarily because of their difference in component composition (Gopalakrishnan, 1984). The present investigation was undertaken to study the changes in flavour components (physico-chemical characters) of clove and nutmeg at different maturity stages. This investigation also aimed at characterising the growth patterns of clove flower buds and nutmeg fruits and developing suitable harvest indices for determining the optimum stage of maturity, for use as a spice as well as for extraction of essential oils.

# **REVIEW OF LITERATURE**

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#### 2 REVIEW OF LITERATURE

Clove and nutmeg are two important tree spices of Kerala which have substantial potential in the Indian and International spice markets. Research on clove and nutmeg were mainly concentrated on the analysis of essential oils at a particular stage of harvest for their constituent components. The literature pertaining to distillation, component analysis of essential oil and related aspects on clove, nutmeg and its mace are reviewed in this chapter.

2.1 Clove

2.1.1 Distillation of clove oil

Experiments were conducted as early in 1946 by Smith on the distillation of clove bud for its oil. He observed that water distillation of clove bud yielded the finest oil for use in the perfumery industry and for flavour purposes with an eugenol content of 85 to 89 per cent. The water distilled oil was colourless and it darkened on ageing. He further observed that eugenol, eugenol acetate and caryophyllene which together constituted 99 per cent of the oil were not the components responsible for the characteristic fresh and fruity note of pure clove bud oil.

Belcher (1965) found that the eugenol acetate content of clove oil is dependent on the time taken for distillation though he has not assigned any reason for this phenomenon. Rapid distillation of clove buds produced an oil having high eugenol content than those normally met with in commercial products. He, therefore, suggested that when clove oils are used mainly as a source of eugenol, a brisk distillation yielding an oil rich in eugenol would be preferable. Whenever the oil was required for olfactory purposes, too rapid a distillation may not be beneficial.

Investigating on the distillation of clove buds, Lewis (1984) observed that the oil was collected in the receiver of the still in two major fractions, one lighter and the other heavier than water. Both fractions when combined gave the whole (genuine) oil.

2.1.2 Oil content of clove flower buds

The steam-volatile, aromatic oil of clove (on dry weight basis) was found as high as 21 per cent in good

quality commercial samples but the usual average was about 17 per cent (Purseglove <u>et al.</u>, 1981)

During the distillation of clove flower buds at two, three and four months of maturity Gopalakrishnan <u>et al</u>. (1982), observed that the concentration of volatile oil was the maximum at the lower maturity stages with slight decrease at the later stages. They opined that the synthesis of volatile oil was very high during the second and third months compared to the fourth month.

2.1.3 Components of clove oil

Marson (1909) identified several components in traces like methyl salicylate, 2 heptanol, furfuryl alcohol and alpha-methyl furfural in clove bud oil.

The volatile oil was described as the predominant flavour principle in clove by Guenther (1961). He listed the major components of clove oil as eugenol, eugenol acetate and caryophyllene.

The steam-volatile constituents of sun dried clove bud from Zanzibar was subjected to gas chromatographic

- 6

analysis by Deyama and Horiguchi (1971). Eugenol(80-87%) beta-caryophyllene (9.12%) and acetyl eugenol (7.33%) were found to be the major components of clove oil. Further, they reported for the first time the presence of several minor components like benzaldehyde, benzyl acetate, benzyl alcohol, m-methoxy benzaldehyde, alphaylangene and chavicol in clove bud oil.

7

Walter (1972) detected beta-caryophyllene as one of the major components of clove bud oil.

The eugenol content in clove buds exhibited a continuous increment with progressive increase in maturity of the buds (Gopalakrishnan, 1984). A decrease in concentration of eugenol acetate with an increase in the maturity of clove bud was also observed. The concentration of caryophyllene was the maximum at medium maturity stage and thereafter it showed a decline. This phenomenon was also observed in the case of minor constituents like alpha-cubebene, alpha-copaene, alpha humulene, benzyl alcohol, gamma-cadinene, delta-cadinene, farnesol, vanillin and asarone. The presence of trace constituents (concentration below 0.3%) was high at lower maturity stages and thereafter declined steadily. The essential oil distilled from fresh cloves during immature stages was found to have a pleasant, fruity odour and during full maturity stage it had a more mellow odour. Gopalakrishnan (1984) also reported for the first time the presence of sesquiterpenes like alpha-cubebene, alpha-copaene, gamma and deltacadinene in clove oil.

Beta-caryophyllene, alpha-humulene and deltacadinene in clove bud oil were reported by Muchalal and Crouzet (1985).

Gaydou and Randriamiharisoa (1987) distilled clove buds collected from Madagascar and subjected the oil to gas chromatographic analysis. The oil was found to contain eugenol (73.5 to 79.7%) beta-caryophyllene (7.3 to 12.4%), alpha-humulene (1.0 to 1.4%) and eugenol acetate(4.5 to 10.7%).

2.1.4 Odour characteristics of clove oil

The presence of vanillin in clove bud oil was reported by Jorissen and Hairs in 1890.

Schimmel <u>et al</u>.(1902) identified the component methyl-n-amyl ketone in clove oil. This component though present only in traces contributed to the characteristic odour of clove oil.

Crystals having vanillin like odour were extracted from clove oil by Van Urk (1928). These crystals when treated with phloroglucinol and hydrochloric acid developed a red colour.

2.1.5 Physical characteristics of clove oil

Variation in the physical properties of clove oil obtained at different maturity stages of clove flower buds were reported by Gopalakrishnan <u>et al</u>. (1982). Refractive index and specific gravity of oil exhibited a gradual increase, while specific rotation showed a slightly more significant increase with advancement in maturity of the spice.

# 2.1.6 Comparative analysis of volatile oil from different plant parts of clove

Comparison of the essential oils obtained from flower bud, stem and leaf of clove was made by Billot and Wells (1975). The study revealed that the chemical constituents were remarkably similar but slight differences existed in percentage of individual constituents. The eugenol content was the maximum in stem oil (87.0 to 92.0%) followed by leaf oil (85.0 to 90.0%) and bud oil (80.0 to 85.0%). The eugenol acetate

content was, however, found to be the maximum in bud oil.

# 2.1.7 Comparative analysis of clove leaf oil obtained at different maturity stages

Gopalakrishnan and Naravanan (1988) conducted experiments on the distillation of oil from the leaves of clove at different maturity stages using a Clevenger apparatus. Gas chromatographic analysis of the bil was carried out to detect the various components present On fresh weight basis, the yield of oil was the in it. maximum (1.9%) at the initial stages of leaf growth (2 to 5 days). In the green leaves, the oil content remained constant from six to thirty days of growth. Further reduction in oil content was noted during the process of yellowing of leaves (30 to 40 days) to 0.5 per cent. The major components reported in the commercial oil were caryophyllene, eugenol and eugenol acetate. The caryophyllene content was found to decrease from 6.3 to 0.2 per cent during leaf maturation. Eugenol content increased from 38.2 to 95.2 per cent during the period of maturation. The eugenol acetate content decreased from 51.2 per cent in tender leaves to 1.5 per cent in dry leaves. The pattern of change

in eugenol content bore an inverse relationship to that of the eugenol acetate content. The variation in flavour of clove leaf oil was attributed to the changes that took place in the composition of oil obtained at different stages of maturity.

2.1.8 Mother-of-clove oil

Distillation of ripe fruits yielded about two per cent 'mother-of-clove' oil (Schimmel <u>et al</u>., 1915). The oil had a brown colour and odour which resembled the bud oil, but was weaker in aroma than the bud oil. It contained 53.0 per cent eugenol and an odourless solid phenol of about 35.0 per cent.

Huneck (1972) identified the crystalline phenol found in 'mother-of-clove' oil as 2 hydroxy, 4,6 dimethoxy-5-methyl acetophenone.

-2.1.9 Moisture content in clove bud

The moisture present in clove bud could be estimated by the toluene distillation method (Gopalakrishnan <u>et al</u>., 1982). An increase in moisture content was observed with advancing maturity of the buds.

2.1.10 Non-volatile ether extract in clove bud

The synthesis of non-volatile ether extract (NVEE) continued throughout the maturation period of clove buds (Gopalakrishnan <u>et al.</u>, 1982). When clove buds of two, three and four months maturity were subjected to analysis, it was observed that NVEE was the maximum at the lower maturity stages with slight decrease in the later stages.

#### 2.2 Nutmeg

The nutmeg tree yields two distinct spices, 'nutmeg' which is the kernel of seed and 'mace' which is the aril that surrounds the seed (Purseglove <u>et al</u>., 1981). The international trade recognises mainly two important types of nutmeg and mace namely East Indian nutmeg and mace and West Indian nutmeg and mace.

## 2.2.1 Oil content of nutmeg and mace

The yield of oil ( on dry weight basis) varied between 7.0 to 16.0 per cent in nutmeg and between 4.0 to 17.0 per cent in mace (Guenther, 1952).

The geographical origin and grade of nutmeg used, together with the type of distillation procedure employed,

can considerably affect the yield and quality of oil. The yield is mainly dependent upon the grade of nutmegs; worm-eaten types, containing little fixed oil and starch, give higher yields of essential oil, while in sound nutmegs the yield will be slightly reduced by retention of some of the essential oil within the non-volatile fixed oil during the distillation process (Purseglove et al., 1981).

Investigations carried out by Gopalakrishnan (1984) using nutmeg of three to six months of maturity revealed that the volatile oil content of nutmeg was more at the medium maturity stage and showed a decline at the full maturity stage. The volatile oil in mace showed a steady decrease as maturity advanced but on per fruit basis the oil content was found to be increasing.

2.2. 2 Components of nutmeg oil

Wallach (1885) reported the presence of limonene and alphapinene in nutmeg oil. Subsequently, Semmler (1890) reported camphene as a component of nutmeg oil.

Several compounds like camphene, limonene, borneol, geraniol, linalool, alpha-terpineol, terpinene-4-ol, myristicin, safrole and myristic acid in nutmeg oil

were reported by Power and Salway in 1907. They identified several phenols like eugenol and isoeugenol in nutmeg oil and reported the presence of camphor. They deduced that this might probably have formed by the oxidation of borneol that was originally present in the oil.

Shulgin <u>et al</u>.(1963) found the presence of sabinene, alpha-terpineol, terpinen-4-ol, geranyl acetate, para-cymene, trans-isoelemicin, eugenol, trans-isoeugenol, methyl eugenol and myristic acid in West Indian nutmeg oil.

Beta-pinene(68.0%) and myristicin (12.0%) were reported as the major components of nutmeg oil by Itty and Nigam (1966). The other components that were identified were dipentene, linalyl acetate, bornyl acetate, linalool, safrole, myristicin and alpha-pinene.

Investigation on nutmeg oils from three to six months old fruits revealed alpha-pinene, beta-pinene and sabinene as the major components (Gopalakrishnan, 1984). These compounds together constituted 64.14 per cent, 81.77 per cent and 77.38 per cent of the total oil, respectively, at the three maturity stages studied.

The concentration of other compounds like delta<sup>3</sup>carene, alpha-terpinene, para-cymene, limonene, cineole, beta-phellandrene, gamma-terpinene, linalool and terpinolene remained more or less steady during maturation. In the case of terpinene-4-ol, a significant reduction in percentage composition was observed during maturation. However, on per unit basis there was a nine-fold increase in concentration of terpinen-4-ol during maturation, showing the continued synthesis of this compound at a slow pace. Safrole which contributed much to the characteristic odour of nutmeg oil showed only marginal increase with fruit maturity. Myristicin and elemicin content decreased at the medium maturity stage and then gradually increased.

2.2.3 Odour characteristics of nutmeg oil

The aromatic ether myristicin was first detected in nutmeg oil by Thoms (1903). Subsequently, Provatroff (1973) found that freshly harvested nutmeg contained 8.0 per cent myristicin, while aged fruits contained 20.0 per cent of this component.

The aromatic ethers myristicin, safrole and elemicin appeared to play a vital role in determining the flavour and drug action of nutmeg (Purseglove <u>et al</u>., 1981).

2.2.4 Physical properties of nutmeg oil

The physical characteristics of East Indian nutmeg oil was studied by Lewis (1984). At a temperature regime of  $20^{\circ}$ C the oil had a specific gravity of 0.87, refractive index of 1.48 and an optical rotation of +10.5°.

Comparing the physical properties of nutmeg oils from three to six months mature fruits, Gopalakrishnan (1984) found that the specific gravity and refractive index of nutmeg oil showed a decline at the mid maturity stage and an increase towards the full maturity stage. The specific rotation of the oil remained more or less steady after an initial increase.

2.2.5 Comparison of nutmeg oils of different geographic origin

According to Clevenger (1935) West Indian nutmeg yielded volatile oils which could be distinguished from the corresponding East Indian oils by their lower specific gravity and refractive indices and higher optical rotation. Baldry <u>et al</u>.(1976) investigated the composition of nutmeg oils of different geographical origin. It was concluded that geographical source of nutmeg influenced the composition of oils and that their composition could be related to the flavour. The West Indian oils were found to be low in alpha-pinene, safrole, myristicin, with higher percentage of sabinene while the reverse order was true for the East Indian Oils which had relatively higher concentration of myristicin (13.5%) compared to the West Indian Oils (less than 1.0%).

2.2.6 Effect of storage on nutmeg oil

The effect of storage on volatile oil composition of nutmeg was studied by Sanford and Heinz (1971). They found that prolonged storage of nutmeg resulted in changes in volatile oil composition of nutmeg. Freshly ground nutmeg was stored in open containers upto 48 hours at  $40^{\circ}$ F. It was observed that more volatile fractions like alpha-pinene, alpha-thujene, camphene, sabinene, beta-pinene, myrcene, delta<sup>3</sup>-carene, alpha-phellandrene, alpha-terpinene, limonene, beta-phellandrene, para-cymene and 1,4-p-menthadiene decreased in percentage while the higher boiling fractions increased. Increase in myristic acid content from 1.9 to 22.8 per cent, methyl

eugenol from 6.9 to 15 per cent, myristicin from 12.1 to 27.1 per cent and elemicin from 7.2 to 15.5 per cent was observed. When whole nutmegs were ground in a mortar and stored at 37-40°F for 11 months in closed containers, similar results were also observed.

2.2.7 Mace oil

The mace oil is a colourless to pale yellow liquid and possesses very similar physico-chemical and organoleptic properties of nutmeg oil(Purseglove <u>et al.</u>, 1981).

2.2.8 Components of mace oil

Forrest and Heacock (1972) conducted analysis of the East Indian mace oil and detected many compounds viz., camphene, limonene, alpha-phellandrene, beta-phellandrene, alpha-pinene, beta-pinene, sabinene, terpinolene, geraniol, linalool, alpha-terpineol, terpinene-4-ol, para-cymene, elemicin, eugenol, trans-eugenol, methyleugenol, myristicin and beta-caryophyllene.

Mace oils obtained from nutmeg fruits of three to six months maturity were studied by Gopalakrishnan (1984). It was found that alpha-pinene, beta-pinene and sabinene

together accounted for 64.50 per cent 56.02 per cent and 60.76 per cent of the total mace oil at 3, 4.5 and 6 months respectively. Terpinen-4-ol which accounts for 4.53 per cent at three months maturity registered a gradual increase to 8.28 per cent during maturation and subsequently decreased to 4.59 per cent at the final stage.

2.2.9 Physical properties of mace oil

The physical properties of mace oils obtained from three to six months mature nutmeg fruits were studied by Gopalakrishnan (1984). He found that specific gravity and refractive index showed an increase upto mid maturity phase and then the values steadily decreased. The specific rotation also showed a steady decline as the maturity increased in mace oil.

2.2.10 Comparison of nutmeg and mace oil

The oils obtained from both nutmeg and mace were found to have nearly similar physico-chemical and organoleptic properties (Guenther, 1952).

Gopalakrishnan (1984) investigated the nutmeg and mace oils obtained from fruits of three to six months maturity. A significant difference observed between the nutmeg and mace oils was that the latter contained relatively higher concentration of myristicin and elemicin at all the maturity levels examined.

2.2.11 Moisture content of nutmeg and mace

The moisture content of nutmeg and mace was found to decrease with increase in maturity of fruits. When three to six months mature nutmeg fruits were examined by Gopalakrishnan (1984), the maximum moisture registered was 72.0 per cent and 88.0 per cent respectively for nutmeg and mace at three months maturity.

2.2.12 NVEE of nutmeg and mace

The non-volatile ether extract(NVEE) showed a sudden increase towards the last phase of growth of nutmeg, registering a maximum of 28.5 per cent at six months maturity (Gopalakrishnan, 1984). Investigations on corresponding mace at three to six months maturity revealed a steady decrease in NVEE from 36.3 per cent at three months. On per fruit basis, the NVEE was found to be steadily increasing from the third to sixth month maturity stages.

MATERIALS AND METHODS

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## 3 MATERIALS AND METHODS

Investigations on quality characterization of clove and nutmeg were carried out at the College of Agriculture, Vellayani during the period January 1988 to May 1989.

Studies on growth and development of clove flower buds and nutmeg fruits and sampling of clove and nutmeg were conducted at the Gokul Estate, Vithura in Trivandrum district. Distillation of clove and nutmeg for their essential oils were conducted at the Department of Horticulture, College of Agriculture, Vellayani. The essential oils so obtained were subjected to physico-chemical analysis at the Division of Foods, Regional Research Laboratory of the Council of Scientific and Industrial Research, Pappanamcode, Trivandrum.

The details regarding experimental material, collection of plant samples, methodology of experiment and analytical techniques adopted are presented in this chapter.

#### 3.1 Experimental material

Bearing trees of clove and nutmeg were chosen from the Gokul estate, Vithura for collecting samples as well as for conducting growth studies. The experimental trees were marked well in advance of their flowering time and observed regularly for their dates of blooming and fruit setting and for tagging flowers/ fruits for conducting further studies and collecting samples.

## 3.2 Collection of plant samples

Fresh samples of clove and nutmeg were taken at monthly intervals from the visual appearance stage of flower bud in clove and from the fruit setting stage in nutmeg. For this purpose, 1000 flower buds of clove were tagged in two separate trees and the samples were collected at monthly intervals. Similarly in nutmeg sufficient number of flowers were tagged on four trees growing side by side and fruit samples were collected from these trees commencing from the fruit setting stage, at monthly intervals. The samples obtained from each nutmeg tree at the same stage were pooled together to form a composite sample.

In clove, the first sample was collected in August, 1988 and thereafter the samples were collected at monthly intervals. During the fourth month stage (November,1988) the flower buds attained full maturity. Sampling was continued during subsequent months and by December,1988 the clove trees flowered profusely and during February, 1989 'mother-of-clove' were obtained from these trees.

Sampling commenced during October 1988 in nutmeg and this was continued until April 1989, when mature nutmeg fruits were obtained.

3.3 Clove

3.3.1 Growth and development of flower buds

The morphological characteristics of flower buds such as length, breadth and girth were measured at sequential intervals in centimeters and average values were computed. The weight of buds were taken using a digital weighing balance (Sartorius make). The volume of buds were measured adopting the water displacement method.

3.3.2 Estimation of moisture content of flower buds

Clove flower buds of uniform maturity were crushed in a mixer-grinder and homogenously mixed for analysis. Moisture content of the bud was determined by the toluene distillation method using a Dean and Stork apparatus (AOAC, 1975).

A known weight of the ground sample was taken in a round bottomed flask and 100 ml toluene was added to it. The flask was then attached to the Dean and Stork apparatus fitted with a reflux condenser. The flask was heated using a heating mantle. When the solvent started boiling, the moisture that was present in clove buds vapourised with toluene, rose up the condenser, got condensed and toluene along with water settled in the trap of Dean and Stork apparatus in such a way that moisture settled below the toluene layer. Distillation was continued till the volume of water collected in the apparatus remained constant. The apparatus was subsequently cooled and the volume of water collected was directly read from the graduated scale of the apparatus. The moisture content of the sample was found out using the following formula:

 $\mathbf{24}$ 

Moisture (per cent) = 
$$\frac{v}{w} \times 100$$

where, v = volume of water collected (ml)

w = weight of sample (clove bud) taken (g)

3.3.3 Estimation of volatile oil

The volatile oil of clove flower buds was estimated by the water distillation method (Clevenger, 1928). The distillation was carried out using a Clevenger apparatus fitted with a trap for collecting volatile oils that are lighter than water. The apparatus consisted of a round bottomed, short necked flask, a trap for collecting oil and a condenser of 'cold-finger type'. A known weight of dried powdered sample was taken in the round bottomed flask. About 500 ml water was added and the Clevenger apparatus was attached to the flask. The mixture was heated using a heating mantle. The volatile oil along with steam condensed and oil together with water collected in the trap as separate layers. The water in the trap was drained at periodic intervals. The distillation was continued for four to six hours until further recovery of oil was not seen. The volume of oil collected was directly read from the trap. Volatile oil content of the material was calculated on a volume to weight basis

using the formula as follows:

Volatile oil (per cent) =  $\frac{v}{w} \times 100$ where v = volume of oil collected (ml) w = weight of sample taken (g)

3.3.4 Estimation of Non-volatile ether extract (NVEE)

The non-volatile ether extract of clove was found out using a soxhlet apparatus. Ground material (2-3g) was accurately weighed and placed in dried extraction thimble. An empty flat bottomed flask was weighed and the soxhlet extractor was connected to it. The thimble was then introduced to the extractor containing the sample.

A condenser was also attached to the extractor. Sufficient quantity of ether was poured through the condenser mount so as to cause a syphoning of ether to the flask. A further quantity of ether was poured so that its level in the extractor remained just below the syphon level. The flask was kept in a warm water bath at 55-60°C. Cold water was circulated through the condenser continuously during the extraction period.

On heating, the solvent ether vapourised and got condensed inside the condenser till the collected ether again syphoned off to the flask. Likewise repeated extractions were carried out until ten syphonings of ether were over. The condenser was later removed and the extractor was disconnected from the flask. Excess ether in the flask was evaporated off by drying the flask in an oven at 100°C for 10 minutes. The flask was then cooled and weighed. The increase in weight of flask gave the quantity of non-volatile ether extract (NVEE) present in the sample.

3.3.5 Physical characteristics of the volatile oil

The physical characteristics of clove oil such as optical rotation and refractive index were also studied. The optical rotation of oil was determined by the polarimetric method using a 'Jasco DIP-370 digital polarimeter. The refractive index of oil was determined using an 'abbe' refractometer (I.S.I., 1969).

3.3.6 Chemical analysis of clove oil

The chemical profile of the volatile oil was determined using a 'Hewlett Packard 5840 A gas chromatograph' with a built-in-electronic integrator, fitted with a column 1.825 x 0.003 m (6' x  $\frac{1"}{8}$  = i.d.)OV-17 (3%) with temperatures programmed from 80°C to 200°C at

the rate of  $5^{\circ}$ C per minute, with nitrogen as the carrier gas at a flow rate of 20 ml per minute.

Flame Ionization Detector was used for this analysis and the temperature was set at  $300^{\circ}$ C. The samples of essential oils were injected at a temperature of  $250^{\circ}$ C. The compounds present in essential oils were identified by comparing their retention times obtained in the gas chromatographic patterns with those of authentic compounds.

#### 3.4 Nutmeg

In the case of nutmeg, sampling was undertaken from fruit setting stage. The first sample was collected during October, 1988 and thereafter sampling was continued at monthly intervals until the fruits attained full maturity which was revealed by the splitting of rind and also the mace enveloping the kernel which developed a deep scarlet colour. Mature fruits were obtained during the month of April, 1989.

3.4.1 Growth and development of fruits

The length and breadth of fruits as well as kernels were recorded in centimetres using Vernier

calipers. In addition, the thickness of rind was also measured. The girth of fruits and kernels were estimated using a piece of string which was subsequently measured on a scale in centimetres. The weight of rind and kernel were recorded separately using a digital weighing balance (Sartorius make). When the mace was easily separable from the kernel, the weight of mace, kernel and rind were separately recorded. The volume of kernel, mace and rind were found out employing the water displacement method.

## 3.4.2 Estimation of moisture and volatile oil content of fruits

The moisture and volatile oil was determined by the method described earlier for clove. In nutmeg, volatile oil was separately distilled from the kernel as well as mace from February, 1989 to April 1989.

3.4.3 Analysis of oil for physico-chemical characters

A comparison of refractive index of nutmeg and mace oils at full maturity stage was carried out by means of an 'Abbe' refractometer.

The nutmeg and mace oils were stored in glass stoppered containers as suggested by Ames and Mathews (1968) in a refrigerator and were later subjected to gas chromatographic analysis. The gas chromatograph ('Hewlett Packard 5840 A' model) which was used for the analysis of clove oil was also employed for the analysis of nutmeg oil. The specifications of the gas chromatograph, viz., column, temperature programming, carrier gas and detector were the same as described earlier in the case of clove. In this case also the compounds present in the oil were identified by comparison of the retention times depicted in the gaschromatographic patterns of the experimental samples with those of authentic compounds.

3.5 Statistical analysis

Statistical interpretation of the data, obtained` from the experiments on clove and nutmeg were carried out. Analyses were done to find out the changes of growth parameters with unit change in maturity periods of flower buds and fruits.

Path coefficient analysis was done to find out the correlation between length, breadth and girth of clove flower buds and nutmeg kernels sampled at a particular period with the respective volatile oil/NVEE obtained from samples of the corresponding period.

The regression relationship for moisture content in clove flower buds as well as nutmeg kernels with the respective oil/NVEE was also found out using statistical methods.

## RESULTS

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#### 4 RESULTS

The results of investigations on quality characteristics of clove and nutmeg at different maturity stages conducted at the College of Agriculture, Vellayani in collaboration with Regional Research Laboratory, Pappanamcode are presented in the following pages.

4.1 CLOVE

4.1.1 Growth and development of clove flower buds

The length, breadth, girth, fresh weight, volume and growth rate per month of clove flower buds were recorded and are presented in Table 1. Different maturity stages of clove are shown in Plates 1 to 5.

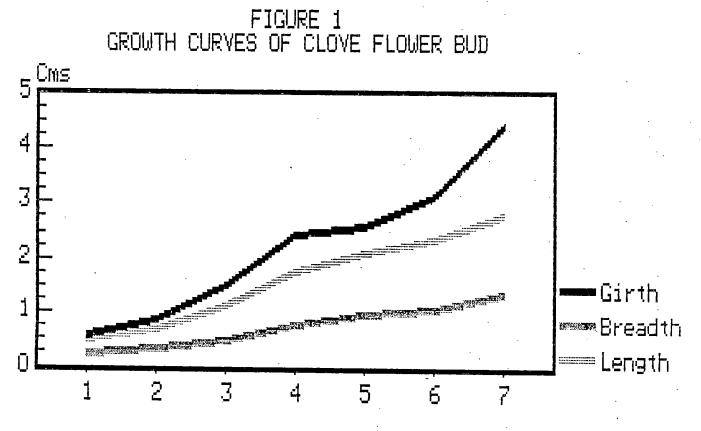
4.1.1.1 Length

The length of clove flower bud increased with increase in maturity of the bud (Fig.1) and reached the maximum (2.78 cm) at the mother-of-clove stage. The rate of increase in length varied from 0.20 cm to 0.64 cm.

Months of				Ler	ngth (a	.m)		Bre	adth (	<b>CB</b> )		Gir	th (cm	)		Fresh	weight(	g)		Volu	ume (ml)	
sampling			1	2	Mean	RI	. 1	2	Mean	RI		2	Mean	RI	1	2	Nean	RI	1,	2	Mean	RI
Pre-anthes.	is														_							
stages	1		0.56	0.40	0.48	-	0.28	0,20	0.24	-	0.66	0,50	0.58	 -	0.01	0.01	0.01	-	0.01	0.01	0,01	· –
	2 ·		0.92	0.44	0.68	0.20	0.40	0.24	0.32	0.08	1.06	0.62	0,84	0.26	0.03	0.01	0.02	0,01	0.03	0.01	0.02	0,01
	3		1.26	0.90	1.08	0.40	0,58	0.40	0.49	0.17	1.78	1.20	.1 , 49	0,65	0,17	0.06	0.12	0.10	0.16	0.06	0.11	0.09
	4	•	1.72	1.72	1.72	0.64	0.84	0,68	0 <b>.76</b>	0,27	2.40	1.88	2.41	0.92	0,24	0,19	0.22	0,10	0,20	_ <b>0,1</b> 0	0,15	0.04
Anthesis		-				·									•				١	t.		
• •	5		2.1	2.08	2.09	0.37	0.92	0.96	0.94	Ó <b>.</b> 18	2.56	2,58	2,57	0.16	0,30	0.28	0.29	0.07	0.43	0.40	0,42	0,27
Post- anthesis	•				· .																	
	6		2.38	2,24	2.31	0.22	1.08	1.02	1.05	0.11	3,26	3.00	3.13	0,56	1.34	0,82	1.08	0.79	1,40	1.00	1,20	0,78
Mother-of-	•										•							•		••	-	
	7		2.80	2.76	2.78	0,47	1.32	1.34	1.33	0,28	4.38	4.40	4,39	1.26	2,63	2.60	2.62	1.54	2.60	2.60	2.60	1.40

Table 1 Growth characteristics of clove at different maturity stages

1 - Sample 1 2 - Sample 2 RI - Rate of increase (ふ (い)



Months of Sampling

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## Plate 1. Growth and development of clove at different maturity stages

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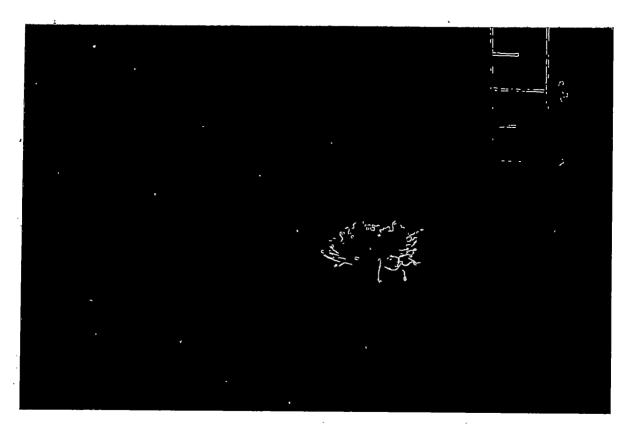


## Plate 2. Clove bud prior to anthesis

## Plate 3. Clove bud at the anthesis stage

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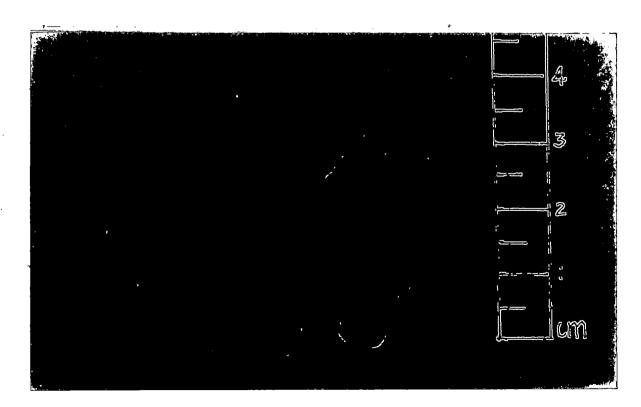


## Plate 4. Growth stages from post-anthesis to "mother-of-clove"

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### Plate5. "Mother-of-clove"





Statistical analysis of the data revealed the relationship Y = -0.0043 + 0.3989X ( $r^2 = 96$  per cent) where Y is length of clove flower bud and X, the period (month of sampling). From the above equation it was deduced that the length of clove flower bud increased by 0.3989 cm for every unit change in period (month).

4.1.1.2 Breadth

The breadth of clove flower buds also increased (Fig.1) with progressive increase in maturity of buds. The rate of increase in breadth ranged from 0.08cm to 0.28 cm. The breadth increased by 0.1850 cm for every unit change in period and was obtained from the equation, Y = -0.0071 + 0.1850X ( $r^2 = 96$  per cent) where Y is the breadth and X, the period (month).

4.1.1.3 Girth

The girth of clove flower buds showed proportionate increase (Fig.1) with advance in maturity and reached the maximum (4.39 cm) at the 'mother-of-clove' stage. The rate of increase in girth ranged from 0.16 to 1.26 cm. An increase of 0.6104 cm was observed for every unit change in period (month) as given by Y = -0.2786 + 0.6104 $(r^2 = 95$  per cent) where Y is the girth and X, the period (month) of sampling.

4.1.1.4 Fresh weight and volume

The fresh weight and volume of the clove flower buds were found to increase with increasing maturity of the buds and a maximum fresh weight (2.62 g) and volume (2.60 ml) per bud were obtained at the 'mother-of--clove' stage.

4.1.2 Moisture content

The moisture content of clove flower buds was estimated using the toluene distillation method and the data are presented in Table 2. It was found that the moisture in clove buds increased as the buds matured (Fig.2). The mean moisture content ranged from a minimum of 63.0 per cent at the first month of sampling to 74.0 per cent at the 'mother-of-clove' stage. The rate of increase was found to be the maximum at the second month of sampling.

4.1.3 Non-volatile ether extract (NVEE)

The NVEE content of clove buds obtained during different periods are depicted in Table 3. The NVEE (on dry weight basis) was found to be the maximum at the initial stage of bud development. It registered a maximum (10.33%) at the first month and progressively

	Preanthe	sis stage	Anthesis	Post- anthesis	'Mother-of- clove'	
1	2	3	4	5	6	7
64.00	66.67	69.30	70.00	71.67	<b>7</b> 3 <b>.3</b> 3	74.00
62.00	66.26	67.50	68.75	70.00	72.00	74.00
63.00	66.47	68.40	69.38	70.84	72.67	.74.00
-	3.47	1.93	0.98	1.46	1.83	1.33
	62.00 63.00	1       2         64.00       66.67         62.00       66.26         63.00       66.47	Preanthesis stage 1 2 3 64.00 66.67 69.30 62.00 66.26 67.50 63.00 66.47 68.40	62.00       66.26       67.50       68.75         63.00       66.47       68.40       69.38	Preanthesis stage       Anthesis         1       2       3       4       5         64.00       66.67       69.30       70.00       71.67         62.00       66.26       67.50       68.75       70.00         63.00       66.47       68.40       69.38       70.84	Preanthesis stageAnthesisPost- anthesis123456 $64.00$ $66.67$ $69.30$ $70.00$ $71.67$ $73.33$ $62.00$ $66.26$ $67.50$ $68.75$ $70.00$ $72.00$ $63.00$ $66.47$ $68.40$ $69.38$ $70.84$ $72.67$

# Table 2 Moisture content of clove flower buds at different maturity stages

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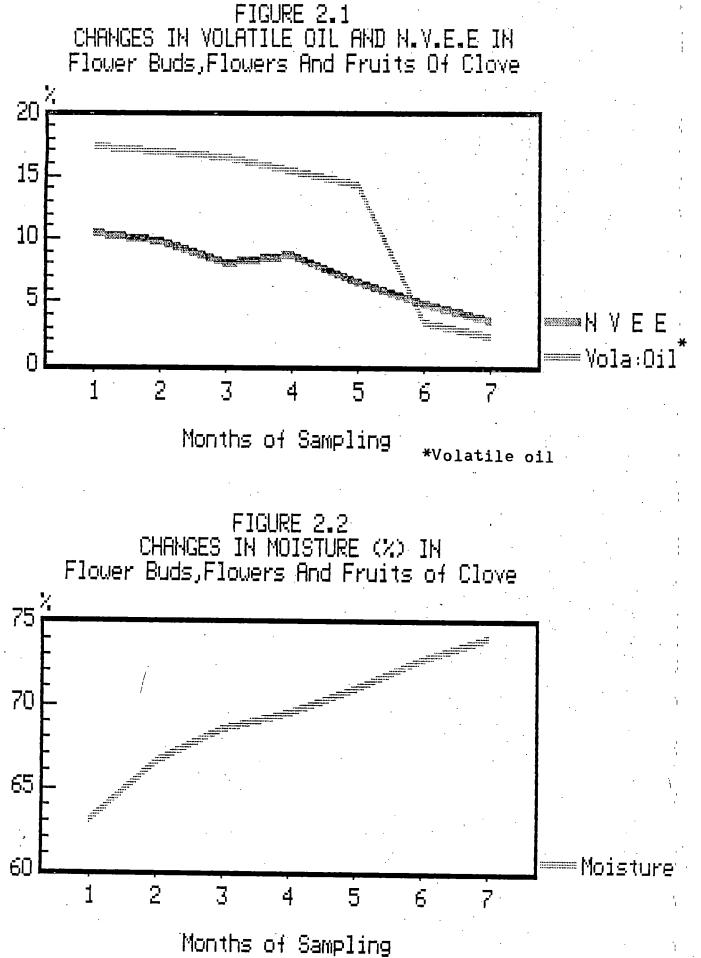
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	Months of sampling		EE (%) on	fresh weig	ght basis	NVEE(	%) on dry	weight bas	 sis
		1 ·	2	Mean	RI	1	2	Mean	RI
Pre-ant stages	hesis								
n	1.	3.76	3.88	3.82		10.44	10,21	10.33	
ñ	2	3.28	3.30	3.29	<b>-</b> 0 <b>.5</b> 3	9.84	9.78	9.81	-0.52
11	3	2.52	2.60	2.56	-0.73	8.21	8.00	8.11	-1.70
<b>11</b>	4	2.67	2.60	2.64	0.08	8.90	8.32	8.61	0.51
Anthesis stage " Post-	s 5	1.83	2.00	1.92	<b>-</b> 0.72	6.46	6.67	6.57	-2.04
anthesis "	6	1.20	1.40	1.30	-0.62	4.50	5.00	4.75	-1.82
Mother-		/e <sup>1</sup>							
f1	7	0.80	1.00	0.90	-0.40	3.08	3.85	3.47	-1.28

Table 3 NVEE of clove at different maturity stages

1- Sample 1 ; 2-Sample 2; RI - Rate of increase.

decreased to 3.47 per cent at the 'mother-of clove' stage (Fig.2).

A significant linear regression relationship was found to exist between NVEE and moisture. The variation in NVEE (84.7%) was explained (Appendix IV) by a regression relationship  $X_{10} = 50.35 - 0.62 X_6$ where  $X_{10}$  was the NVEE content (%) and  $X_6$  was the moisture content (%).

4.1.4 Direct and indirect effects of growth parameters on NVEE

The data presented in Table 4 reveals the influence of growth parameters (length, breadth, girth and weight) on the NVEE content in clove flower buds.

The correlation between length and non-volatile ether extract (NVEE) percentage was r = -0.9213 of which -1.1352 was the direct contribution of length and the remaining was the contribution of length indirectly through other growth parameters. The indirect effects through girth and weight were negative while that of breadth was positive.

Length	Breadth	Girth	Fresh weight	Correlation
- <u>1,1352</u>	1.1329	<b>-0</b> ,7198	-0.2092	-0.9213
<b>-</b> 1.1277	1.1405	-0.7297	-0.2190	-0.9359
-1.1099	1.1305	<u>-0.7361</u>	<b>-</b> 0.2347	-0,9503
-0.8982	0.9445	-0.6536	<u>-0.2644</u>	-0.8717
	- <u>1,1352</u> -1.1277 -1.1099	- <u>1,1352</u> 1.1329 -1.1277 <u>1.1405</u> -1.1099 1.1305	-1.1352   1.1329   -0.7198   -1.1277   1.1405   -0.7297   -1.1099   1.1305   -0.7361	-1.1352 $1.1329$ $-0.7198$ $-0.2092$ $-1.1277$ $1.1405$ $-0.7297$ $-0.2190$ $-1.1099$ $1.1305$ $-0.7361$ $-0.2347$

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Table 4	Direct and	indirect	effects of	f arowth	parameters	on
	NVEE conter	nt of clow	/e	5-0	P	011

Residual effects = 0.2834

The diagonal values (underlined) are the direct effects and the horizontal values are the indirect effects.

**\*\*** Significant at 1 per cent level

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The correlation between breadth and NVEE was  $r = -0.9359^{**}$  and its direct effect was positive (1.1405). The indirect effects through length, girth and weight of flower buds (which were negative) masked the direct effect of breadth (positive) resulting in negative correlation.

The correlation between girth and NVEE was r = -0.9503 of which the direct contribution of girth was -0.7361. The remaining was the contribution indirectly through length, breadth and weight of flower bud. The indirect effect through length and weight were negative while the indirect effect through breadth was positive.

The correlation between weight and NVEE was -0.8717 of which -0.2644 was the direct effect of weight of flower bud. The indirect effects through length and girth were negative and that through breadth was positive.

From the above results it is clear that breadth influenced directly and indirectly in a positive way while the direct and indirect effect of the other growth parameters were negative. The direct effect of growth parameters contributed to 72.0 per cent variation in NVEE content of clove buds.

#### 4.1.5 Volatile oil of clove

Table 5 depicts the percentage of volatile oil obtained by steam distillation of clove at different maturity stages.

The percentage of volatile oil obtained on dry weight basis was found to register the maximum value of 17.40 per cent at the first month of sampling followed by a progressive decrease registering a low volume of 2.23 per cent at the 'mother-of-clove' stage (Fig.2).

A significant linear regression relationship of the form,  $X_8 = 109.91 - 1.41 X_6$  (where  $X_8$  is oil percentage and  $X_6$  is the moisture percentage) was found to exist between oil and moisture. The variation in oil (67.50%) was explained by the above regression relationship (Appendix IV).

4.1.6 Direct and indirect effects of growth parameters on volatile oil

The result of the above analysis is presented in Table 6. Path co-efficient analysis was conducted to study

Volatil	e oil (%)	on fresh w	Volatile oil(%) on dry weight basis				
1	2	Mean	RI	1	2		RI
					· <u> </u>		
6.21	6.67	6.44		17.25	17.55	17。40	
5.56	5.71	5.64	-0.80	16.68	16.92	16.80	-0.60
5.00	5,36	5.18	<b>-</b> 0.46	16.29	16.49	16.39	<u>-</u> 0.41
4.52	4.84	4.68	-0,50	15.07	15.49	15,28	-1.11
4.00	4.33	4.17	-0,51	14.12	14.43	14.28	-1.00
-	0.94	0.90	-3.27	3.19	3.36	3.28	-11.00
r .							
0.57	0.59	0,58	<b>-</b> 0.32	2.19	2.27	2,23	-1.05
	1 6.21 5.56 5.00 4.52 4.00 0.85	1       2         6.21       6.67         5.56       5.71         5.00       5.36         4.52       4.84         4.00       4.33         0.85       0.94	1       2       Mean         6.21       6.67       6.44         5.56       5.71       5.64         5.00       5.36       5.18         4.52       4.84       4.68         4.00       4.33       4.17         0.85       0.94       0.90	4       Mean       R1         6.21       6.67       6.44          5.56       5.71       5.64       -0.80         5.00       5.36       5.18       -0.46         4.52       4.84       4.68       -0.50         4.00       4.33       4.17       -0.51         0.85       0.94       0.90       -3.27	$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	12MeanRI12 $6.21$ $6.67$ $6.44$ $17.25$ $17.55$ $5.56$ $5.71$ $5.64$ $-0.80$ $16.68$ $16.92$ $5.00$ $5.36$ $5.18$ $-0.46$ $16.29$ $16.49$ $4.52$ $4.84$ $4.68$ $-0.50$ $15.07$ $15.49$ $4.00$ $4.33$ $4.17$ $-0.51$ $14.12$ $14.43$ $0.85$ $0.94$ $0.90$ $-3.27$ $3.19$ $3.36$	12MeanRI12Mean $6.21$ $6.67$ $6.44$ $17.25$ $17.55$ $17.40$ $5.56$ $5.71$ $5.64$ $-0.80$ $16.68$ $16.92$ $16.80$ $5.00$ $5.36$ $5.18$ $-0.46$ $16.29$ $16.49$ $16.39$ $4.52$ $4.84$ $4.68$ $-0.50$ $15.07$ $15.49$ $15.28$ $4.00$ $4.33$ $4.17$ $-0.51$ $14.12$ $14.43$ $14.28$ $0.85$ $0.94$ $0.90$ $-3.27$ $3.19$ $3.36$ $3.28$

Table 5 Volatile oil content of clove at different maturity stages

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	Length	Breadth	Girth	Fresh weight	Correlation
Length	-0.4627	-0.8748	1.2215	-0.7229	-0.8389**
Breadth	-0.4597	<u>-0.8806</u>	1.2384	-0.7567	-0.8586**
Girth	<b>-</b> 0 <b>.</b> 4524	<b>-</b> 0.8729	1.2492	-0.8113	<b>-</b> 0.8874 <sup>**</sup>
Fresh weight	-0.3661	-0.7293	1.1092	- <u>0.9137</u>	-0.8999**

Table 6 Direct and indirect effect of growth parameters on clove oil

Residual effects = 0.3770

The diagonal values (underlined) are the direct effects and the horizontal values are the indirect effects.

\*\* Significant at 1 per cent level.

Å S the direct and indirect effects of length, breadth, girth and weight of clove buds on the volatile oil content.

The correlation between length and oil was r = -0.8389 of which -0.4627 was the direct contribution of length and remaining was the contribution of length indirectly through the other parameters. The indirect of breadth and weight of flower buds were negative while that of girth was positive.

The correlation between breadth and oil content was -0.8586 in which the direct effect of breadth was -0.8806. The difference between direct effect and correlation was marginal, which was evidently due to the indirect effects of breadth through the other growth parameters.

The correlation between girth and oil content was -0.8874 but its direct effect was positive (1.2492). Here the indirect effect of girth through the other growth parameters studied (which were negative) masked the direct effect of girth (positive) resulting in a negative correlation.

The direct effect of weight on volatile oil and its correlation were negative and nearly equal, thereby giving an implication that the correlation was mainly attributed due to the fresh weight of flower buds.

From the above results it is obvious that girth influenced directly and indirectly in a positive manner the volatile oil content, while the direct and indirect effects of other growth parameters were negative. The direct effect of the growth parameters studied accounted for 62.3 per cent of the variation in oil content.

4.1.7 Physical properties of clove oil

The physical properties of clove oil, namely, optical rotation and refractive index were studied and are presented in Table 7.

4.1.7.1 Optical rotation

The optical rotation in clove oil extracted from unopened flower bud was maximum  $(-4^{\circ} 19')$  at the third month of sampling. At the flowering stage it was found to decrease and the optical rotation showed a substantial increase at the fruit maturity stages and it was the highest at the 'mother-of-clove' stage( $-9^{\circ} 28'$ ).

Physical properties	Months of sampling										
	1	2	3	4	5	6	7				
Optical rotation <sup>*</sup>	-2 <sup>0</sup> 18	-1°60'	-4 <sup>0</sup> 19	-3 <sup>°</sup> 76	-2°28	-6°32'	-9 <sup>0</sup> 28				
Refractive index <sup>*</sup>	1.524	1.517	1.514	1.521	1.525	1.524	1.523				

Table 7 Physical properties of clove oil at different maturity stages

\* Mean values of two samples

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4.1.7.2 Refractive index

The refractive index of clove oil was 1.524 at the initial maturity stage (1st month) which fell at the 2nd, 3rd and 4th months of sampling, rose slightly at the 5th month (1.525) which fell progressively upto the 'mother-of-clove' stage.

4.1.8 Changes in flavour components of clove oil at different maturity stages

The clove oil obtained by steam distillation of clove buds was subjected to gas chromatographic analysis. The data presented in Table 8 depicts the changes in major aromatic components of clove oil. The results of the analysis are given below.

4.1.8.1 Alpha-cubebene

The maximum alpha-cubebene concentration during the pre-anthesis stage was seen at the third month of maturity of clove (2.50%). At the anthesis stage it dropped to 1.12 per cent and thereafter at the 'mother-ofclove' stage, the highest value of 3.56 per cent was recorded.

Components of clove oil	Months of sampling									
(concentration in %)	Pre-anthesis stages				Anthesis	Post- anthesis	'Mother-of- clove'			
	1	2	3	4	5	6	7			
Alpha-cubebene	0.49	0.78	2,50	1.67	1.12	1.19	3.56			
Alpha-copaene	0.69	0.84	2,28	1.54	0.94	1.36	3.36			
Beta-caryophyllene	10.21	14.02	28.26	14.82	10.23	19.13	23,32			
Eugenol	65.22	36.18	56.32	51,96	73.12	66.78	54.70			
Cis-isoeugenol	Trace	1.17	1.78	2,50	2.41	Trace	2.75			
Trans <b>-</b> isoeugenol	0.94	0.68	1.58	1.64	1.17	2.21	4.95			
Eugenol acetate	20.92	41.16	4.57	22.56	6.28	2.09	1.55			
Isoeugenol acetate	. –	-	-	-	-	0.91	2.59			

Table 8 Flavour characteristics of clove oil at different maturity stages

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# 4.1.8.2 Alpha-copaene

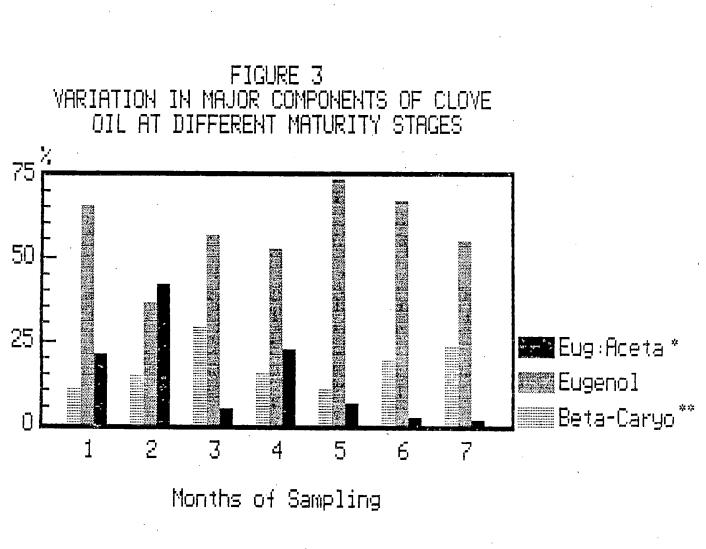
The concentration of alpha-copaene showed a trend almost similar to that of alpha-cubebene. During the pre-anthesis stages the maximum concentration (2.28%) was seen at the third month of maturity. The opened flowers had a low alpha-copaene concentration (0.94%) and a high of 3.36 per cent was reached at the 'mother-ofclove' stage.

## 4.1.8.3 Beta-caryophyllene

The beta-caryophyllene concentration was found to increase from the first month of sampling (Fig.3). It reached a maximum concentration (28.26%) at the third month and subsequently decreased till the fifth month (anthesis stage). Further increase was observed from the sixth month and finally attained a concentration of 23.32 per cent at the 'mother-of-clove' stage.

4.1.8.4 Eugenol

The eugenol concentration was found to be relatively high (65.22%) at the first month of sampling. Since then, the concentration decreased and increased alternately until a value of 73.12 per cent (maximum) was observed



\* Eugenol acetate
\*\* Beta-caryophyllene

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at the anthesis stage. A slight decline was observed at the final sampling stages, attaining a value of 54.70 per cent at the 'mother-of-clove' stage (Fig.3).

## 4.1.8.5 Cis-isoeugenol

The cis-isoeugenol concentration showed a gradual increase registering only traces at the first month and attained a value of 2.50 per cent at the fourth month stage. Again, at the sixth month stage only traces of this compound was detected and finally attained a rather high value (2.75%) at the 'mother-of-clove' stage.

# 4.1.8.6 Trans-isoeugenol

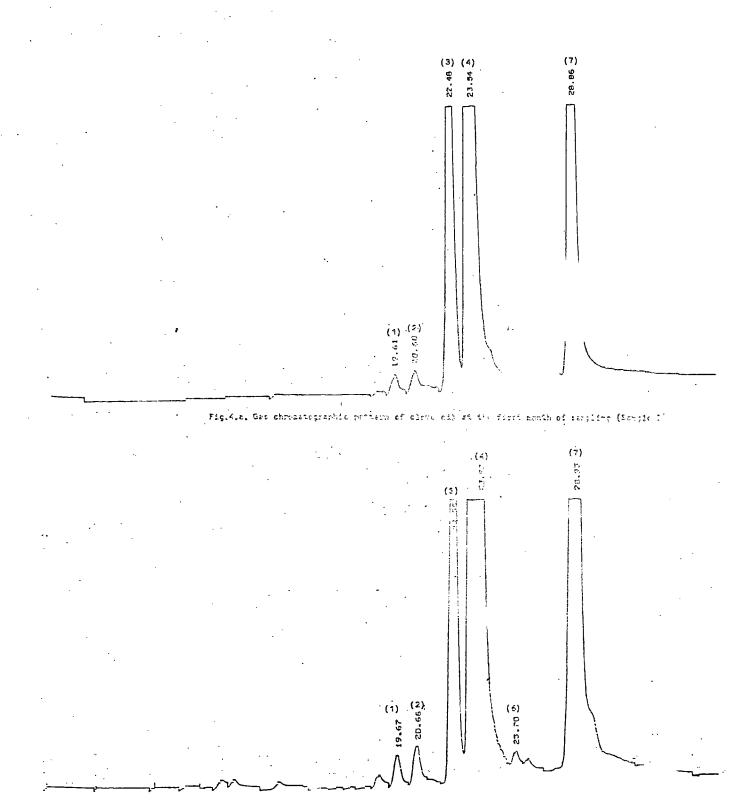
The concentration of trans-isoeugenol showed alternating decreasing and increasing trends upto the anthesis stage. During the 6th and 7th month periods, rather high values (2.21 and 4.95 per cent respectively) were observed in the oil.

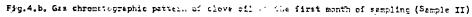
#### 4.1.8.7 Eugenol acetate

The level of eugenol acetate was found to increase from 20.92 per cent(1st month) to 41.16 per cent (2nd month) and then showed a steep fall to 4.57 per cent (3rd month) (Fig.3). The level of this component, however, increased

# Components of clove oil

- (1) Alpha-cubebene
- (2) Alpha-copaene
- (3) Beta caryophyllene
- (4) Eugenol
- (5) Cis-isoeugenol
- (6) Trans-isoeugenol
- (7) Eugenol acetate
- (8) Isoeugenol acetate





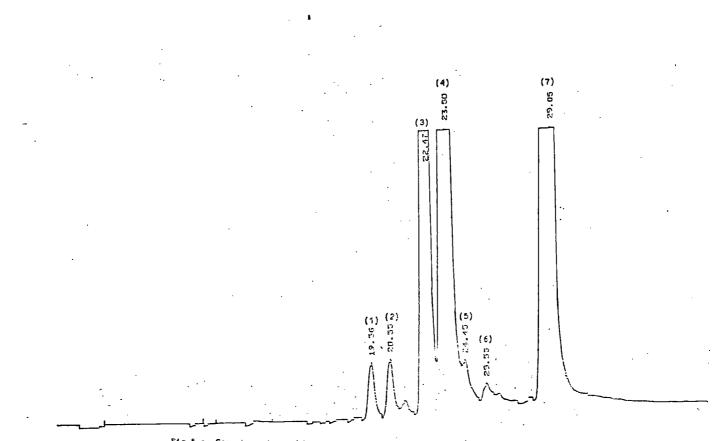
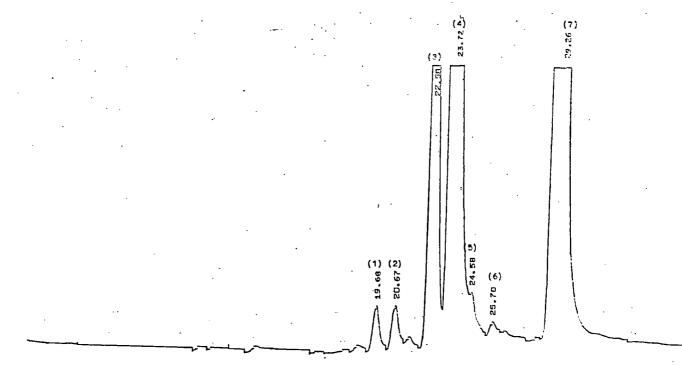
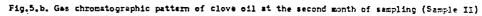


Fig.5.a. Ge: chromatographic pattern of clove cil at the second month of sampling (Sample I)





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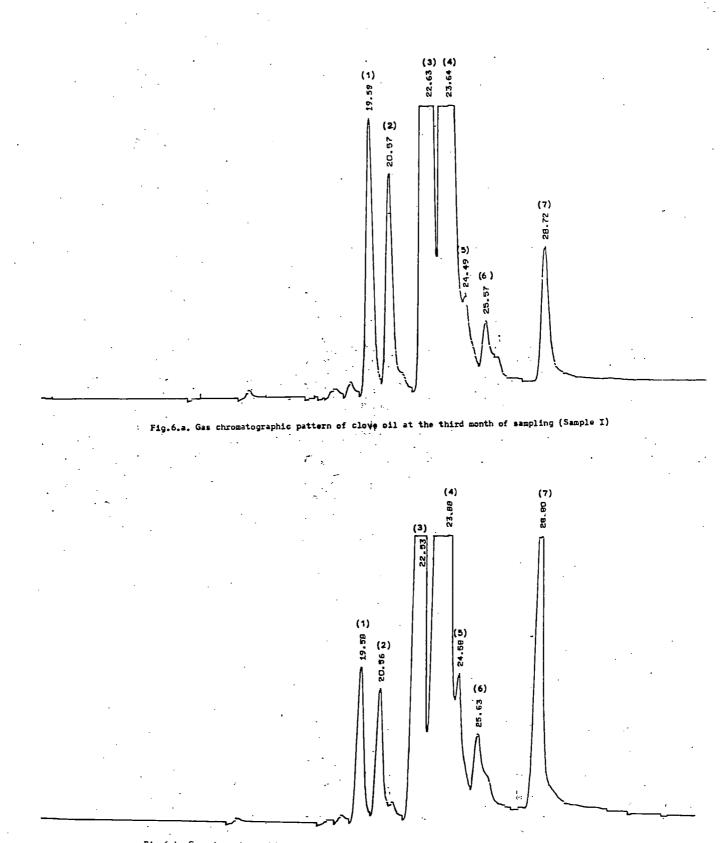


Fig.6.b. Gas chromatographic pattern of clove oil at the third month of sampling (Sample II)

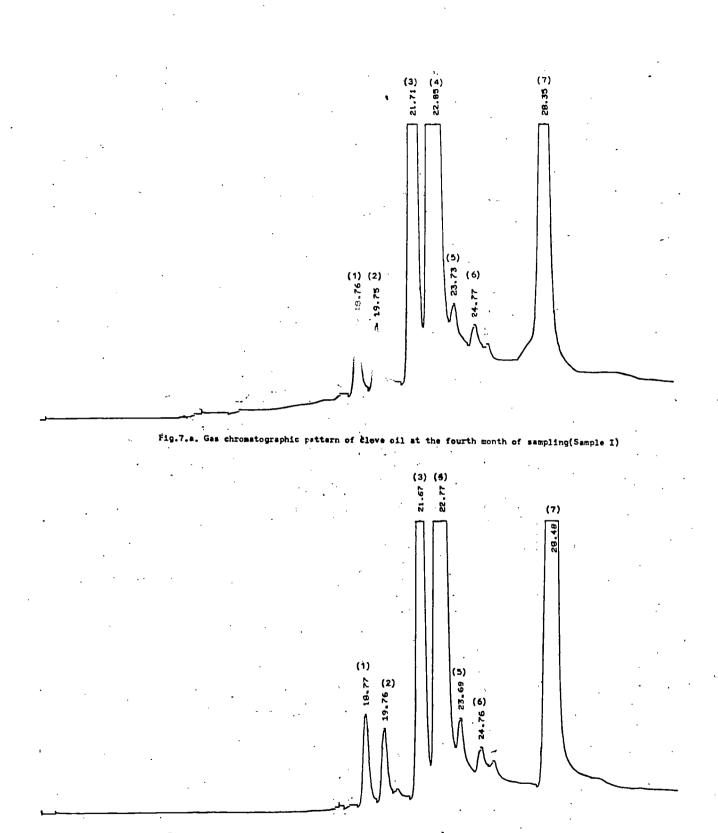


Fig.7.b. Gas chromatographic pattern of clove oil at the fourth month of sampling (Sample II)

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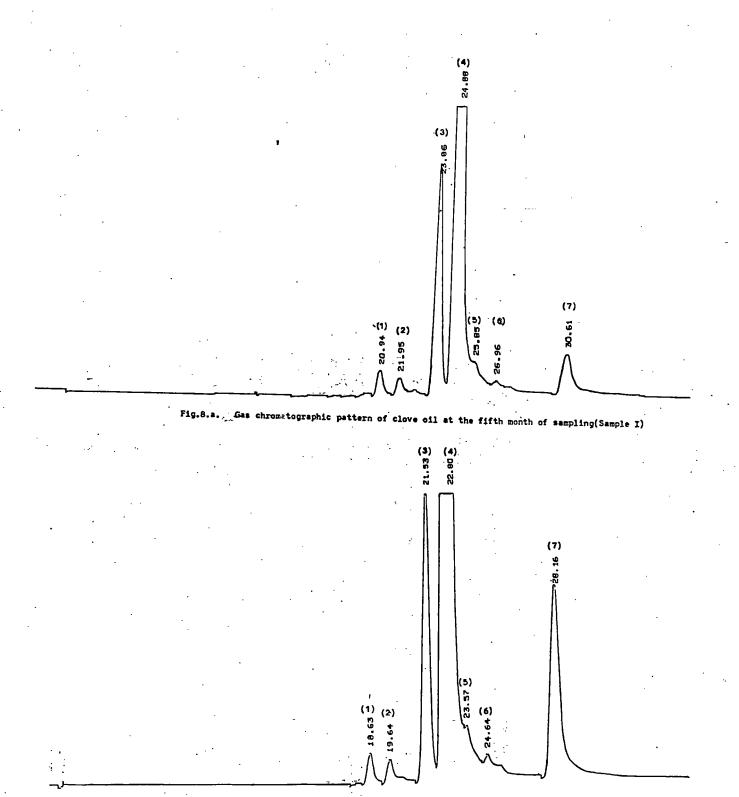
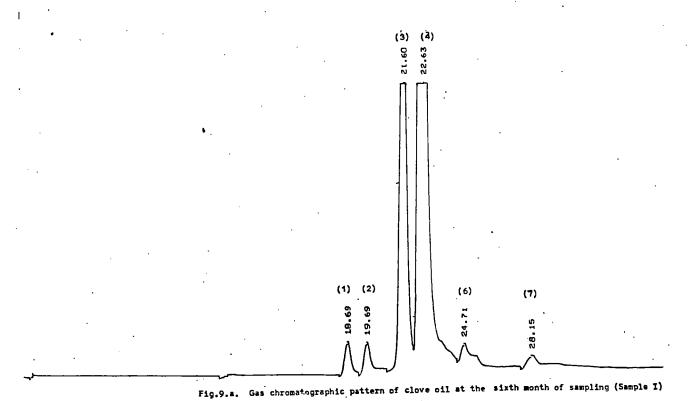
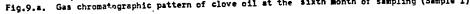
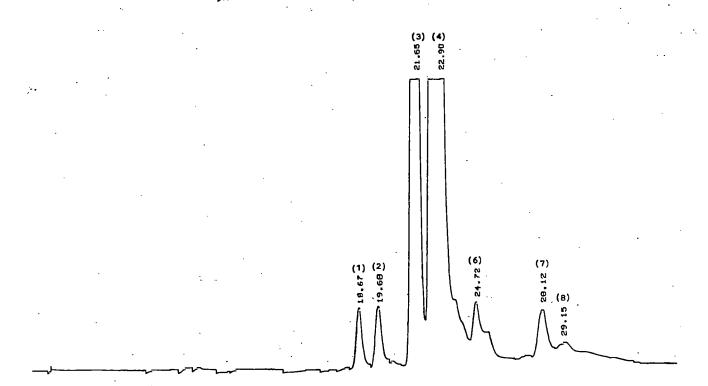
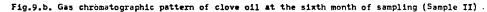


Fig.8.b. Gas chromatographic pattern of clove oil at the fifth month of sampling (Sample II)









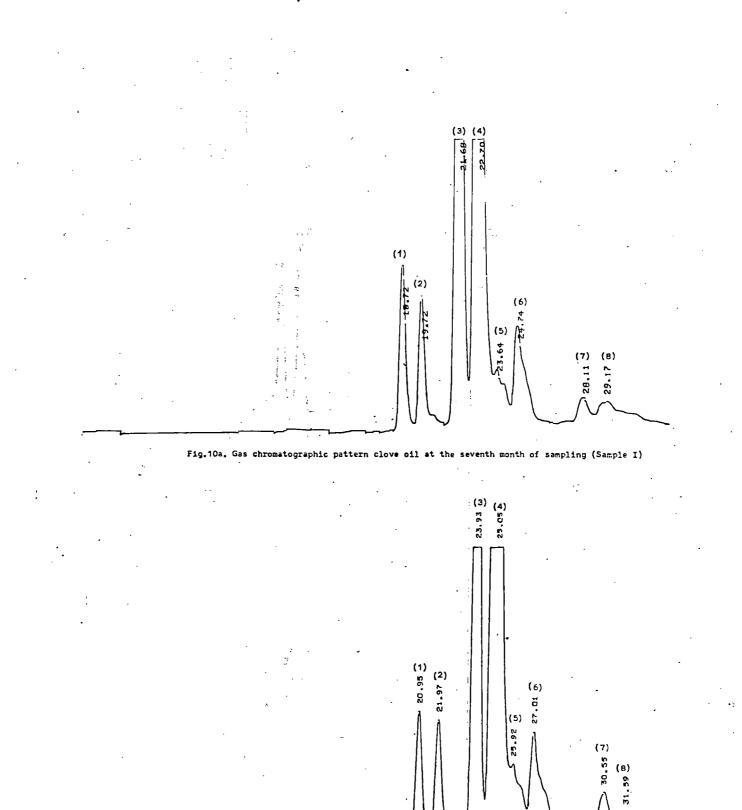


Fig.10.b. Gas chromatographic pattern of clove oil at the seventh month of sampling (Sample II)

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to 22.56 per cent (4th month) and thereafter gradually declined to 1.55 per cent at the final harvesting ('mother-of-clove') stage.

4.1.8.8 Isoeugenol acetate

The gas chromatographic patterns did not reveal the presence of this compound until the anthesis stage of clove. A level of 0.91 per cent was detected at the post-anthesis stage which rose to 2.59 per cent at the 'mother-of-clove' stage.

The gas chromatographic patterns of clove oil are depicted in Figures 4 to 10.

4.2 NUTMEG

4.2.1 Growth and development of nutmeg fruits

In nutmeg, the growth characteristics were studied separately for the whole fruit, kernel and mace. The length, breadth, girth, weight, volume and growth rate of these characters were recorded at monthly intervals and are presented in Table 9. The nutmeg fruits at various maturity stages are depicted in Plate 6.

Month of sampling	Length (cm)	RI (cm)	Breadth (cm)	RI (cm)	Girth (cm)	RI (cm)	Fresh weight (g)	RI (g)	Volume (ml)	RI (ml)
1	1.90		1.20		3.80		1.04		1.00	
2	3.30	1.40	2.20	1.00	6.90	3.10	2.64	1.60	1.47	0.47
3	4.10	0.80	2.83	0.63	9.00	2.10	11.57	8.93	12,56	11.09
4	4.80	0.70	3.80	0.97	12.08	3.08	28.10	16.53	24.75	12.19
5	5.51	0.71	4.78	0.98	14.96	2.88	55.36	27.26	55,50	30.75
6	5.80	0.29	<b>5.4</b> 0 ·	0.62	16.45	1.49	67.44	12.08	66.00	10,50
7	6.30	0.50	5.90	0.50	17 <b>.</b> 50	1.05	75.39	7.95	71.97	5.97

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Table 9 Growth characteristics of nutmeg fruits at different maturity stages

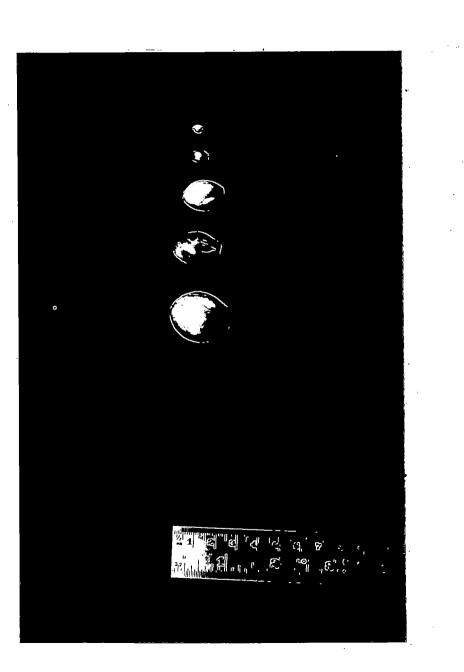
RI - Rate of increase

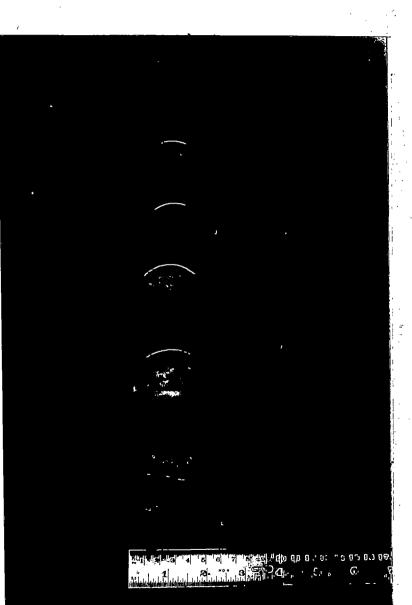
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# Plate 6. Nutmeg fruits at different growth stages

Plate 7. Nutmeg kernels at different growth stages





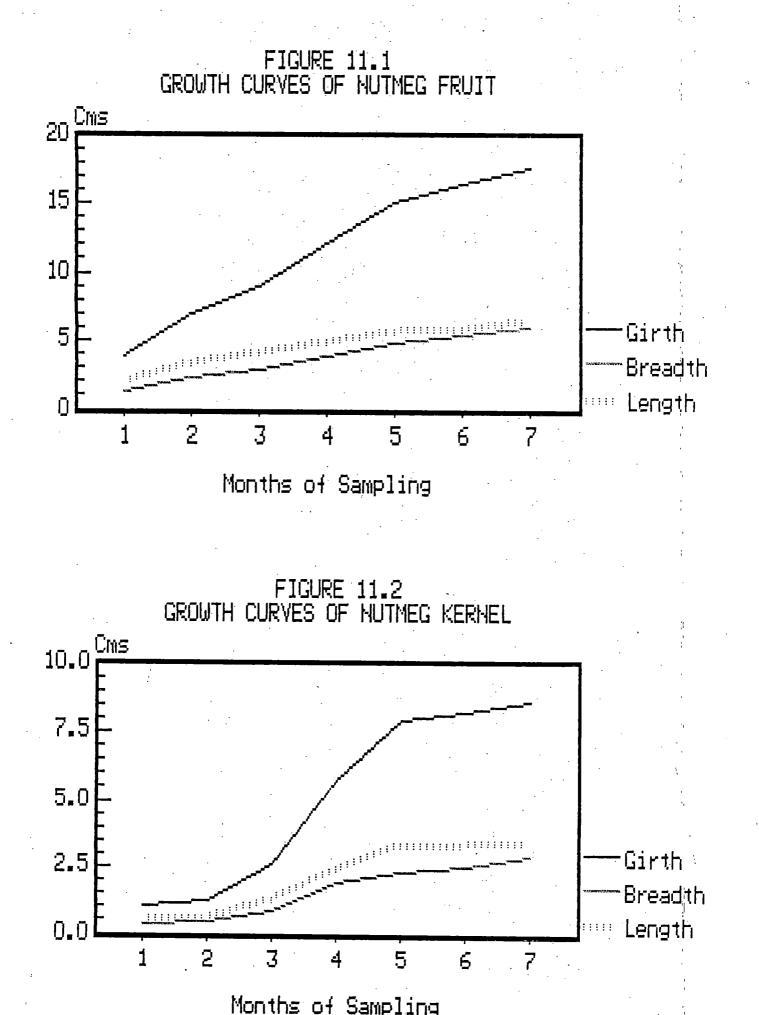
## 4.2.1.1 Length

The length of nutmeg fruits increased with increase in maturity (Fig.11) and reached the maximum (6.30 cm) at the final stage of harvest. The rate of increase varied from 0.29 cm to 1.40 cm and it was relatively high between the first and second months of sampling, which declined at the final maturity stages. Statistical analysis of the data revealed the relationship,  $Y = 1.7286 + 0.7004 X (r^2 = 95 per cent)$  where Y is the length of nutmeg fruits and X is the period (month). This relationship indicated that the length of nutmeg fruits increased by 0.7004 cm for every unit increase in period.

# 4.2.1.2 Breadth

The breadth of nutmeg fruit was found to increase progressively as maturity of the fruit advanced(Fig.11). The breadth recorded at full maturity stage was 5.90 cm and the rate of increase ranged from 0.50 to 1.00 cm, the highest being observed between first and second months.

The breadth increased by 0.8108 cm for every unit change in period as evident from the equation,  $Y = 0.5229 + 0.8018 X(r^2 = 99 \text{ per cent})$  where Y is the



breadth and X is the period (month).

4.2.1.3 Girth

The girth of nutmeg fruit increased with increase in age and reached the maximum (17.50 cm) at the final stage (7th month) of sampling. The rate of increase ranged from 1.05 cm to 3.10 cm, the highest being recorded between the first and second months of sampling (Fig.11).

The girth was found to increase by 2.3629 cm for every unit change in period and was evident from the relationship Y =  $2.0757 + 2.3629X(r^2 = 98 \text{ per cent})$  where Y is the girth and X is the period (month).

4.2.1.4 Fresh weight and volume

The fresh weight and volume of nutmeg fruits were found to increase with advancement of maturity, and reached the maximum value (75.39 g and 71.97 ml respectively) at the seventh month of sampling. The highest rate of increase in fresh weight and volume (27.26g and 30.75 ml respectively) were recorded between the fourth and fifth months of sampling.

4.2.2 Growth and development of nutmeg kernel

The data pertaining to growth and development of kernels are depicted in Table 10. Nutmeg kernels at

Table 10 Growth characteristics of nutmeg kernel at different maturity stages

Months of sampling	Length (cm)	RI (cm)	Breadth (cm)	RI (cm)	Girth (cm)	RI (cm)	Fresh weight (g)	RI (g)	Volume (ml)	RI (ml)
1	0.40	<b></b>	0.30		1.00		0.08		0.17	
2	0.50	0.10	0.40	0.10	1.25	0.25	0.13	0.05	0.19	0.02
3	1.20	0.70	. 0.80	0.40	2.60	1.35	0.30	0.17	0.33	0.14
4	2.37	1.17	1.90	1.10	5.78	3.18	3.80	3.50	3.95	3.62
5	3.25	0.88	2.35	0.45	7.93	2.15	9.19	5.39	8.00	4.05
6	3.30	0.05	2.47	0.12	8.20	0.27	9.64	0.45	8.50	0.50
7	3,35	0.05	2.90	0.43	8.60	0.40	10.02	0,38	10.30	1.80
	RI - Rate	of incr	:ease			- <u></u>				

រ ប different maturity stages are shown in Flates 7 and 8. 4.2.2.1 Length

The length of nutmeg kernel increased with advancing maturity of the fruits (Fig.11). The kernel length reached the maximum of 3.35 cm at the final stage of sampling. The rate of increase in length varied from 0.05 cm to 1.17 cm.

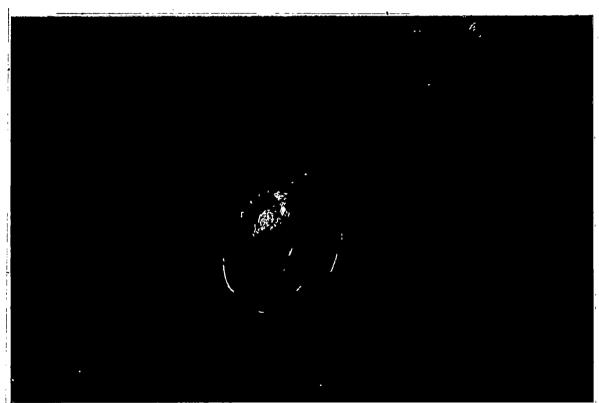
The length was found to increase by 0.5893 cm for every unit change in period as indicated by the statistical model Y = -0.3043 + 0.5893X ( $r^2$  = 91 per cent) where Y is the length of kernel and X is the period(month) of sampling.

#### 4.2.2.2 Breadth

The breadth of nutmeg kernel reached a maximum value of 2.90 cm at final stage (seventh month) of harvest (Fig.11). The rate of increase recorded ranged from 0.10 to 1.10 cm. The statistical model Y = -0.3386 + 0.4818X( $r^2 = 95$  per cent) where Y is the breadth of kernel and X is the period (month), revealed that the breadth increased by 0.4818 cm for every unit increase in period.

Plate 8. Nutmeg kernel with mace

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#### 4.2.2.3 Girth

The girth of kernels showed a rate of increase ranging from 0.27 cm to 3.18 cm. The maximum girth (8.60 cm) was recorded at the final maturity stage (Fig.11). The increase in girth was 1.5011 cm for every unit change in period as given by the equation Y = -0.9529 + 1.5011X ( $r^2 = 93$  per cent) where Y is the girth of kernel and X is the period (month).

# 4.2.2.4 Fresh weight and volume

The fresh weight and volume of kernel were found to increase with increase in maturity of the nutmeg fruits. The maximum fresh weight (10.02g) and volume (10.30 ml) were recorded at the final sampling stage.

4.2.3 Growth characteristics of mace

The mace was easily separable from kernels at the final three stages of sampling and the physical parameters like fresh weight and volume were recorded from the mace so separated from kernels. The fresh weight of mace recorded at these stages were 0.97, 1.73 and 2.29 g and their corresponding volumes were 1.38, 2.00 and 2.10 ml respectively. 4.2.4 Moisture content of nutmeg

The moisture content of rind and kernel were recorded separately and are presented in Table 11. The moisture in mace was also determined at the final three stages of sampling.

4.2.4.1 Moisture content of rind

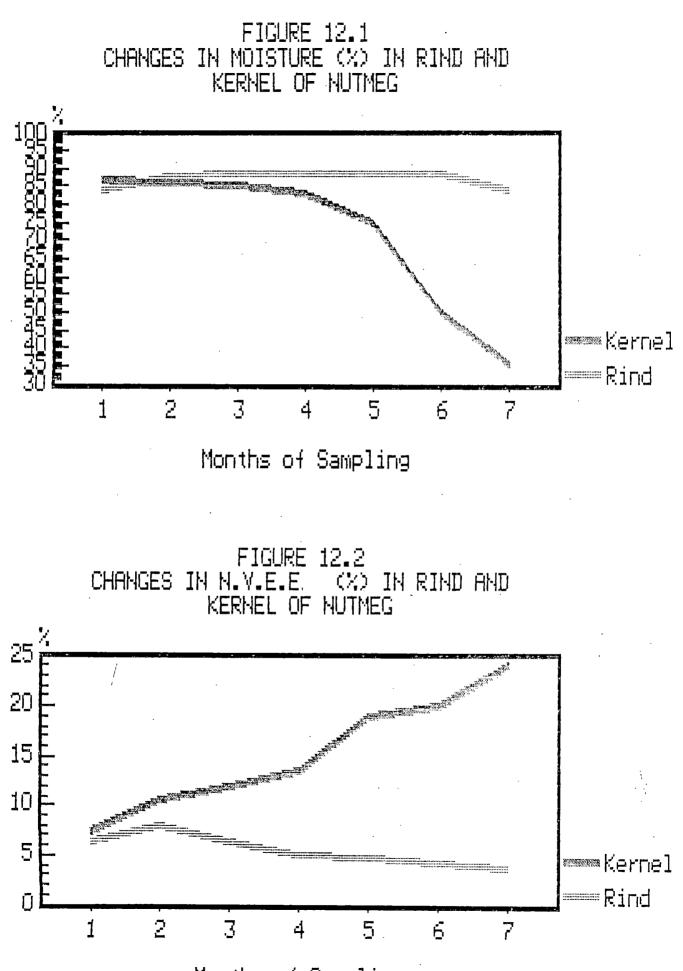
The moisture content of rind was found to increase upto the third month (84.00 to 88.00%) and thereafter remained steady until the 6th month (88.00%) which further dropped to 84.00 per cent at the final stage (seventh month) of sampling (Fig.12).

4.2.4.2 Moisture content of kernel

The moisture content of kernel was the maximum (86.67%) at the first month and then was progressively reduced to 36.00 per cent at the final stage of sampling (Fig.12).

4.2.4.3 Moisture content of mace

The moisture content of mace was found to decrease with increase in maturity. During the final three maturity stages (5th, 6th and 7th months) the moisture



Months of Sampling

Months of	Rind		Kernel		
sampling	Moisture (%)	RI (%)	Moisture (%)	RI (%)	
1	84.00		86.67		
2	87.50	3.50	85.71	-0.96	
3	88.00	0,50	85.00	-0.71	
4	88.00	0.00	83.33	<b>-1.</b> 67	
5	88.00	0.00	75.00	-8.33	
6	88.00	0.00	50.00	-25,00	
7	84.00	_4.00	36.00	-14.00	
•	<b>—</b> • • • •		· .		

Table 11 Moisture content of rind and kernel of nutmeg fruit at different maturity stages

# RI - Rate of increase

values registered were 70.00, 68.00 and 50.00 per cent respectively.

4.2.5 NVEE of nutmeg fruits

The non-volatile ether extract content in nutmeg fruits (rind as well as kernel) were estimated and are presented in Table 12.

The NVEE of mace was determined at the final three stages of sampling when the mace was easily separable from kernel.

4.2.5.1 NVEE of rind

The non-volatile ether extract (NVEE) on fresh weight basis in rind was found to decrease from an initial high value (1.00%) to a low value at the 6th month (0.50%). When the NVEE was computed on dry weight basis it was found to increase from an initial value of 6.25 per cent to 8.00 per cent at the second month and thereafter progressively declined and reached a minimum of 3.75 per cent at the final stage (Fig.12). The regression relationship obtained between NVEE and moisture content was however non-significant.

#### 4.2.5.2 NVEE of kernel

The NVEE of kernel (on dry weight basis) showed an increasing trend from 7.50 per cent at the first month and reached 23.95 per cent at the seventh month of sampling (Fig.12). The rate of increase ranged from 1.00 to 3.95 per cent.

A significant linear regression relationship was found to exist between moisture content of kernel and NVEE. Variation in NVEE (80.73%) was explained by the regression relationship,  $X_{13} = 33.84 - 0.26 X_7$  where  $X_{13}$ is the NVEE(%) in kernel and  $X_7$  is the moisture content(%) (Appendix V).

# '4.2.5.3 NVEE of mace

The NVEE of mace was estimated and recorded at the final three stages of sampling when the mace was easily separable from the kernel. The NVEE percentage ( on dry weight basis) was found to decrease with ageing of the fruits. The value obtained at the final three stages were 32.10, 28.13 and 23.00 per cent respectively. However on fresh weight basis the NVEE percentage was found to increase at the final stages of sampling and the

values registered were 9.63, 9.00 and 11.50 per cent respectively.

4.2.6 Direct and indirect effect of growth parameters on NVEE in nutmeg

The data presented in Table 13 shows the influence of growth parameters, viz; length, breadth, girth and weight of kernel on NVEE content of nutmeg kernel.

The correlation between length and NVEE was  $r = 0.9364^{**}$  and its direct effect was 6.8542. The indirect effect of length through girth was -12.4064 which nullified the direct effect of length and indirect effects of breadth and fresh weight (all positive).

The correlation between breadth and NVEE was  $r = 0.9542^{**}$  and its direct effect was 4.4578. The reduced value for correlation was mainly due to the indirect effect of breadth through girth (-12.3554) masking the direct effect of breadth.

The correlation between girth and NVEE was  $r = 0.9458^{**}$  and its direct effect was -12.4263. A positive correlation between girth and NVEE was obtained because the direct effect of girth was dominated by the indirect effects of other growth parameters (all negative). Table 13 Direct and indirect effects of growth parameters on NVEE in nutmeg

	Length	Breadth	Girth	Fresh weight	Correlation
Length	6.8542	4.4105	-12.4064	2.0781	0.9364**
Breadth	6.7815	4.4578	<del>-</del> 12.3554	2,0704	0.9543**
Girth	6.8432	4.4323	-12.4263	2.0966	0.9458**
Fresh weight	6.6191	4.2888	-12,1069	2.1519	0.9529**

Residual effects = 0.1786

The diagonal values(underlined) are the direct effects and the horizontal values are the indirect effects.

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\*\* Significant at 1 per cent level

The correlation between weight and NVEE was  $r = 0.9529^{**}$  and the direct effect of weight was 2.1519. The indirect effect of fresh weight through girth was rather high (-12.1069), the other indirect effects through length and breadth being positive.

From the above results it is evident that the length, breadth and fresh weight of nutmeg fruits influenced directly in a positive way while the direct and indirect effects of girth was negative. The direct effects of growth parameters studied attributed to 82.00 per cent of the variation in NVEE of fruits.

4.2.7 Volatile oil from nutmeg fruit

Two types of volatile oil were obtained from nutmeg fruits. The kernel of the fruit yields nutmeg oil and mace oil is extracted from the mace.

4.2.7.1 Nutmeg oil

Table 14 depicts the percentage of nutmeg oil obtained by distillation of nutmeg kernel at the different maturity stages.

The percentage of nutmeg oil obtained (on dry weight basis) was found to register a maximum value of 20.01 per cent at the second month of sampling and

Months of sampling									
1	2	3	4	5	6	7			
2.56	2.86	1.74	1.59	2.12	3.78	4.65	- <u>-</u>		
0.00	0.30	-1.12	-0.15	0.53	1.66	0.87			
19.20	20.01	11.60	9.54	8.48	7.56	7.27			
0.00	0.81	-8.41	-2.06	-1.06	-0.92	-0.29	-		
	0.00 19.20	2.56 2.86 0.00 0.30 19.20 20.01	1     2     3       2.56     2.86     1.74       0.00     0.30     -1.12       19.20     20.01     11.60	$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	$\begin{array}{c ccccccccccccccccccccccccccccccccccc$		

Table 14 Volatile oil in nutmeg at different maturity stages

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thereafter it progressively reduced, registering a value of 7.27 per cent at the final stage of sampling (Fig.13).

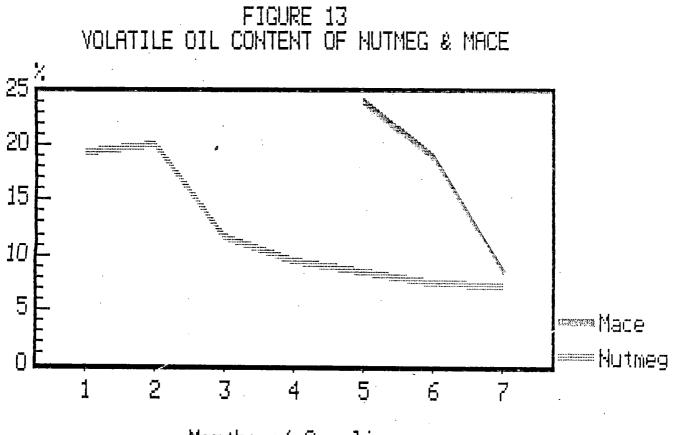
The regression relationship  $X_9 = -0.735 + 0.177 X_7$ between moisture  $(X_7)$  and oil  $(X_9)$  was however found to be non-significant. This regression relationship explained for 44.05 per cent of the variation in oil (Appendix V).

4.2.7.2 Mace oil

The mace oil was found to decrease with increase in maturity of fruits (Fig.13). The percentage of mace oil obtained on fresh weight basis during the final three stages of sampling were 7.14, 6.09 and 4.39 per cent respectively. The corresponding values on dry weight basis were 23.8, 19.03 and 8.78 per cent respectively.

4.2.8 Direct and indirect effects of growth parameters on nutmeg oil

The results of the above analysis are presented in Table 15. The growth parameters studied for this path co-efficient analysis were length, breadth, girth and weight of kernel.



Months of Sampling

s of sampling

·	Length	Breadth	Girth	Fresh weight	Correlation
Length	- <u>1.1712</u>	-1.4110	1.7855	-0.1177	<b>-</b> 0.9144 <sup>**</sup>
Breadth	· <b>-</b> 1 <b>.</b> 1588	- <u>1.4261</u>	1.7781	-0.1173	-0.9241**
Girth	<b>-</b> 1 <b>.</b> 1694	-1.4179	1.7883	-0.1188	-0.9178**
Fresh weight	-1.1311	-1,3720	1.7424	- <u>0.1220</u>	-0.8827**

Table 15 Direct and indirect effects of growth parameters on nutmeg oil

Residual effects = 0.3818

The diagonal values (underlined) are the direct effects and the horizontal values are the indirect effects.

\*\* Significant at 1 per cent level.

The correlation between length and nutmeg oil was -0.9144<sup>\*\*</sup> of which -1.1712 was the direct contribution of length. The remaining was the contribution of length indirectly through the other growth parameters. The indirect effects through breadth and fresh weight were negative while that of girth was positive.

The correlation between breadth and oil was  $r = -0.9241^{**}$  of which the direct effect of breadth was -1.4261. The indirect effects through length and weight were negative while through girth was positive.

The correlation obtained between girth and nutmeg oil was  $r = -0.9178^{**}$  of which the direct effect of girth was positive (1.7883). Here, the indirect effects on girth through length, breadth and weight of kernel dominated the direct effect of girth resulting in a negative correlation.

The correlation value estimated between fresh weight of nutmeg kernel and oil was  $r = -0.8827^{**}$  of which the direct effect of weight was -0.1220, the remaining being the contribution of weight indirectly through the other growth parameters. The indirect effects through length and breadth were negative while that of girth was positive.

The above results clearly indicate that girth influenced directly as well as indirectly in a positive manner while the direct and indirect effects of length, breadth and weight of kernel influenced the oil content in a negative manner. Of the variation in oil, 62 per cent was attributed to the direct effect of the growth parameters studied.

4.2.9 Changes in flavour components of nutmeg oil at the different maturity stages

Volatile oil of nutmeg obtained by steam distillation at the different maturity stages were stored in glass stoppered bottles in a refrigerator and were later subjected to gas chromatographic analysis. The oil obtained from the first month's sample was however not subjected to gas chromatography because the oil was not sufficient for analysis. Table 16 depicts the changes in components of nutmeg oil at different maturity stages. The results of the analysis are given below.

4.2.9.1 Alpha-pinene

The alpha-pinene concentration in nutmeg oil was found to increase upto the third month of sampling when it reached a maximum value (15.81%). Thereafter it showed

Compound (%)			Months of s	ampling	-	
· · · · ·	2	3	4	5	6	7
Alpha pinene	7.98	15.81	9.98	8,38	5.00	15.00
Beta-pinene + Sabinene	28.01	30.29	37.63	34.51	36.30	29.49
Kyrcene	1.58	4 00	0 40 × 1	0	2.08	
Pnellandrene	1.93 I	· 4.99	2.19	2,85	0.80 I	. 4.58
Limonene	7.14	7.10	5.34	4.78	6.28	5.95
lpha-terpinene	1.54	trace	trace	1.26	trace	2,38
Cineole	7.38	8.99	4.05	5.25	2.91	6.89
Gamma-terpinene	4.39	3.18	1.55	2.82	3.23	2.41
Alpha-copaene	0.05	0.27	. 0,34	0,31	0.70	0,33
Linalool	trace	0.20	5,34	1.03	0,57	0.63
Fenchyl alcohol + Cis-sabinen hydrate	0.17	0.90	0,65	0.97	0.60	1.17
Cis-p-menth-2-en-ol	0.16	0.61	0.39	0.61	0,34	1.23
Terpinene-4-ol	6.01	16.09	10.07	14.89	9.15	17.26
Alpha_terpineol	0.67	3.01	1.55	2.02	1.33	trace
Geraniol	1.41	0.63	0.34	0.60	1.04	0.73
Borneol	0.73	0.39	0.27	0.44	1,20	0.32
Satrole	2.27	1.11	1.00	1.25	1.27	0.59
Eugenol	2.08	0.26	0,35	0_11	0.85	0.86
Cis-isoeugenol	0.63	0.23	0.35	0,21	1.15	0.49
Trans-isoeugenol	1.31	0.07	0.69	0.04	0.77	trace
<b>Ayristicin</b>	6.31	4.66	7.54	9.22	4.66	1.08
Elemicin	3.59	truce	trace	trace	8.96	5.61

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Table 16 Flavour characteristics of nutmeg oil at different maturity stages

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a gradual reduction in concentration upto the sixth month. At the last month of sampling a spurt in concentration was observed ( 15.00 per cent).

## 4.2.9.2 Beta-pinene + Sabinene

The concentration of these two compounds (together) were found to increase upto the third month when it reached 37.63 per cent dropped to 34.51 per cent at the fourth month and subsequently increased to 36.30 per cent at the sixth month. Thereafter it decreased to 29.49 per cent at the seventh month of sampling.

#### 4.2.9.3 Myrcene + Phellandrene

These compounds were seen separately in the chromatograms at second and sixth months whereas in the chromatograms of other samples they occurred as a single peak. Their concentration was found to increase at the third month (4.99%), declined until the sixth month and finally rose to 4.58 per cent at the seventh month of sampling.

#### 4.2.9.4 Limonene

The percentage concentration of this component decreased from a high value of 7.14 per cent at the second month of sampling to 4.78 per cent at the fifth month. Subsequently it increased to 6.28 per cent (sixth month) and thereafter decreased slightly to 5.95 per cent at the seventh month.

4.2.9.5 Alpha-terpinene

This compound was detected only in some of the chromatograms (2nd, 5th and 7th months) studied. The maximum value (2.38%) was observed at the final stage of harvest.

4.2.9.6 Cineole

The cineole concentration was found to increase and decrease alternatively. The maximum value of 8.99 per cent was observed at the third month of sampling. At the final stage it recorded a value of 6.89 per cent.

4.2.9.7 Gamma-terpinene

The concentration of gamma-terpinene decreased from a higher value 4.39 per cent during the second month of sampling upto the fourth month (1.55%) and then increased at the sixth month (3.23%) and thereafter declined at the seventh month (2.41%).

4.2.9.8 Alpha-copaene

This component showed an increasing trend upto the fourth month of sampling (0.05 to 0.34%) and then reduced

slightly at the fifth month (0.31%). The maximum concentration was attained at the sixth month (0.70%) and thereafter it dropped to 0.33 per cent at the last stage of maturity.

4.2.9.9 Linalool

The linalool concentration increased from traces (2nd month) to 5.34 per cent (4th month) and thereafter fell progressively during the last stages of sampling.

4.2.9.10 Fenchyl alcohol + Cis - Sabinene hydrate

The concentration of these two compounds (together) did not reveal any definite trend during the various growth maturity stages studied. Alternating high and low values were observed, the minimum being observed at the second month (0.17%) and the maximum at the seventh month of sampling (1.17%).

4.2.9.11 Cis-p-menth-2-en-ol

Here again, the concentration was found to increase and decrease alternately until it reached the maximum concentration (1.23%) at the final harvesting stage. 4.2.9.12 Terpinen-4-ol

An alternate increasing and decreasing trend in concentration of this compound was observed in the

chromatograms. The minimum value was observed at the second month (6.01%) and the maximum at the seventh month (17.26%) of maturity.

# 4.2.9.13 Alpha-terpineol

The concentration of alpha-terpineol also showed an alternate increasing and decreasing trend until the sixth month of sampling. However during the last stage of sampling, only traces of this compound could be detected.

#### 4.2.9.14 Geraniol

The geraniol concentration decreased from 1.41 per cent (maximum) at the second month to 0.34 per cent (minimum) at the fourth month. Then its concentration increased and reached 1.04 per cent at the sixth month and there-after reduced to 0.73 per cent at the final stage of sampling.

### 4.2.9.15 Borneol

The concentration of borneol dropped from 0.73 per cent during the second month to 0.27 per cent at the fourth month, then increased to 1.20 per cent (maximum) at the sixth month and subsequently fell to 0.32 per cent at the final harvesting stage.

## 4.2.9.16 Safrole

The safrole concentration in nutmeg oil decreased from 2.27 per cent (maximum) during the second month of sampling upto the later maturity stages and the minimum value (0.59%) was observed at the seventh month of sampling.

4.2.9.17 Eugenol

The concentration of eugenol showed a decline from an initial high value (2.08%) to a low value (0.11%) at the fifth month. Thereafter, the concentration remained almost steady at the last two sampling periods(0.85 to 0.86%).

4.2.9.18 Cis-isoeugenol

The concentration of this compound showed alternating decreasing and increasing trends. The lowest value was registered at the fifth month (0.21%) and the highest concentration (1.15%) at the sixth month of sampling. 4.2.9.19 Trans-isoeugenol

This compound also showed alternating decreasing and increasing trends from a initial high concentration of 1.31 per cent to only traces at the peak maturity stage.

#### 4.2.9.20 Myristicin

The myristicin concentration decreased from an initial value of 6.31 per cent during the second month to 4.66 per cent at the third month. Then the concentration increased to 9.22 per cent (maximum) at the fifth month finally dropping to 1.08 per cent (minimum) at the seventh month period:

4.2.9.21 Elemicin

The elemicin concentration was 3.59 per cent at the second month of sampling. Only traces of this compound was detected in the chromatograms at the 3rd, 4th and 5th months. However at the sixth and seventh months of sampling rather high concentrations (8.96% and 5.61% respectively) were recorded.

The gas chromatographic patterns of nutmeg oil are depicted in Figures 14 to 19.

4.2.10 Changes in flavour components of mace oil at different maturity stages

The mace was separated from the nutmeg kernels and distillation was carried out to extract mace oil at the final three stages of sampling.

# Components of nutmeg oil

(1)	Alpha pinen <b>e</b>
(2)	Beta-pinene + Sabinene
(3)	Myrcene
(4)	Phellandrene
(5)	Limonene
(6)	Alpha-terpinene
(7)	Cineole
(8)	Gamma-terpinene
(9)	Alpha-copaene
(10)	Linalool
(11)	Fenchyl alcohol + Cis-sabinene hydrate
(12)	Cis-p-menth-2-en-ol
(13)	Terpinen-4-ol
(14)	Alpha-terpineol
(15)	Geraniol
(16)	Borneol
(17)	Safrole
(18)	Eugenol
(19)	Cis-isoeugenol
(20)	Trans-isoeugenol
(21)	Myristicin
(22)	Elemicin

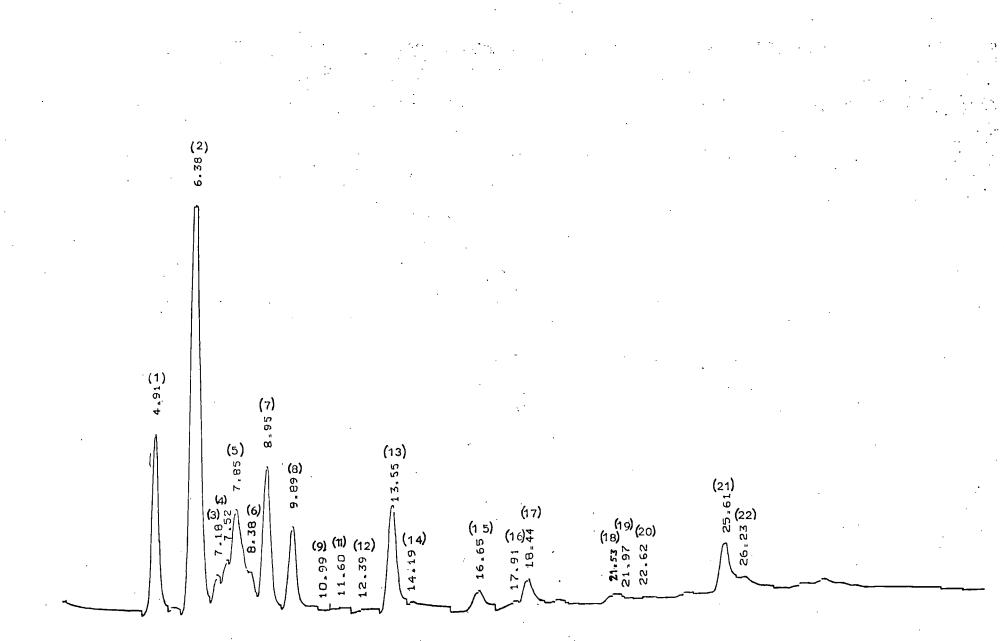


Fig.14. Gas chromatographic pattern of nutmeg oil at the second month of sampling.

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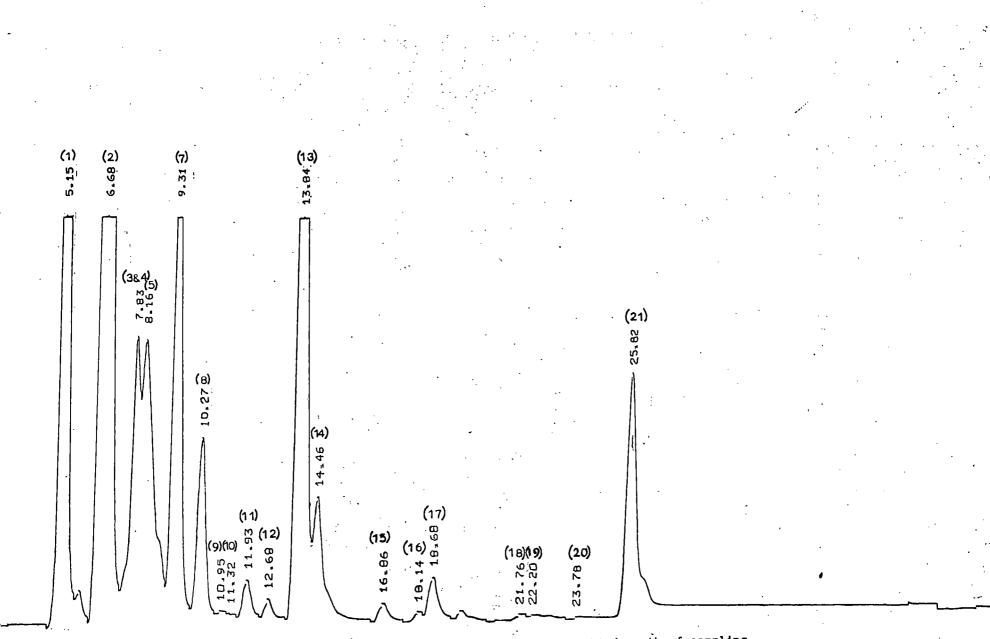


Fig.15. Gas chromatographic pattern of nutmeg oil at the third month of sampling

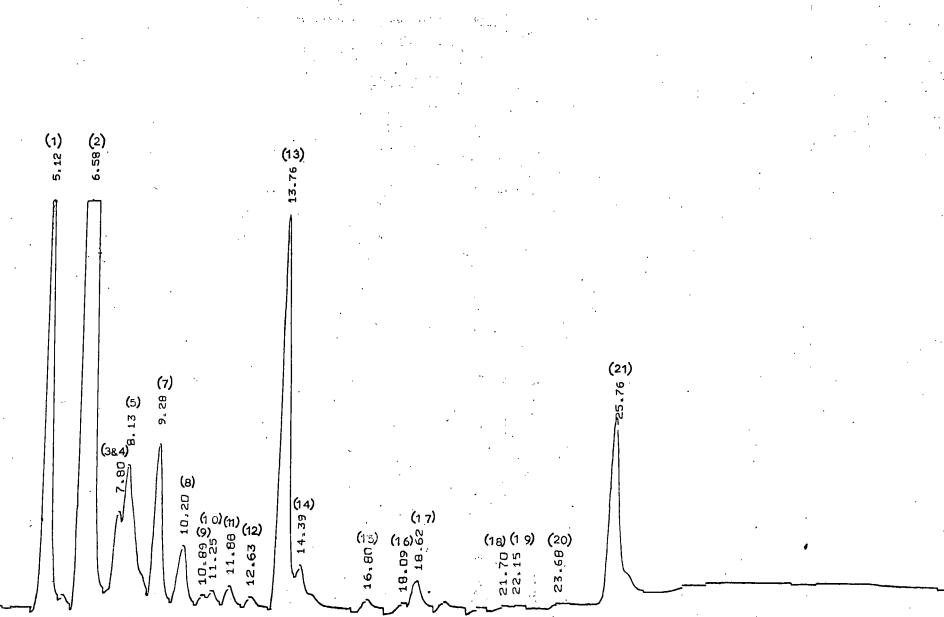


Fig.16. Gas chromatographic pattern of nutmeg oil at the fourth month of sampling

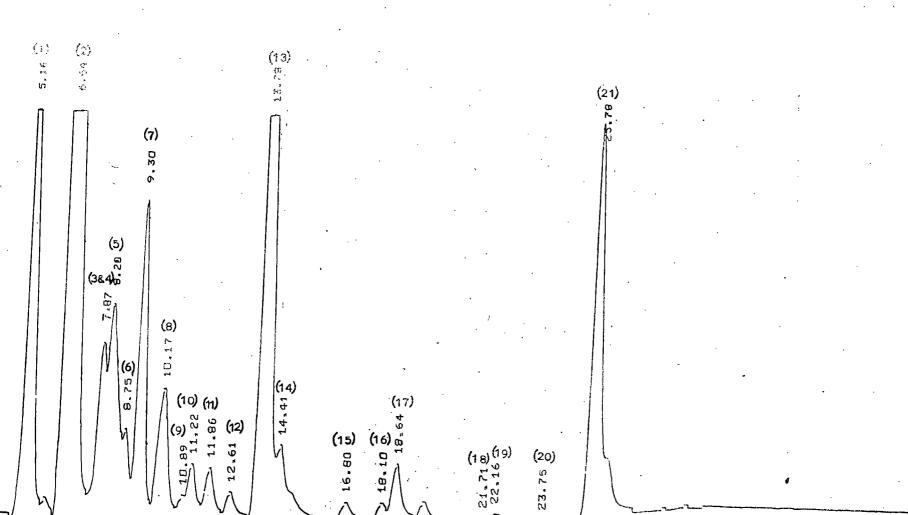


Fig.17. Gas chromatographic pattern of nutmeg oil at the fifth month of sampling

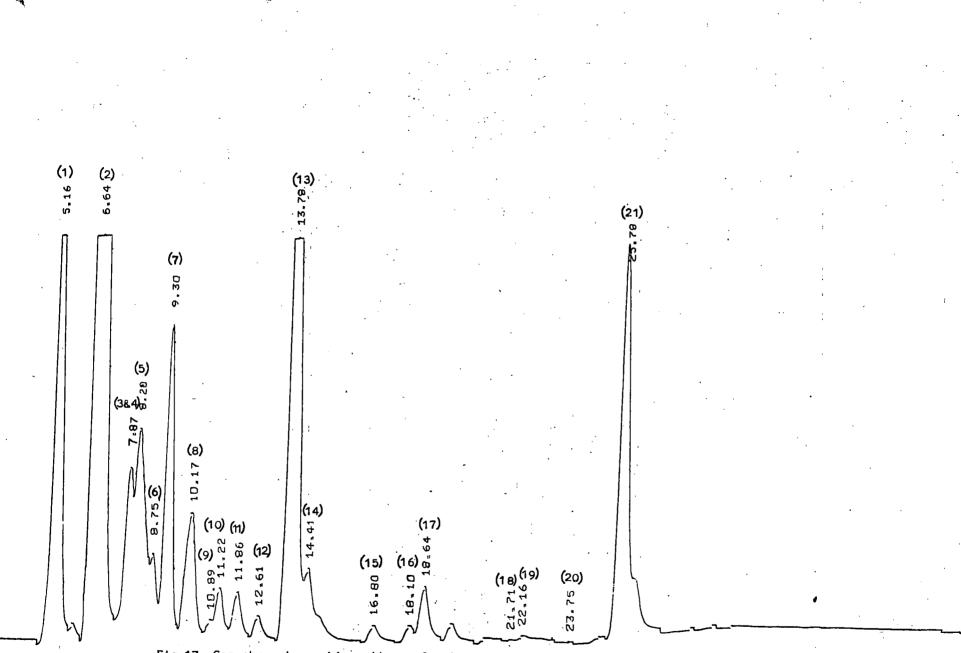


Fig.17. Gas chromatographic pattern of nutmeg oil at the fifth month of sampling

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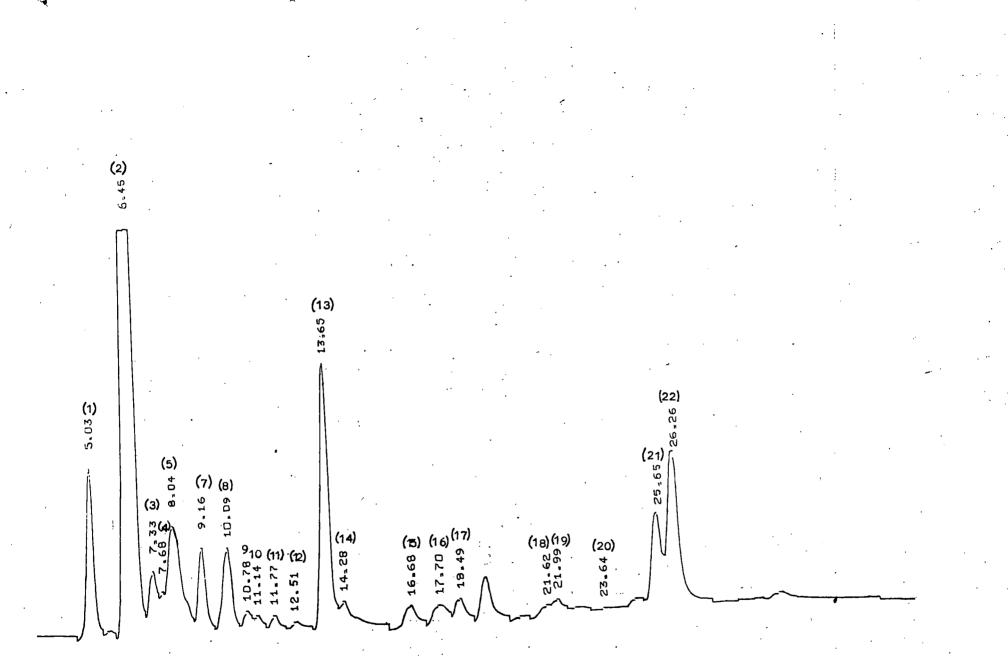


Fig.18. Gas chromatographic pattern of nutmeg oil at the sixth month of sampling

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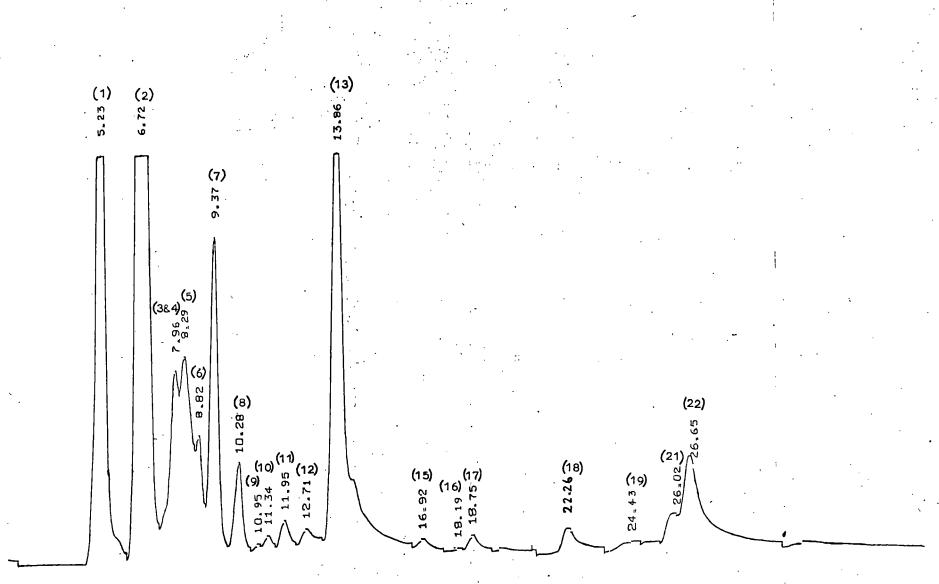


Fig.19 Gas chromatographic pattern of nutmeg oil at the seventh month of sampling

The oil obtained from mace was subjected to gas chromatographic analysis. The pattern of changes in the constituent components of mace oil at the three stages studied are depicted in Table 17. The results of the analysis are given below.

4.2.10.1 Alpha-pinene

Alpha-pinene concentration was found to increase slightly from 5.08 per cent in the first sample to 5.25 per cent in the second and subsequently reducing to 3.54 per cent in the Last stage.

#### 4.2.10.2 Beta-pinene + Sabinene

The concentration of these two compounds increased from 17.45 per cent (5th month) to a maximum of 29.75 per cent (6th month) and further dropping to 19.47 per cent at the final stage.

#### 4.2.10.3 Myrcene

The concentration of myrcene rose from an initial value of 0.11 per cent (5th month) to a high of 4.19 per cent (6th month) and subsequently declined to 0.91 per cent at the 7th month of sampling.

Compound (%)	<del></del>	g	
	5	6	7
Alpha-pinene	5.08	5.25	
Beta-pinene + Sabinene	17.45	29.75	3,54
Myrcene	0.11	4.19	19,47
Alpha-phellandrene	0.38		0.91
Limonene	1.52	3.06	1.26
Alpha-terpinene	0.65	4.31	6.21
Cineole		6.30	trace
Gamma-terpinene	1.47	7.62	4.61
Alpha-copaene	2.18	5.59	2.79
Linalool	0.58	0.52	0.83
Fenchyl alcohol + Cis-sabinene hydrate	1,52	0.51	0.44
Dis-p-menth-2-en-ol	1,60	1.35	1.14
[erpinen=4=ol	1.17	0.97	0.82
	23,23	22.97	20,80
lpha-terpineol	4.22	2.67	3.02
eraniol	1.54	0.86	1.14
orneol	0.46	0,29	0.43
afrole	3.28	0.62	1.97
ugenol	1.32	0.35	3,09
is-isoeugenol	1.46	0.06	
rans-isoeugenol	0.72	0.14	trace
yristicin	27.25	0.40	0.93
lemicin	Trace		7.94
· · · · · · · · · · · · · · · · · · ·		0.68	. 15.87

Table 17 Flavour characteristics of mace oil at different maturity stages

#### 4.2.10.4 Alpha-phellandrene

The alpha-phellandrene concentration was found to increase from 0.38 to 3.06 per cent at the sixth month. However at the final stage, the concentration decreased to 1.26 per cent.

#### 4.2.10.5 Limonene

The limonene concentration showed an increasing trend as the maturity of mace increased and reached a maximum concentration (6.21%) at the final stage of harvest.

4.2.10.6 Alpha-terpinene

The concentration of this compound showed a spurt from 0.65 per cent (5th month) to 6.30 per cent at the 6th month of sampling. Thereafter it reduced substantially and only traces of this compound was detected at the final stage.

4.2.10.7 Cineole

The concentration of cineole increased from 1.47 per cent (5th month) to 7.62 per cent at the 6th month and subsequently decreased to 4.61 per cent at the last sampling stage.

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# 4.2.10.8 Gamma-terpinene

The concentration of this compound showed an increase from 2.18 per cent to 5.59 per cent during the first and second sampling periods and at the final sampling it decreased to 2.79 per cent.

4.2.10.9 Alpha-copaene

The alpha copaene concentration showed a slight decrease from 0.58 per cent (5th month) to 0.52 per cent (6th month) and finally rose to 0.83 per cent at the 7th month of sampling.

4.2.10.10 Linalool

The concentration of this compound showed a progressive decreasing trend from an initial value of 1.52 per cent to a low value of 0.44 per cent at the final stage.

4.2.10.11 Fenchyl alcohol + Cis-Sabinene hydrate

These two compounds also showed a decrease in concentration from 1.60 to 1.14 per cent with increase in maturity of the mace.

# 4.2.10.12 Cis-p-menth-2-en-ol

The concentration of this compound showed a progressive decrease from 1.17 to 0.82 per cent as the maturity of mace advanced from the 5th to 7th month stage.

4.2.10.13 Terpinen-4-ol

The terpinen-4-ol concentration also showed a gradual decline from 23.23 to 20.80 per cent as the maturity of mace progressed.

4.2.10.14 Alpha-terpineol

The alpha-terpineol concentration decreased from 4.22 per cent at the 5th month to 2.67 per cent at the 6th month and since then increased slightly to 3.02 per cent at the final maturity stage.

4.2.10.15 Geraniol

The geraniol concentration also showed a decrease from an initial level of 1.54 per cent to 0.86 per cent and finally attained a concentration of 1.14 per cent.

#### 4.2.10.16 Borneol

The concentration of this compound showed a decrease from an initial concentration of 0.46 per cent to 0.29 per cent and then an increase to 0.43 per cent was observed at the final stage of harvest.

4.2.10.17 Safrole

The safrole concentration also decreased from 3.28 to 0.62 per cent and subsequently registered a value of 1.97 per cent at the final stage.

4.2.10.18 Eugenol

The level of eugenol in mace oil showed a slump from 1.32 per cent (5th month) to 0.35 per cent (6th month) whereas a sharp increase in concentration to 3.09 per cent was seen at the final stage.

4.2.10.19 Cis-isoeugenol

The Cis-isoeugenol content in mace oil showed a progressive decrease from an initial concentration of 1.46 per cent and only traces of this compound was observed at the final stage of harvest. 4.2.10.20 Trans-isoeugenol

This compound recorded a value of 0.72 per cent at the initial stage and subsequently dropped to 0.14 per cent

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# 4.2.10.21 Myristicin

The myristicin concentration showed a steep decline from 27.25 per cent at the initial stage to 0.40 per cent at the 6th month of sampling. Its concentration however increased to 7.94 per cent at 7th month of harvest.

4.2.10.22 Elemicin

The concentration of this compound showed a proportionate increase with increase in maturity of mace. Only traces of elemicin were detected at the 5th month stage, which slightly rose to 0.68 per cent at the 6th month and finally increased to 15.87 per cent at the 7th month of harvest.

The gas chromatographic patterns of mace oil are depicted in Figures 20 to 22.

# Components of mace oil

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(1)	Alpha pinen <b>e</b>
(2)	Beta-pinene + Sabinene
(3)	Myrcene
(4)	Phellandrene
(5)	Limonene
(•6)	Alpha-terpinene
(7)	Cineole
(8)	Gamma-terpinene
(9)	Alpha-copaene
(10)	Linalool
(11)	Fenchyl alcohol + Cis-sabinene hydrate
(12)	Cis-p-menth-2-en-ol
(13)	Terpinen-4-ol
(14)	Alpha-terpineol
(15)	Geraniol
(16)	Borneol
(17)	Safrole
(18)	Eugenol
(19)	Cis-isoeugenol
(20)	Trans-isoeugenol
(21)	Myristicin
(22)	Elemicin

(22) Elemicin

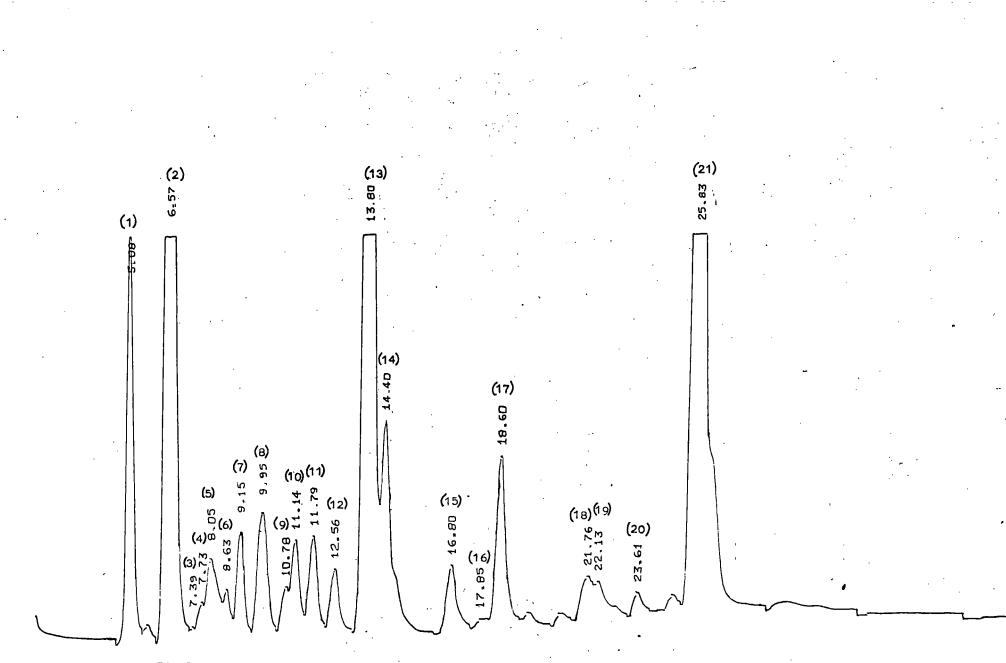


Fig.20. Gas chromatographic pattern of mace oil at the fifth month of sampling

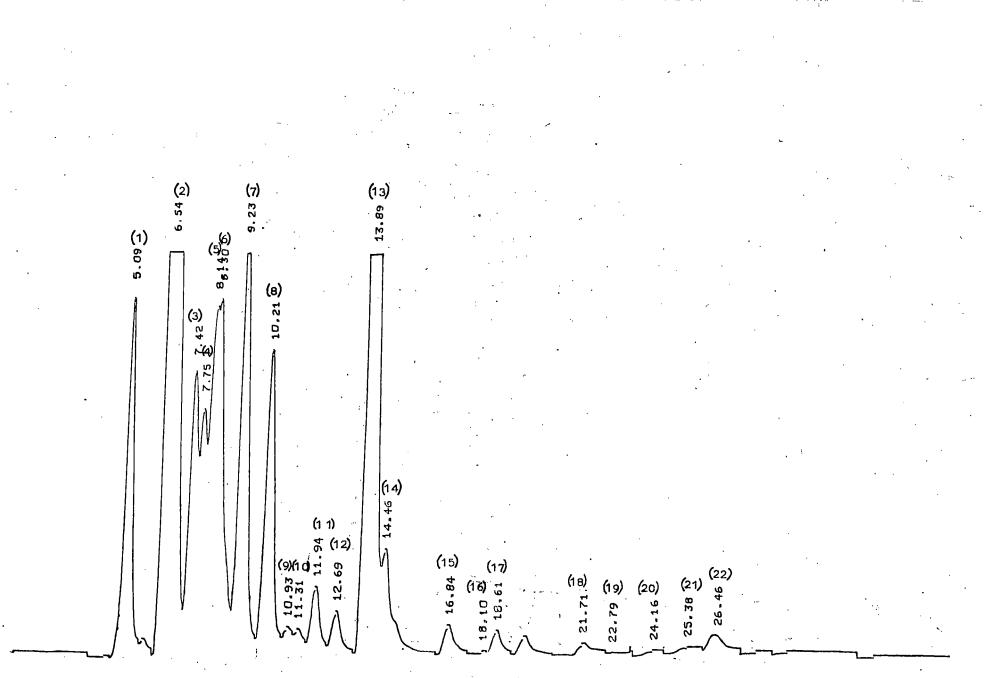


Fig.21 Gas chromatographic pattern of mace oil at the sixth month of sampling

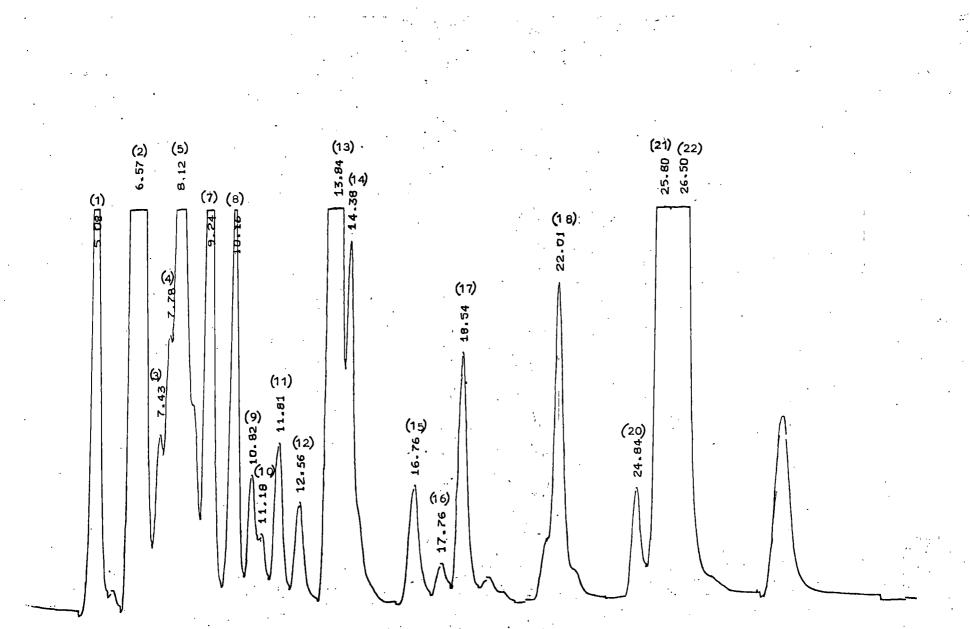


Fig.22 Gas chromatographic pattern of mace oil at the seventh month of sampling

# DISCUSSION

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#### 5 DISCUSSION

Clove and nutmeg, two important spice crops of Kerala, yield essential oils which are widely used for aromatic, culinary and medicinal preparations. Research on the quality aspects of these oils so far, concentrated mainly on extraction and characterization of oil from mature stages of the spice. The present study probed into detailed analysis of the components present in these spice oils collected from different maturity stages and their variation at these stages. In addition to the above aspect, the variation in moisture content, non-volatile ether content and growth parameters of clove flower buds/nutmeg fruits at different maturity stages were also investigated.

5.1 Clove

5.1.1 Growth and development of clove

The growth parameters studied namely, length, breadth, girth, fresh weight and volume showed increasing trends in all cases with increase in maturity of the crop. Maximum growth was observed for all parameters at the 'mother-of-clove' stage. The rate of growth was rather low for length and breadth, whereas the rate was comparatively high for girth and at later stages of maturity for fresh weight and volume. The pre-anthesis stages were characterized by low growth rate as far as fresh weight and volume were concerned.

5.1.2 Moisture content, NVEE and volatile oil of clove

The moisture content in clove bud increased as the bud matured.

The non-volatile ether extract (NVEE), on dry weight basis was found to be more at the initial stage of growth of clove flower bud and then decreased progressively as maturity of clove bud advanced. Gopalakrishnan <u>et al</u>. (1982) also observed that NVEE was the maximum at lower maturity stages with slight decrease at the last phase.

The oil content obtained at the harvesting stage of clove flower bud (represented by the fourth month of sampling) was 15.28 per cent on dry weight basis. Purseglove <u>et al.(1981)</u> have also cited an oil content of 17.00 per cent in commercial samples (dried flower buds) of clove.

The oil content in clove was found to decrease with increasing maturity of clove bud. This finding is in conformity with the results obtained by Gopalakrishnan <u>et al.</u> (1982) wherein the maximum percentage of volatile

oil was obtained at the lower maturity stages.

The distillation of ripe fruits yielded a volatile oil known as 'mother-of-clove oil'. The recovery obtained was 2.23 per cent. Earlier investigations, as reported by Schimmel <u>et al</u>. (1915) revealed an oil content of 2.00 per cent, when 'mother-of-clove' was subjected to steam distillation.

The refractive index of clove oil was found to decrease slightly after the first month of sampling and subsequently increased at the fourth month and remained more or less constant until the final stages of maturity. 5.1.3 Flavour components of clove oil

The present investigation attempted to characterise the major components of the clove oil by gas chromatography. The fourth month of sampling (prior to anthesis)corresponds to the commercial harvesting stage.

Eugenol is found to be the major component of clove oil. At the commercial harvesting stage, its content was found to be 51.96 per cent. This is a rather low value compared to the eugenol content of 80 to 87 per cent in Zanzibar clove oil (Deyama and Horiguchi, 1971). In

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Madagascar clove buds, Gaydou and Randriamiharisoa (1987) also observed a high eugenol content (73.5 to 79.7%) for the commercial samples.

Maximum eugenol concentration (73.12%) was observed at the flowering phase. Harvesting of clove done at this stage would enable extraction of volatile oil which may be more acceptable for medicinal purpose.

Eugenol content of 54.70 per cent was estimated at the 'mother-of-clove' stage which is in conformity with an earlier report of 53.00 per cent eugenol at the same stage (Schimmel et al., 1915).

A greater concentration (22.56%) of eugenol acetate was observed at the fourth month of sampling. Contrary to this Gopalakrishnan <u>et al</u>. (1982) reported only a low concentration of this compound (9.00%) at the corresponding stage of sampling. Similar observations were also made by Gaydou and Randriamiharisoa (1987) wherein a concentration ranging from 4.50 to 10.70 per cent of eugenol acetate was detected in Madagascar clove bud oil.

A decrease in eugenol during the second and fourth months of sampling and a corresponding increase in eugenol acetate during the same periods suggest a possible inter-conversion of eugenol to eugenol acetate. A reversal of this phenomenon was observed at the third month, wherein there was a decline in the concentration of eugenol acetate and a simultaneous increase in the concentration of eugenol.

The concentration of cis-isoeugenol and transisoeugenol revealed rather erratic trends at the various stages of sampling. This could be due to the possible interconversion of these two compounds.

The beta-caryophyllene content was 14.82 per cent at the commercial harvesting stage of clove. Investigations by Deyama and Horiguchi (1971) in Zanzibar clove, Gopalakrishnan <u>et al</u>. (1982) in Indian clove and Gaydou and Randriamiharisoa (1987) in Madagascar clove, revealed more or less similar concentration of beta-caryophyllene in clove oil (9.12, 11.20 and 7.30 to 12.40% respectively).

### 5.2 Nutmeg

5.2.1 Growth and development of nutmeg fruit and kernel

Investigations on the growth of fruit and kernel in nutmeg showed increasing trends for length, breadth, girth, fresh weight and volume with increase in maturity. The rate of growth for length and breadth was more towards lower maturity stages of the fruit. For fresh weight and volume, the maximum increase in growth was noticed between fourth and fifth months of maturity in both kernel and fruit. A prominent sigmoid pattern of growth was observed for girth of nutmeg kernel. A similar sigmoid pattern of growth had been reported earlier as characteristic to both fruit and kernel in nutmeg (Nazeem, 1979).

# 5.2.2 Moisture content in nutmeg

The moisture content in rind of nutmeg fruits showed a steady increase upto the third month and thereafter remained steady. However a decrease in moisture percentage was observed at the final stage of sampling. This may be due to the fact that at the final harvesting stage, fruit splitting had taken place and rinds dried up which on exposure to wind resulted in water loss from the fruits.

The moisture content in kernel and mace progressively decreased with increasing maturity of fruits. Such trends had been reported earlier by Gopalakrishnan (1984).

## 5.2.3 NVEE in nutmeg and mace

The non-volatile ether extract (NVEE) in nutmeg, on dry weight basis showed a steady increasing trend registering a maximum of 23.95 per cent at the fruit splitting stage. The NVEE content in mace however was found decreasing with increasing maturity of fruit. Similar trends for nutmeg fruits had been reported earlier by Gopalakrishnan (1984). He obtained a value of 28.5 per cent at the final harvesting stage for NVEE in nutmeg.

# 5.2.4 Nutmeg and mace oil

In the present investigation, an yield of oil ranging from 7.27 per cent to 19.20 per cent was obtained for nutmeg on dry weight basis. This result falls in line with the reports of Guenther (1952) for the yield of nutmeg oil, ranging from 7.00 to 16.00 per cent.

In nutmeg, the volatile oil on dry weight basis was more at the second month of fruit maturity. The content decreased steadily as the maturity advanced. The volatile oil content in mace on dry weight basis showed more or less a similar trend. This increase in oil content for nutmeg and mace with an advance in maturity of the spice had been reported earlier by Gopalakrishnan (1984).

5.2.5 Components in nutmeg oil

Gas chromatographic analysis of nutmeg oils revealed the presence of several flavour components. The variation in major components (Table 18) of nutmeg oil is depicted in Fig.23. The flavour components can be broadly classified as follows, as elucidated by Guenther (1949).

- Monoterpene hydrocarbons which include alphapinene, beta-pinene, sabinene, myrcene, phellandrene, limonene, alpha-terpinene and gamma-terpinene.
- 2. Oxygenated monoterpenes which include cineole, linalool, fenchyl alcohol, cis-sabinene hydrate, cis-p-menth-2-en-ol, terpinene-4-ol, alphaterpineol, geraniol and borneol.

Most of the compounds identified in this category belong to alcohols, the exception being cineole which is an oxide and cis-sabinene hydrate.

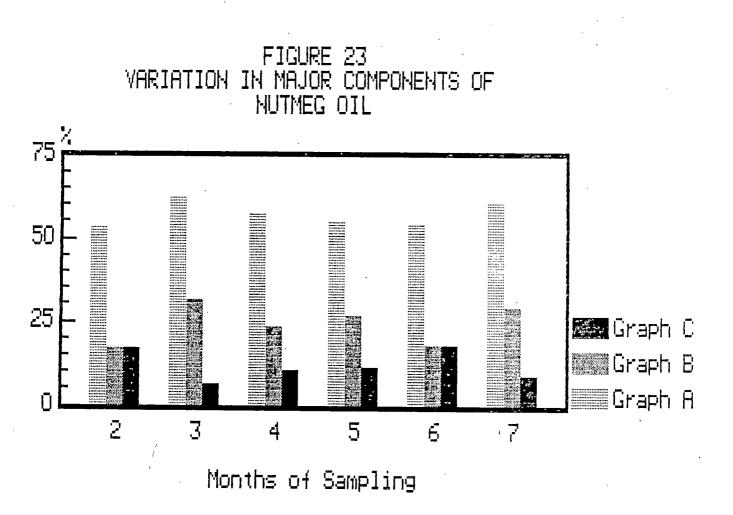
Category of compounds(%)			Month c	of sampling		
	2	3	4	5	6	7
Monoterpene hydrocarbons	52.57	61.37	56.69	54.60	53.69	59.81
Oxygenated				1		
monoterpenes	16.53	30.82	22.66	25.81	17.14	28.23
Sesquiterpene	0.05	0.27	0.34	0.31	0.70	0.33
Aromatic phenols and phenol ethers	16.19	6.33	9.93	10.83	17.66	8.63

Table 18 Components in nutmeg oil

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- A Monoterpene hydrocarbons
- B Oxygenated monoter Penes
- C Aromatic phenol and phenol ethers

 Sesquiterpene which include alpha-copaene
 Aromatic phenols and phenol ethers which include safrole, eugenol, cis-isoeugenol, trans-isoeugenol, myristicin and elemicin.

### Monoterpene hydrocarbons

The major components in this category are betapinene and sabinene which together accounted for a concentration ranging from 28.01 to 37.63 per cent. This concentration is much lower than the one in the earlier report of Itty and Nigam (1966) wherein they observed a level of 68.00 per cent in nutmeg oil. Gopalakrishnan (1984) stated that alpha-pinene, beta-pinene and sabinene are the major components of nutmeg oil and he reported a total concentration of 77.38 per cent for these components. From the present investigation, the total concentration for these components obtained at the corresponding stage was only 44.49 per cent. If the total monoterpene hydrocarbon concentration is taken into account then the highest concentration recorded was at the third month of maturity followed by the final harvesting stage.

## Oxygenated monoterpenes

Terpinen-4-ol is the major compound in this group and its highest concentration was at the final harvesting stage.

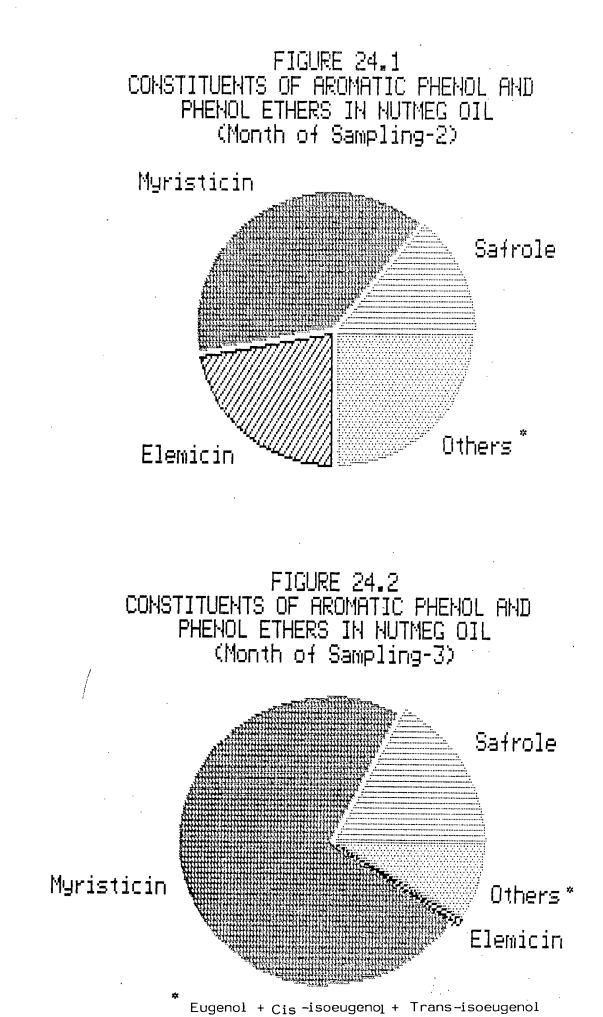
The total oxygenated monoterpene concentration showed a trend almost similar to the monoterpene hydrocarbons.

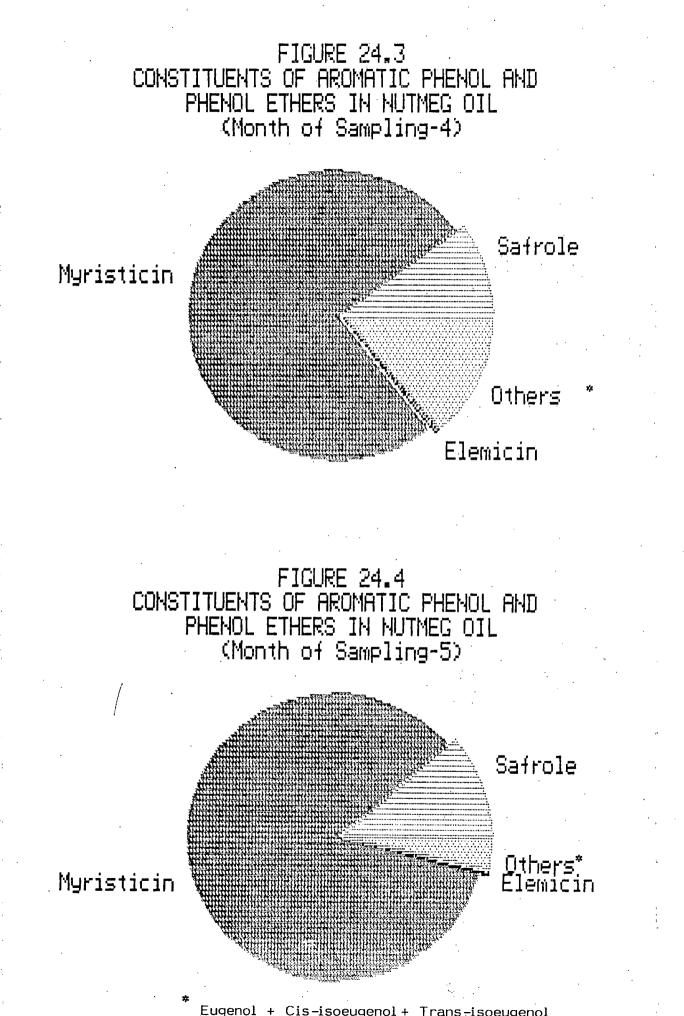
### Sesquiterpene

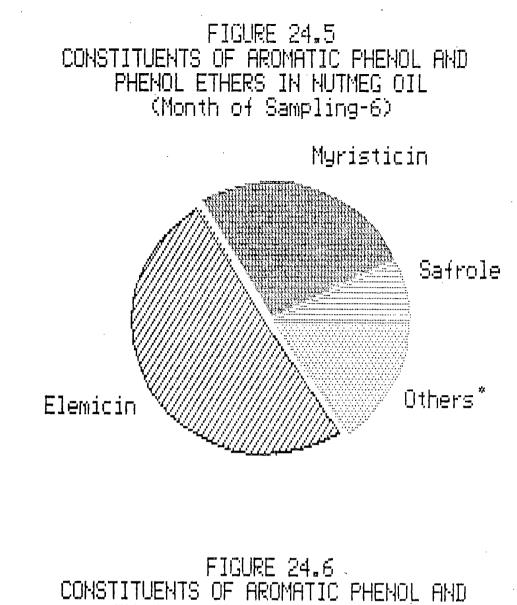
The only sesquiterpene identified in the present analysis was alpha-copaene whose concentration was rather low (less than 1%).

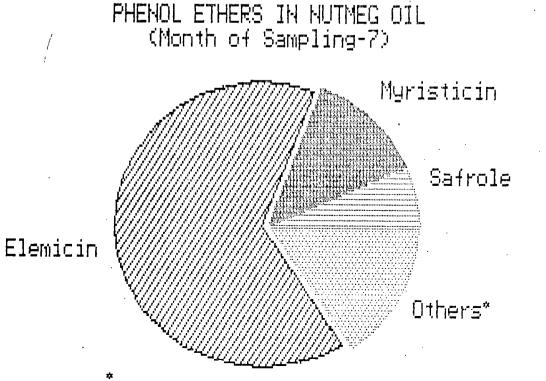
#### Aromatic phenols and phenol ethers

In this group, the highest concentration recorded was for myristicin followed by elemicin and safrole. According to Purseglove <u>et al</u>., (1981) the aromatic ethers, myristicin, safrole and elemicin appeared to play a vital role in determining the flavour and drug action of nutmeg. Itty and Nigam (1966) had reported a myristicin concentration of 12.00 per cent in nutmeg oil. Provatroff (1973) reported the concentration of this compound as 8.00 per cent. In the present analysis it has been found that the myristicin concentration was the maximum (19.22%) at the fifth month of sampling (Fig.24) and then decreased to a low of 1.08 per cent at final harvesting stage. Elemicin showed an increased synthesis as the harvesting stage of the spice approached. Safrole however showed a general decrease from the initial









~ Eugenol + Cis-isoeugenol + Trans-isoeugenol

concentration especially at the younger stages. When the total aromatic phenol and phenol ether content was taken into account, it was found that the concentration of these class of compounds were the highest (17.66%) at a stage prior to the final harvest. Hence harvesting at this stage may be advisable for extraction of nutmeg oils that are used for medicinal purposes.

# 5.2.6 Components in mace oil

The mace oil was extracted at the final three stages of sampling. The components in mace oil (Table 19) were similar to those obtained in nutmeg oil. Forrest and Heacock (1972) analysed East Indian mace oil and reported similar compounds in the oil.

# Monoterpene hydrocarbons

Among the monoterpene hydrocarbons, beta-pinene and sabinene together constituted the major components followed by limonene and alpha-pinene. Higher concentration of alpha-pinene, beta-pinene and sabinene were recorded at the stage prior to final harvest. The total concentration of alpha-pinene, beta-pinene and sabinene recorded at the final harvesting stage was found to be 23.01 per cent. A much higher concentration (60.76%) of

Table 19 Components in	mace	oil
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Category of compounds(%) -	Mont	h of sampling	
	5	6	7
Monoterpene hydrocarbons	27.37	58,45	34.18
Oxygenated monoterpenes	35,21	37.24	32.40
Sesquiterpene	0.58	0.52	0.83
Aromatic phenols and phenol ethers	34.03	2,25	29.80

these compounds together was reported by Gopalakrishnan (1984) for mace oil distilled during the commercial stage of harvesting. The monoterpene hydrocarbon concentration in the present investigation recorded the highest value at the stage prior to final harvest.

# Oxygenated monoterpene

Terpinen-4-ol constituted the major compound in this category recording a value of 20.80 per cent at the fruit splitting stage. Gopalakrishnan (1984) reported a rather low value of 4.59 per cent at similar stage of sampling. The maximum total oxygenated monoterpene concentration was observed prior to the final stage of sampling.

# Sesquiterpene

Alpha-copaene, the only sesquiterpene identified had a concentration of less than one per cent.

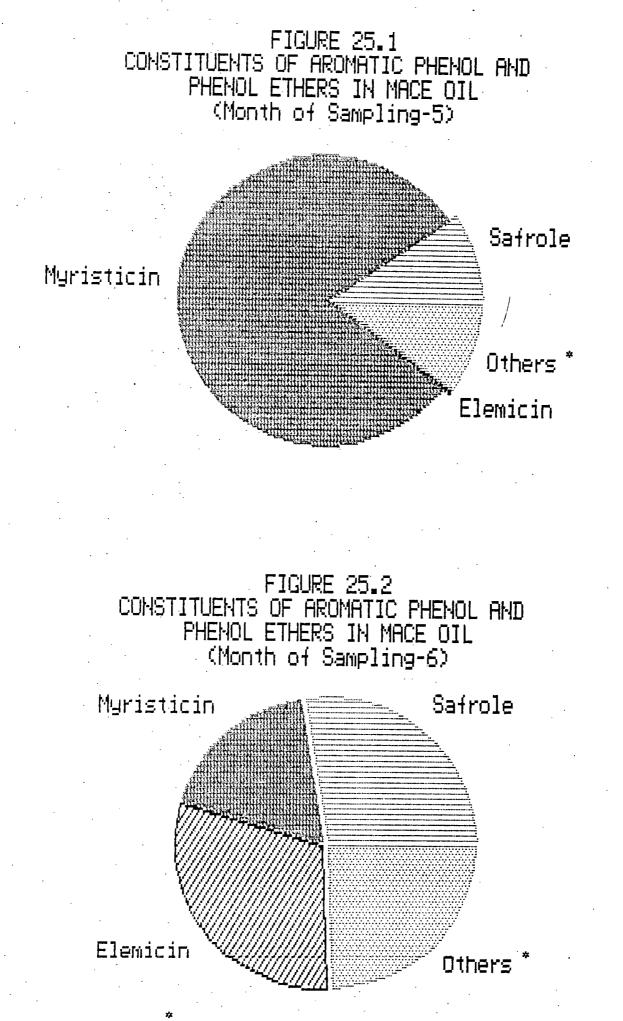
Aromatic phenol and phenol ethers

The aromatic ethers are of great importance, since they contributed to the flavour characteristics of mace oil. Among the aromatic ethers, myristicin was found to decrease

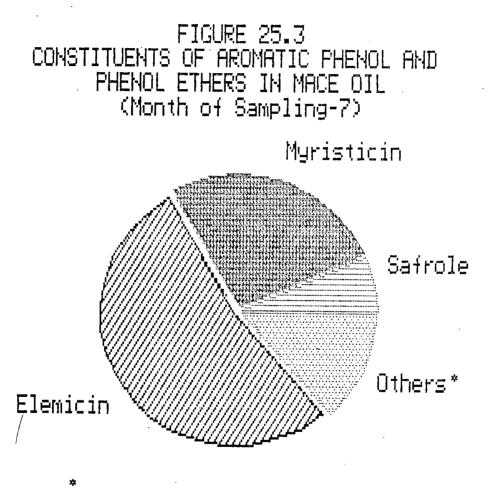
substantially at the stage prior to the final sampling. This aspect needs further investigation for confirmation. The elemicin concentration in mace oil was found to be the highest at the fruit splitting stage (Fig.25). Safrole synthesis, however, was found to decrease at the stage prior to harvest which subsequently showed an increase at the final stage. When the total aromatic phenol and phenol ether concentration was taken into account it was found that their concentration showed a trend similar to myristicin wherein a sudden decrease was seen one month prior to harvesting. Since the highest concentration of aromatic compounds was observed two months prior to fruit splitting, it may be worthwhile to harvest the fruits at this stage for extraction of mace oil.

# 5.2.7 Comparison of nutmeg and mace oil

A comparison of nutmeg and mace oils reveals that the components are more or less similar in both oils. However, differences do exist in the concentration of these components. In general, it is seen that monoterpene hydrocarbons are less in mace oil. A major difference was that nutmeg oil at the fruit splitting stage recorded 8.63 per cent for aromatic phenols and phenol ether



- Eugenol + Cis-isoeugenol + Trans-isoeugenol



Eugenol + Cis-isoeugenol + Trans-isoeugenol

concentration. The mace oil at the corresponding stage had a value of 29.80per cent for this category of compound. Mace oil at this stage had an elemicin concentration of 15.87 per cent compared to 5.61 per cent in nutmeg oil. Gopalakrishnan (1984) observed similar differences wherein he found that myristicin and elemicin were more in mace oil compared to nutmeg oil. Analytical results of the present investigation also revealed high value of myristicin at the fruit splitting stage in mace oil (7.94%) and a low value in nutmeg oil (1.08%).

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# SUMMARY

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#### 6 SUMMARY

Investigations were carried out at the College of Agriculture, Vellayani from January 1988 to May 1989, to gather information on the quality characteristics of clove and nutmeg at different maturity stages.

The results of this study are summarised as follows:

#### CLOVE

- 6.1 The moisture content of clove flower buds increased with increase in maturity and it was the maximum at the 'mother-of-clove' stage.
- 6.2 The non-volatile ether extract (NVEE) on dry weight basis, was found to be the maximum at the initial growth stage of clove buds.
- 6.3 The volatile oil content was the maximum at the first month of sampling, thereafter, decreasing progressively.
- 6.4 Among the physical properties of clove oil, optical rotation showed marked variation at different maturity stages. In the pre-antnesis stages, the optical rotation showed alternate decrease and increase while after anthesis, it was found to increase steadily.

- 6.5 Eugenol, which is the major component of clove oil, was the maximum at the flowering stage.
- 6.6 During the pre-anthesis stages, the volatile oil had a high eugenol content at the initial stage of sampling.
- 6.7 Eugenol acetate, another major component of clove oil was the maximum at the second month. Its concentration diminished at the final maturity stages.
- 6.8 The concentration of beta-caryophyllene rose from the first month to the third month of sampling and fell as the budsapproached the anthesis stage. Its concentration subsequently rose at the 'mother-of-clove' stage.

#### NUTMEG

- 6.9 The moisture content of nutmeg rind registered an increase whereas that of kernel and mace was found to decrease with increase in maturity.
- 6.10 The non-volatile ether extract(NVEE) showed a general decreasing trend in rind and mace while NVEE in kernel showed an increasing trend with increase in maturity.

6.11 The percentage recovery of nutmeg and mace oils on dry weight basis was the maximum at the immature stages and showed decrease as the maturity advanced.

- 6.12 The aromatic phenol ethers (safrole, myristicin and elemicin), which contribute to the fragrance and drug action of nutmeg oil, was the maximum (17.66%) one month prior to the fruit splitting stage, while in mace oil, it was the maximum (34.03%) two months prior to the final harvest.
- 6.13 The level of monoterpene hydrocarbons (alpha\_ pinene, beta-pinene, sabinene, myrcene, phellandrene, limonene, alpha-terpinene and gamma-terpinene) fluctuated between 52.57 and 61.37 per cent at the different harvesting stages in nutmeg oil, whereas in mace oil, the level of these compounds registered a high (58.45%) at the sixth month of harvest.
- 6.14 The concentration of oxygenated monoterpenes (cineole, linalool, fenchyl alcohol, cis-sabinene hydrate, cis-p-menth-2-en ol, terpinene-4-ol, alpha-terpineol, geraniol and borneol) showed



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\* Original not seen.

APPENDICES

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## Table A

Test of significance of fitted regression of growth parameters on sampling period of clove

· · · · ·			MS	
Source	df	Length	Breadth	Girth
Regression	1	8.912**	1.917**	20.862**
Deviation from regression	12	0.028	0.007	0.094
Total	13	0.711	0.153	1.691

**\*\*** Significant at 1% level

## Table B

Test of significance of regression coefficient of growth parameters on sampling period of clove

Dependent character	Regression coefficient	S.E	t-test
Length	0.399	0.022	17.870**
Breadth	0.185	0.011	17.230**
Girth	0.610	0.041	14.920**

**\*\*** Significant at 1% level

# Table A

Test of significance of fitted regression of growth parameters on sampling period of nutmeg fruit

Source	df	M S			
	Length	Length	Breadth	Girth	
Regression	1	13.734**	18.000**	156.327**	
Deviation from regression	5	0.132	0.031	0.636	
Total	6	2.399	3.026	26.584	

\*\* Significant at 1% level

#### Table B

Test of significance of regression coefficient of growth parameters on sampling period of nutmeg fruit

Dependent character	Regression coefficient	S.E	t_ test
Length	0.700	0.069	10.200 <sup>**</sup>
Breadth	0.802	0.034	23.950**
Girth	2.363	0.151	15.680**

**\*\*** Significant at 1% level

## Tabie A

Test of significance of fitted regression of growth parameters on sampling period of nutmeg kernel

Source	df		MS		
		Length	Breadth	Girth	
Regression	1	9.723**	6.499**	63.090**	
Deviation from regression	5	0.184	0.074	1.022	
Total	6	1.774	1.145	11.366	

**\*\*** Significant at 1% level

## Table B

Test of significance of regression coefficient of growth parameters on sampling period of nutmeg kernel

Dependent character	Regression coefficient	S.E.	t-test
Length	0.589	0.081	7.270**
Breadth	0.482	0.051	9.380**
Girth	1.501	0.191	7.860**

**\*\*** Significant at 1% level

Table A

Test of significance of regression of NVEE and oil content on moisture in clove

S	4.E	M.S	
Source	df -	NVEE	Oil content
Regression	1	4.822**	24.910**
Deviation from regression	- 12	0.073	1.004
Total	13	0.438	2.843
<del></del>	** Significa	nt at 1% level	

Coefficient of determination of NVEE = 0.847Coefficient of determination of oil = 0.675

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Table B

Test of significance of regression coefficient of NVEE and oil content on moisture in clove

Dependent character	Regression coefficient	S.E.	t
NVEE	0.621	0.270	2.300*
Oil content	1.411	1.002	1.408

\* Significant at 5% level

#### Table A

Test of significance of regression of NVEE and oil content on moisture in nutmeg kernel

Sauraa		M.S.	
Source	df	NVEE	Oil content
Regression	1	24.050**	11.117
Deviation from regression	- 5	1.148	2.824
Total	6	4.965	4.206

**\*\*** Significant at 1% level

Coefficient of determination of NVEE = 0.8073Coefficient of determination of oil = 0.4405

#### Table B

Test of significance of regression coefficient of NVEE and oil content on moisture in nutmeg kernel

Regression Coefficient	S.E	t
0.260	1.071	0.243
0.177	1.680	0.105
	Coefficient	Coefficient 5.E 0.260 1.071

# QUALITY CHARACTERISTICS OF CLOVE AND NUTMEG AT DIFFERENT STAGES OF MATURITY

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MANOJ, A. M.

## ABSTRACT OF A THESIS

submitted in partial fulfilment of the requirement for the Degree MASTER OF SCIENCE IN HORTICULTURE Faculty of Agriculture Kerala Agricultural University

Department of Horticulture COLLEGE OF AGRICULTURE VELLAYANI, THIRUVANANTHAPURAM

#### ABSTRACT

The present investigation was carried out at the College of Agriculture, Vellayani during 1988-89.

The objectives of this study were to characterise the growth pattern of clove flower buds and nutmeg fruits and to develop suitable harvest indices for these crops based on quality characteristics at different stages of maturity.

The moisture content in clove buds and rind of nutmeg fruits increased with increase in maturity. However, moisture percentage in nutmeg kernel and mace was found to decrease as maturity advanced.

The non-volatile ether extract (NVEE), on dry weight basis, was found to decrease on maturity advanced in clove buds and in mace while in nutmeg kernel the NVEE showed an increasing trend.

The volatile oil in clove, nutmeg and mace was more at the immature stages and it progressively decreased at the peak harvesting stages. Eugenol, the chief component in clove oil was the maximum at the flowering stage. Hence for extraction of clove oil which is intended for use in medicine, dentistry and other pharmaceutical uses, it is advisable to harvest clove buds at the anthesis stage.

The aromatic ethers which are the chief components that determine the flavour and drug action in nutmeg oil was the maximum one month prior to the fruit splitting stage. In mace oil it was found to be high two months prior to fruit splitting stage. So if nutmeg and mace oils are intended for medicinal purpose, then it may be worthwhile to harvest nutmeg fruits at the 6th month for extracting kernel oil and at the 5th month for extracting mace oil.

Considering the fact that the nutmeg fruit yield both nutmeg and mace oil, harvesting separately at different maturity stages for extraction of the different oils is practically difficult. The aromatic phenol and phenol ethers showed a very low value in mace oil one month prior to fruit splitting. So in practice the fruit may be harvested two months prior to fruit splitting for extraction of both nutmeg and mace oils. However the high moisture content at this stage makes drying a problem. So such a practice of early harvesting of nutmeg fruits may be resorted to in places where facilities are available for extraction of oil from the fresh kernel and mace immediately after harvesting.