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THE YIELD AND QUALITY OF ESSENTIAL OILS DISTILLED FROM CARDAMOMS GROWN IN PAPUA NEW GUINEA

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SUMMARY

A total of 37 cardamom samples were taken from Chimbu, Western Highlands, East New Britain and North Solomons Provinces for essential oil distillation and subsequent chemical analysis for cineole and terpinyl acetate, the major flavour components found in cardamom oil. Oil yields as high as 8.1% v/w (mean of 28 samples was 7.Ø%) were obtained and yields compared very favourably with those obtained elsewhere in the world.

The composition of essential oils distilled from the Mysore race, from all Provinces, compared very well with that required by the food processing industry.

Recommendations are made with a view to the possible establishment of a small, viable essential oil industry in Papua New Guinea.

CONTENTS

1. INTRODUCTION

Cardamoms of commerce are the dried ripe fruits of a perennial herb, Elettaria cardamomum Maton. The plant thrives in the moist tropics at elevations between 750 and 1500 m, with an average temperature of 22°C and a mean annual rainfall exceeding 3000 mm (Brown, 1968).

The seeds contain a volatile oil which has an odour described as warm - spicey, aromatic (Arctander, 1960). Whole cardamoms are divided into two races on the basis of their physical appearance and chemical composition. The cured fruits of the Mysore type are three-cornered and ribbed and tend to be longer than the Malabar type, being about 21 mm in length (Purseglove, et al, 1981). This race possesses a relatively harsh aroma. The Malabar race has a more pleasant, mellow aroma and its cured fruits are generally rounded in shape and about 18.5 mm in length.

The major use of the whole spice is in food preparation. In Asia it is used for flavouring meat and vegetable dishes, in the Arab countries for addition to coffee and in Europe for flavouring baked goods and a variety of processed foods.

An essential oil may be obtained from cardamom seeds by steam distillation. Cardamom oil is one of the oldest essential oils known, its distillation being described in reports as early as 1540. The cured fruits are subjected to coarse crushing and sieving to separate the oil-rich seeds for distillation. The yield and properties of the oil obtained depend on the type of spice used, its age and pre-distillation treatment and to a large extent on the distillation technique employed. The essential oil obtained is an almost colourless or pale yellow liquid which darkens on exposure to daylight.

Like the spice itself, the oil is used for the flavouring of various food products such as cakes, meat products, seasonings, beverages and in perfumery (Arctander, 1960 and Lewis, et al, 1974). It also finds use medicinally in tonic, carminative and purgative preparations (Guenther, 1952).

In Papua New Guinea cardamom plantings are found in the majority of Provinces. The plantings are extremely varied, in scale and nature of management, and also with respect to their climatic and altitudinal situation. The main plantings are in Chimbu (both plantation at Karimui and smallholder plantings at Karimui and Northern Chimbu), Southern Highlands (relatively recent smallholder blocks), Western Highlands and East New Britain. The total production estimated for 1985 is 300 t, of which 225 t will be exported as whole cardamom and 75 t as decorticated seed. Prices have fallen considerably since their 1984 peak and in 1985 are expected to average K8,000/t and K7,500/t (as seed) respectively.

Currently, a market is available for all grades of cardamom. However, as new plantings in India reach maturity, lower grade material may become increasingly difficult to market and in the foreseeable future there will be large quantities of smallholder produce which will be of poor quality (Bakani, 1983).

In this situation an alternative outlet for low grade cardamoms would be beneficial. Distillation of the essential oil from decorticated seed represents one such alternative and offers the additional advantages of much lower domestic and export freight costs, a greater value - added within PNG and hence, potentially, a better price for PNG producers.

The work described in this Technical Report was commissioned by the National Crop Advisor (Spices) to provide data on the yield and quality of essential oils distilled from export graded cardamoms obtained from the major growing areas in Papua New Guinea. This information may be used in an economic appraisal of the viability of a small - scale essential oil distillation plant.

Additionally, cardamoms harvested from trials of the highest - yielding Malabar clones have been assessed for their yield and quality of essential oil This work is presented in Appendix II.

2. MATERIALS AND METHODS

2.1 Reference Compounds and Reagents

(a) oc - Terpinyl acetate (extra UC), 97% v/v, Bush Boake Allen, London, UK.

(b) 1,8 - Cineole (Unilab), 98% v/v, Ajax Chemicals, Sydney, Australia.

(c) Methanol (AR grade), BDH, Dorset, UK.

(d) Acetonitrile (HPLC grade), Waters Associates, Sydney, Australia.

(e) Water, double distilled in glass.

2.2 Apparatus

(a) Clevenger-type volatile oil apparatus incorporating a 2 L flask and 4.0 mL graduated oil separation column.

(b) Forced-draught drying oven capable of maintaining a temperature of 70 $\pm$ 0.5 C. Blue M, Illinois, USA.

(c) High Performance Liquid Chromatograph:-

Model 510 solvent delivery system,
Model U6K injector,
Model 401 differential refractometer,
Model SE120 millivolt recorder,
Millipore solvent filtration apparatus with a 0.45 µm membrane filter, Bondapak C18 column (30cm).

Waters Associates, Sydney, Australia.

2.3 Recovery and Determination of the Essential Oil.

Samples of whole cardamoms and decorticated seed were obtained from cardamom processors and exporters in the major growing areas of Papua New Guinea. The samples were mostly taken from bags of sorted, export grade cardamoms prior to packaging for consignment.

The essential oil was recovered from samples by means of distillation in a Clevenger-type water still (refer appendix I for analytical procedure) prior to determination of the major flavour components by high performance liquid chromatography (HPLC).

Although cardamoms are steam distilled commercially, the Clevenger still was chosen as it affords a simple laboratory-scale method for recovering essential oils in a comparative study. Reproducability of data using this method is superior to that obtained by small-scale steam distillations (Green, 1984).

Moisture determinations were carried out on 10.0 g portions of all samples. Moisture was calculated as the weight loss on drying of the samples in a forced-draught oven at 70° C overnight. An experiment showed that there was no loss of volatile oil during this process.

3. RESULTS

In the period May 1984 to January 1985 a total of 28 whole cardamom (13 Mysore and 15 Malabar), 7 seed and 2 husk samples were collected from Western Highlands, Chimbu, East New Britain and North Solomons Provinces. The volatile oil was recovered from each of these samples by water distillation and the yield of oil recorded. The composition of these essential oils was then determined by HPLC.

3.1 Essential Oil Yields

Purseglove, et al (1981) reported that the highest yielding race is Mysore. The seeds of this type reach maturity when the capsules gain the desirable full green colour, whereas Malabar seeds reach maturity only when the capsules have begun to yellow. In Papua New Guinea cardamoms are normally harvested when green as this trait is desired by the consumer of whole cardamoms. Oil yields of 3.5 - 7% are usually found in cardamoms imported by the major consuming countries. Yields as high as 11% have been obtained from freshly harvested and decorticated seeds.

In Papua New Guinea, laboratory distillations by Price (1973) and Piper (1978) yielded 3.7 - 5.9%, v/w, oil calculated on a moisture - free basis. These results were obtained from samples taken from relatively young plants. Oil yields would be expected to improve with age of the plant.

3.1.1 Mysore Type

Distillation of Mysore cardamoms gave essential oil yields in the range 6.1 - 7.7% v/w (mean 7.1%) on a moisture-free basis (refer Table 1). A similar spread of results was observed in oils derived from cardamoms from all Provinces and there was no consistent relationship between grade and oil yield, although it is to be expected that split capsules will lose the more volatile components from the oil during lengthy periods of storage. Clevenger (1934) reported that whole cardamoms lost 10% and decorticated seeds 30% of their volatile oil content over a period of 8 months.

3.2 Moisture Content.

Moisture remaining in cardamoms after drying is indicative of the efficiency of the drying process. Providing levels are below 10 - 12%, the growth of moulds during storage and transit will be minimised. However, over-drying or use of incorrect temperatures will cause splitting of capsules and subsequent loss of volatile oil by evaporation. Consequently, higher oil yields may have been obtained from cardamoms dried to low moisture levels (e.g. BCC produce) had drying been less severe.

The relatively high moisture levels found in KSC (May 1984) samples (Table 1) were due to early difficulties experienced with a newly-installed oil-fired drier. Reference to the January 1985 moisture value indicates that these problems had been overcome. A similar drier was installed by KGK in 1984 and this probably accounts for the increased moisture levels found in the cardamoms sampled in November.

3.3 Composition of Essential Oils.

Purseglove, et al (1981) stated that the principal quality determinant for cardamoms is the content and composition of the volatile oil, which governs the odour and flavour of the spice. The Malabar type is described as having the most pleasant and mellow aroma and flavour character, while the Mysore type has a harsher aroma and flavour character. This difference in character may be attributed to differences in the composition of the volatile oil. The Mysore type is reported as possessing a higher content of cineole, the major contituent of eucalyptus oil and this causes the harsh aroma of this race. The Malabar type, however, has a higher content of terpinyl acetate and other esters which impart a pleasent "fruity" aroma to the volatile oil. Cardamom oils of relatively low cineole and high ester content are the most valued in food flavouring (Lewis, et al, 1974) and the optimum ratio of cineole/terpinyl acetate is estmated to be 30:40% v/v (Robinson 1984). Although other minor components are important in contributing to the true cardamom flavour, this study was concerned with determining only the major two components. These accounted for 68-86% of the essential oil in all cases.

3.3.1 Mysore Type

Essential oils recovered from the race defined above as Mysore were higher in terpinyl acetate, containing 30-40% v/v cineole (mean 33%) and 32-47% v/v terpinyl acetate (mean 41%) (refer Table 3). The ratio of the two major flavour components is contrary to that reported in the literature (Arctander, 1960; Lewis, 1966; Berhard, 1971 and Purseglove, 1981), where it is stated that the Mysore race has a higher cineole content. However, this result confirms that of Piper (1978) who also showed East New Britain Mysore to contain a higher proportion of terpinyl acetate and Malabar to be higher in cineole.

KSC cardamoms were of consistent composition throughout the grades, although the January 1985 sample contained approximately equal amounts of cineole and terpinyl acetate. A seasonal variation in composition may be indicated here, but requires confirmation. East New Britain Mysore cardamoms and those growing along the Wahgi Valley also show a favourably high proportion of terpinyl acetate in the distilled oil. The single Mysore sample obtained from DPI in the North Solomons yielded an oil relatively high in cineole. However, cardamoms grown in the region are predominently of the Malabar race and this high proportion of cineole is probably due to the sample being harvested from mixed race material (refer Table 4; very high cineole content in Malabar samples from this area).

Table 3. Composition of Mysore Type Essential Oils.

Origin	Grade	% Composition, v/v 1,8-Cineole	oc - Terpinyl Acetate	Total
Chimbu				
KSC (May 1984)	1	32	46	78
	2	32	42	74
	3	32	44	76
	4	31	47	78
	Seed	32	42	74
	Split Capsule	32	36	68
KSC (January 1985)	Unsorted	36	34	70
East New Britain				
BCC (August 1984)	A	30	45	75
	C	35	41	76
BCC (January 1985)	Unsorted	32	36	68
Western Highlands				
CIL (September 1984)	A	35	43	78
	B	32	39	71
	Seed 1	29	48	77
	Seed 2	27	42	69
	Husk	17	51	68
	"Dust"	19	53	72
DPI (September 1984)	A	32	43	75
North Solomons				
DPI (December 1984)	A	40	32	72

3.3.2 Malabar Type

Cardamoms of the Malabar type yielded essential oils proportionately and consistently higher in cineole than terpinyl acetate (refer Table 4). Ranges in composition were 42-56%, v/v (mean 48%) and 28-38%, v/v (mean 33%) respectively. Again, the ratio of these two compounds is the reverse of that normally found in the Malabar type overseas.

The Malabar race grown in Papua New Guinea contains a mixture of cineole and terpinyl acetate in a ratio which is unlikely to be acceptable to the food flavouring industry.

Table 4. Composition of Malabar Type Essential Oils.

Origin	Grade	% Composition, v/v 1,8 - Cineole	α-Terpinyl Acetate	Total
Chimbu				
DPI (December 1984)	Hot air dried	44	30	74
	Solar dried	47	30	77
DPI (January 1985)	A	42	32	74
East New Britain				
BCC (August 1984)	A	42	34	76
	B	43	36	79
	C	47	38	85
	Mixed Seed	41	43	84
BCC (January 1985)	A	42	30	72
	B	48	30	78
	Mixed Seed	34	40	74
KGK (August 1984)	A	50	35	85
	B	51	34	85
	C	47	37	84
	Seed	34	44	78
KGK (November 1984)	A	55	28	83
	B	56	30	86
	Seed	39	42	81
North Solomons				
DPI (December 1984)	1	52	30	82
	2	50	34	84

3.3.3 Decorticated Seed and Husk.

Essential oils distilled from seed exhibited a depressed cineole/terpinyl acetate ratio when compared with oils derived from the corresponding whole cardamoms. This was due to a greater loss of the more volatile cineole (bp 175°C) than the ester (bp 220°C).

Loss of some volatile oil prior to distillation therefore leads to recovery of an essential oil which does not truly reflect the character of the cardamom at harvest. This emphasises the basic requirements for distillation of cardamoms after a minimal post-harvest delay and immediately after decortication.

Examination of the anaytical data for husk samples (Table 3) again demonstrates how cineole is preferentially lost by evaporation, leading to a low cineole/terpinyl acetate ratio and to a low oil yield.

4. DISCUSSION

High yields and a favourable compostion of the Mysore type of cardamom oil were found in Chimbu, Western Highlands and East New Britain Provinces. Therefore, the decision of choosing a location in which to site a pilot-scale still remains one of a logistic nature. Several points need to be considered here. Firstly the smallholder benefits most from any added value since he is more likely to be producing unmarketable low grade cardamoms. On the other hand, the Karimui Spice Company would benefit from the reduced freight costs of transporting an oil rather than a greater weight of whole spice, but then the product quality is high enough to justify export without further processing.

Another important consideration is the provision of steam source as this is integral to the distillation plant. If such a source was currently available, then the cost of a pilot-scale still would be much reduced. Steam is required for the processing of pyrethrum flowers and the plant at Kagamuga Natural Products is equipped with this facility. The factory is close to smallholder cardamom blocks along the Waghi Valley and may prove to be the optimum site for the establishment of a pilot-scale essential oil still. One other steam source alternative is the plant at Ramu Sugar where ethanol is currently distilled.

The viabity of an essential oil industry would depend on other factors such as continuity of supply of raw material and the availability of additional crops suitable for production of essential oils (*e.g.* ginger, citronella, lemongrass and massoi bark). If adequate quantities of such crops are available, this would enable the still to be utilised during periods of low cardamom production.

5. CONCLUSIONS

The evidence presented in this report shows that Papua New Guinea is capable of producing cardamoms with a volatile oil content as high, or higher than cardamoms grown in other countries.

Essential oils distilled from cardamoms of the Mysore type had a composition very similar to that desired by the food processing industry, while the Malabar type produced an oil which is unlikely to be favoured, due to a high content of 1,8 - cineole. The composition of the two races was found to be the reverse of that reported in cardamom oils distilled overseas.

Seed grade material generally contained low quantities of volatile oil, emphasising the fundamental requirement for essential oil distillation immediately after husk removal. High yield and quality of essential oils also depends on the distillation technique, particularly in terms of completeness of oil recovery.

Smallholders, being the major producers of low grade cardamoms, would benefit most from processing of a proportion of their crop into an essential oil. The external appearance of the cardamom husk (*e.g.* colour) is unimportant in terms of oil content and quality of the seeds enclosed.

RECOMMENDATIONS

The following recommendations are made:-

1. Laboratory distillations are practical for a comparative yield/quality study, but do not give the best idea of what may be achieved commercially. Pilot-Scale (50 - 100kg) trials should therefore be established. The trials should be closely supervised by an experienced operator, possibly a representative of the Tropical Development and Research Institute.

2. Trials should receive analytical support from Chemistry Section, DPI.

3. The possibility of locating trials at Kagamuga Natural Products or Ramu Sugar should be investigated.

4. The establishment of a trial still and hire of an operator may prove to be a costly exercise. It would therefore be advisable first to carry out basic market research to determine the size of the market for cardamom oil, current and forecast prices and possible specific quality requirements of consumers. This information would best be obtained from an essential oils dealer who would be conversant with current market trends.

5. If the decision is taken to proceed with trial distillations, further technical studies should be carried out by the Chemistry Section on seasonal changes in cardamom oil yield and quality in the chosen growing area. Chemical investigationsshould also be carried out to determine yield and quality data for additional essential oil crops.

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APPENDIX I

Analytical Procedure

Whole cardamom and decorticated seed samples collected for essential oil recovery were prepared for distillation by passing through the grinder unit of a Kenwood mixer. The unit was cleaned prior to grinding by passing a small portion of the sample to be ground. Loss of volatile oil due to the generation of heat was minimised by passing the sample at a slow rate. Sample preparation was carried out immediately before distillation and the weighed portion transfered directly to the distillation flask. All samples were distilled in duplicate.

Prepared samples (50 g) were water distilled in a clevenger-type glass apparatus, using double distilled water (1 L), anti-bumping granules (2-3) and silicone anti-foaming agent (1-2 drops). The essential oils were recovered by distilling to constant volume. In no case was this of shorter duration than three hours.

The oil yield was recorded and the distilled oil transfered to a glass screw-capped vial prior to analysis by high performance liquid chromatography.

Determination of 1,8 - cineole and α-terpinyl acetate.

The major two flavour components in cardamom oil were determined using a differential refractometer equipped with reference and flow cells. Signals generated by the passage of each component through the flow cell were transmitted to a millivolt recorder and the recorded peak heights compared to those derived from authentic reference compounds.

Oilsamples were prepared for analysis by diluting 100 µL portions (measured using a 100 µL syringe) to standard volume in 5.0 mL volumetric flasks with methanol.

Terpinyl acetate and cineole reference compounds (100 µL each) were similarly diluted in a 10 mL volumetric flask for use as a working standard.

The following conditions were used for the HPLC determination:-

Column: µBondapak C_{18},

Detector: Differential Refractometer (attenuation 4x),

Mobile Phase: 75% v/v acetonitrile/distilled water at 2.0 mL/min,

Injection volume: 5.0 µL,

Chart Speed: 10 mm/min.

The concentrations of the two components in sample solutions were calculated by comparison of peak height measurements with those of the working standard.

APPENDIX II

Malabar Clonal Trials at Raunsepna, East New Britain.

A selection of the highest yielding Malabar clones were planted at Raunsepna, in the Bainings area of East New Britain in October 1981.

Samples were taken from the November 1984 harvest and dried in the laboratory at 54°C for two hours and then overnight at 43°C. The dried cardamoms were then subjected to distillation and HPLC analysis of the essential oils.

Essential oil yields in the range 6.2 - 8.1% v/w (mean 7.1%) (refer Table 5) were obtained from the Malabar clones and these were comparable to cardamoms grown commercially.

Consistent with the results obtained above in 3.3.2, the essential oils were found to have a harsh medicinal aroma and contained a high proportion of cineole. The cineole content was higher than in any other cardamoms analysed (maximum 65% v/v). By comparison, a commercial sample of eucalyptus oil was found to contain 85% v/v cineole.

Whole cardamom samples from these trials should be despatched overseas for assessment as this high cineole content may lead to the whole spice being unpopular among consumers.

Further analyses should be carried out at yearly intervals to determine whether the essential oil content changes as the plants mature.

Table 5. Data from Clonal Trials, Raunsepna

Clone	* % Oil Yield, v/w	1,8 - Cineole	oc-Terpinyl Acetate	Total
C1 /T2	7.1	58	24	82
C2 /T7	7.7	56	29	85
C3 /T9	6.6	63	23	86
C4 /T14	7.6	54	26	80
C5 /T4	7.3	57	25	82
C6 /T15	6.3	58	26	84
C7 /T10	7.2	58	25	83
C8 /T3	7.7	65	23	88
C9 /T6	6.9	59	25	84
C10/T8	6.2	55	27	82
C11/T1	6.2	53	28	81
C12/T13	N/A	N/A	N/A	N/A
C13/T12	8.1	55	28	83
C14/T16	7.9	53	25	78
C15/T5	7.0	57	26	83
CUS/T11	7.0	59	27	86

* Oil yields are expressed on a moisture - free basis.

N/A = Not analysed due to insufficient material.

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