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AN INVESTIGATION INTO THE CHEMICAL CONSTITUENTS OF QUEENSLAND-GROWN GINGER

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SUMMARY

The effects of time of harvest and solvent on the yield and quality of oleoresin and volatile oil from Queensland-grown ginger were examined. The harvest times studied extended from May to October in 1962 and from March to August in 1963. The solvents used to extract the oleoresin were 95 per cent. ethyl alcohol (1962) and 100 per cent. acetone (1963).

The yield of oleoresin extracted from dried ginger varied with both time of harvest and type of solvent used. Time of harvest also had an influence on the yield of volatile oil. When acetone was used as a solvent, there were only small differences in yield of oleoresin on a green-weight basis over the period March to August. There was little variation in volatile oil content during the period April to October.

Extraction with alcohol gave more than three times the amount of oleoresin as extraction with acetone, but the acetone extract was more acceptable to the trade. The volatile oil recovery by steam distillation was not of sufficiently good quality to be of commercial value.

I. INTRODUCTION

In Australia, the ginger plant (*Zingiber officinale*) is grown commercially on clay-loam in the south-eastern corner of Queensland. Since 1958, mechanization of the industry both in the field and in the factory has led to an increased output and markets additional to that of confectionery ginger, which previously had absorbed the bulk of the ginger crop, are now being sought.

The part of the plant which is of commercial value is the underground stem or rhizome, which is used in confectionery trade in a sugar-impregnated form or sold in the dried form for use as spice and the chemical extraction of flavours. The bulk of the world's supplies of dried ginger is at present produced in the

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West Indies, India and West Africa. According to the Bulletin of the Imperial Institute (Anon. 1926), Jamaican ginger was of a relatively uniform high grade; Indian ginger was on the whole of somewhat lower quality, although certain kinds such as Calicut ginger realised prices on the world markets approaching those of Jamaican ginger; and the ginger produced in Sierra Leone and Nigeria was of a lower grade.

According to the British Pharmaceutical Codex (1959), the pungency of ginger is due to the oleoresin, which consists of a mixture of terpenes, ketones (chiefly zingerone), and saturated aliphatic aldehydes (principally n-heptaldehyde). The odour is due to volatile oil, of which the sesquiterpene zingiberine is the principal constituent, in addition to other terpenes and terpene alcohols. The oleoresin is extracted from the ground dried rhizome with alcohol or acetone and is used as a flavouring essence, mainly in the soft drink trade. The volatile oil is recovered by steam distillation of either the green or the dried rhizome and is used as a flavour adjunct and, to a lesser extent, in perfumery.

The experiments reported in this paper were carried out to determine (a) the optimum harvest time for ginger to obtain maximum yields of oleoresin and volatile oil; (b) the relative efficiency of alcohol and acetone in extracting the oleoresin from the dried ginger; (c) whether the alcohol-extracted or acetone-extracted oleoresin produced the better carbonated beverages; (d) how the best of the oleoresin obtained from Buderim-grown ginger compared with oleoresin produced from imported ginger; and (e) if sawdust mulching of the ginger plants affected the flavour of the oleoresin obtained.

II. 1962 TRIAL

The original aim of this trial was to determine the optimum time for harvesting ginger to obtain the maximum yield of volatile oil and alcohol-extracted oleoresin, but subsequent developments in experimental work and in the ginger trade itself led to a widening in scope of the project.

(a) Experimental

Harvesting.—The first harvest was made on May 3 from a plant crop in the Nambour area. The experimental area consisted of 156 randomized sample plants to provide 12 harvests, each of 13 plants. The trial extended over 22 weeks and harvests were made at fortnightly intervals. The plants of each harvest were packed individually and then sent to the laboratory for examination.

Sample Preparation.—On arrival, each ginger plant was treated separately. The 'hands' of the plant were broken up so that all adhering soil could be removed by washing under cold water, and the washed pieces were mechanically cut into $\frac{1}{8}$-in. thick slices. These slices were then loaded in single layers onto trays in a cross-draught dehydrator and the green weight of each sample was noted. The samples were then dried to consistent weight at an air temperature of 120°F, which was chosen to minimize loss of volatile oil during drying. When constant weight had been reached, the dry weight of each sample was recorded

and the drying ratio calculated. The dried slices of each plant were then collected separately and ground in a laboratory hammer-mill to a particle size similar to that of dried ginger sold commercially as a spice. Each sample was then collected and stored in a ground-glass-stoppered bottle prior to chemical determinations, which were carried out within five days to minimize error due to loss in storage.

Analytical Procedures.—The extraction of oleoresin with ethyl alcohol was carried out in a Soxhlet extractor, using 10 g of the sample. The extraction was complete when the solvent surrounding the ginger in the extraction thimble was colourless. This was established in preliminary investigations. The solvent was subsequently evaporated under vacuum, but due to the viscous nature of the oleoresin it was impossible to reduce the product to exactly the same viscosity during each solvent evaporation. However, every effort was made to prepare comparable extracts and therefore reduce experimental errors to a minimum. The volatile oil was determined in a modified Dean and Stark moisture determination apparatus. It was found that the volatile oil could be extracted just as efficiently from the green product.

(b) Results

The results of the 1962 trial are given in Tables 1 and 2 and Figures 1–3. Significant differences in yield of both oleoresin and volatile oil were obtained throughout the entire period studied, the differences in oleoresin being most significant. Based on green weight, the yield of oleoresin remained fairly constant from the beginning of May until the end of June, after which there was a fall until the end of the trial in October. From mid May onwards there was little variation in yield of volatile oil.

TABLE 1

PERCENTAGE OF ALCOHOL-EXTRACTED OLEORESIN BASED ON GREEN GINGER WEIGHT FROM MAY 3, 1962, TO OCTOBER 4, 1962

Treatment	Harvest Date	Mean Percentage	Significant Differences
1	May 3	3·1477	1< 3**, 1 < 4*
2	May 17	3·0577	1 > 8, 9, 10, 11, 12**
3	May 31	3·5962	2 < 3, 4**
4	June 14	3·4808	2 > 8, 9, 10, 11, 12**
5	June 28	3·1108	3 > 5, 6, 7, 8, 9, 10, 11, 12**
6	July 12	2·9569	4 > 5, 6, 7, 8, 9, 10, 11, 12**
7	July 26	3·0523	5 > 8, 9, 10, 11, 12**
8	Aug. 9	2·1992	6 > 8, 9, 10, 11, 12**
9	Aug. 23	2·2631	7 > 8, 9, 10, 11, 12**
10	Sept. 6	2·4600	8 > 12*
11	Sept. 20	2·0308	9 > 12**
12	Oct. 4	1·8831	10 > 11, 12**

* Significant at 5 per cent. level.
** Significant at 1 per cent. level.

TABLE 2

PERCENTAGE OF VOLATILE OIL BASED ON GREEN GINGER WEIGHT FROM MAY 3, 1962, TO OCTOBER 4, 1962

Treatment	Harvest Date	Mean Percentage	Significant Differences
1	May 3	0·3408	1 < 3, 4, 5, 7, 8, 9, 10, 11, 12**
2	May 17	0·3955	2 > 6**
3	May 31	0·4185	3 > 5, 6, 9**, 3 > 12*
4	June 14	0·4100	4 > 6**, 4 > 5, 9*
5	June 28	0·3792	5 < 7**, 5 < 8, 10*
6	July 12	0·3569	6 < 7, 8, 10, 11**, 6 < 12*
7	July 26	0·4185	7 > 9**, 7 > 12*
8	Aug. 9	0·4138	8 > 9*
9	Aug. 23	0·3792	9 < 10*
10	Sept. 6	0·4092	
11	Sept. 20	0·3954	
12	Oct. 4	0·3892	

* Significant at 5 per cent. level. ** Significant at 1 per cent. level.

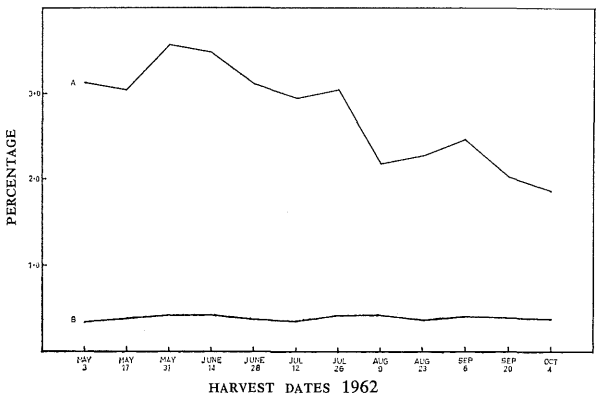

Fig. 1.—Percentages of oleoresin (A) and volatile oil (B) calculated on a green-weight basis, 1962.

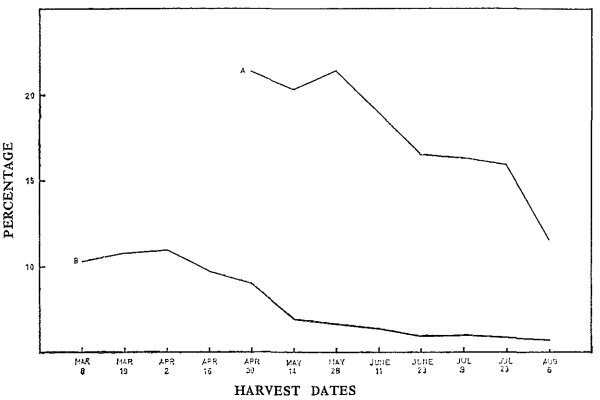

Fig. 2.—Comparative yields of oleoresin calculated on a dry-weight basis. A, alcohol extract, 1962; B, acetone extract, 1963.

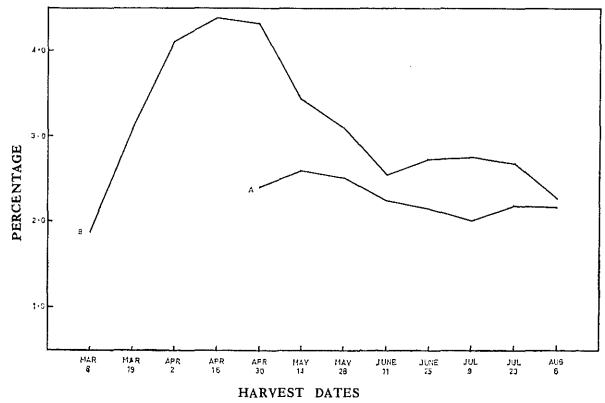

Fig. 3.—Comparative yields of volatile oil calculated on a dry-weight basis. A, 1962; B, 1963.

III. 1963 TRIAL

At the request of the ginger industry, a similar trial was commenced in 1963, using acetone to extract oleoresin. Because of the significant difference obtained in the yields of oleoresin over the period May to October in 1962, it was decided to commence harvesting earlier in the year to determine whether there was a gradual build-up in the percentage of oleoresin simliar to that experienced at the end of May 1962. As R. E. Leverington (unpublished data) reported the optimum time to harvest ginger for the confectionery trade to be about mid March, it was decided to commence the trials about this time.

(a) Experimental

Samples were again taken from a plant crop. Six randomized plants per harvest were considered sufficient for statistical analyses of results. Dehydration methods and determination of oleoresin were similar to that described in the 1962 experiment. Volatile oil was determined on green ginger in a similar manner to that described for dried ginger.

(b) Results

The results of the 1963 trial are set out in Tables 3–7 and Figures 2–4. Significant differences similar to those in the 1962 season occurred. When the oleoresin was determined on a dry-weight basis the highest percentage was obtained during March and April, after which percentages decreased. There was an identical trend in the drying ratio.

TABLE 3

DRYING RATIOS OF GINGER HARVESTED IN 1963

Treatment	Harvest Date	Mean Drying Ratio	Significant Differences
1	Mar. 8	13·233	1 > 4, 5, 6, 7, 8, 9, 10, 11, 12**
2	Mar. 19	12·850	2 > 4, 5, 6, 7, 8, 9, 10, 11, 12**
3	Apr. 2	12·500	3 > 4, 5, 6, 7, 8, 9, 10, 11, 12**
4	Apr. 16	9·850	4 > 6, 7, 8, 9, 10, 11, 12**
5	Apr. 30	10·317	5 > 6, 7, 8, 9, 10, 11, 12**
6	May 14	8·317	6 > 8, 9, 10, 11, 12**, 6 > 7**
7	May 28	7·300	
8	June 11	6·133	7 > 12**, 7 > 8, 9, 10, 11**
9	June 25	6·267	
10	July 9	6·317	
11	July 23	6·317	
12	Aug. 6	5·976	

* Significant at 5 per cent. level.

** Significant at 1 per cent. level.

TABLE 4

PERCENTAGE OF ACETONE-EXTRACTED OLEORESIN BASED ON DRIED GINGER WEIGHT FROM MARCH 8, 1963, TO AUGUST 6, 1963

Treatment	Harvest Date	Mean Percentage	Significant Differences
1	Mar. 8	10·2333	1 > 6, 7, 8, 9, 10, 11, 12**, 1 > 5*
2	Mar. 19	10·7167	2 > 5, 6, 7, 8, 9, 10, 11, 12**
3	Apr. 2	10·9000	3 > 5, 6, 7, 8, 9, 10, 11, 12**, 1 > 4*
4	Apr. 16	9·8667	4 > 6, 7, 8, 9, 10, 11, 12
5	Apr. 30	9·0667	5 > 6, 7, 8, 9, 10, 11, 12**
6	May 14	6·9833	6 > 12**, 6 > 9, 10, 11**
7	May 28	6·6500	7 > 12*
8	June 11	6·3500	
9	June 25	5·933	
10	July 9	5·9667	
11	July 23	5·7167	
12	Aug. 6	5·3667	

* Significant at 5 per cent. level.

** Significant at 1 per cent. level.

TABLE 5

PERCENTAGE OF ACETONE-EXTRACTED OLEORESIN BASED ON GREEN GINGER WEIGHT FROM MARCH 8, 1963, TO AUGUST 6, 1963

Treatment	Harvest Date	Mean Percentage	Significant Differences
1	Mar. 8	0·7767	1 < 4, 8, 9, 10**, 1 < 7, 11, 12*
2	Mar. 19	0·8417	2 < 4, 8**
3	Apr. 2	0·8700	3 < 8**, 3 < 4*
4	Apr. 16	1·0033	4 > 6**, 4 > 5*
5	Apr. 30	0·8800	5 < 8*
6	May 14	0·8383	6 < 8**
7	May 28	0·9167	8 > 11, 12*
8	June 11	1·0250	
9	June 25	0·9483	
10	July 9	0·9433	
11	July 23	0·9033	
12	Aug. 6	0·8983	

* Significant at 5 per cent. level.

** Significant at 1 per cent. level.

TABLE 6

PERCENTAGE OF VOLATILE OIL BASED ON DRY GINGER WEIGHT FROM MARCH 8, 1963, TO AUGUST 6, 1963

Treatment	Harvest Date	Mean Percentage	Significant Differences
1	Mar. 8	1·8833	1 < 2, 3, 4, 5, 6, 7, 8, 9, 10, 11**, 1 < 12*
2	Mar. 19	3·0833	2 < 3, 4, 5**, 2 > 8, 12**, 2 > 11*
3	Apr. 2	4·1000	3 > 6, 7, 8, 9, 10, 11, 12**
4	Apr. 16	4·3833	4 > 6, 7, 8, 9, 10, 11, 12**
5	Apr. 30	4·3167	5 > 6, 7, 8, 9, 10, 11, 12**
6	May 14	3·4333	6 > 8, 9, 10, 11, 12**
7	May 28	3·1000	7 > 8, 12**, 7 > 11*
8	June 11	2·5500	9 > 12*
9	June 25	2·7333	10 > 12*
10	July 9	2·7500	11 > 12*
11	July 23	2·6833	
12	Aug. 6	2·2667	

* Significant at 5 per cent. level.

** Significant at 1 per cent. level.

TABLE 7

PERCENTAGE OF VOLATILE OIL BASED ON GREEN GINGER WEIGHT FROM MARCH 8, 1963, TO AUGUST 6, 1963

Treatment	Harvest Date	Mean Percentage	Significant Differences
1	Mar. 8	0·1433	1 < 2, 3, 4, 5, 6, 7, 8, 9, 10, 11, 12**
2	Mar. 19	0·2417	2 < 3, 4, 5, 6, 7, 8, 9, 10, 11, 12**
3	Apr. 2	0·3300	3 < 4, 5, 6, 7, 8, 9, 10, 11**, 3 < 12*
4	Apr. 16	0·4467	4 > 12**
5	Apr. 30	0·4200	5 > 12*
6	May 14	0·4117	7 > 12*
7	May 28	0·4250	9 > 12**
8	June 11	0·4167	10 > 12**
9	June 25	0·4383	11 > 12*
10	July 9	0·4383	
11	July 23	0·4267	
12	Aug. 6	0·3783	

* Significant at 5 per cent. level.

** Significant at 1 per cent. level.

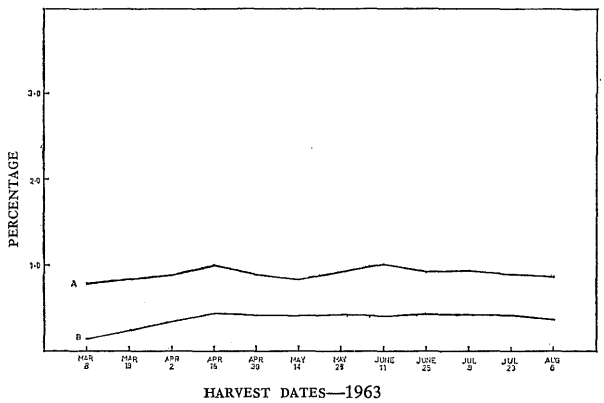

Fig. 4.—Percentages of oleoresin (A) and volatile oil (B) calculated on a green-weight basis, 1963.

When oleoresin was calculated on a green-weight basis, the period of maximum percentage of oleoresin appeared to extend from mid April to mid June. This finding is in line with results obtained during 1962, except that the initial stage is not shown for this year. It was observed that the recovery of oleoresin when using this method was significantly lower than when using alcohol as the solvent.

IV. TASTE EVALUATION

Samples of acetone-extracted and alcohol-extracted resins from Queensland ginger were compared commercially with standard extracts from Jamaican ginger. The oleoresin was incorporated into a carbonated beverage and submitted to a taste panel. The tests showed that beverages made from both the acetone and the alcohol extracts from Queensland ginger were a little weaker in flavour but of the same degree of pungency as beverages made from Jamaican oleoresin. There was a slight preference for the acetone-extracted oleoresin from Queensland ginger, but both this and the alcohol extract had good general appearance and flavour characteristics and were quite promising.

Another investigation was carried out to determine the effect of sawdust mulching on the flavour of the oleoresin. Sawdust mulching is a recommended procedure in ginger cultivation in Queensland and a large proportion of the crop is grown under these conditions. No off-taste was detected in acetone-extracted oleoresin from ginger grown under sawdust mulch when this was tested commercially.

The volatile oil tested was of inferior quality, having little basic ginger flavour.

V. DISCUSSION

The most important fact emerging from these results was the large difference in recovery between the acetone and alcohol extracts of oleoresin from Queensland-grown ginger. This is in contrast to the behaviour of Jamaican, African and Indian ginger tested by the authors (unpublished data), in which the yield of acetone-extracted oleoresin was only slightly lower than the yield of alcohol extract. The difference in yields, however, was never comparable to that obtained with Queensland ginger. It should be noted that the acetone and alcohol extracts compared by us were made in different years.

The method of extracting oleoresin is of major economic importance with Queensland-grown ginger. Although trade preference is for the acetone-extracted oleoresin, acetone is considerably dearer than alcohol. Figures 1, 2 and 4 show clearly the differences in oleoresin obtained by these two methods.

Because of the conflicting factors involved, exploratory work on the extraction by mixed solvents and their effect on flavour was carried out. Results indicate that the 50/50 mixture yields approximately 60 per cent. more oleoresin than 100 per cent. acetone, and the extract has a satisfactory flavour. A 25/75 cent. alcohol and the cost of extraction is still further reduced. Further trials to obtain an oleoresin which has a flavour completely satisfactory to the trade and has a higher recovery as well as cheaper production costs than is possible with 100 per cent. acetone are under way.

Because of the unfavourable reports received on the quality of the volatile oil, it is unlikely that any commercial production of this will be initiated. If, however, the product is later found suitable for manufacturing purposes it could be extracted from crushed green ginger. From Figure 4 it appears that there is a build-up of oil in the green ginger until the middle of April and the oil content then remains fairly constant until the end of the harvest. This is supported by the results shown in Figure 1 when the harvesting was started after April. As current trials include March and April in the harvesting period, it will be possible to compare results with those obtained in 1963.

A graphical representation of the oleoresin content throughout the harvesting period based on dry-weight basis is shown in Figure 2 and trends can be seen in both methods of extraction. The results of the acetone extraction indicate a gradual increase to a peak value early in April, followed by a rather rapid decrease until the middle of May. The yield then drops off more slowly but continuously until August, when the trial was terminated. From this graph the best time to harvest, if acetone is to be the extraction solvent, would be from mid March to mid April. Because the trial using alcohol as the extracting solvent did not commence until May, the period during which the oleoresin content was increasing was not observed. A fairly steady oleoresin content was maintained throughout May, the value being approximately three times that obtained with acetone at approximately the same times in the following year. From June onwards there was a sharp decrease in the yields obtained using alcohol as the solvent. This decrease continued to the end of the trial in October. From this graph it can be seen that harvesting of ginger for oleoresin which is to be extracted with alcohol should be completed by the end of May, but the starting point has not been determined.

VI. ACKNOWLEDGEMENTS

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