

## EFFECT OF MATURITY ON SOME CHEMICAL CONSTITUENTS OF TURMERIC (*CURCUMA LONGA L.*)

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**Abstract :** The effect of maturity on rhizome yield, essential oil content and composition, and curcumin I, II and III of the main Sri Lankan cultivar of *Curcuma longa L.* is reported. The optimum time of harvest for maximum rhizome yield, was found to be 9 months. The oil content in bulbs was higher than that of finger rhizomes. The optimum time for harvest for maximum yield of turmeric oil was found to be 7.5 - 8.0 months. Essential oil composition varied with maturity, sesquiterpenes (ar-turmerone and turmerone) increased while monoterpenes (1,8 cineole and  $\alpha$ -phellandrene) declined in both bulbs and finger rhizomes. Monoterpene content was lower in the mother sets during the early stages of growth. Curcumin (curcumin, demethoxy-curcumin, bis-demethoxycurcumin also termed curcumin I, II and III respectively) content was monitored using t.l.c.-uv spectrophotometry and a technique based on t.l.c.-uv densitometry which was developed during this study. Curcumin I content was highest, however curcumin III content was more than curcumin II. Maturity did not affect the ratio of curcumins to any great extent. Advanced maturity resulted in a decline in total curcumin content. Maximum curcumin per bush was attained about 9 months after planting.

### 1. Introduction

*Curcuma longa L.* (Zingiberaceae) known as Turmeric, is cultivated widely throughout the tropics. The rhizome of this plant is conspicuous by its yellow pigment comprising the curcumins. It is an important food adjuvant and is used in indigenous medicine.<sup>1</sup>

Although turmeric rhizome is used the world over as a spice and food colour, the effect of maturity on essential oil content and ratio of curcumins has not been reported in any detail in the literature. According to Krishnamurthi *et al.*<sup>5</sup> the maximum colour varies with maturity and falls to nearly half its value if harvest is delayed.

The objectives of this study were to determine the effect of maturity on:— (i) curcumin content, (ii) the ratio of curcumins, (iii) volatile oil content, and (iv) volatile oil composition, in order to determine the time of optimum harvest considering volatile oil and curcumin yield.

## 2. Experimental

### 2.1 Plant material

Rhizomes of the major local turmeric cultivar were planted in an experimental plot at Ceylon Institute of Scientific and Industrial Research in two trials in June 1983 and June 1984. Seven beds were prepared (3m x 1.3m). Dried cowdung was applied ( $2\text{tha}^{-1}$ ) on the plot. A spacing of 0.3m x 0.3m was maintained between each seed rhizome. Sampling was carried out by harvesting four bushes (selected at random) from each bed at regular intervals of 15 days from the fourth month after planting to the end of the tenth month.

### 2.2 General procedure

Rhizomes (1kg to 2kg) were washed and pre-dried in the sun for 2h. All experiments were conducted separately on finger rhizome and mother sets (bulbs). Fresh rhizomes were analysed for moisture content (Dean & Stark entrainment method), oil content (Section 2.3) and oil composition (Section 2.4).

Fresh rhizomes were boiled for 10 min., sliced and dried in forced draft air oven at  $60^{\circ}\text{C}$  for 24 h. and powdered in a micro-hammer mill. This powder was analysed for curcumins (Section 2.5).

### 2.3 Oil content

Fresh rhizomes were frozen at  $-20^{\circ}\text{C}$  and ground to fine particle size using a Waring blender along with ice. The oil was extracted by water distillation using a Clavenger arm. The cooling water in the condenser was maintained at  $5^{\circ}\text{C}$  using a Fryka-Term FT 800 cooling water circulator.

### 2.4 Oil composition

The volatile oil was analysed using gas liquid chromatography. Peak identification was carried out by retention data, peak enrichment and GC/MS data. Peak area normalization was used for quantification. The instrument used was a Varian 2440 instrument equipped with a Varian 9176 strip chart recorder through a Pye-Unicam DP-88 computing integrator. The operating parameters are given in Table 1. GC/MS data were recorded using a Finnigen 4000 series GC/MS system under the conditions given in Table 2. Sesquiterpene content was computed by addition of these constituents as obtained from the gas liquid chromatogram.

Table 1 – Operating parameters for glc

Instrument model	– Varian 2440
Recorder model	– Varian 9176
Integrator model	– Pye Unicam – DP 88 (Computing integrator)
Detector	– FID
Column length	– 3 m
Column diameter	– 3mm
Liquid phase	– Carbowax 20M
Solid support	– Chromasorb W (80 – 100 mesh)
Packing material	– 10% carbowax 20M on chromasorb W
Programming	– 60 – 210°C (4°C min <sup>-1</sup> and hold)
Injector temperature	– 200°C
Detector temperature	– 240°C
Carrier gas (He)	– 30 ml min <sup>-1</sup>
Hydrogen supply	– 25 ml min <sup>-1</sup>
Air supply	– 55 ml min <sup>-1</sup>
Recorder Setting	– 1mV
Chart speed	– 5 mm min <sup>-1</sup>

Table 2 – Conditions for GC/MS analysis

## GC Separation

Column length	– 2 m
Column diameter	– 2 mm
Liquid phase	– OV – 351
Solid support	– Chromosorb W (80 – 100 mesh)
Injector temperature	– 275°C
Detector	– 240°C
Programming	– 80 – 240°C (4°C min <sup>-1</sup> and hold)
Carrier gas (He)	– 25 ml min <sup>-1</sup>

## MS details

Ionization voltage	– 70 V
Ionization current	– 300 mA
Accelerating voltage	– 2 – 20 V
Scan-speed	– 44 AMU sec <sup>-1</sup> (33 – 450)
Resolution	– 2500 at 1000 mass

## 2.5 Curcumin content

Oleoresin was extracted from dried, ground turmeric (0.5g) by refluxing with 95% ethanol (100 ml) for 2.5 h. Two solutions  $X_1$  (0.5 g 25 ml<sup>-1</sup>) and  $X_2$  (0.4 g 100 ml<sup>-1</sup>) were prepared from the above solution. The curcumin content of the oleoresin was determined by two methods both based on t.l.c. using  $3 \times 10^{-4}$  m silica gel G- 60 plates with  $\text{CHCl}_3 : \text{C}_2\text{H}_5\text{OH} (25:1)^3$  as the developing solvent.

In the first of these two methods, t.l.c.—uv spectrophotometry was used, 30  $\mu\text{l}$  of solution  $X_1$  was spotted along with 1.6  $\mu\text{g}$  of individual curcumins (curcumin, dcmethoxycurcumin, bis-demethoxy-curcumin also known as curcumin I, II and III respectively) as standards. The curcumins were determined after extracting the yellow spot areas with  $\text{C}_2\text{H}_5\text{OH}$  by stirring the scrapings of the t.l.c. spot in a vortex mixer for 5 min (3 times). Absorbance was measured using a Varian 634 S spectrophotometer at 429, 424 and 419 nm for curcumin I, II and III respectively.

Second quantitative technique was based on t.l.c. —uv densitometry. In this technique 20  $\mu\text{l}$  of solution  $X_2$  along with 0.8  $\mu\text{g}$  of each individual curcumins was spotted on the t.l.c. plate. The plate was scanned using Camag t.l.c. — hptlc variable wavelength densitometer equipped with a strip chart recorder connected to a Pye-Unicam CDPI computing integrator. Operating parameters are given in Table 3. The coefficient of variation of this method was in the range of 4.0 — 5.5%. Although there was a linear relationship between  $\mu\text{g}$  curcumin and peak area, a standard curve was not used for the calculation as small variations in peak area were observed from plate to plate; instead peak areas of standards (on the same plate) were used for calculations.

Table 3 — Operating parameters for t.l.c. — uv densitometry

Light source :- Tungsten

Wave length	—	485 nm
Band width	—	30 nm
Slit width	—	12 mm
Scan speed	—	1 mm s <sup>-1</sup>
Sensitivity	—	6
Recorder setting	—	500 mV
Chart speed	—	50 mm min <sup>-1</sup>

### 3. Results

#### 3.1 Rhizome yield

Both experiments (in 1983/84 as well as 1984/85) yielded very similar results. Table 4 gives the effect of maturity on rhizome yield where a steady increase was observed until 9 months.

Table 4 — Effect of maturity on rhizome yield

Age (months)	Dry Weight*		Total
	M	F	
4	8.8	6.8	15.6
4.5	9.2	14.6	23.8
5.0	10.6	24.5	35.1
5.5	15.2	30.5	45.7
6.0	17.6	46.7	64.3
6.5	22.3	62.9	85.2
7.0	18.9	67.8	86.7
7.5	19.6	70.0	97.6
8.0	22.5	100.9	123.4
8.5	21.4	100.3	121.7
9.0	27.3	104.9	132.2
9.5	21.3	92.7	114.0
10.0	21.9	89.2	111.1

\*Mean for both years

M — Mother set (bulbs)

F — Finger rhizomes

#### 3.2 Oil content

Effect of maturity on volatile oil content is given in Table 5. Volatile oil content (% dry basis) was highest at 5 months and then declined. The oil content of the mother sets in most instances were higher than that of the fingers.

Table 5 — Effect of maturity on volatile oil content

Age (months)	Volatile oil (ml. 100g <sup>-1</sup> , dry basis)	
	M	F
4.0	5.8	5.5
4.5	7.3	7.7
5.0	16.1	9.6
5.5	5.9	6.5
6.0	5.0	4.7
6.5	5.1	4.9
7.0	5.4	4.8
7.5	5.7	5.2
8.0	5.7	4.4
8.5	4.9	4.0
9.0	5.2	3.8
9.5	5.6	4.0
10.0	5.6	3.9

M — Mother set

F — Finger rhizome

### 3.3 Oil composition

A typical gas chromatogram of turmeric oil is given in Figure 1. Effect of maturity on selected essential oil components of turmeric is given in Tables 6 and 7.

Comparative study for oil from mother sets and fingers revealed that monoterpene content ( $\alpha$ -phellandrene, 1,8 cineole and  $\alpha$ -Terpinene) at the early maturities was higher in the fingers ( $\sim 27\%$ ) than in the mother sets ( $\sim 15\%$ ), while at the same time sesquiterpene content (ar-turmerone and tumerone) was higher in the mother sets. As maturity progressed these differences narrowed.

The monoterpene content of the oil from fingers declined markedly with maturity from 27% to less than 10%, while sesquiterpene content increased from 54.5 — 80%. A similar trend, to a lesser degree, was observed in the mother sets where sesquiterpene content increased from 67.5 to 84%.

Table 6 — Effect of maturity on (selected) essential oil components (Mother Sets)

Constituents	Age (Months)												
	4.0	4.5	5.0	5.5	6.0	6.5	7.0	7.5	8.0	8.5	9.0	9.5	10.0
$\alpha$ -phellandrene	8.9	7.2	2.9	6.1	3.1	4.0	1.9	5.1	1.4	1.9	1.7	1.5	1.2
1,8-cineole	3.9	3.2	0.7	2.1	2.1	2.2	2.3	2.2	0.4	0.7	0.9	0.8	0.5
ar-turmerone	26.2	32.6	42.3	37.8	38.8	41.5	46.9	40.9	46.2	46.2	43.7	42.9	41.9
Turmerone	20.0	24.8	34.2	30.6	36.9	36.5	35.9	33.9	39.1	38.9	32.4	33.6	33.3
Sesquiterpene alcohol	8.5	7.9	8.3	7.3	5.4	4.4	3.5	1.4	2.6	1.2	3.4	3.7	4.8

Results are expressed as % total oil.

Table 7 — Effect of maturity on (selected) essential oil components (Finger rhizomes)

Constituents	Age (Months)												
	4.0	4.5	5.0	5.5	6.0	6.5	7.0	7.5	8.0	8.5	9.0	9.5	10.0
$\alpha$ -phellandrene	15.1	13.9	15.5	11.5	14.9	14.5	12.3	10.7	9.9	9.8	3.4	6.9	1.4
1,8-cineole	6.3	5.3	3.6	2.0	4.9	2.5	3.6	2.8	1.9	2.2	1.1	1.5	0.4
ar-turmerone	23.7	24.0	39.0	39.4	36.0	42.0	37.9	39.6	38.6	39.2	39.4	40.1	36.8
Turmerone	17.6	16.7	28.7	32.6	30.6	30.8	31.4	33.0	34.5	37.1	32.9	34.7	36.6
Sesquiterpene alcohol	6.0	6.0	2.1	4.9	2.5	+	2.5	0.8	1.3	0.7	3.3	2.6	5.7

Results are expressed as % total oil

+, trace



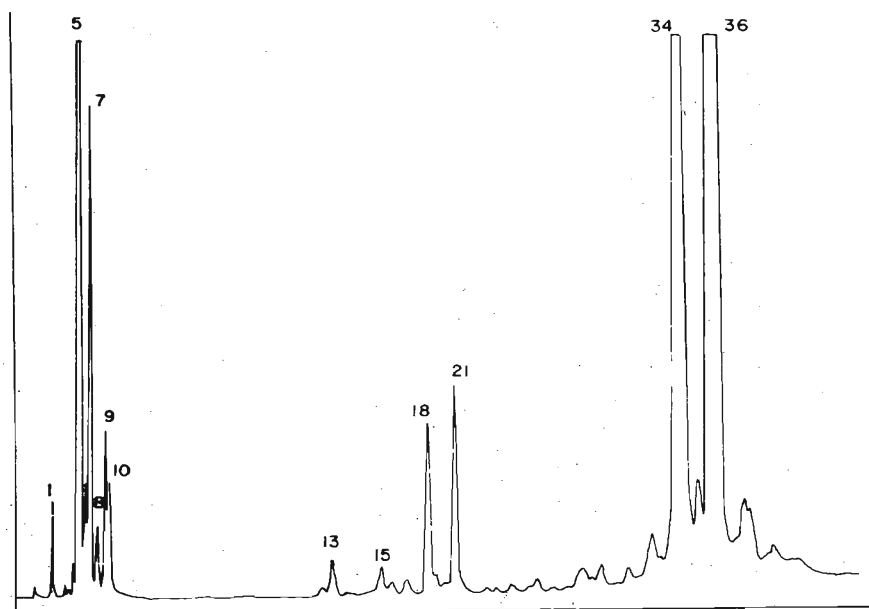


Figure 1. A TYPICAL GAS LIQUID CHROMATOGRAM OF TURMERIC OIL

- |                     |                           |                 |                        |
|---------------------|---------------------------|-----------------|------------------------|
| 1. $\alpha$ -pinene | 5. $\alpha$ -phellandrene | 7. 1,8 Cineole  | 8. $\gamma$ -terpinene |
| 9. p-cymene         | 10. $\alpha$ -terpinolene | 18. Zingiberene | 21. bisabolene         |
| 34. ar-turmerone    | 36. turmerone             |                 |                        |

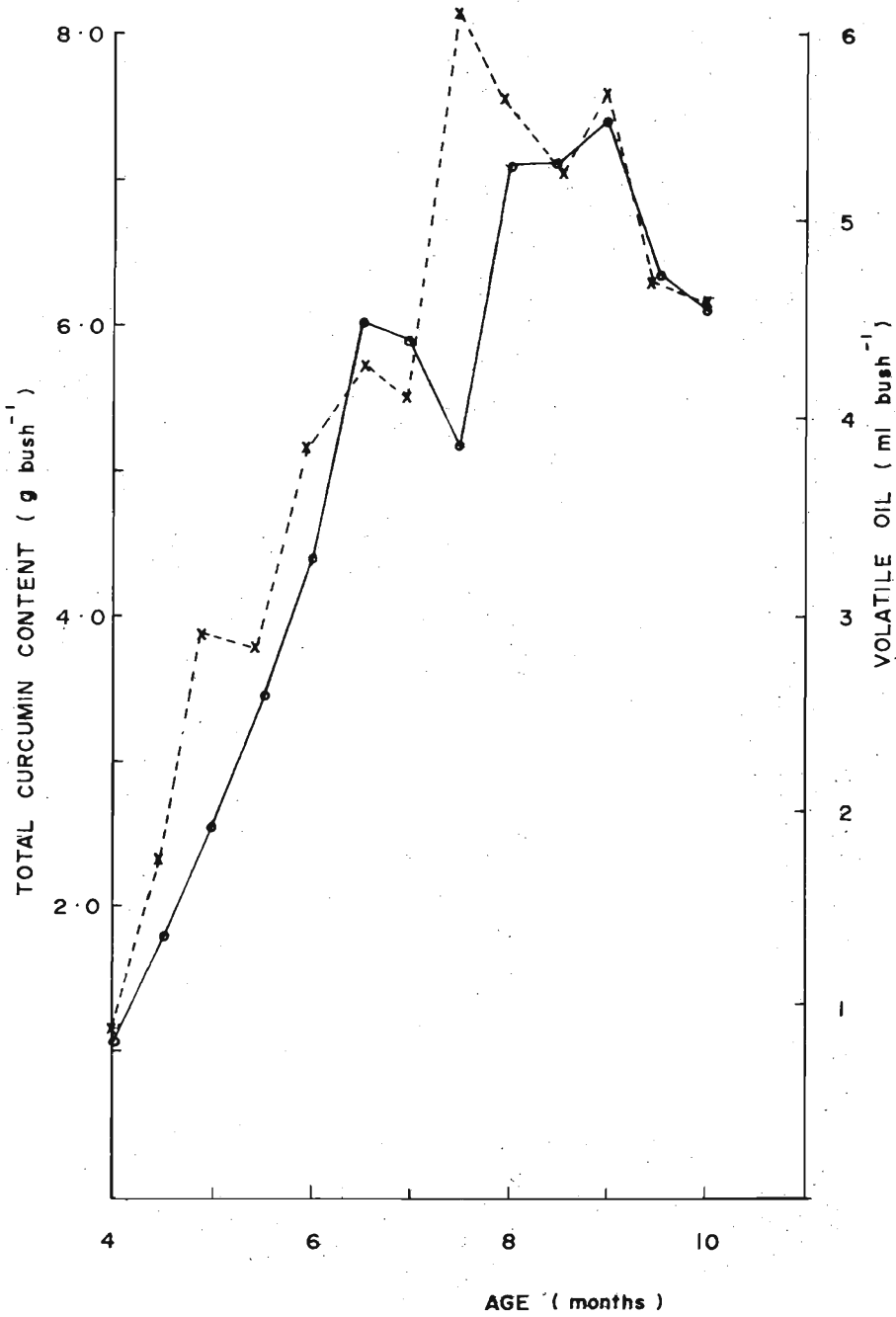


Figure 2. EFFECT OF MATURITY OF TURMERIC RHIZOME ON TOTAL OIL AND CURCUMIN PER BUSH

○—○ CURCUMIN CONTENT  
X- - -X OIL CONTENT

Table 8 — Effect of maturity on curcumin content

Age (months)	Curcumin content (g. 100 g <sup>-1</sup> )							
	Mother rhizomes				Finger rhizomes			
	C-I	C-II	C-III	Total	C-I	C-II	C-III	Total
4.0	4.16	1.14	1.60	6.90	4.78	1.38	1.65	7.81
4.5	4.02	1.16	1.64	6.82	4.99	1.37	1.72	8.08
5.0	4.05	1.39	1.81	7.25	4.23	1.39	1.76	7.38
5.5	4.21	1.46	1.76	7.43	4.30	1.50	1.90	7.70
6.0	3.87	1.26	1.63	6.76	4.21	1.27	1.53	7.01
6.5	3.44	1.30	1.79	6.53	3.77	1.39	1.69	6.95
7.0	3.45	1.15	1.52	6.12	4.06	1.26	1.69	7.01
7.5	3.22	1.11	1.44	5.87	3.26	1.10	1.35	5.71
8.0	2.86	1.18	1.45	5.49	3.26	1.19	1.37	5.82
8.5	2.90	0.93	1.27	5.10	3.48	1.09	1.43	6.00
9.0	2.79	0.95	1.15	4.89	3.41	1.10	1.39	5.80
9.5	2.71	0.91	1.23	4.85	3.29	1.10	1.30	5.60
10.0	2.82	1.04	1.22	5.08	3.32	1.04	1.23	5.59

Results are a mean of both experimental years.

C. — Curcumin

### 3.4 Curcumin content

Effect of maturity on curcumin content is given in Table 8. There is decline in curcumin content both in mother sets and fingers with maturity. However total curcumin content per bush reached maximum at 9 months (Figure 2). In most instances the finger rhizomes contain higher curcumin content than mother rhizomes.

## 4. Discussion

When the data of both the years were pooled and analysed, it was observed that rhizome yield (dry basis) gradually increased with maturity and reached a maximum around 9 months. After this period yields declined.

Despite oil content being higher at early maturities, as the total yield per bush is low, the maximum oil content per bush is attained between 7.5 to 8.0 months after planting (Figure 2).

At the fully matured stage about 80% of the oil comprises sesquiterpenes. The results on oil composition show two main trends.

- (1) Monoterpene content is higher at the outset in the finger rhizome than the mother sets. The reverse is true for sesquiterpenes.
- (2) Differences narrowed as maturity was reached.

Though it is generally believed that bulbs have more colour than fingers, our results proved fingers generally contain slightly higher levels of curcumin than the mother sets (fully mature mother rhizomes contain  $\sim$  5% curcumin whereas fingers contain 5.6%).

Curcumin content reaches a maximum around 5.5 months and 4.5 to 5.0 months respectively for the mother sets and fingers thereafter declines with maturity. The total curcumin per bush reaches its highest at about 9 months after planting and this appears to be the ideal time for harvest for curcumin extraction (Figure 2).

The ratio of curcumins: demethoxycurcumin:bis-demethoxycurcumin has been estimated previously<sup>2,3,4,6</sup> using t.l.c.—uv spectrophotometry. No work with t.l.c.—uv densitometer has been previously reported. In this study both methods yielded similar results.

Our results showed that curcumin I:II:III ratio does not vary to any great extent during maturation. The ratio differs from results reported from other parts of the world.<sup>2,3,4,6</sup>

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