PEEL OIL CONTENT OF 'VALENCIA' ORANGES[1]

R. HENDRICKSON
J. W. KESTERSON

Florida Citrus Experiment Station
Lake Alfred
AND
S. V. TING

State of Florida, Department of Citrus
Florida Citrus Experiment Station
Lake Alfred

ABSTRACT

The influence of maturity brought about an increasing quantity of oil per unit of peel surface area as well as per unit of fresh weight. Cultural practices such as rates and sources of magnesium, manganese, potassium, and nitrogen did not significantly influence peel oil content. The oil content of the peel increased progressively from the stem end to the stylar end and noted oil yield, in any large lot of fruit, to be correlated with the quantity of surface area. A very significant relationship confirmed by regression analysis showed that the quantity of oil per unit of surface area was related to size of fruit. The larger the fruit, the greater the quantity of peel oil found per unit of peel area. There is an indication that bud wood selection probably has a greater influence on oil yield than previously anticipated.

INTRODUCTION

This investigation was initiated to provide fundamental information on the quantity of oil in Florida 'Valencia' oranges. Despite the economic importance of this essential oil, the only previous study pertinent to the oil content of Florida oranges was published in 1916 by Hood (4). 'Valencia' trees at 4 locations were sampled biweekly from November 1 to December 18, each sample comprising 12 oranges. The quantity of oil recovered by distillation from a water slurry of the peel, that had been removed by knife and ground in a food chopper, was converted to a percentage. Volatile oil contents ranged from 0.24% to 0.53% in this study. The author concluded that a considerable increase in oil content had occurred as maturity advanced, that oil content was retarded in its increase by a period of rainfall, and that rust mite damaged fruit eventually regained normal amounts of oil by maturity.

In 1917, Wilson and Young (7) determined the volatile oil content of citrus by triple-grinding whole fruit in a food chopper and measuring the oil condensed by distilling an aliquot with additional water. Four samples of California 'Valencia' oranges ranged from 0.71% to 1.20% volatile oil. They recovered less oil from separated peel, Hood procedure (4), than from whole fruit. More recently, Braverman and Monselise (3), studying the oil content of Palestine oranges and grapefruit, could not establish a seasonal pattern nor find any relationship between it and rainfall. A thin layer was peeled from the fruit in a somewhat similar overall procedure to that of Hood (4). Samisch (5) found the coarse fruit of 5-year-old 'Shamouti' trees to have slightly more oil than smooth fruit of the same diameter (0.96% *vs* 0.94%). As fruit size decreased, a respectively greater percentage of oil was found in one trial. The fruit were halved and the pulp removed prior to completing the usual volatile oil distillation.

Bartholomew and Sinclair (1,2) studied the volatile oil content of California 'Valencia' oranges by removing discs of peel as in the present investigation. The 100 discs, removed from 50 or 100 fruit, were chopped in a Waring blendor with water and the volatile oil recovered by distillation. The authors found little variation in mean yield of oil per unit of peel area regardless of maturity of fruit size. They found the oil content of the peel to increase progressively from the stem end to the stylar end and noted oil yield, in any large lot of fruit, to be correlated with the quantity of surface area.

The present study describes an improved technique for determining the peel oil content of citrus and comments on the accumulated results obtained by analyzing numerous lots of Florida 'Valencia' oranges during the 1968-69 season.

MATERIALS AND METHODS

Sampling.—The influence of maturity on the

Florida Agricultural Experiment Station Journal Series No. 3360.
[1]Cooperative research, University of Florida, IFAS and State of Florida, Department of Citrus.

oil content of 'Valencia' oranges was investigated by picking fruit samples at approximately monthly intervals from 2 adjacent trees that were sampled from September 24, 1968, to June 5, 1969. These trees, all about 31 years old, located at the Lake Alfred Citrus Experiment Station, received a standard fertilizer program. Each sample comprised 16 fruit picked at intervals around the circumference of the trees between 2 and 8 feet from the ground. One additional fruit was picked at each sampling for a whole fruit analysis of oil content.

The effect of various fertilizer elements on the yield of oil in the peel of 'Valencia' oranges was determined by sampling the blocks and plots of co-workers. An 8-fruit sample was taken from each of the 3 replications in a 15 block experiment wherein manganese was applied at different rates using various sources. Large, small, irregular, and peel-damaged fruit were avoided.

Similarly, 36 plots of 'Valencia' trees in a magnesium experiment, using 4 sources, 3 levels, and replicated 3 times, and 28 plots in a potassium experiment, using 3 sources, 2 levels, and replicated 4 times were sampled April 30, 1969. Also, 16 plots of a nitrogen-potassium experiment of factorial design with 4 levels were sampled April 2, 1969.

To determine the influence of size on quantity of oil in the peel of fruit, samples (8 fruit) were randomly taken from bins representing 6 established sizes in the packinghouse. Samples were taken February 18, 1969, February 26, 1969, and March 31, 1969, each from 12 to 15-box lots of commercially grown fruit from the Haines City area of Florida.

The oil content of fruit picked from trees that had been sprayed with a plastic sealer (3% Pinolene) 3 months prior to harvest was also determined and compared with that from control trees.

Analytical technique. — Determine the net weight of a 16-fruit sample, measure the diameter of the major and minor axes, then spray each fruit with Krylon® and allow it to dry. Cut 2 or 4 discs per fruit in the unblemished equator region using a No. 13 cork borer (2 cm diameter). When the sample comprises 8 fruit, cut 4 discs per fruit. The discs are cut free using a dissecting knife and dropped immediately into 300 ml of previously tared 99% technical isopropanol. Determine the net weight of alcohol plus 32 discs and blend in a one-quart stainless steel sealed jar using a Waring Disintegrator at medium speed for a total of 3 minutes. Transfer and seal the sample in a one-pint Mason jar then shake for a minimum of 16 hours.

To determine the volatile oil content, stir contents of the one-pint jar magnetically while pipetting about 20 ml into a tared 500-ml round-bottom distilling flask, then reweigh. The pipette should have a minimum of taper and a wide opening to drain completely. Add 5 to 10 ml additional isopropanol, 25 ml of water, a few Alundum chips, 4 drops of Dow-Croning Antifoam B®, then distill and titrate with 0.100 N bromide-bromate by the Scott-Veldhuis procedure 6). Alternatively, the clear serum of the equilibrated Mason jar sample can be distilled (eliminates bumping) and titrated, provided a 3% correction is subtracted for the missing insolubles.

A whole fruit sample is analyzed for volatile oil content by cutting the orange into 2 or more smaller pieces under alcohol and chopping to a smooth slurry in the Waring Disintegrator. A total of 700 ml of isopropanol is used and the sample sealed in a one-quart Mason jar.

Calculations.—Major and minor fruit axes measurements were entered into mathematical formulas for spheres, prolate spheroids, and oblate spheroids to calculate individual surface areas which were then averaged for the sample. The volume of peel oil in ml per 100 square cm of peel surface was calculated on the basis that each disc comprised 3.30 square cm of surface area, and that 'Valencia' orange oil has 96.3% volatile oil responding to the bromide-bromate procedure. Pounds of peel oil per ton of fruit were calculated, by determining the average volume of oil per unit weight of fruit, then extrapolating for the equivalent volume in a ton of fruit and finally determining its weight using 0.843 as the density of Florida 'Valencia' oil.

Results and Discussion

Seasonal changes in oil content.—The results of analyzing a one-fruit and 16-fruit sample from each of 2 trees during the 1968-69 season are presented in Table 1. Although no data are shown for the comparison of fruit weight to surface area changes during the sampling

Table 1.--Influence of maturity on quantity of peel oil in 'Valencia' oranges.

Sample picking date	16-fruit disc sample: Mean* fruit wt.	16-fruit disc sample: Mean* surface area	16-fruit disc sample: Peel oil per 100 sq. cm.		1 whole fruit: Peel oil per 100 sq. cm.		Peel oil: Per ton of fruit	Peel oil: In whole fruit
Tree	B	B	A	B	A	B	A & B	A & B
	g	cm^2	ml	ml	ml	ml	lbs	%
9/24/68	104	106	0.89	0.84	-	-	14.8	0.74
10/8/68	117	116	0.83	0.85	0.90	0.71	14.1	0.70
10/22/68	132	130	0.99	0.93	0.98	0.65	15.3	0.76
11/25/68	147	151	0.96	0.85	1.00	0.98	15.0	0.75
12/16/68	141	133	0.97	0.96	0.96	0.78	15.2	0.76
1/13/69	177	156	0.93	1.00	1.25	1.21	14.3	0.72
2/10/69	148	139	1.01	0.99	0.69	1.08	15.6	0.78
3/10/69	157	143	1.04	0.94	0.94	1.06	15.2	0.76
4/21/69	185	158	1.31	1.21	1.16	1.18	18.3	0.92
5/15/69	177	156	1.38	1.36	1.06	1.34	19.7	0.98
6/5/69	181	152	1.31	1.45	1.51	1.42	19.6	0.98

*Similar values were obtained for fruit of Tree A.

period, this ratio increased continually and at maturity was 20% greater than that found at the initial sampling. The weight to surface area has a profound influence on the total surface area of a one-ton fruit sample and the ultimate yield of peel oil obtained from this quantity of fruit. Other factors being equal, the ml of oil per unit of surface area must consequently increase 20% from September to June to maintain the same yield of oil per ton of fruit. The results shown in Table 1 for ml of peel oil per 100 square cm demonstrated that oil content did increase at a rate sufficient to make up for the changing ratio of weight to surface area. The same results also provide evidences of sampling and tree-to-tree variation that occurred for the 2 respective sample sizes.

A linear regression was calculated for the influence of maturation upon peel oil content per 100 square cm. The equation was as follows:

$$Y = 0.76 + 0.002 X$$

In this equation, Y is equivalent to ml of peel oil per 100 square cm and X equivalent to the days of maturity beyond September 1. This equation indicates that oil content was increasing an average of 6% per month. Although this linear regression showed a very significant correlation coefficient, peel oil content per 100 square cm so sharply increased in April as to imply the need for a more precise equation. Further study in the next season will clarify this point.

The natural variation that occurred when analyzing only a portion of the fruit picked by a standard pattern from 'Valencia' trees led to standard deviations of greater magnitude as sample size decreased. Since the larger samples were analyzed by removing peel discs from the equator of the fruit, there was the possibility that the percentage of oil in this area would not represent the mean equivalent for the total surface. Linear regression plots of the data obtained by both disc and whole fruit analyses throughout the season led to parallel lines, each

Table 2.--Summary of peel oil analyses of 8-fruit 'Valencia' samples picked from experimental fertilizer plots.

Fertilizer experiment varying	Date picked	No. of samples	Fruit wt.(g.) Avg.	Range	ml peel oil/ 100 cm^2 Avg.	Range	Pounds peel oil/ ton fruit Avg.	Range
Manganese	5/16/69	45	137	119 174	1.16	0.98 1.37	17.0	13.7 19.7
Magnesium	4/30/69	36	224	209 256	1.48	1.22 1.67	20.8	16.3 22.4
Potassium	4/30/69	29	167	105 166	1.56	1.38 1.80	22.7	20.0 28.0
Nitrogen-potassium	4/2/69	16	207	196 223	1.10	1.04 1.18	16.3	14.8 17.7

showing a very signficant correlation. Statistically, it was indicated that disc analyses found 0.04 ml of peel oil per 100 square cm of surface area more than whole fruit analyses. Although a mean correction of 4% is statistically suggested, the authors have avoided substracting it until new data again signifies the need.

When the peel oil results of this maturity study were converted to yield of oil per ton of fruit (Table 1) there were less accentuated differences, but still a very noticeable upward trend as the season advanced. Although this lot of fruit had almost 20 pounds of oil per ton of fruit at maturity, Florida processors usually recover no more than 4 to 6 pounds of oil per ton of 'Valencia' fruit.

The percent of peel oil in the fruit (Table 1) was surprisingly higher than that found by many earlier investigators who peeled their fruit. Essentially, the results indicated that oil content was increasing at a more rapid rate than fruit weight.

Cultural practice vs oil content.—The summarized analytical results for 126 'Valencia' plots on various replicated fertilizer programs are presented in Table 2. No significant rela-

Table 3.--Peel oil content of 'Valencia' oranges from different areas.

Fruit from	Picking date	Fruit per sample	No. of samples	Average Fruit wt. g.	Average Oil per 100 cm^2 ml.	Average Oil/ton fruit lbs.	Average Oil in fruit %
Haines City	3/31/69	8	7	180	1.17	15.1	0.76
CES, Block 17	4/30/69	8	29	167	1.56	24.0	1.20
IRFL	5/16/69	8	45	137	1.16	16.3	0.82
California	7/4/69	8	2	216	1.51	20.2	1.01
CES, Block R Plastic-sprayed	7/9/69	16	2	221	1.23	16.0	0.80
CES, Block R Non-sprayed	7/9/69	16	2	200	1.38	18.7	0.94

tionship was found that correlated fertilizer practice with oil content of the fruit.

Oil content of fruit from different regions.—In Table 3 are found the average fruit weight, oil per 100 square cm of peel area, peel oil per ton of fruit, and percentage of oil in 'Valencia' oranges from California and different areas of Florida. The California samples were taken from carefully handled, unwashed fruit flown into this area for pesticide residue trials at the Citrus Experiment Station's piolt plant. The oil content of this fruit was considerably greater per unit of peel area than the highest reported (1.29 ml per 100 square cm) by Bartholomew and Sinclair (1) for the Inland district of California. These 2 fruit samples are inadequate to draw broad conclusions since they did not meet the usual size and weight statistics that Floridians consider to be standard for California fruit. Oil content was within the range of values for Florida fruit. The high quantity of oil present in Florida fruit suggests that efficiency of oil recovery is probably lower than many processors have estimated in the past.

Plastic sealer vs oil content of fruit.—Samples picked from a tree sprayed with a plastic sealer (3% Pinolene) yielded less oil as determined by the present method in contradiction to a theory that the spray might seal more oil in its fruit.

Effect of fruit size on yield of oil.—There was a very significant relationship confirmed by regression analysis that quantity of oil per unit of surface area was related to size of fruit. The larger the fruit, the greater was the quantity of peel oil found per unit of peel area. Regression curves for the 3 packinghouse trials are shown in Figure 1.

Although insufficient information is available at this writing, the wide range of values for percent of oil in mature 'Valencia' oranges (Tables 2 and 3) suggests that some condition, other than climate or physiology, such as 'cultivar' or bud wood difference, may be more responsible for the differences found. This possibility will be evaluated in the following season since the economic return for the recovery of orange oil could justify its consideration.

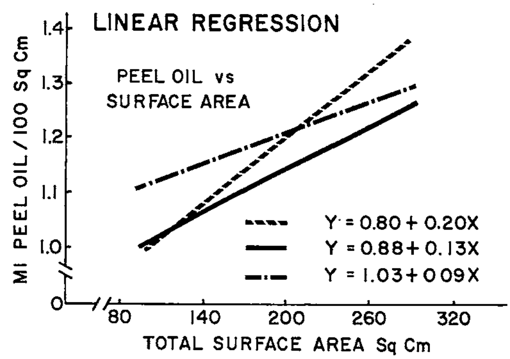

Figure 1.—Linear regressions showing the relationships found between uantity of iil per unit of peel surface and mean total surface area for each of three 12 to 15-box lots of packinghouse 'Valencia' fruit.

LITERATURE CITED

1. Bartolomew, E. T., and W. B. Sinclair. 1946. Factors influencing the volatile oil content of the peel of immature and mature oranges. Plant Physiol. 21: 319-31.
2. Bartholomew, E. T., and W. B. Sinclair. 1946. Volatile oil content of the peel of oranges. Calif. Citrograph 31: 293, 329.
3. Braverman, J. B. S., and G. G. Monselise. 1940. Studies in citrus oils of Palestine. Hadar 13: 225-26.
4. Hood, S. C. 1916. Relative oil yield of Florida oranges. Ind. & Eng. Chem. 8: 709-11.
5. Samisch, Z. 1940. The production of citrus oil as a home industry in Palestine. Hadar 13: 67-70.
6. Scott, C. W., and M. K. Veldhuis. 1966. Rapid estimation of recoverable oil in citrus juices by bromate titration. J. Ass. Affic. Agric. Chem. 49: 628-33.
7. Wilson, C. P., and C. O. Young. 1917. A method for the determination of the volatile oil content of citrus fruits. Ind. & Eng. Chem. 9: 959-61.