*Processing Section*

# COMPARISON OF CHARACTERISTICS IN COMMERCIAL AQUEOUS EXTRACTS OF ORANGE PULP PRODUCED FROM MIDSEASON AND LATE SEASON FRUIT

## I. INTRODUCTION AND SOME CHARACTERISTICS IN AQUEOUS EXTRACTS OF ORANGE PULP[1]

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Procedures for the water extraction of fruit solids from orange pulp were incorporated during the 1957-58 citrus season as an additional process in the production of frozen orange concentrate by some commercial plants in Florida. This additional process was introduced primarily because of the extra yield of orange solids obtainable. Such an increase in yield of fruit solids, naturally, has always been important and of interest to management. It became of greater importance during the 1957-58 processing season because of the high cost of oranges which resulted from a short supply brought about by the severe freezes of that season. Also, the elimination of the second finisher in a double juice-finishing operation by the substitution of a "pulp washing" process was of interest to some companies as a possible means for improving the quality of frozen orange concentrate. A majority of the concentrate plants in Florida produced aqueous extracts of orange pulp by various pulp washing procedures during the 1958-59 citrus season and added such extracts to the evaporator feed juice during the production of orange concentrate.

Many questions were asked about equipment and procedures for the recovery of fruit solids from orange pulp, as well as the effect of pulp washing on yield of solids and the quality of frozen orange concentrate. To obtain data on which to base answers to such questions, a study of the water extraction of orange solids from pulp was undertaken at the Citrus Experiment Station in April 1958. Results from this investigation have been discussed on several occasions (1, 3) and also published (2, 4).

The purpose of this series of four papers is to present and discuss data obtained from the examination of commercial samples of aqueous extracts of orange pulp which were collected during the 1958-59 season from some Florida plants. Characteristics of extracts of orange pulp produced from midseason and late season fruit are compared. These data make possible a comparison of these properties with those found in samples of orange juices obtained from the commercial plants at the same time. A discussion of the effect of some pulp washing procedures on these characteristics is also presented. Finally, some conclusions, based upon the results of this investigation, are given concerning the use of aqueous extracts of orange pulp in the production of frozen orange concentrate.

### EXPERIMENTAL PROCEDURE

*Collection of Commercial Samples.*—Water extracts of orange pulp and samples of pulp, both before and after extraction, were collected from 9 plants from January 28 to February 11, 1959, at which time some of the commercial midseason pack of frozen orange con-

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centrate was being processed. Samples of orange juice also were taken in these plants immediately after the first juice-finishing operation. Although 5 plants were using centrifuges to remove water-insoluble solids from the very pulpy extracts, samples of extracts both before and after centrifugation were obtained from only 3 plants. When some of the late season pack of frozen orange concentrate was being processed during May 1959, similar samples of water extracts, pulps, and orange juices were obtained from the same concentrate plants.

All samples were filled in No. 1 plain tin cans at the plants, the cans closed, and the products immediately and rapidly frozen by immersion in a mixture of dry ice and isopropyl alcohol. During transportation to this Station the samples were kept frozen and then stored at -8°F. until examined.

*Analytical Procedures.*—After removal of the samples from storage at -8°F. and thawing, 35 samples of orange pulp, 23 water extracts, and 17 orange juices were examined for the 8 characteristics discussed in this paper. All Brix values were determined using a refractometer. Total acid, pH, and ascorbic acid in the aqueous extracts and orange juices were determined by conventional methods. The Brix/acid ratios were calculated. Relative viscosities of the centrifuged extracts and juices were measured at 26°C with an Ostwald pipette. The orange juices and the water extracts of orange pulp were also examined for color and flavor.

## Results and Discussion

*Processes and Equipment Used in Commercial Plants.*—When the commercial samples were obtained, information in considerable detail was furnished by all companies, except one, concerning the equipment and process being used for water extraction of orange pulp. Both paddle finishers, with either brushes or paddles, and screw finishers were used in the various pulp extraction processes with the number in a series ranging from 3 to 6; rotating screens were employed in 2 plants. Food Machinery In-Line juice extractors and Brown juice extractors were utilized in 5 and 4 of the plants, respectively. A brief description of the different pulp washing processes in use when the midseason and late season samples were obtained from the plants is given in Tables 1 and 2. Letter codes used in many of the tables in this series of 4 papers

Table 1. Procedures used for water extraction of orange pulp in some commercial plants during processing of the 1958-59 midseason pack of frozen orange concentrate

| Plant number | Code used in tables[1] | Extraction process | Soluble solids - ° Brix: In pulp, Before extraction | In pulp, After extraction | In water extract |
|---|---|---|---|---|---|
| 1 | CF | Countercurrent flow through finishers | 12.2 | 6.4 | 4.0 |
| 2 | CF | Countercurrent flow through finishers; however, extract and juice mixed and then centrifuged before evaporation | 9.6 | 3.4[2] | 7.5[3] |
| 3 | CFC* | Countercurrent flow through finishers and extract centrifuged | 10.6 | 2.4 | 4.9 |
| 4 | CFRC* | Countercurrent flow through finishers, extracts recycled and final extract centrifuged | 12.0 | 5.2 | 7.7 |
| 5 | CFRC* | Countercurrent flow through finishers, extracts recycled and final extract centrifuged | 10.2 | 3.6 | 7.5[4] |
| 6 | CS. | Countercurrent flow through rotating screen | 11.0 | 7.6 | 8.6 |
| 7 | SFR | Rotating screen and finisher used in double juice finishing operation; water added after rotating screen and extract recycled | 9.4 | 5.0 | 6.5 |
| 8 | WDF | Water added after first finisher in double juice finishing operation | 10.4 | 8.2 | 7.9 |
| 9 | NOC* | Not observed, but extract centrifuged | 10.8 | 4.0 | 7.7 |

[1] C = countercurrent flow of water and pulp; and C* = centrifuge used.

[2] Soluble solids in pulp after centrifugation of mixture of extract and juice.

[3] Sample of extract before it was mixed with juice for centrifugation.

[4] Sample of extract before centrifugation.

to indicate the different pulp extraction processes are also given in Tables 1 and 2.

In plants 1, 2, 3, 4, and 5 (Table 1) the pulp was discharged consecutively from each of a series of juice finishers and water was added to the pulp which entered the last finisher. Then the water extract was pumped successively from each of the finishers, in a countercurrent fashion, to the other finishers in the series; also, in plants 4 and 5 the water extract was recycled through several finishers. A countercurrent flow of water extract from sections of a rotating screen was employed in plant 6 as the pulp passed through the screen. The extraction process in plant 7 consisted of recycling the water extract through a double finishing process using a rotating screen and a juice finisher. Water was added between the first and second finishers of a double finishing operation in plant 8. In plants 3, 4, 5, and 9 centrifuges of various types were used to reduce the amount of pulp in the water extract prior to mixing with juice for evaporation. However, it was not possible to get a sample of extract after centrifugation from plant 5, whereas such samples were taken in the other 3 plants. In plant 2 the water extract and the juice were mixed and then centrifuged before evaporation. Therefore, the sample of pulp discharged from the centrifuge in this plant contained pulp from both the water extract and the evaporator feed juice. Also, it was not possible to obtain a sample of water extract after centrifugation from plant 2.

Some changes in the pulp washing process had been made in 3 of the 9 plants when the late season samples of orange juices, pulps, and water extracts were collected during May. In plants 5 and 9 (Table 2) centrifuges were not being used and in one of these some changes had been made in the recycling procedures. The water extract was being centrifuged in plant 2, whereas previously the extract and juice were mixed and then centrifuged before evaporation. As indicated in Table 2, samples of water extract and orange pulp were not obtained from plants 1 and 9, respectively.

*Use of Pilot Plant Unit for Recovery of Fruit Solids from Orange Pulp.*—The rapidity with which soluble solids may be extracted from orange pulp was demonstrated (4) with a pilot plant extraction unit. This unit consisted of pulp feeding equipment and a series of four small Chisholm-Ryder Sepro-sieve screw finishers equipped with 0.020 inch screens. When used, pulp was fed into the first finisher and discharged from the fourth finisher; water was added to the fourth finisher and the extracts were then pumped successively back into each

Table 2. Procedures used for water extraction of orange pulp in some commercial plants during processing of the 1958-59 late season pack of frozen orange concentrate

| Plant number | Code used in tables[1] | Extraction process | Solubl solids - ° Brix: In pulp: Before extraction | After extraction | In water extract |
|---|---|---|---|---|---|
| 1 | CF | Countercurrent flow through finishers | 10.4 | 4.4 | Footnote[2] |
| 2 | CFC* | Countercurrent flow through finishers and extract centrifuged | 11.0 | 3.4 | 6.1 |
| 3 | CFC* | Countercurrent flow through finishers and extract centrifuged | 11.2 | 3.6 | 6.2 |
| 4 | CFRC* | Countercurrent flow through finishers, extracts recycled and final extract centrifuged | 11.0 | 3.6 | 7.2 |
| 5 | CFR | Countercurrent flow through finishers and extract recycled | 10.4 | 5.2 | 8.0 |
| 6 | CS | Countercurrent flow through rotating screen | 10.8 | 7.4 | 7.3 |
| 7 | SFR | Rotating screen and finisher used in double juice finishing operation; water added after rotating screen and extract recycled | 10.8 | 5.8 | 7.0 |
| 8 | WDF | Water added after first finisher in double juice finishing operation | 11.0 | 8.2 | 6.2 |
| 9 | NO | Not observed | Footnote[2] | 3.6 | 7.4 |

[1] C = countercurrent flow of water and pulp; and C* = centrifuge used.

[2] Sample not obtained.

of the other three finishers with the final extract being discharged from the first finisher.

Juice from a Food Machinery In-Line extractor was passed through a screw finisher and the discharged pulp fed to the pulp extractor unit. Results showed that the Brix value of orange pulp was reduced from 11.7° to 3.1° when pulp was extracted as it passed through the four finishers. The extract increased from 1.6° to 7.2° Brix. The time required for the pulp to pass through the unit was approximately three minutes. The percentage of fruit solids recovered from the pulp also increased (4) as a larger number of finishers were used in the extraction process.

The effect of the water/pulp ratio used on the recovery of soluble solids was also determined. As this ratio was increased from 0.50 to 2.00, the percentage of soluble solids recovered increased from 51 to 81 per cent. In commercial operations it is desirable to keep this ratio as low as possible to avoid the evaporation of excessive amounts of water. Countercurrent flow of extract and pulp through processing equipment and recycling of extract are used to increase the soluble solids in the extract so that the water/pulp ratio may be maintained as low as possible.

*Soluble Solids in Orange Pulp and Water Extracts.*—Presented in Table 1 are the Brix values of pulps from midseason oranges, both before and after water extraction. The soluble solids in the pulp after extraction ranged from 2.4° to 8.2° Brix; these values show the relative efficiencies of the various extraction processes for the recovery of soluble solids from the pulp. Two of the lower Brix values were found in the discharged pulps from plants 2 and 3 where the process consisted of a countercurrent flow through a series of finishers; the third lower value was in pulp from plant 5 where, in addition to this process, recycling of the extract was used. Centrifuges were also employed in these three plants. Data are given in Table 2 for pulps from late season oranges and show a range from 3.4° to 8.2° Brix in these pulps after extraction. Three of the lower Brix values were found in the discharged pulps from plants 2, 3, and 4 where the process consisted, as previously, of a countercurrent flow through a series of finishers and the use of a centrifuge; also, in plant 4 the extract was recycled. The highest Brix value was in the extracted pulp from plant 8, where water was added after the first finisher in a double finishing operation; this was the same as when pulps from midseason oranges were extracted as shown by data in Table 1.

The Brix values of the midseason water extracts, as shown in Table 1, ranged from 4.0° to 8.5°. However, 6 of the 9 samples

Table 3. Some characteristics of water extracts of orange pulp obtained from nine commercial plants during the processing of the 1958-59 midseason and late season packs of frozen orange concentrate

| Plant number | Pulp extraction process | | Brix value | | Acid as citric % | | | | Ascorbic acid mg/100 ml | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | Initial | | 12° Brix basis | | Initial | | 12° Brix basis | |
| | Mid | Late | Mid | Late | Mid | Late | Mid | Late | Mid | Late | Mid | Late |
| 1 | CF | - - | 4.0 | - - | 0.36 | - - | 1.08 | - - | 19 | - - | 59 | - - |
| 2 | CF | CFC* | 7.5 | 6.1 | 0.48 | 0.32 | 0.77 | 0.63 | 35 | 21 | 57 | 42 |
| 3 | CFC* | CFC* | 4.9 | 6.2 | 0.32 | 0.38 | 0.78 | 0.74 | 23 | 21 | 58 | 42 |
| 4 | CFRC* | CFRC* | 7.7 | 7.2 | 0.48 | 0.45 | 0.75 | 0.75 | 37 | 22 | 59 | 37 |
| 5 | CFRC* | CFR | 7.5 | 8.0 | 0.56 | 0.47 | 0.90 | 0.71 | 32 | 25 | 52 | 38 |
| 6 | CS | CS | 8.6 | 7.3 | 0.56 | 0.39 | 0.78 | 0.64 | 34 | 27 | 48 | 44 |
| 7 | SFR | SFR | 6.5 | 7.0 | 0.40 | 0.38 | 0.74 | 0.65 | 32 | 23 | 61 | 40 |
| 8 | WDF | WDF | 7.9 | 6.2 | 0.54 | 0.37 | 0.82 | 0.71 | 37 | 23 | 57 | 46 |
| 9 | NOC* | NO | 7.7 | 7.4 | 0.47 | 0.41 | 0.73 | 0.67 | 34 | 21 | 53 | 35 |
| | Minimum | | 4.0 | 6.1 | 0.32 | 0.32 | 0.73 | 0.63 | 19 | 21 | 48 | 35 |
| | Maximum | | 8.6 | 8.0 | 0.56 | 0.47 | 1.08 | 0.75 | 37 | 27 | 61 | 46 |

were in the range of 6.5° to 7.9° Brix indicating the desirability of obtaining as much soluble solids as possible in the water extracts, so as to eliminate evaporating excessive amounts of water. Processes used in plants 2, 5, and 9 resulted in the desirable combination of a high Brix value in the extract and a low Brix value in the extracted pulp. The late season water extracts of orange pulp had Brix values which ranged from 6.1° to 8.0°, as indicated in Table 2, and a high Brix value in the extract together with a low value in the extracted pulp were found in samples from plants 2, 3, 4, and 9.

*Chemical and Physical Characteristics of Water Extracts of Orange Pulp.*—Results reported in the tables in this series of papers show various characteristics of water extracts of orange pulp, and wherever possible, analytical values have been calculated to a 12° Brix basis for comparison purposes; minimum and maximum values are also indicated. Data obtained from the examination of the orange juice samples are also listed. A comparison of these values with those of the water extracts, calculated to 12° Brix basis, shows the relative amounts of substances, such as ascorbic acid and others, in these products. If the quantity of any substance is shown to be higher in the 12° Brix extract than that in a 12° Brix orange juice, then the amount of this substance in orange concentrate made from juice and extract will be slightly higher than that in concentrate made only from the juice.

A comparison of some characteristics in the aqueous extracts of orange pulp from midseason and late season fruit may be made by considering the data given in Tables 3 and 4. It should be mentioned that the Brix values, total acid, Brix/acid ratio, and pH of citrus juices are chiefly dependent upon the maturity of the fruit and ascorbic acid content varies in different citrus varieties. On a 12° Brix basis, the acid in the late season water extracts was about the same or slightly lower than that found in the midseason extracts and this resulted in a greater Brix/acid ratio in most of the late season extracts; however, the pH was slightly lower in the

Table 4. Some characteristics of water extracts of orange pulp obtained from nine commercial plants during the processing of the 1958-59 midseason and late season packs of frozen orange concentrate

| Plant number | Pulp extraction process | | Brix/acid ratio | | pH | | Relative viscosity[1] | |
|---|---|---|---|---|---|---|---|---|
| | Mid | Late | Mid | Late | Mid | Late | Mid | Late |
| 1 | CF | - - | 11.1 | - - | 3.6 | - - | 2.58 | - - |
| 2 | CF | CFC* | 15.6 | 19.0 | 4.1 | 4.0 | 4.39 | 4.97 |
| 3 | CFC* | CFC* | 15.3 | 16.3 | 4.0 | 3.6 | 2.16 | 2.18 |
| 4 | CFRC* | CFRC* | 16.0 | 16.0 | 4.0 | 3.9 | 13.78 | 5.55 |
| 5 | CFRC* | CFR | 13.4 | 17.0 | 3.9 | 3.8 | 23.90 | 30.40 |
| 6 | CS | CS | 15.4 | 18.7 | 3.9 | 4.0 | 23.00 | 1.68 |
| 7 | SFR | SFR | 16.3 | 18.4 | 4.0 | 3.9 | 18.33 | 4.00 |
| 8 | WDF | WDF | 14.6 | 16.8 | 4.0 | 3.9 | 2.98 | 3.34 |
| 9 | NOC* | NO | 16.4 | 18.0 | 3.9 | 3.6 | 3.99 | 2.00 |
| | Minimum | | 11.1 | 16.0 | 3.6 | 3.6 | 2.16 | 1.68 |
| | Maximum | | 16.4 | 19.0 | 4.1 | 4.0 | 23.90 | 30.40 |

[1] Relative viscosity of centrifuged extract determined at 26°C using an Ostwald pipette.

late season extracts than that in the midseason samples. As expected, the ascorbic acid was lower in the late season extracts than in those produced when midseason fruit was used. The relative viscosity of 4 midseason water extracts ranged from 13.78 to 23.90 and that of the other 5 samples were in a range of 2.16 to 4.39. Only 1 of the late season extracts had a very high viscosity of 30.40, while the other 7 samples fell within the range of 1.68 to 5.55.

Variations in the extraction procedures in the different plants, as described in Tables 1 and 2, are believed to have little effect upon characteristics of the aqueous extracts, such as total acid and ascorbic acid, provided comparisons are made on a 12° Brix basis. However, variations in the water/pulp ratio used by different plants resulted in a wide range of initial values in the extracts for such characteristics as total acid, Brix values, and others. From data shown in Table 4, it is apparent that the extracts having the very high relative viscosities were obtained from plants using recycling procedures. Such procedures resulted in extraction from the orange pulp of larger amounts of water-soluble pectin, as shown in Table 3 of Part II in this series of papers, than that extracted from pulp in the plants where the extracts were not recycled; also in plants 4, 6, and 7 more water-soluble pectin was extracted from the pulp of midseason oranges than from pulp when late season fruit was used.

Data presented in Table 5 indicate that the use of centrifuges in 4 plants to remove pulp and water-insoluble solids from water extracts of orange pulp had little effect upon the Brix value, acid, pH, or ascorbic acid in these products. The causes of the large increase in the relative viscosity of the extract from plant 4 after centrifugation and the presence of an objectionable off-flavor in this sample will be discussed later.

Comparison of data in Table 6 for water extracts of orange pulp with that for the orange juices shows that the percentage of acid was less in the extracts than in the juices, which generally resulted in higher values for both Brix/acid ratio and pH in the extracts. Only slight differences in ascorbic acid were found between the juices and the extracts on a 12° Brix basis, and the amounts of ascorbic acid in both the extracts and juices were in the same range of 35 to 46 mg/100 ml when late season fruit was used. The relative viscosity of the water extracts, except 5 having high values, was in the range of 1.68 to 5.55, as compared to 1.45 to 2.62 for the centrifuged orange juices from both midseason and late season fruit.

Table 5. Effect of centrifugation for removal of pulp on some characteristics of water extracts of orange pulp during the processing of the 1958-59 midseason and late season packs of frozen orange concentrate

| Characteristics | Season | Plant No. 2 CFC* | | Plant No. 3 CFC* | | Plant No. 4 CFRC* | | Plant No. 9 NOC* | |
|---|---|---|---|---|---|---|---|---|---|
| | | Values before and after centrifugation | | | | | | | |
| | | Before | After | Before | After | Before | After | Before | After |
| Brix value | Mid | - - | - - | 5.2° | 4.9° | 9.0° | 7.7° | 7.8° | 7.7° |
| | Late | 6.2 | 6.1 | 6.2 | 6.2 | 7.0 | 7.2 | - - | - - |
| Acid as citric - % | Mid | - - | - - | 0.35 | 0.32 | 0.57 | 0.48 | 0.47 | 0.47 |
| | Late | 0.34 | 0.32 | 0.38 | 0.39 | 0.45 | 0.45 | - - | - - |
| Brix/acid ratio | Mid | - - | - - | 14.9 | 15.3 | 15.8 | 16.0 | 16.6 | 16.4 |
| | Late | 18.2 | 19.1 | 16.3 | 16.3 | 15.6 | 16.0 | - - | - - |
| pH | Mid | - - | - - | 3.9 | 4.0 | 3.9 | 4.0 | 3.9 | 3.9 |
| | Late | 3.9 | 4.0 | 3.6 | 3.6 | 3.9 | 3.9 | - - | - - |
| Ascorbic acid - mg/100 ml | Mid | - - | - - | 24 | 23 | 41 | 37 | 35 | 34 |
| | Late | 22 | 21 | 20 | 21 | 22 | 22 | - - | - - |
| Relative viscosity[1] | Mid | - - | - - | 2.23 | 2.16 | 3.25 | 13.78 | 4.26 | 3.99 |
| | Late | 3.92 | 4.97 | 2.10 | 2.18 | 3.62 | 5.55 | - - | - - |
| Objectionable off-flavor | Mid | - - | - - | No | No | Yes | Yes | No | Yes |
| | Late | No | No | No | No | No | No | - - | - - |

[1] Relative viscosity of centrifuged extract determined at 26°C using an Ostwald pipette.

Table 6. Minimum and maximum values for some characteristics in commercial samples of orange juices and water extracts of orange pulp produced from midseason and late season fruit

| Characteristics | Water extracts of orange pulp | | | | Orange juices | | | |
|---|---|---|---|---|---|---|---|---|
| | Midseason | | Late season | | Midseason | | Late season | |
| | Min. | Max. | Min. | Max. | Min. | Max. | Min. | Max. |
| Brix value | 4.0° | 8.6° | 6.1° | 8.0° | 11.2° | 13.6° | 10.7° | 12.4° |
| Acid as citric - % | 0.32 (0.73)[1] | 0.56 (1.08)[1] | 0.32 (0.63)[1] | 0.47 (0.75)[1] | 0.83 | 1.13 | 0.69 | 0.90 |
| Brix/acid ratio | 11.1 | 16.4 | 16.0 | 19.0 | 11.5 | 15.1 | 13.1 | 15.5 |
| Ascorbic acid - mg/100 ml | 19 (48)[1] | 37 (61)[1] | 21 (35)[1] | 27 (46)[1] | 49 | 67 | 35 | 46 |
| pH | 3.6 | 4.1 | 3.6 | 4.0 | 3.7 | 3.9 | 3.4 | 3.8 |
| Relative viscosity[2] | 2.16 | 23.90 | 1.68 | 30.40 | 1.73 | 2.62 | 1.45 | 2.23 |

[1] Values in parentheses calculated to 12° Brix basis for comparison purposes.

[2] Relative viscosity of centrifuged extract or juice determined at 26°C using an Ostwald pipette.

*Color and Flavor in Water Extracts of Orange Pulp.*—All of the water extracts had a slight milky appearance and a slight yellow or greenish-yellow color. Extracts which had been recycled during the extraction process were poorer in appearance and color.

Very little orange aroma and flavor were detectable in any of the aqueous extracts of orange pulp. A peel aroma of moderate intensity was evident in all samples, similar to that often noticed in commercial extracted orange juice; this type of aroma is removed when juice is concentrated. Some astringency, varying in intensity from slight to moderate, was found in all of the extracts and slight bitterness was noticed in only 1 of the midseason extracts. Objectionable off-flavors were found in 3 of the midseason extracts as indicated in Table 1 in Part III of this series of papers.


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## II. PECTIC SUBSTANCES AND RELATED CHARACTERISTICS IN AQUEOUS EXTRACTS OF ORANGE PULP[1]


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Pectic substances in orange concentrate, such as water-soluble pectin and pectinesterase, are important because they determine to a great extent the degree of gelation which may occur in this product, as well as the amount and stability of the cloud in the reconstituted juice. Therefore, it was essential that data be obtained concerning these pectic and other related substances which are removed from orange pulp by water extraction. The characteristics in orange juices and aqueous extracts of orange pulp which were determined and are discussed in this paper were pulp content, water-insoluble solids, pectinesterase activity, cloud, and the water-, ammonium oxalate-, and sodium hydroxide-soluble pectic fractions. Flavonoids, as hesperidin, were also


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