Table 4. Ripening of tomatoes after 4 days at 21°C as influenced by cultivar, mulching, and postharvest practices.

	Percent of green tomatoes								
	Walter cultivar			Homestead cultivar			Total[z]		
Postharvest treatment	Mulch	Uncovered	Avg.	Mulch	Uncovered	Avg.	Mulch	Uncovered	Avg.
Washed	49.5	65.2	56.3	45.9	68.7	56.3	57.6	67.0	57.3
Washed, waxed	52.0	65.5	58.7	42.7	65.2	54.0	57.3	65.4	57.4
Washed, waxed, ethylene	17.0	44.5	30.7	12.7	19.6	16.5	15.2	32.2	22.7
Washed, ethylene	21.5	40.5	30.5	12.7	24.5	18.6	17.2	33.0	25.1
Average	35.0	54.2	44.0	28.6	44.5	36.6	36.8	49.3	

[z]Average of Walter and Homestead cultivars.

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VISCOMETRY AFFECTED BY PECTIC CONSTITUENTS IN 45° BRIX FROZEN CONCENTRATED ORANGE JUICE[1]

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Abstract. **Three viscometers were investigated for their practicability in measuring consistency of non-Newtonian frozen concentrated orange juice at the 45° Brix level. Viscosity measurements by the tube viscometer and Brabender Visco-Corder were highly correlated ($r^2 = 0.91$). Viscosity measurements by the Brookfield LVT viscometer were not as highly correlated to either the tube ($r^2 = 0.80$) or to the Brabender ($r^2 = 0.79$). The pecentage of apparent viscosity retained as measured by the Brabender at different shear rates indicated that several of the orange concentrates approached Newtonian fluids. Pectic fractions, total pectins, and water-insoluble solids were determined on samples to establish consistency correlations.**

Average values for the percentage of total pectin soluble in water, ammonium oxalate, and sodium hydroxide, representative of 200 commercial samples, were 46.9, 12.2, and 40.9%, respectively. Water-insoluble solids ranged from 466 to 838 mg/100 g of concentrate.

Consistency or viscosity measurements of frozen concentrated orange juice (FCOJ) are important to the designer of mechanical equipment in citrus processing plants and are of value to the technologist as a quality factor that must be kept under control. Most liquid type foods have a non-Newtonian behavior and FCOJ falls into this category because of its heterogeneous mixtures of water-insoluble solids (suspended solids) and the colloidal pectic substances. Fruit puree and FCOJ usually exhibit pseudoplastic properties (4, 11) because their apparent viscosity decreases as the shear-rate increases.

Flow behavior properties of citrus juices and concentrates have been studied using various inexpensive equipment ranging from pipettes to expensive cone-plate viscometers (2, 4, 7, 11). Three types of viscometers have been used at this Center for measuring the resistance of citrus juices and concentrates to flow. Ezell (3) meas-

Florida Agricultural Experiment Stations, Journal Series No. 5126.

[1]Cooperative research of the University of Florida, IFAS, Agricultural Research and Education Center, Lake Alfred and State of Florida Department of Citrus. Appreciation is expressed to the Polk Community College Data Processing Center for use of their computer facilities.

ured apparent viscosity of concentrated orange and grapefruit juices with a Brookfield LVF viscometer and relative viscosity with a 200 ml pipette. A more sophisticated instrument than the pipette is the tube viscometer (10). This is a set of stainless steel tubes with different size bores and tube lengths to assure laminar flow. The viscometer tube is connected to a cup or reservoir that is kept filled with the sample to maintain a constant flow rate. Huggart et al. (5) obtained consistency measurements of citrus concentrates with a Brabender Visco-Corder paddle-cup type viscometer. Consistency measurements obtained by this instrument at various shear rates produce a simple method of characterizing the flow behavior properties of orange concentrate.

The purposes of this study were to correlate viscosity measurements by these 3 viscometers and to relate the effect of pectic constituents and water-insoluble solids on the apparent viscosities using samples of FCOJ.

Materials and Methods

Concentrates were 200 commercial samples of 45° Brix FCOJ collected from 22 Florida processing plants during the 1971-72 citrus season.

Apparent viscosity data were determined by means of the Brabender Visco-Corder (5), tube viscometer (10), and Brookfield Synchro-Lectric viscometer Model LVT (3). All measurements on the concentrates were made at 78°F and expressed as apparent viscosity in centipoises. Apparent viscosity data were calculated using formulas in the above references.

Measurements by the Brabender Visco-Corder in Brabender units were made at 50 and 200 rpm with sensitivity cartridge range of 125 centimeter-grams.

The average flow rate determined from 3 separate measurements (g/sec) on each FCOJ sample with the tube viscometer (1/8 inch I. D. bore and 12.5 inch length) was used for calculating the apparent viscosity.

Most measurements (195 of 200 samples) by the Brookfield viscometer were made at 30 rpm with spindle No. 2. Readings of the other 5 samples were made either with spindle No. 2 at 12 rpm or with spindle No. 3 at 30 rpm in order to obtain a dial reading.

Pectins and water-insoluble solids were determined (8, 9) on 18 composite and 3 individual samples, representative of the 200 FCOJ for mid- and late season packs. The range into which these composite samples belonged (Table 2) was determined by its apparent viscosity as measured by the tube viscometer. Four additional composites were prepared from mid- and late season packs from 2 processing plants, one plant represented FCOJ samples with low consistency measurements and the other with high consistency measurements.

Statistical Analysis. Simple (r) or multiple (R) correlations were determined between apparent viscosities by different viscometers. The various chemical determinations on the FCOJ samples were considered as independent variables and viscosity as the dependent variable in simple and linear regression analysis (1). The analyses of the data were by an automatic stepwise multiple linear regression program (NCR Century Statistical Analysis System) that calculated a simple correlation matrix before developing the multiple regression equations. Viscosities by the 3 instruments were compared separately to the chemical data. The Brabender 200 rpm/50 rpm viscosity ratio (% retention) was also run as a dependent variable. The partial F for inclusion and deletion was set at 2.5. Transformations (squares and cubic functions) of independent variables were included in the statistical analyses.

Results and Discussion

Arbitrary instruments such as consistometers, pipettes, etc. for measuring viscosity of non-Newtonian fluids are simple both in construction and use, but give results in arbitrary units applicable only to the particular instrument. The minimum and maximum apparent viscosity values obtained by the 3 instruments for 200 FCOJ samples are shown in Table 1. Values obtained by the Brookfield were always numerically higher than those values found by the Brabender which in turn were greater than the values found by the tube viscometer. The greatest numerical difference between the low and high values was found with the Brookfield viscometer because the spindles and shear rates (rpm) were changed for 5 samples to obtain readings on the instrument's scale.

The frequency distribution of viscosity data obtained from 200 FCOJ samples is shown in Table 2; apparent viscosity was determined using the tube viscometer. Midseason was represented with 79 samples and late season with 121 samples of FCOJ. The frequency distribution of

Table 1. Apparent viscosity range for FCOJ samples in centipoises as measured by 3 viscometers.

Instruments	Min.	Max.	Av.
Tube	21	256	70
Brabender	120	565	286
Brookfield	149	2,173	523

samples according to tube viscometry showed that 96% of the mid- and 100% of the late season samples were under 150 centipoises. The 200 samples measured by the Brookfield instrument, not shown in tabular form, revealed that 98% were under 1000 centipoises at 78°F. In 1956-57 Ezell (3) reported that only 69% of frozen concentrated orange juice (42° Brix) samples examined fell under 1000 centipoises when measured by the Brookfield at 86° F. Improved processing procedures that mainly include uniform and sufficient heat-treatment for the inactivation of the pectic enzyme and the lower pulp levels of the juice today are responsible for this lower apparent viscosity in FCOJ.

Water-soluble and total pectin of those samples falling within the designated *centipoise* range as determined by the tube are shown in Table 2. Generally, the apparent viscosity increased as H_2O-soluble and total pectins increased. Similar quantities of total pectin were found for midseason (292-471 mg) and late season (309-459 mg) samples (Table 2).

The minimum and maximum values for the pectic fractions and water-insoluble solids in the samples are shown in Table 3. Samples with higher apparent viscosities in the midseason composites (100 centipoises and up) contained greater quantities of H_2O-soluble pectin (188-237 mg) than did the late season (181-193 mg) ones, but slightly greater NaOH-soluble pectin was found in the late season samples as indicated by the maximum value (223 mg) in Table 3. Very little differences were found for the $(NH_4)_2C_2O_4$-soluble pectin in samples for either mid- or late season. The average whole season values for the percentage of total pectin soluble in water, ammonium oxalate, and sodium hydroxide were 46.9, 12.2 and 40.9%, respectively. Corresponding values reported for the 1953-54 citrus season (8) were 37.9, 22.7, and 39.4% with the larger differences in the percentages of the water- and ammonium oxalate-soluble fractions. Heat treatment used in processing FCOJ today protects the

Table 2. Frequency distribution of apparent viscosities and corresponding pectins for 79 midseason and 121 late season samples of 45° Brix FCOJ obtained during the 1971-72 citrus season. (Pectins expressed in mg/100 g conc).

Tube	Midseason			Late season		
viscosity cps	sample %	H2O-sol. pectin	total pectin	sample %	H2O-sol. pectin	total pectin
21- 39	2.5	133	292	23.1	152	309
40- 49	10.1	159	318	19.8	164	337
50- 59	12.7	176	347	18.2	152	335
60- 69	12.7	155	350	12.4	164	350
70- 79	16.4	165	378	5.8	159	367
80- 89	7.5	181	373	7.5	179	380
90- 99	6.3	184	384	4.1	191	428
100-119	15.2	188	404	5.8	193	431
120-149	12.7	199	425	3.3	181	459
183	1.3	205	422	none	none	none
236	1.3	237	457	none	none	none
256	1.3	229	471	none	none	none

Table 3. Minimum and maximum values for the pectic fractions and water-insoluble solids found in FCOJ samples.

Chemical constituents	Midseason min.	Midseason max.	Midseason av.	Late season min.	Late season max.	Late season av.
H_2O-insol. solids-mg/100g	466	838	653	584	696	644
H_2O-sol. pectin-mg/100g	133	237	184	152	193	171
$(NH_4)_2C_2O_4$-sol. pectin-mg/100g	35	59	46	36	62	47
NaOH-sol. pectin-mg/100g	120	184	154	116	223	160
Total pectin-mg/100g	292	471	385	309	459	377

H_2O-soluble pectin from deesterification caused by pectinesterase, thus preventing this pectic fraction from conversion to insoluble pectinates and pectates. Partially and completely demethylated pectins readily combine with the calcium in citrus juice to form calcium salts of pectinic and pectic acids producing a highly viscous concentrate.

Although the average amounts of water-insoluble solids for both mid- and late season samples (653 and 644 mg) were approximately the same, the difference in spread between the low and high values for this component was much greater in the midseason (466-838 mg) samples than in the late season (584-696 mg) samples.

The Brabender is very useful for determining the percentage apparent viscosity retained (AVR) (5), an indication of the flow behavior properties of an individual sample. Apparent viscosities of a sample are measured at 2 shear rates. The viscosity measured at 200 rpm is divided by the viscosity measured at 50 rpm to give the AVR. A quotient of 1.00 indicates a Newtonian liquid, retaining the same viscosity or consistency at a shear rate of 200 as that found at 50 rpm. The AVR in the FCOJ samples range from 50.8 to 93.6% (Fig. 1) and were plotted against total pectin. Generally, those samples with the higher percentage AVR also contained the lesser amounts of H_2O-soluble and total pectin.

Table 4. Simple correlations between 3 viscometers and of viscometers to several chemical constituents of commercial FCOJ samples.

	Tube 1/8-inch dia.	Brabender 50 rpm	Brabender 200 rpm	Brookfield[z] sp #2 @ 30 rpm
	r[y]	r	r	r
Viscosities				
Tube	-	0.97	0.96	0.90
Brabender 50 rpm		-	0.98	0.88
Brabender 200 rpm			-	0.89
Chemical constituents				
H_2O-insol. solids	0.82	0.82	0.82	0.80
H_2O-sol. pectin	0.93	0.93	0.94	0.83
$(NH_4)_2C_2O_4$-sol. pectin	0.66	0.70	0.73	0.56
NaOH-sol. pectin	0.51	0.58	0.63	0.39
Total pectin	0.85	0.89	0.92	0.72

[z]Viscosities of 195 of the 200 FCOJ samples were measured with spindle #2 at 30 rpm.

[y]Required r's for significance 1% level = 0.50
df = 24 5% level = 0.39.

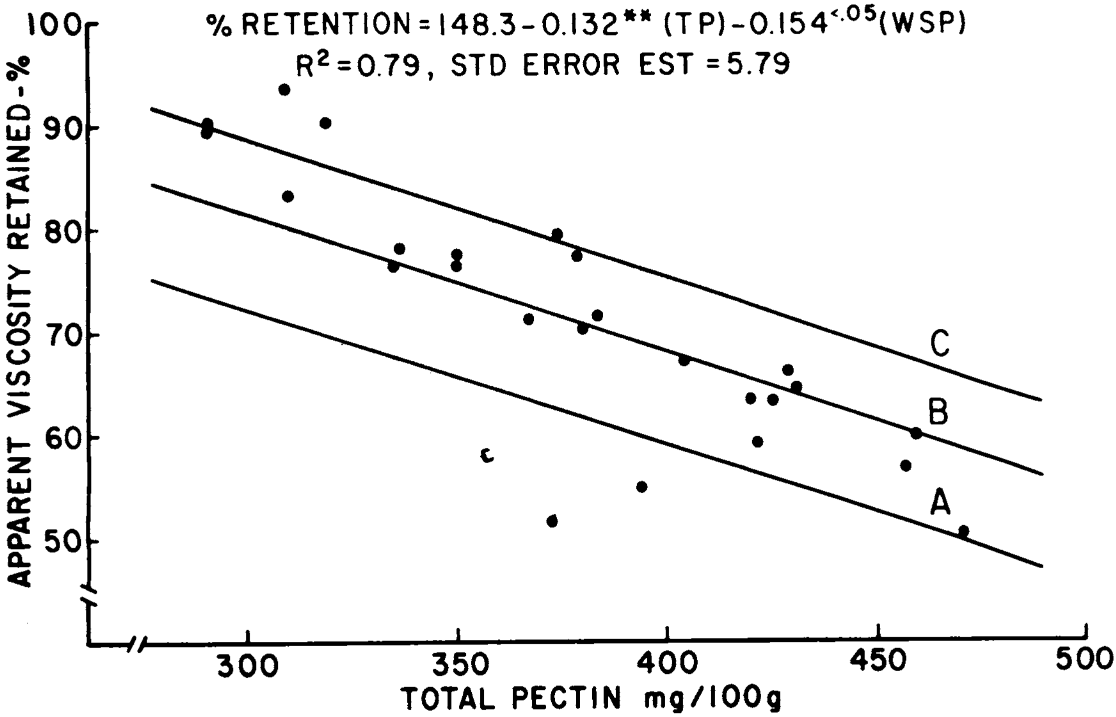

Fig. 1. Relation of the percentage of apparent viscosity retention to total pectin in prepared composites from FCOJ measured by the Brabender. The predictive equation lines are shown for % retention based on total pectin (TP) and A [high (237 mg)], B [medium (177 mg)], and C [low (130 mg)] H_2O-soluble pectin (WSP). The singular correlations of total pectin and H_2O-soluble pectin to % retention were 0.88 and 0.85, respectively. Slope coefficients in multiple regression were significant at the 1 and 10% levels, respectively for total pectin and H_2O-soluble pectin.

Simple correlations. The correlations between viscometers were as good or exceeded acceptable levels of Kramer and Twigg (6) with the tube and Brabender instruments giving the most closely related readings for viscosity (Table 4). The chemical determinations that were highly correlated to viscosity were water-soluble pectin, total pectin, and water-insoluble solids (Table 4). All of the chemical determinations were significantly correlated at the 5% level to the viscosity readings of all 3 viscometers.

Multiple correlations. Stepwise multiple regression developed statistically significant equations relating the chemical determinations to viscosity as measured by the 3 viscometers. The coefficients of determination (R^2) for these correlations were as follows: Tube = 0.96, Brabender at 50 rpm = 0.94, Brabender at 200 rpm = 0.94, and Brookfield = 0.77. When the viscosity data range for each viscometer was divided into 3 equal parts (low, medium, and high viscosity values), the errors in predicting viscosity compared to measured viscosity were more uniform and smaller for the Brabender and tube viscometers than for the Brookfield instrument (Table 5). The equations for predicting viscosity that yielded the lowest prediction errors (Tube and Brabender at 200 rpm) are presented below.

1. Viscosity (Tube) = 198.65 + 0.75** (NaOH-sol. pectin)^3 + 3.95** (H_2O-sol. pectin)^3 — 2.07** (H_2O-sol. pectin), Std. Error Est. = 13.06.
2. Viscosity (Brabender 200 rpm) = — 170.57 + 0.0094** (H_2O-sol. pectin)^2 + 1.25** (NaOH-sol. pectin), Std. Error Est. = 29.11.

The other 2 predictive equations had the following R^2 and Std. Error Est.: Brabender 50 rpm, R^2=0.94, Std. Error Est.= 68.99; Brookfield, R^2=0.77, Std. Error Est.=254.5.

These data indicate that viscosity of FCOJ samples could be predicted within about 10% error from the NaOH- and H_2O-soluble pectin determinations. The equations also suggest that

Table 5. Av. errors in predicting FCOJ viscosities from pectin and water-insol. solids levels for 3 viscometers.

Vis. range	Tube	Brabender 50	Brabender 200	Brookfield
	% errors[z]			
Low[y]	8.2	11.0	8.4	27.4
Med.	7.1	9.3	6.5	16.9
High	10.9	7.9	6.2	21.1

[z]% error from observed readings.

[y]Viscosity readings divided into 3 equal parts of total range.

these 2 pectic fractions are probably very important in determining the viscosity of orange juice concentrate.

The multiple correlation program was also run for the rates of the AVR values against the chemical constituents and a multiple R^2 of 0.79 was obtained. The regression equation for this relationship and the graphical representations are shown in Fig. 1. The percentage apparent viscosity retained (AVR) data were plotted against total pectins (Fig. 1) but the regression equation lines are shown for low, medium and high H_2O-soluble pectin levels (Fig. 1). Apparently the pseudoplastic characteristic of FCOJ in respect to viscosity is related to increased total pectin and possibly more specifically to H_2O-soluble pectin.

Conclusions

1. Viscometer to viscometer correlations were high, particularly Tube and Brabender.

2. Most of the simple correlations of chemical constitutents to viscosity were high, particularly water-soluble pectin, total pectin, and water-insoluble solids.

3. Multiple correlation equations predicting viscosity of 45° Brix FCOJ gave the following results: Tube viscometer, $R^2 = 0.96$, std. error est. $= 13.06$; Brabender at 50 rpm, $R^2 = 0.94$, std. error est. $= 68.99$; Brabender at 200 rpm, $R^2 = 0.94$, std. error est. $= 29.11$; Brookfield, $R^2 = 0.77$, std. error est. $= 254.5$.

a. Percent prediction error

$$\frac{\text{Y observed-Y predicted}}{\text{Y observed}}$$

least for Brabender at 200 rpm.

b. The 2 variables entered most in the multiple equations were H_2O-soluble and NaOH-soluble pectins; the only other variable entered was water-insoluble solids in the Brookfield equation.

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