stand the low pH found in citrus waste and when this is found, automatic pH control will probably be necesary to prevent killing the cover crop during clean up periods. This same control is necessary with all the other methods likewise. The use of a different type of pipe joint or the use of a smaller pump permitting longer periods of operation would eliminate or reduce the nuisance of ponding of waste at each pipe joint. The use of easily moved pipe is, however, necessary since any area will eventually become saturated.

Additional study of all these methods of waste treatment is needed and efforts are continuing to find the funds required for such a study.

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NOTES ON FACTORS ASSOCIATED WITH GELATION IN FROZEN CONCENTRATED ORANGE JUICE

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Frozen orange concentrate has been observed to gel when stored at temperatures higher than 0° F. (−17.8° C.). This phenomenon has been studied by a number of investigators (4, 9, 11, 12) and they generally agree that the basic factors involved are the pectin concentration, sugar content, pectinesterase activity, pH, and divalent ion content. In the preparation of the concentrate, the sugar content and pH are quite uniform. Baker and Goodwin (2) observed that pectin gels will form over a wide range of pH values. These include the pH range of 42° Brix commercial orange concentrate. Based on the results of Roberts and Gaddum (10), Wenzel, Moore, Rouse, and Atkins (12) came to the conclusion that citrus juices contain more than the optimum amount of calcium necessary for the formation of enzyme-demethylated pectin gels.

Recent investigations by Atkins, Rouse, Huggart, Moore, and Wenzel (1) have shown that heating the juice to inactivate the enzyme will stabilize the concentrate with respect to gelation.

Rouse (11) has shown that the loss of cloud and gelation in citrus juice are related to the low methoxyl pectin produced by the action of pectinesterase on pectin. Baker and Goodwin (2) have found that as the methoxyl content decreased from 7.1% to 4.5%, the amount of pectin required for gel formation in a given sugar solution also decreased. At 4.5% methoxyl content, the minimum amount of pectin was required. Other factors such as molecular weight of the pectin, the extent of esterification, and the distribution of the ester groups on the pectinic acid molecule have been listed by Kertesz (5) as important factors in the gel forming power of low methoxyl pectin.

The purpose of this study is to explore the relationship between pectinesterase activity and pectin content in commercial frozen orange concentrates. The observations made from the data obtained in the preliminary study are tentative, since the number of samples and analyses that are necessary in this type of study are insufficient to draw positive conclusions.

EXPERIMENTAL METHODS

During the 1950-51 Valencia season samples of commercial concentrate were obtained from

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processing plants of the four largest packers. These samples were stored for eight months at different temperatures and examined for "total" and "soluble" pectin, and "total" and "soluble" calcium and magnesium, pectinesterase activity, and degree of gelation.

"Soluble" pectin is defined as the pectin in solution in the juice serum and was obtained from the juice passing through No. 1 Whatman filter paper. "Total" pectin was extracted by essentially the same method as that described by McCready and McComb (7) except a 0.3% Versene solution was used instead of 0.5%. The pulp was not comminuted. "Soluble" calcium and magnesium were determined by filtering the juice and ashing the filtrate. Calcium was estimated in the ash by the oxalate-permanganate method and magnesium as magnesium pyro-phosphate. "Total" calcium and magnesium were determined on the ash of the whole juices.

During the 1951-52 season, another series of samples were collected. These were obtained from six different commercial processing plants. Samples were obtained from each plant at the beginning of the Valencia season and another sample at the end of the season. Only samples which had not been stabilized by heating were considered. During this season a total of 12 samples were obtained and examined for "water-soluble," "oxalate-soluble," and "alkali-soluble" pectin, degree of gelation, and pectinesterase activity.

The concentrates were examined for gelation after storage for 96 hours at 35° F. (1.7° C.). The degree of gelation was expressed by designations proposed by Olsen, Huggart and Asbell (9).

The pectinesterase activity was determined after the concentrate had been reconstituted to 12° Brix by the method of McDonnell, Jansen, and Lineweaver (8).

The methods of analysis of pectic substances used with the 1951-52 samples differed somewhat from those used the preceding year. Pectins were extracted for analysis in three steps: (a) distilled water, (b) 0.2% ammonium oxalate, and (c) 0.05N sodium hydroxide. This is similar to the method described by Dietz and Rouse (3) except that the juice pulp was not broken up in a blendor. This extraction of the pectin with different solutions permitted to some degree a separation and classification of the pectic substances.

The extraction with distilled water removed the high molecular weight pectinic acids that were sufficiently methylated to be rendered soluble in water. This fraction is found largely in the liquid phase of the juice and contributes to its body or viscosity.

The "oxalate-soluble" fraction includes the pectinic acids of sufficiently low methoxyl content to make them insoluble in water. The low ester pectinic acids of this fraction are formed for the most part by the action of pectinesterase on the water-soluble pectins. This fraction is generally considered to be most effective in the formation of the low-ester-pectin-calcium gel in the concentrate. The low concentration of divalent cations that can exist with the oxalate ion render these pectinic substances soluble during the extraction process.

The "alkali-soluble" fraction is ill defined but probably includes the pectic substances not soluble in the previous extractions.

Pectin was determined on the different extracts by the carbazole method of McCready and McCombs (6, 7). The results by this method are expressed as percent anhydrous galacturonic acid. The precision of this method of estimating the galacturonic acid is $\pm$ 2%, while that of the extraction process is about $\pm$ 10%. The limitations of the methods are recognized, but were used because they were relatively rapid and simple.

Results

The results of the examination of the four samples of commercial concentrate for pectin, pectinesterase, degree of gelation, calcium and magnesium are shown in Table 1. Samples are listed in order of their degree of gelation, sample L having the greatest tendency to gel. Table 2 gives the results of the examination of the 12 commercial orange concentrate samples obtained during the 1951-52 season from six different processing plants.

Discussion

In Table 1, there is some correlation between gelation and total pectin and pectinesterase activity. With an increase in "total" pectin and pectinesterase activity there was an increase in tendency to gel. There was no correlation between "soluble" pectin and gelation. "Soluble" pectin decreased when concentrate was stored at temperatures above 0° F. (−17.8° C.), and Table 1 shows that the

Table I

Factors related to gelation of concentrate sampled 1950-51 season.

Examination	Sample Code S	O	H	L
Pectin, pectinesterase, degree of gelation				
Total pectin [c]	0.241	0.249	0.257	0.282
Soluble pectin 0° F. [c] [d]	0.0636	0.0457	0.0748	0.0610
Soluble pectin 10° F. [c] [d]	0.0443	0.0263	0.0360	0.0235
Soluble pectin 20° F. [c] [d]	0.0208	0.0152	0.0208	0.0192
Pectinesterase activity [e]	13.42	16.4	24.97	34.71
Degree of gelation [f]	none	very slight	slight	semi gel
Calcium and magnesium				
Total calcium [c]	0.032	0.0281	0.029	0.029
Soluble calcium 0° F. [c] [d]	0.0272	0.0291	0.0238	0.0270
Soluble calcium 20° F. [c] [d]	0.0260	0.0264	0.0226	0.0232
Total magnesium [c]	0.047	0.040	0.040	0.039
Soluble magnesium [c]	0.044	0.042	0.041	0.034
Loss of soluble pectin during storage at elevated temperature				
Grams lost 10° F. [c] [d]	0.0193	0.0194	0.0388	0.0375
Percentage loss 10° F. [d]	30.4	42.5	51.9	61.5
Grams lost 20° F. [c] [d]	0.0428	0.0305	0.0540	0.0418
Percentage loss 20° F. [d]	67.3	66.8	72.1	68.6
Soluble calcium and pectin loss relationship				
Grams pectin lost 20° F. [c] [d]	0.0428	0.0305	0.0540	0.0418
Grams calcium needed to react with pectin lost	0.0049	0.0061	0.0034	0.0048
Grams calcium lost by analysis	0.0012	0.0027	0.0012	0.0036

[c] Grams per 100 g. of 42° Brix concentrate.
[d] Analyzed after 8 months storage at the constant temperature indicated.
[e] Pectinesterase activity per ml. of concentrate reconstituted to 12° Brix. Expressed as (PE.u.) ml. x 10^4.
[f] Degree of gelation in concentrate after 96 hours storage at 35° F. (1.7° C.).

rate of decrease followed the order of stability. At 10° F. (−12.2° C.) the loss of "soluble" pectin was more rapid in samples that were less stable. At 20° F. (−6.7° C.) storage, the percentage loss of "soluble" pectin was about the same in all samples. "Soluble" calcium decreased with increased storage temperature, and most of the calcium and magnesium in the concentrate was in solution in the serum. The loss of calcium with increase in storage temperature would be expected since the demethylated pectin would react with the calcium to form an insoluble pectinate or pectate. However, the data presented in Table 1 do not show that the loss of calcium is equivalent to the loss of pectin. Magnesium present in the juice will also react with the pectinic or pectic acids to form insoluble pectinates or pectates which may account for the loss of calcium being less than equivalent to the loss of pectin.

The pectinesterase activities given in Table 2 follow the order of tendency to gel except for sample L. It should also be noted that this sample contained more water-soluble, oxalate-soluble, and total pectin than any of the other samples, and these may have compensated for the low enzyme value. However, there does not appear to be any close correlation between the different pectin fractions and the stability of the concentrate towards gelation. In Table 2 the values show that in general there was an increase in tendency to gel with an increase in total pectin. Also when the sum of the water

Table 2

Factors related to gelation of concentrate sampled during 1951-52 season.

Sample code	Degree of gelation f/	Pectin-esterase activity e/	Water-soluble pectin c/	Oxalate-soluble pectin c/	Alkali-soluble pectin c/	Total pectin c/	Water + oxalate-soluble pectin c/
V*	None	22.1	0.073	0.071	0.163	0.307	0.144
T*	None	22.0	0.079	0.058	0.143	0.280	0.137
Z	Very slight	23.7	0.065	0.069	0.126	0.260	0.130
T	" "	23.7	0.096	0.074	0.130	0.300	0.170
Y*	" "	26.6	0.062	0.076	0.166	0.304	0.142
X	Slight	28.0	0.061	0.073	0.160	0.294	0.134
X*	"	30.6	0.074	0.083	0.159	0.314	0.157
V	"	30.3	0.090	0.077	0.157	0.324	0.167
L	Semi gel	24.0	0.102	0.083	0.172	0.357	0.183
LB*	" "	38.2	0.083	0.079	0.190	0.352	0.162
Y	" "	38.6	0.088	0.072	0.178	0.338	0.160
LR*	" "	42.7	0.080	0.071	0.196	0.346	0.151

c/ Grams per 100 g. of 42° Brix concentrate.
e/ Pectinesterase activity per ml. of concentrate reconstituted to 12° Brix. Expressed as (PE.u.) ml. x 10^4.
f/ Degree of gelation in concentrate after 96 hours storage at 35° F. (1.7° C.).
*/ Collected at the end of the 1951-52 Valencia season.

and oxalate-soluble fractions are considered there is an increase in degree of gelation with an increase in these two fractions.

If the data in Table 2 were arranged in order of increasing values for water-soluble plus oxalate-soluble pectin, it is apparent that samples having the same amounts of water-soluble plus oxalate-soluble pectin had less gel stability as the pectinesterase activity increased. A high pectinesterase activity and a low water plus oxalate-soluble pectin fraction was as effective in forming a gel as a high water plus oxalate-soluble fraction with a low pectinesterase activity.

The results obtained indicate in a preliminary way that gelation of frozen orange concentrate under unfavorable storage conditions is related to the pectinesterase enzyme activity and the amounts of the various pectin fractions present, but these variations are small in comparison to normal variations between samples.

SUMMARY

The data presented in this paper are the result of analyses of samples of orange concentrate obtained from citrus processing plants during the 1950-51 and 1951-52 canning seasons. Although data are limited they indicate there is a loss of soluble pectin and divalent ions when the concentrate is stored at temperatures higher than 0° F. (−17.8° C.). The degree of gelation increased as the rate of pectin loss increased. Samples containing about the same amounts of water plus oxalate-soluble pectin decreased in stability with an increase in pectinesterase activity. There was some indication that a high pectinesterase activity with low water plus oxalate-soluble pectin was as effective in producing a gel as a high water plus oxalate-soluble fraction with low pectinesterase activity. Stability to gelation is related to the pectinesterase activity; as the pectinesterase activity increased the tendency towards gelation increased.

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LIME OIL AS A FACTOR IN DETERIORATION OF FLAVOR OF LIME JUICE

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The citrus industry as a whole has turned the bulk of its harvest from the fresh market to that of processed products. Since this change was made rather suddenly and recently, information providing a basis for complete quality control is not yet available. The citrus fruit that has most recently made its way into the processing industry is the lime. This late arrival was caused mostly by the failure to process an acceptable product under the older methods as well as the limited supply of raw material which prevented financing of large scale research.

Lime juice is now reaching the market in an excellent, processed form. The industry is becoming a factor in the citrus trade of the country. Even now, however, processing techniques are based on information provided for the handling of other fruits, as well as a crude system of trial and error. Therefore, research