ELSEVIER

Food Chemistry **51** (1994) 153–157

0308-8146/94/$7.00

Physical–chemical characteristics of retail pasteurized orangeades

Roldán Marín, Betzabé Sulbarán de Ferrer,* Alexis Ferrer & Graciela Ojeda de Rodríguez

Laboratorio de Alimentos, Departamento de Química, Facultad de Ciencias, Universidad del Zulia, Grano de Oro, Módulo 2, Maracaibo, Venezuela

(Received 26 May 1993; revised version received and accepted 28 January 1994)

The objective of this work was to determine the physical–chemical characteristics of four different brands of commercial orangeades sold in Maracaibo, Venezuela. The analyzed parameters were: pH, titratable acidity, soluble solids, suspended solids, relative density, ash, ascorbic acid, reducing and non-reducing sugars and °Brix/acid ratio. The values of the parameters were compared with the limits established by the Comisión Venezolana de Normas Industriales (Covenin). Results indicated that all parameters, except ascorbic acid, reducing and non-reducing sugars and ash, were within the Covenin limits. The concentration of ascorbic acid was below the required value, reducing and non-reducing sugars were at higher concentrations than the limits and the ash for two of the brands was slightly lower than the limit. The compositions of the orangeades turned out to be very heterogeneous. They differed in at least four parameters. Some brands differed in all the parameters.

INTRODUCTION

Pasteurized orangeades are products prepared by adding potable water and natural sweeteners to non-fermented orange juice or a concentrate of non-fermented orange juice, that has been subjected to a heat process or an appropriate pasteurization process. The juice is extracted by mechanical means applied to the endocarp of ripe, fresh, healthy and clean oranges of the *Citrus sinensis* (L) species. This process assures its conservation in appropriate containers at a temperature not greater than 8°C (Covenin, 1981; Redd *et al.*, 1986).

The pasteurized orangeades should contain not less than 44% or more than 62% orange juice with 9% soluble solids, or its equivalent, derived from the juice concentrate. Concentration is determined in a refractometer at 20°C, correcting for acidity and expressed in °Brix on the international scale for sucrose (Covenin, 1981).

Pasteurized orangeades, of whatever brand, should maintain and conserve their physical–chemical quality in a state comparable to the raw material used in their fabrication (Kramer & Twigg, 1973). As pasteurized orangeades are a popular consumer drink in this country, it is necessary to investigate their quality.

At present, there are a number of brands of pasteurized orangeades sold in Venezuela, all of which must possess specified physical-chemical characteristics, as established by the Comisión Venezolana de Normas Industriales (Covenin), such as: pH (min. 3·2–max. 3·7), titratable acidity (max. 1·2% w/w as citric acid), soluble solids (min. 4°Brix), suspended solids (max. 12·4% v/v), relative density (max. 1·0654), ash (min. 0·162% w/w, max. 0·420% w/w), ascorbic acid (min. 35 mg/100 ml), reducing and non-reducing sugars (max. 11·8% w/v) and °Brix/acid ratio (min 13, max. 28) (Covenin, 1981).

The objective of this paper is to determine the aforementioned physical–chemical parameters, with respect to four commercial brands of pasteurized orangeades consumed regionally, in order to determine their compliance with the quality control norms established by the Ministerio de Fomento of Venezuela in the Covenin norms.

MATERIALS AND METHODS

Materials

Four brands of commercial pasteurized orangeades were chosen from those with the widest consumption in Maracaibo. These were identified with the letters A, B, C and D for analysis. So that the analysis could be performed 'blind', the samples were coded immediately after purchase.

The size of the sample was calculated according to Snedecor and Cochran (1980), based on the analysis of

* To whom correspondence should be addressed.

Table 1. Physical–chemical characteristics of brand A orangeade

Parameter	$\bar{X}$	s	CV(%)
pH	3·24	0·0718	2·22
Titratable acidity (% w/w citric acid)	0·481	0·0076	1·58
Soluble solids (°Brix)	12·02	0·0719	0·60
Suspended solids (% v/v)	1·00	0·0182	1·83
Relative density	1·0526	0·0008	0·08
Ash (% w/w)	0·120	0·0109	9·07
Ascorbic acid (mg/100 ml)	12·6	0·5119	4·06
Reducing and non-reducing sugars (% w/v)	12·0	0·2920	2·43
°Brix/acid	24·9	0·5300	2·13

$\bar{X}$, Mean values; s, standard deviation; CV, coefficient of variation.
Values are average of 30 determinations.

Table 3. Physical–chemical characteristics of brand C orangeade

Parameters	$\bar{X}$	s	CV(%)
pH	3·51	0·1042	2·97
Titratable acidity (% w/w citric acid)	0·494	0·0470	9·51
Soluble solids (°Brix)	13·17	0·6285	4·77
Suspended solids (% v/v)	2·40	1·2832	53·39
Relative density	1·0563	0·0017	0·16
Ash (% w/w)	0·163	0·0147	8·99
Ascorbic acid (mg/100 ml)	26·0	6·3395	24·39
Reducing and non-reducing sugars (% w/v)	13·4	0·5152	3·83
°Brix/acid	26·9	2·8450	10·59

$\bar{X}$, Mean values; s, standard deviation; CV, coefficient of variation.
Values are average of 30 determinations.

two brands and five samples per brand, and it turned out to be 15. Three different production lots were analyzed for each brand, with five samples of one liter each per lot.

Brand A was packed in plastic, and the other brands in PurePak cartons. The samples were purchased in stores throughout Maracaibo (western Venezuela).

Analytical methods

The samples were kept under refrigeration during transport. In the laboratory, the volume was measured and the samples were then refrigerated in tightly sealed containers, until the subsequent analysis which was performed the same day as purchase. The physical–chemical parameters were determined on the basis of the Covenin norms (1981).

The pH, titratable acidity as citric acid, reducing and non-reducing sugars were determined according to the norms established by Covenin (1977a, 1979, 1983), equivalent to the methods of the AOAC (1980). Relative density was determined by the AOAC (1980) method. Soluble solids were determined according to Covenin (1977*b*), using a Bausch & Lomb refractometer, with corrections for temperature and acidity. Suspended solids were determined according to Covenin (1978), using a Dynac II centrifuge at 1400 rpm, with a 170 mm tip radius horizontal rotor, The ascorbic acid content was determined by titration, using 2,6-dichloroindophenol, having first verified the absence of interfering substances (Covenin, 1982). Ash content was determined by the method proposed in the *Quality Control Manual for Citrus Processing Plants* (Redd *et al.*, 1986). Each analysis was carried out in duplicate for the 60 samples of orangeades. Comparison between brands was done using the Duncan Multiple Range test. Comparison of the statistical relevance of analyzed samples to the Covenin norms was carried out with the Chi-square test (Snedecor & Cochran, 1980).

Table 2. Physical–chemical characteristics of brand B orangeade

Parameters	$\bar{X}$	s	CV(%)
pH	3·46	0·1524	4·40
Titratable acidity (% w/w citric acid)	0·491	0·0146	2·97
Soluble solids (°Brix)	11·95	0·1752	1·47
Suspended solids (% v/v)	2·51	0·2693	10·74
Relative density	1·0516	0·0006	0·06
Ash (% w/w)	0·163	0·0093	5·68
Ascorbic acid (mg/100 ml)	11·7	1·0075	8·61
Reducing and non-reducing sugars (% w/v)	12·1	0·0990	0·83
°Brix/acid	24·3	0·6929	2·85

$\bar{X}$, Mean values; s, standard deviation; CV, coefficient of variation.
Values are average of 30 determinations.

RESULTS AND DISCUSSION

Tables 1–4 show the averages ($\bar{X}$), standard deviations (s) and coefficients of variation (CV) with respect to the physical–chemical parameters for the four brands of orangeades. The reproducibility of the methods used was very high, as measured by the closeness between the values of the duplicate tests. The coefficient of variation was less than 4% and ash had the greatest values (results not shown). Where the tables show high CV values, as for example in orangeade C, with suspended

Table 4. Physical–chemical characteristics of brand D orangeade

Parameters	$\bar{X}$	s	CV(%)
pH	3·23	0·0450	1·39
Titratable acidity (% w/w citric acid)	0·476	0·0122	2·56
Soluble solids (°Brix)	12·29	0·2231	1·82
Suspended solids (% v/v)	0·95	0·0681	7·15
Relative density	1·0528	0·0009	0·09
Ash (% w/w)	0·120	0·0108	9·02
Ascorbic acid (mg/100 ml)	10·6	0·1756	1·65
Reducing and non-reducing sugars (% w/v)	12·3	0·4416	3·60
°Brix/acid	25·8	0·6314	2·45

$\bar{X}$, Mean values; s, standard deviation; CV, coefficient of variation.
Values are average of 30 determinations.

Table 5. Mean values of physical–chemical characteristics of orangeades

Parameter	Orangeade brand				
	A	B	C	D	Covenin
pH	3·24 a	3·46 b	3·51 b	3·23 a	3·2–3·7
Titratable Acidity (% w/w citric acid)	0·481 ab	0·491 bc	0·494 c	0·476 a	≤1·2
Soluble solids (°Brix)	12·02 a	11·95 a	13·17 c	12·29 b	≥4·0
Suspended solids (% v/v)	1·00 a	2·51 b	2·40 b	0·95 a	≤12·4
Relative density	1·0526 b	1·0516 a	1·0563 c	1·0528 b	≤1·0654
Ash (% w/w)[a]	0·120 a	0·163 b	0·163 b	0·120 a	0·162–0·420
Ascorbic acid (mg/100 ml)	12·6 b	11·70 ab	25·99 c	10·62 a	≥35
Reducing and non-reducing sugars (% w/v)[a]	12·0 a	12·09 a	13·44 c	12·28 b	≤11·8
Reducing and non-reducing sugars (% w/w)	11·4 a	11·50 a	12·72 c	11·66 b	—
°Brix/acid[a]	24·9 a	24·32 a	26·87 c	25·78 b	13–28

Mean values are average of 30 determinations. Means in a row with different coefficients are significantly different ($p > 0·05$).
[a]A Chi-square test showed significant differences ($p < 0·05$).

solids (53·39%) and ascorbic acid (24·39%), these values actually express the difference between lots. Although this might be partially explained by differences in raw materials used, such as juice concentrates, caused by differences in maturity and variety of oranges (Bernardi *et al.*, 1991), it is assumed, in this work, that the large variability in samples is due to manufacturing defects.

Table 5 shows the comparative statistics of the averages of the physical-chemical parameters of the four brands of orangeades. It also indicates the values required by the Covenin norms (1981). The statistics for reducing and non-reducing sugars have been written using two different units: % w/v, which is the unit required by the Covenin norm; and w/w, which is used for comparison with soluble solids. On comparing the average values of the physical–chemical parameters with those recommended by Covenin, it was observed that the pH, titratable acidity, soluble solids, suspended solids, relative density and the °Brix/acid ratio complied with the norms. All the measured values of the first five parameters met the required values. Although the average °Brix/acid ratio of all brands complied with the norm, the Chi-square test showed significant differences among the brands when compared to the norms ($p < 0·05$). Brand C is lower in quality than the rest of the brands in terms of the °Brix/acid ratio; 30% of the samples were outside this norm, and this was significant.

The range of pH (3·23–3·51) corresponds to the literature (Mata, 1982; Fonseca, 1984). Titratable acidity (0·48–0·49%) was slightly lower than that found by Mata (1982) in orangeades produced in the central region of the country (0·56%). There were marked differences between lots with respect to titratable acidity in orangeade C. The value for titratable acidity is similar in all the orangeades. Among the organic acids present in the orangeades, citric acid is predominant and the most characteristic of all. It is known that processing plants adjust the acidity by the addition of citric acid. The value found for the orangeades was relatively low with respect to the maximum expressed in the norm, 1·2% w/w, and reflects the low acid content of the oranges used in the manufacture of orangeades, and also the fact that in Venezuela, public acceptance is high for low acidic orangeades.

The concentration of soluble solids (11·95–13·17 °Brix) corresponds to data reported in the literature by Prieto (1985), while other investigators (Mata 1982; Fonseca, 1984) report values slightly higher (12·6–14·4 °Brix). The Covenin norm (1977*b*) seems to be inadequate since the minimum value (4 °Brix) is very low compared with the averages of the different brands of orangeades. These values for soluble solids reflect the fact that these drinks are very sweet. On comparing the averages of soluble solids with the total for reducing and non-reducing sugars, it can be observed that the values are similar, showing that the sugars practically correspond to the soluble solids detected by refractometry. It has been reported (Primo, 1979; Kimball, 1991) that the sugars should represent about 75–80% of the total soluble solids.

The content of suspended solids in orangeades B, C and D shows great variation between lots, with greater values for orangeade C, which gave a CV of 53·39% (Tables 1–4). However, the range 0·95–2·51% v/v coincides almost completely with that reported by Fonseca (1984). It should be noted that the norm establishes 12·4% as a maximum, which suggests that the orangeades sold have been clarified, in spite of the fact that in other countries these orangeades would have very little commercial value (Durán *et al.*, 1976). Regardless of the low content of solids in suspension, the formation of colloidal particles was frequently observed in orangeades under refrigeration.

The values for relative density (1·0516–1·0563) were so reproducible that significant differences were detected in the third decimal place. The average values were very similar and the coefficient of variation was the lowest of all parameters. The results were very similar to those reported in other Venezuelan orangeades (Fonseca, 1984; Prieto, 1985). The °Brix/acid relationship (24·4–26·9) showed a high degree of homogeneity with respect to the profile of predominant flavors in the orangeades, sweetness and acidity. This also shows that this parameter is controlled by the processing plants. The fact that the average value is close to the maximum

value of 28 confirms the preference for sweetness. In earlier years (Mata, 1982; Fonseca, 1984) orangeades have been reported with a °Brix/acid ratio closer to 28, including some that exceeded this value (exceeding the norm).

Lack of compliance with Covenin norms was found with respect to ash, total reducing and non-reducing sugars, and ascorbic acid content.

In general, the levels of ash were low compared to the norm, and two of the brands were well below this norm. Both Duncan and Chi-square tests showed significant differences ($p < 0.05$), Brands B and C being the best among them. It has been reported that orangeades produced in earlier years (Amedh, 1983; Prieto, 1985) have maintained the ash content to within the Covenin norms, ranging between 0·34 and 0·44%.

With respect to the content of reducing and non-reducing sugars, as shown in Table 5, all brands showed average values slightly higher that the norm. However, the Chi-square test detected significant differences among the brands when compared to the norms ($p < 0.05$), brand A having relatively better quality.

The most noteworthy finding is that the percentage of non-reducing sugars (sucrose) was between 73·3 and 76·5% of the total sugar content (values not reported), while in the literature (Prieto, 1985) the percentage of non-reducing sugars has oscillated between 39·5 and 50·5%. As the level of sucrose, as a percentage of sugar, should never exceed 55% (Kimball, 1991), the addition of sucrose is strongly suggested from this result. This also causes the subsequent dilution of the product and a decrease of its nutrient value. Kimball (1991) also reports that sugar and invert sugar can be added to pasteurized orange juice to raise the Brix to a normal level, but such addition must be declared on the label, and this was not the case. Water and carbohydrate additions are considered to be the main types of adulteration of orange juice, in use, worldwide.

All the brands of orangeades showed an ascorbic acid content much less than the norm (10·6–25·9 mg/100 ml), and in the cases of A, B, and D, it was less than 36% of the value required by law. In addition, none of the measured values of this parameter met the required value. Since ascorbic acid, with respect to nutritive value, is the most important component of citrus drinks, it is vitally important to exercise strict control over the processing plants. Primo (1979) reported that, in the USA, 60% of the daily ingestion of vitamin C came from orange juice. A standard serving (6 oz or 170·1 g) of orange juice should contain 100% of the recommended amount of this vitamin (Nagy *et al.*, 1993). In a similar manner, orangeade is the principal source of vitamin C in the Venezuelan population. Analyses carried out on Venezuelan orangeades in 1982 and 1983 (Fonseca, 1984) also showed levels below the norm (8·15–18·05 mg/100 ml), which demonstrates that the situation has been deficient for sometime. Nagy *et al.* (1993) have reported that losses of vitamin C, up to 10%, may occur during the pasteurization process. Losses are also probably due to the loss of stability of vitamin C on exposure to oxygen in air, and to heat, or because of inappropriate transport and storage of the raw material (Fennema, 1987; Kennedy *et al.*, 1992). Even under refrigeration, losses of 12–23% have been reported in juices stored open, during 1 or 2 weeks respectively (Shaw & Moshonas, 1991). This shows a great susceptibility to oxidation which increases at higher temperatures (Kennedy *et al.*, 1989), although the presence of glucose and fructose contribute to stability. Skurray *et al.* (1988) determined the content of vitamin C in orange juice coming from major exporting countries and found values of 41·4–111·4 mg/100 ml. On comparing these results with those obtained in this study, it is observed that Venezuelan orangeades show a much lower content. However, Venezuelan orangeades contain only 60% natural orange juice; by definition, the rest should only be water.

Based on the results it may be inferred that there is considerable variability in the manufacture of orangeades in Venezuela and major deviations from the Covenin norms occur. All the brands failed to comply with the norms in at least two of the physical–chemical parameters. Significant differences were found among the brands and between the brands and the Covenin norms. Major adulteration of the product is indicated by its very low acidity, very high percentage of sugar with respect to soluble solids, and extremely high percentage of sucrose with respect to total sugar.

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