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Chemical characterization of pineapple juices and nectars. Principal components analysis

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Characterization of pineapple derivates is achieved using several parameters and comparisons with natural samples. Eleven types of physico-chemical measurements are carried out on each sample: °Brix, pulp, titratable acidity, pH, organic acids, free sugars, vitamin C, formol index, ash, minerals and pectic substances. This allows 23 parameters to be studied. Samples analysed include: fresh pineapple juice (home-made), pineapple commercial juices and nectars. Statistical correlation analysis and principal components analysis (PCA) show that the organic acids, sugars, potassium levels and formol index are the most useful parameters for quality control analysis of pineapple juices and nectars.

INTRODUCTION

Pineapple fruit is consumed either fresh or in the processed form. Pineapple juices and nectars are generally prepared by redilution of concentrates imported from different countries of tropical or subtropical areas. In Europe, commercial juices are made 100% from concentrate and nectars are considered to have at least 40% of juice.

Many analytical and statistical methods have been proposed for the characterization of fruit derivates; generally, the concentrations of different elements are measured and those results compared to a data base analyzing natural samples (Aristoy *et al.*, 1989; Krueger & Krueger, 1992; Richard *et al.*, 1984). This comparison can be done using different statistical tests (Brown & Cohen, 1983; Lifschitz *et al.*, 1971; Richard *et al.*, 1984). This study is aimed at reducing the number of explanatory parameters on pineapple juices and nectars composition, and finding a specific quality index (Cámara, 1992; Hodgson & Hodgson, 1993).

MATERIAL AND METHODS

Samples

1. Natural pineapple juice (home-made) (ZN): five batches of freshly extracted juice were analysed (five fruits each batch).
2. Pineapple commercial juices: 100% made from concentrate. Samples from five different producers (three batches each), tetrapack-packaged, were tested.
3. Pineapple nectars, made from concentrate, at least 40% juice. These were from four different producers packed either in glass bottles or small cans, three batches each.

Table 1 shows the commercial samples' abbreviations used in this study.

Methods

The following determinations were carried out: *pulp*; *°Brix*, soluble solids, by refractometric method; *pH*; *titratable acidity*, reported as citric acid (AOAC, 1984); *organic acids*: citric and malic acid by HPLC (Cámara, 1992); *sugars*: fructose, glucose and sucrose by HPLC (Cámara, 1992); *pectic substances* (Blumenkrantz & Asboe-Hansen, 1973); *formol index* (Cámara, 1992); *vitamin C* by fluorimetric method (Brubacher, 1985); *ash* (AOAC, 1990); *mineral elements:* Na, K, Ca, Mg, Cu, Fe, Mn and Zn by atomic absorption spectrophotometry (Torija, 1981); *phosphorus* by colorimetric method (Cámara, 1992).

Statistical analysis

Correlation analysis

This was done taking as statistically significant those correlations with a significance level $p < 0{\cdot}05$ (based on the Student's t distribution), with a confidence level of 95% (Lebart, 1985; STSC, 1986).

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Table 1. Abbreviations used in this study for commercial samples

Producers	Samples	Packaging system		
		Tetrapack	Glass bottles	Small cans
0	Juice 100%	Z0	—	—
1	Juice 100%	Z1		
	Nectar		—	NL1
2	Juice 100%	Z2		
	Nectar		NC2	NL2
3	Juice 100%	Z3		
	Nectar		NC3	NL3
4	Juice 100%	Z4		
	Nectar		NC4	—
5	Juice 100%	Z5		
	Nectar		NC5	NL5

Z, juice 100%; N, nectar; NC, nectars packaged in glass bottles; NL, nectar packaged in small cans.

Multivariate principal components analysis (PCA)
This was based on the correlation analysis and computerized in the statgraphic program, (Lebart, 1985; STSC, 1986).

RESULTS AND DISCUSSION

All tables show the mean value of three analyses (X) and standard deviation $n-1$ (SD) of the results obtained from different batches of each sample analysed.

The pH, acidity, organic acids, vitamin C and formol index results are shown in Table 2 related to natural and commercial pineapple juices and Table 3 related to nectars. Pineapple juice pH is also known to vary with growing location, harvest time, fruit maturity and other similar factors which affect the fruit (Hodgson & Hodgson, 1993). All the samples analysed had pH levels similar between themselves and similar to natural juice (ZN: 3·660 of pH), except the juice Z4 with a pH of 4·260.

In all the commercial juices analysed the ratio total organic acids/titratable acidity was close to 1, except in the Z1 sample.

For commercial nectars this ratio was close to 1 only in samples NL1, NC5 and NL5. It is notable that citric acid may legally be added to nectars.

Fresh pineapple juice has a citric acid/malic acid ratio close to 2, as in most of the commercial samples, except Z2, NL2, NC2 and NL1 (Cámara, 1992; Hodgson & Hodgson, 1993; Krueger & Krueger, 1992).

All the samples analysed had lower levels of vitamin C than the natural juice (ZN: 84·2 mg ascorbic acid/100 ml) as this vitamin can easily be destroyed by the technological process applied. The juice Z0 and most of the nectars analysed were labelled as containing added vitamin C or ascorbic acid.

Commercial juices show similar formol indices as natural juice (ZN: 12.378), excepting for sample Z3 with a formol index below RSK values. Only the nectars NL1, NC2, NL5, NC5 have the correct formol index (40% of natural juice, 4·95).

If we considered the ratio °Brix/acidity, often used for industrial classification of these products, the high levels found in the commercial juices are caused by a low acidity of these products compared with natural juice. Nectars have also higher levels of °Brix, by the addition of carbohydrates, and lower levels of acidity than natural juice.

In Table 4 and 5 are shown the °Brix, pulp, sugars and pectic substances of the juices and nectars.

Natural juice (ZN) and commercial samples (juices and nectars) show similar levels of °Brix (ZN: 11·8 °Brix), that means that carbohydrates has been added to correct the effect of the concentrate dilution on this parameter. The pulp and pectic substance levels of natural juice (ZN) are higher than in juices and nectars analysed; the difference is a consequence of the extraction procedure applied.

The total sugar content of samples analysed ranged from 8·50% of ZN to 12·8% of NC4; these values are similar to those observed by Li & Schuhmann (1983).

Sucrose is the main sugar in natural (ZN: 4·13 g sucrose/100 ml) and in commercial juices (Hodgson & Hodgson, 1993). Fructose and glucose are present in these samples in similar proportions (1:1) (Krueger & Krueger, 1992). The sample Z2 glucose and fructose contents relative to sucrose are unusually high. The sugar levels of nectars analysed are variable; most of them mentioned in their labels the addition of glucose.

Ash, and mineral element results are shown in Table 6, for natural (ZN) and commercial juices and in Table 7 for nectars. It is noteworthy that mineral concentrations of pineapple juices vary with the composition of growing soil and the irrigation water.

Both natural (ZN: 0·386 g ash/100 ml) and commercial juices, show similar levels of ash which are higher than nectars, (except Z2: 0·163 g ash/100 ml with lower levels), because nectars contain only 40% of natural juice. The major mineral elements are K, Mg, P, Cu and Zn, but Z2 and Z3 mineral values are below normal for K (Krueger & Krueger, 1992).

To conduct the correlation analysis, the mean values of variables: °Brix, vitamin C, formol index, pH, ash, potassium, fructose, glucose, sucrose, citric acid, malic acid, pectic substances and pulp,shown in Tables 2–7, were used.

Table 2. pH, acidity (g citric acid/100 ml), organic acids (g/100 ml), vitamin C (mg ascorbic acid/100 ml), and formol index levels of natural and commercial pineapple juices

	Samples					
	ZN $X \pm SD$	Z0 $X \pm SD$	Z1 $X \pm SD$	Z2 $X \pm SD$	Z3 $X \pm SD$	Z4 $X \pm SD$
pH	3·66 ± 0·021	3·48 ± 0·095	3·57 ± 0·177	3·00 ± 0·020	3·83 ± 0·146	4·26 ± 0·085
Acidity	0·959 ± 0·078	0·643 ± 0·032	0·572 ± 0·053	0·689 ± 0·014	0·458 ± 0·011	0·612 ± 0·011
Citric acid	0·864 ± 0·254	0·431 ± 0·096	0·514 ± 0·072	0·576 ± 0·078	0·317 ± 0·151	0·514 ± 0·153
Malic acid	0·461 ± 0·104	0·229 ± 0·049	0·235 ± 0·025	0·167 ± 0·034	0·182 ± 0·070	0·258 ± 0·010
Vitamin C	84·2 ± 9·695	58·0 ± 4·954	14·2 ± 3·964	8·52 ± 1·440	44·8 ± 3·560	15·8 ± 3·244
Formol index	12·4 ± 2·626	14·3 ± 1·925	14·1 ± 2·941	10·9 ± 2·253	7·36 ± 1·090	12·2 ± 0·643

X = mean value of three batches analysed on triplicate.
SD = standard deviation (n–1).

Table 3. pH, acidity (g citric acid/100 ml), organic acids (g/100 ml), vitamin C (mg ascorbic acid/100 ml), and formol index levels of pineapple nectars

	Samples							
	NL1 $X \pm SD$	NC2 $X \pm SD$	NL2 $X \pm SD$	NC3 $X \pm SD$	NL3 $X \pm SD$	NC4 $X \pm SD$	NC5 $X \pm SD$	NL5 $X \pm SD$
pH	3·87 ± 0·050	3·27 ± 0·287	3·63 ± 0·296	3·38 ± 0·043	3·44 ± 0·139	3·44 ± 0·006	3·26 ± 0·011	3·29 ± 0·368
Acidity	0·647 ± 0·006	0·408 ± 0·009	0·346 ± 0·015	0·458 ± 0·011	0·412 ± 0·029	0·508 ± 0·030	0·588 ± 0·016	0·605 ± 0·027
Citric acid	0·365 ± 0·028	0·397 ± 0·108	0·483 ± 0·070	0·443 ± 0·071	0·411 ± 0·193	0·190 ± 0·008	0·442 ± 0·055	0·523 ± 0·060
Malic acid	0·325 ± 0·022	0·241 ± 0·177	0·146 ± 0·032	0·210 ± 0·041	0·147 ± 0·057	0·086 ± 0·008	0·200 ± 0·025	0·187 ± 0·032
Vitamin C	10·2 ± 0·581	19·0 ± 2·549	23·9 ± 6·802	39·9 ± 2·843	57·5 ± 5·567	84·9 ± 3·939	21·0 ± 2·473	17·0 ± 0·888
Formol index	6·07 ± 2·024	5·11 ± 1·705	4·29 ± 0·044	3·31 ± 0·352	3·31 ± 0·704	4·07 ± 0·820	6·49 ± 0·846	7·34 ± 1·345

X = mean value of three batches analysed on triplicate.
SD = standard deviation (n–1).

Table 4. °Brix, pulp (g/100 ml), sugars (g/100 ml), and pectic substances (g galacturonic acid/100 ml) levels of natural and commercial pineapple juices

	Samples					
	ZN $X \pm SD$	Z0 $X \pm SD$	Z1 $X \pm SD$	Z2 $X \pm SD$	Z3 $X \pm SD$	Z4 $X \pm SD$
°Brix	11·8 ± 1·852	12·6 ± 0·288	11·7 ± 0·264	11·7 ± 0·100	11·4 ± 0·100	12·8 ± 0·321
Pulp	8·779 ± 3·569	4·800 ± 0·452	5·70 ± 0·528	4·11 ± 0·899	4·78 ± 0·661	4·47 ± 0·618
Fructose	2·51 ± 0·551	2·44 ± 0·264	2·44 ± 0·273	3·19 ± 0·258	2·19 ± 0·220	2·37 ± 0·363
Glucose	2·31 ± 0·455	2·57 ± 0·295	2·21 ± 0·279	3·31 ± 0·104	2·18 ± 0·245	2·59 ± 0·304
Sucrose	4·13 ± 0·692	4·85 ± 0·447	4·29 ± 0·447	4·84 ± 1·227	4·74 ± 0·583	5·51 ± 0·721
Total sugars	8·95 ± 1·303	9·61 ± 0·785	8·94 ± 0·723	11·2 ± 1·060	9·11 ± 0·824	10·5 ± 0·782
Pectic substances	0·279 ± 0·087	0·092 ± 0·008	0·095 ± 0·009	0·070 ± 0·013	0·148 ± 0·026	0·137 ± 0·024

X = mean value of three batches analysed on triplicate.
SD = standard deviation (n–1).

Table 5. °Brix, pulp (g/100 ml), sugars (g/100 ml), and pectic substances (g galacturonic acid/100 ml) levels of pineapple nectars

	Samples							
	NL1 $X \pm SD$	NC2 $X \pm SD$	NL2 $X \pm SD$	NC3 $X \pm SD$	NL3 $X \pm SD$	NC4 $X \pm SD$	NC5 $X \pm SD$	NL5 $X \pm SD$
°Brix	15·7 ± 0·173	12·8 ± 0·200	11·7 ± 0·305	12·4 ± 0·115	12·2 ± 0·200	14·5 ± 0·058	13·4 ± 0·100	13·2 ± 0·305
Pulp	7·133 ± 0·223	4·69 ± 0·570	2·03 ± 0·223	4·54 ± 0·385	4·53 ± 0·541	3·24 ± 0·090	3·86 ± 0·406	5·35 ± 0·303
Fructose	4·31 ± 0·364	2·92 ± 0·182	2·09 ± 0·337	2·49 ± 0·555	1·89 ± 0·024	6·35 ± 0·263	4·90 ± 0·007	5·18 ± 0·519
Glucose	5·99 ± 0·391	3·76 ± 0·408	2·62 ± 0·424	3·19 ± 0·571	2·76 ± 0·071	6·21 ± 0·250	5·50 ± 0·110	6·25 ± 0·744
Sucrose	0·343 ± 0·041	2·38 ± 0·795	3·58 ± 0·642	2·65 ± 1·039	3·51 ± 0·346	0·213 ± 0·036	1·07 ± 0·116	0·874 ± 0·123
Total sugars	11·86 ± 0·861	9·94 ± 0·624	8·97 ± 0·802	9·38 ± 1·338	9·11 ± 0·459	12·8 ± 0·547	11·5 ± 0·201	12·47 ± 1·334
Pectic substances	0·081 ± 0·019	0·142 ± 0·017	0·107 ± 0·013	0·048 ± 0·009	0·156 ± 0·032	0·063 ± 0·005	0·074 ± 0·011	0·079 ± 0·005

X = mean value of three batches analysed on triplicate.
SD = standard deviation (n–1).

Table 6. Ash (g/100 ml) and mineral elements (mg/100 ml) levels of natural and commercial pineapple juices

	Samples					
	ZN $X \pm SD$	Z0 $X \pm SD$	Z1 $X \pm SD$	Z2 $X \pm SD$	Z3 $X \pm SD$	Z4 $X \pm SD$
Ash	0·386 ± 0·053	0·339 ± 0·042	0·318 ± 0·015	0·163 ± 0·042	0·235 ± 0·009	0·414 ± 0·041
Na	2·24 ± 0·855	3·56 ± 1·403	5·12 ± 3·207	1·52 ± 0·389	9·66 ± 0·799	8·57 ± 1·570
K	124 ± 9·572	113 ± 9·114	101 ± 9·904	40·2 ± 4·471	69·9 ± 6·316	132 ± 9·536
Ca	11·5 ± 4·288	16·2 ± 1·958	18·5 ± 3·143	9·64 ± 0·802	14·2 ± 0·597	17·5 ± 1·051
Mg	15·4 ± 5·105	15·7 ± 1·399	12·3 ± 1·453	6·96 ± 1·111	12·5 ± 0·382	17·3 ± 1·273
P	3·16 ± 0·261	3·55 ± 0·347	4·01 ± 0·350	2·45 ± 0·213	1·87 ± 0·623	3·79 ± 0·402
Cu	0·059 ± 0·019	0·062 ± 0·016	0·062 ± 0·012	0·031 ± 0·006	0·028 ± 0·004	0·052 ± 0·016
Fe	0·265 ± 0·028	0·278 ± 0·031	0·184 ± 0·027	0·129 ± 0·015	0·209 ± 0·054	0·325 ± 0·196
Mn	0·295 ± 0·072	1·26 ± 0·186	0·661 ± 0·363	0·686 ± 0·059	1·18 ± 0·157	0·962 ± 0·224
Zn	0·074 ± 0·013	0·106 ± 0·021	0·090 ± 0·017	0·039 ± 0·008	0·042 ± 0·004	0·062 ± 0·006

X = mean value of three batches analysed on triplicate.
SD = standard deviation (n–1).

Table 7. Ash (g/100 ml) and mineral elements (mg/100 ml) levels of pineapple nectars

	Samples							
	NL1 $X \pm SD$	NC2 $X \pm SD$	NL2 $X \pm SD$	NC3 $X \pm SD$	NL3 $X \pm SD$	NC4 $X \pm SD$	NC5 $X \pm SD$	NL5 $X \pm SD$
Ash	0·189 ± 0·036	0·082 ± 0·008	0·181 ± 0·034	0·171 ± 0·010	0·211 ± 0·014	0·147 ± 0·005	0·119 ± 0·009	0·125 ± 0·023
Na	1·72 ± 0·253	2·37 ± 0·515	32·0 ± 9·791	8·09 ± 0·797	13·2 ± 3·484	11·7 ± 1·382	3·67 ± 1·602	2·00 ± 1·139
K	40·9 ± 3·415	21·3 ± 2·882	21·5 ± 2·404	36·7 ± 1·556	37·3 ± 2·705	33·8 ± 3·748	37·9 ± 4·942	35·1 ± 2·280
Ca	9·75 ± 0·516	10·2 ± 1·905	9·19 ± 3·538	13·8 ± 1·007	14·8 ± 1·353	6·75 ± 0·157	5·40 ± 2·937	8·12 ± 2·042
Mg	6·56 ± 0·332	5·42 ± 0·595	4·28 ± 1·565	10·0 ± 0·430	10·8 ± 1·158	5·07 ± 0·189	4·25 ± 0·827	4·72 ± 0·204
P	0·727 ± 0·092	1·04 ± 0·089	0·535 ± 0·031	1·86 ± 0·152	0·534 ± 0·083	1·71 ± 0·287	1·44 ± 0·225	1·26 ± 0·165
Cu	0·028 ± 0·002	0·026 ± 0·004	0·017 ± 0·004	0·028 ± 0·011	0·028 ± 0·006	0·026 ± 0·004	0·029 ± 0·014	0·030 ± 0·015
Fe	0·441 ± 0·085	0·094 ± 0·010	0·300 ± 0·026	0·168 ± 0·041	0·337 ± 0·103	0·397 ± 0·026	0·113 ± 0·018	0·140 ± 0·152
Mn	0·358 ± 0·020	0·247 ± 0·005	0·137 ± 0·036	0·758 ± 0·117	0·487 ± 0·123	0·467 ± 0·029	0·283 ± 0·117	0·317 ± 0·036
Zn	0·033 ± 0·006	0·030 ± 0·009	0·021 ± 0·003	0·033 ± 0·003	0·030 ± 0·006	0·028 ± 0·003	0·057 ± 0·008	0·036 ± 0·004

X = mean value of three batches analysed on triplicate.
SD = standard deviation (n–1).

Table 8. Correlation matrix between general variables

	°Brix	pH	Acidity	Citric acid	Malic acid	Ash	K	Formol index	Vitamin C	Fructose	Glucose	Sucrose	Pectic substances	Pulp
°Brix	1·0000 0·0000													
pH	0·1151 0·6952	1·0000 0·0000												
Acidity	0·0630 0·8306	0·0774 0·7926	1·0000 0·0000											
Citric acid	−0·4577 0·0998	−0·0041 0·9889	0·7034 0·0050	1·0000 0·0000										
Malic acid	0·0248 0·9330	0·3553 0·2125	0·7434 0·0023	0·7017 0·0052	1·0000 0·0000									
Ash	−0·3059 0·2875	0·6633 0·0097	0·5168 0·0584	0·4797 0·0826	0·5335 0·0495	1·0000 0·0000								
K	−0·2708 0·3490	0·5736 0·0320	0·6078 0·0211	0·4827 0·0811	0·5725 0·0324	0·9588 0·0000	1·0000 0·0000							
Formol index	−0·3091 0·2822	0·2187 0·4526	0·6657 0·0094	0·5327 0·0499	0·4687 0·0909	0·7505 0·0020	0·8491 0·0001	1·0000 0·0000						
Vitamin C	−0·0470 0·8733	0·0298 0·9196	0·2041 0·4841	0·0021 0·9943	0·0782 0·7904	0·2582 0·3725	0·2273 0·4346	−0·0626 0·8317	1·0000 0·0000					
Fructose	0·7363 0·0027	−0·2913 0·3123	0·1178 0·6884	−0·3657 0·1985	−0·2482 0·3923	−0·5080 0·0637	−0·3872 0·1713	−0·2663 0·3574	0·0523 0·8590	1·0000 0·0000				
Glucose	0·8382 0·0002	−0·2720 0·3469	0·0096 0·9741	−0·3971 0·1598	−0·2104 0·4703	−0·6228 0·0174	−0·5345 0·0489	−0·4229 0·1319	−0·1138 0·6985	0·9390 0·0000	1·0000 0·0000			
Sucrose	−0·7919 0·0007	0·2831 0·3267	0·1365 0·6417	0·4217 0·1331	0·1660 0·5706	0·6882 0·0067	0·6407 0·0136	0·6148 0·0193	−0·0060 0·9838	−0·8301 0·0102	−0·9219 0·0000	1·0000 0·0000		
Pectic substances	−0·3802 0·1800	0·3434 0·2293	0·4298 0·1251	0·6060 0·0216	0·6541 0·0112	0·5237 0·0546	0·4939 0·0726	0·2660 0·3581	0·4343 0·1208	−0·4515 0·1051	−0·4925 0·0736	0·4041 0·1518	1·0000 0·0000	
Pulp	0·1207 0·6809	0·2299 0·4292	0·7548 0·0018	0·5587 0·0378	0·8838 0·0000	0·4330 0·1219	0·4898 0·0754	0·4183 0·1366	0·1691 0·5633	−0·0810 0·7832	−0·0506 0·8635	0·0190 0·9485	0·5711 0·0329	1·0000 0·0000

Correlation coefficient/significance level.

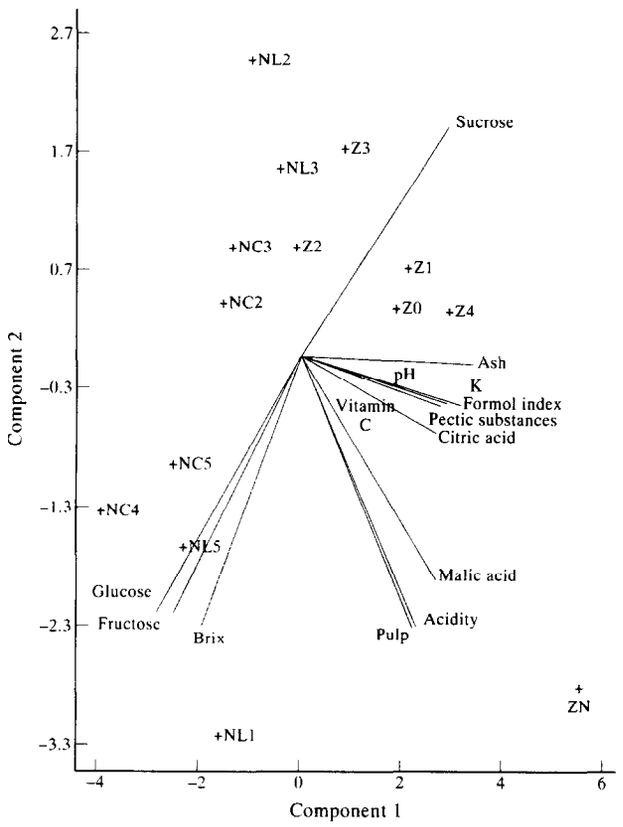

Fig. 1. Principal components analysis of the composition of natural pineapple juice, commercial juices and nectars. Biplot of first two principal components.

The correlation matrix (Table 8) between variables shows significant correlations between most of the variables considered. Only vitamin C showed no significant correlations with any of the other parameters considered.

The PCA (Fig. 1), based on the correlations mentioned above, allows reduction of the number of explanatory variables of the experimental results; the lines intersecting at (0,0) represent the experimental variables.

The first two axes of the main plane represent 67% of the variance of the system; the percentage of variance explained by each component is shown in Table 9.

Table 9. Percentage of variance explained by each component

Component number	Percent of variance	Cumulative percentage
1	46·53080	46·53080
2	21·37120	67·90799
3	10·33326	78·23525
4	9·15722	87·39247
5	6·96209	94·35456
6	2·27845	96·63301
7	1·33963	97·97264
8	0·75634	98·72898
9	0·61609	99·34507
10	0·39073	99·73580
11	0·15855	99·89435
12	0·06699	99·96134
13	0·03845	99·99979
14	0·00021	100·00000

The samples closest to the natural juice (ZN) are the juices Z0, Z1 and Z4. They are characterized by high levels of acids and minerals. The juices Z2 and Z3 are represented closer to their corresponding nectars (NC2, NL2, NC3, NL3). Samples NC4, NC5 and NL5 are represented together in the main plane and characterized by high levels of sugars. Nectar NL1 showed independent behaviour and is the nectar closest to natural juice, which indicates a similar composition.

CONCLUSIONS

The fresh pineapple juice is characterized by high levels of pulp, acids, sucrose and potassium. The commercial juices Z0, Z1 and Z4 show similar composition to natural juice; on the other hand, the juices Z3 and especially Z2, show similar compositions to nectars; this could indicate processing and storage abuse and also adulteration. The nectar NL1 show independent behaviour and can be considered as the nectar with a composition most similar to natural juice.

Vitamin C, Na and Fe are not useful parameters for characterization of pineapple juices and nectars.

The statistical analysis elucidated as useful quality indices for pineapple juices and nectars:

— ratio of citric acid/malic acid = 2.
— ratio of fructose/glucose = 1.
— potassium and formol index values.
— fructose + glucose/sucrose = 1 (only for juices).

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