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Preparation of pomegranate jellies

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SUMMARY – The production of processed foods derived from pomegranate juice is of essential interest, due to the possibility of giving an alternative use to this crop, which is generally consumed fresh. This would provide substantial benefits to the growers. One of these new products that we have assayed, is pomegranate jelly. In order to manufacture this food different jellifying agents were analysed, Agar rendering the best results. Once this objective was fulfilled and the production optimised, the influence of different parameters such as pH and storage temperature were investigated. The main objective of this work was focused on establishing the optimum conditions for obtaining a product with a stable colour, since it is well known that colour is one of the most important quality factors for consumers.

Key words: Jellies, pomegranate, juice, colour, pH, anthocyanins.

RESUME – *"Préparation des gelées de grenades". La production d'aliments transformés provenant de jus de grenade est d'un intérêt essentiel, dû à la possibilité d'apporter une utilisation alternative à cette culture, qui est généralement consommée en frais. Ceci apporterait des bénéfices substantiels aux agriculteurs. L'un de ces nouveaux produits que nous avons testés, est la gelée de grenades. Afin de fabriquer ce produit alimentaire, différents agents gélifiants ont été analysés, l'Agar donnant les meilleurs résultats. Une fois cet objectif atteint et la production optimisée, on a examiné l'influence de différents paramètres tels que le pH et la température de stockage. Le principal objectif de ce travail était axé sur la détermination des conditions optimales pour obtenir un produit ayant une couleur stable, car il est bien connu que la couleur est l'un des facteurs de qualité les plus importants pour les consommateurs.*

Mots-clés : *Gelées, grenade, jus, couleur, pH, anthocyanines.*

Introduction

The main objective of this study, continuing along the same line of obtaining maximum industrial benefit from pomegranate fruit, has been to make a new product: pomegranate jelly, from pomegranate juice. In order to reach this objective, fruits representing very different varietal groups were chosen: (i) Mollar (sweet commercial variety, used for fresh consumption); and (ii) Borde (bitter variety, normally used as a rootstock and with great possibilities for food use).

Jelly refers to a product made from fruit juice with a semi-solid or gelatinous consistency (Madrid *et al.*, 1994). The ingredients used in making jellies are: juices and fruit pulp, sweeteners, acids (citric, lactic, tartaric, malic and ascorbic), sulphurous anhydride, benzoic acid or sodium, potassium or calcic benzoate, antifoams, thickeners and natural jellifiers (agar-agar, pectins, carob gum, alginates and carrageenates, colourings, essences and authorised flavourings) (Código Alimentario Español, 1988).

The experiments conducted were fundamentally aimed at studying colour, and were therefore based on the analysis of anthocyanins, phenolic compounds responsible for the characteristic red colour of this fruit. Among the parameters that most influenced colour were pH and temperature.

The effect of the pH is of utmost importance as it can cause great modifications in the anthocyanin colour, therefore this study focused on the pH and its interaction with temperature. At very acid pHs, the cation flavilio is the only form present in significant quantity. As the pH rises, a proton is lost, a water molecule is gained and a carbinol (colourless) pseudobase is formed, which is in tautomeric equilibrium with the chalcone form (colourless). If the pH continues to rise, the quinoidal base of the blue colour is formed.

Six anthocyanins have been described in the pomegranate, which coincide with those found in our study. They are the derivates 3,5-diglycoside and 3 delphinidin cyanidin and pelargonidin glycosides (Du *et al*., 1975; Kriventsov and Arendt, 1981; Gil *et al.*, 1995).

The food industry uses different types of thickeners and jellifiers, also called hydrosoluble gums, hydrocholloids, hydrophilic colloids or simply gums. These products allow some rheological characteristics to be modified and adapted to the taste of the consumer or simply to solve technological manufacturing problems (Maestre Albert, 1983).

In general, the thickeners and jellifiers are polysaccharides that, as they are in solution, can increase the viscosity of the liquid products and/or form gels. They are also used to stabilise suspensions or emulsions in order to increase the water retention capacity or make some foods smoother and more palatable. They are essentially plant products although some are of animal origin and of a proteic nature (caseinates and gelatines). Given their abundance and diversity, the choice of a gum or a mixture of gums is rather difficult as it is hard to predict their behaviour when submitted to possible manufacturing operations; likewise it is complex to determine the appropriate dose (Maestre Albert, 1983)

Materials and methods

Plant material

The Mollar or Mollar de Elche varieties were provided by the agricultural processing company AURORA de Albatera (Alicante, Spain), at the end of September 1996.

The Borde or bitter variety was provided by the Higher Polytechnical School of Orihuela (Escuela Politécnica Superior de Orihuela) harvested in October 1996.

Juice extraction and analysis

In order to obtain kernels from inside the pomegranate, it is cut in two equal halves. It is squeezed gently by hand in order to loosen the kernels and then hit with a pestle until the fruit is empty.

The juice is extracted from the kernels with a hand press.

The juice is shaken by hand to make it homogeneous and the pH is determined immediately afterwards, being pH 4.25 for Mollar and 2.91 for Borde.

Jelly manufacture

As soon as the pomegranate juice is extracted, sodium benzoate is added (0.1%) to avoid mould and yeast formation.

The Mollar juice was divided into four batches to obtain the different jellies: (i) Batch 1 (Mollar pH 4); (ii) Batch 2 (Mollar pH 3.5); (iii) Batch 3 (Mollar pH 3); and (iv) Batch 4 (Borde pH 3).

The composition is the same for all jellies, made up of pomegranate juice and agar (2%). In order to acidify the pH of the Mollar juice, citric acid was added to batches 2 and 3, to obtain the pH values 3.5 and 3, respectively.

Half of each batch was stored at 25°C, in an air oven, and the other half at 5°C, in a refrigerator, for a period of 3 months.

Anthocyanin extraction

Five grams of jelly were extracted with 5 ml of an extracting solution made up of methanol:formic acid:water (25:1:24), shaken for 15 min at room temperature.

It was then filtered through glass wool and vacuum-concentrated to desiccation point in a vapour rotor (40°C).

The concentrate was redissolved in 5 ml of formic acid (3%), and separated through a Sep-Pak C-18 cartridge. The methanolic fraction was concentrated to desiccation point and redissolved in 1 ml of a methanol:formic acid 5% (80:20) mixture for its HPLC analysis.

Chromatographic analysis of anthocyanins

A Merck-Hitachi high-efficiency liquid chromatographer was used, made up of an L-6200 pump, a vis-UV L-7420 sight and an automatic AS-2000A injector with a Lichrochart 100 RP-18 (125×4 mm, particle size 5 µm) column; mobile phase: formic acid at 5% (solvent A) and methanol (solvent B). The elution was carried out with a flux of 1 ml/min, with a gradient whose initial conditions were 15% B, increasing to 30% B 15 min later, isocratic elution for 5 min and increasing to 95% B after 25 min. The detection was carried out at 520 nm.

Colorimetric analysis of jellies

The colorimetric measurements were taken directly from the jellies (2 mm thick) through a Minolta 508i spectrophotometer on a white surface.

Results

Mollar pomegranate

The photocolorimetric analyses indicated that the L* and b* colour components hardly varied in either batch at either of the 2 temperatures assayed (Tables 1 and 2). However, there was a slight decrease in component a*, when stored at 25°C, which means that the fruit turns very brown, and moreso at a higher pH. Furthermore, the lower the pH, the redder the initial colour of the jelly is. When stored at 5°C, no alterations of the initial values were observed.

Table 1. Colour of Mollar pomegranate jelly at 25°C

Time	L*	SD	a*	SD	b*	SD	H
pH 3							
Day 0	32.14	0.20	17.72	0.48	-0.81	0.16	-2.60
Day 53	36.57	0.27	3.92	0.08	2.99	0.19	37.32
pH 3.5							
Day 0	33.09	0.56	14.24	0.22	-0.81	0.05	-3.24
Day 53	31.81	1.34	2.92	0.48	4.34	0.02	56.04
pH 4							
Day 0	35.75	0.06	8.42	0.22	3.23	0.01	20.97
Day 38	30.76	1.75	2.47	0.76	5.99	0.06	67.63

On analysing the results obtained from the anthocyanin analysis, it can be observed that during the processing there is a loss in the concentration of the different anthocyanins (Table 3).

Table 2. Colour Mollar pomegranate jelly at 5°C

Time	L*	SD	a*	SD	b*	SD	H
pH 3							
Day 0	32.14	0.20	17.72	0.48	-0.81	0.16	-2.60
Day 76	33.08	0.28	12.86	1.85	-1.10	0.11	-4.89
pH 3.5							
Day 0	33.09	0.56	14.24	0.22	-0.81	0.05	-3.24
Day 76	31.80	0.47	7.99	0.53	0.19	0.01	1.33
pH 4							
Day 0	35.75	0.06	8.42	0.22	3.23	0.01	20.97
Day 76	32.08	0.25	6.96	0.10	3.96	0.04	29.64

Table 3. Degradation of anthocyanins during the elaboration of the jelly

Anthocyanins	µg/g juice	µg/g jelly
Dp 3,5-dglc	6.66	0.35
Cy 3,5-dglc	34.63	11.61
Pg 3,5-dglc	3.25	0.72
Dp 3-glc	5.56	0.33
Cy 3-glc	42.93	15.20
Pg 3-glc	5.00	1.93
Total	98.03	30.14

The degradation kinetics of the anthocyanins is greater in the jellies whose pH is 4, and is similar in the other two (Tables 4 and 5). However there is an almost total loss of anthocyanins after 53 days when stored at 25°C (Table 4), whereas this loss percentage is never reached when stored at 5°C (Table 5), or in longer storage periods.

Table 4. Alteration of total Mollar pomegranate anthocyanins during storage at 25°C

Time (days)	µg anthocyanin/g jelly		
	pH 3	pH 3.5	pH 4
0	53.29	43.75	30.72
5	30.22	26.33	4.60
17	8.87	10.49	3.62
26	4.94	7.68	3.34
38	1.67	5.01	2.20
53	0.33	0.70	

Borde pomegranate

It is also observed in this variety that preservation at a lower temperature produces less colour alteration, as analysed by photocolorimetry (Table 6).

Regarding the loss of anthocyanin concentration during processing, a degradation is observed (Table 7), as in the Mollar variety, although the final anthocyanin concentration is greater in the Borde variety, due to a greater anthocyanin concentration in this fruit.

Table 5. Concentration of total anthocyanins in Mollar pomegranates at 5°C

Time (days)	μg anthocyanin/g jelly		
	pH 3	pH 3.5	pH 4
0	53.29	43.75	30.72
11	36.36	31.49	11.26
20	51.99	41.29	12.77
32	42.56	37.80	8.93
47	36.99	33.03	7.40
56	26.02	25.27	5.12
76	22.92	18.98	3.80

Table 6. Colour of Borde pomegranate jelly at 25 and 5°C

Time	L*	SD	a*	SD	b*	SD	H
25°C							
Day 0	28.79	0.61	19.55	0.42	-2.47	0.06	-7.19
Day 76	30.68	1.24	7.88	0.13	1.62	0.14	11.62
5°C							
Day 0	28.79	0.61	19.55	0.42	-2.47	0.06	-7.19
Day 53	28.53	0.14	16.64	0.10	-2.81	0.04	-9.59

Table 7. Degradation of anthocyanins during the elaboration of jelly with Borde pomegranates

Anthocyanins	μg/g juice	μg/g jelly
Dp 3,5-dglc	39.05	19.95
Cy 3,5-dglc	41.34	21.44
Pg 3,5-dglc	5.34	0.50
Dp 3-glc	50.40	14.66
Cy 3-glc	66.46	21.96
Pg 3-glc	16.13	0.92
Total	218.72	79.44

During storage there is a loss of total anthocyanin concentration that is much more pronounced in those stored at higher temperatures (Table 8). However the amount of measurable anthocyanins extends up to 76 days when stored at 25°C, unlike the other variety tested.

Discussion of results

Temperature is the factor that fundamentally affects the conservation of the colour of pomegranate jelly during storage, due to the instability of the cation flavilio. This phenomenon has already been described in other foods such as strawberry juice (Bakker *et al.*, 1992).

In our particular case in jelly making, approximately 50% of total anthocyanins present in pomegranate juice is lost in both varieties. Likewise it can be observed that jellies kept at 5°C suffer less pigment degradation. However it is noteworthy that, although the degradation kinetics is similar in both varieties at 5°C, when the jellies made with Mollar are at pH 3 or 3.5, and storage is at a higher temperature, losses are quicker. Likewise, the Mollar variety presents a quicker degradation when jellies are made with non-acidified juices (pH 4). Therefore, the acidification of the juice produces a pronounced improvement in the colour of these products, both initially and during storage. This was expected, bearing in mind the great influence exerted by the pH in shifting the equilibrium towards the cationic form of the anthocyanin.

Table 8. Loss of total anthocyanins during the storage of jelly made with Borde pomegranates

Time (days)	μg anthocyanins/g jelly	
	Borde (25°C)	Borde (5°C)
0	79.44	79.44
11	53.73	83.80
20	31.82	95.14
32	12.61	85.48
47	4.32	69.65
56	2.56	49.02
76	1.15	37.61

However, during storage, certain colour differences can be observed, which indicates that the pH is not the only factor responsible for this characteristic. For example, in jellies made with Mollar pomegranates and stored at 25°C, the batch with a pH of 3.5 is more stable. However, visually, the batch with a pH of 3 presents the most attractive colour. This point is confirmed by the parameters CIEL*a*b*.

Finally, it has been proved that the acidification of the Mollar pomegranate juice brings the results closer to the jelly made with Borde pomegranates, although the latter variety provides products that are redder and more stable.

The explanation of the differences found should be sought, not in the composition of anthocyanins (the same for both varieties), but in the presence of other chemical or biochemical compounds that may influence the degradation of pigments.

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