

Preparation and characterization of jellies with reduced sugar content from date (*Phoenix dactylifera* L.) and lemon (*Citrus limon* L.) by-products

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Preparation and characterization of jellies with reduced sugar content from date (*Phoenix dactylifera* L.) and lemon (*Citrus limon* L.) by-products.

Abstract — Introduction. The increase in diabetes and obesity has increased the demand for reduced sugar products such as jams and jellies. Four jelly formulations were prepared using date juice which was enriched with pectin and lemon flavors. **Materials and methods.** Reduced quantities of sugars (45% and 55%) were added to the juice at different pH (3 and 3.5). The prepared jellies were evaluated for physico-chemical and sensory properties. **Results and discussion.** The water activity values for jellies ranged between 0.767 and 0.804, making them safe from the development of the majority of bacteria. The addition of less quantity of sugar, as well as the decreased pH, resulted in significantly firmer jellies, with higher adhesiveness, chewiness and cohesiveness. Sensory evaluation showed that the prepared jellies averaged 4.17–5.47 and 4.59–5.67 for taste and firmness, respectively, in a 7-point hedonic scale consumer acceptance study. The most appreciated jellies were those prepared with the lowest sugar content, with a slight preference for that with a pH of 3.5. Significant differences were not found between scores for the other sensory attributes (color, transparency, brightness, odor and springiness).

Tunisia / *Phoenix dactylifera* / *Citrus limon* / by-products / fruit products / jams / carbohydrate content / reducing sugars / quality

Préparation et caractérisation de gelées à teneur en sucre réduite, obtenues à partir de sous-produits de datte (*Phoenix dactylifera* L.) et de citron (*Citrus limon* L.).

Résumé — Introduction. L'augmentation des cas de diabète et d'obésité a augmenté la demande pour des produits pauvres en sucre dans les confitures et gelées. Quatre formulations de gelée ont été préparées en utilisant du jus de datte enrichi en pectine de citron. **Matériel et méthodes.** Des quantités réduites de sucres (45 % et 55 %) ont été ajoutées au jus étudié, ajusté à des pH différents (3 et 3,5). Les propriétés physico-chimiques et sensorielles des gelées préparées ont été évaluées. **Résultats et discussion.** Les valeurs d'activité de l'eau des gelées ont varié de 0,767 à 0,804, les protégeant du développement de la plupart des bactéries. L'addition de sucre en quantité moindre, aussi bien que la diminution du pH, a eu pour conséquence d'obtenir des gelées sensiblement plus fermes, avec une adhérence, une masticité et une cohésivité plus élevées. Lors d'une étude hédonique d'acceptation du consommateur évaluée par une échelle de 7 points, l'évaluation sensorielle a montré que les gelées préparées obtenaient, respectivement, une moyenne de 4,17 à 5,47 et de 4,59 à 5,67 pour les caractéristiques de goût et fermeté. Les gelées les plus appréciées ont été celles préparées avec la plus basse teneur en sucre, avec une légère préférence pour celle ayant un pH de 3,5. Des différences significatives n'ont pas été trouvées entre les scores des autres caractéristiques sensorielles (couleur, transparence, teinte, odeur et élasticité).

Tunisie / *Phoenix dactylifera* / *Citrus limon* / sous-produit / produit à base de fruits / confiture / teneur en glucides / sucre réducteur / qualité

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Received 26 January 2009
Accepted 16 June 2009

Fruits, 2010, vol. 65, p. 21–29
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DOI: 10.1051/fruits/2009038
www.fruits-journal.org

RESUMEN ESPAÑOL, p. 29

1. Introduction

Dates (*Phoenix dactylifera* L.) are considered as a major fruit crop in Tunisia with a production of about 0.12 Mt in 2007¹. In addition, Tunisia ranks first as an exporter of dates in value, with a predominance of the Deglet Nour variety, constituting about 60% of the total production. About 30 000 t of dates are lost per year, during picking, manufacture or storage. These by-products, classified as second grade dates, are generally discarded or partially integrated into animal feed [1].

A few studies have been carried out to develop new processed products such as jams [2, 3], jellies [4–6], chutney [7] and other applications [8, 9]. Results indicated that, globally, these products possessed good quality attributes and were well accepted by consumers.

However, the majority of these products had high content of sugar. Due to consumer preferences for low sugar content products for health reasons, there is an increased interest in food products with low or reduced sugar content [10–12].

Food products with reduced sugar content had dissolved solids below 55% (35% to 55%) compared with reference food [13, 14]. An alternative for gel production with lower quantities of sucrose is to use low methoxyl pectin in the presence of calcium [15].

On the other hand, like dates, lemon (*Citrus limon* L.) production in Tunisia is also very significant. In fact, it reached nearly 31 000 t in 2007¹. Citrus juice industries generate a considerable quantity of by-products, reaching about 50% of the original quantity of the whole fruit [16].

These by-products are rich in many natural compounds such as flavonoids [17], acids [18], aromatic compounds which confer upon the fruit its special flavor [19], and fiber, especially pectin [20]. Pectin is widely used in the food industry as a thickening and gelling agent for the preparation of jams and jellies [21].

Consequently, it could be interesting to valorize lemon by-products which could be

used as a source of pectin and flavor, for the preparation, without commercial pectin addition, of date-lemon jellies with reduced sugar content.

In our study, a new process was developed on a laboratory scale for the preparation of date-lemon jellies by date juice enrichment of lemon pectin and flavors. Jellies with reduced sugar content (45% and 55%) were prepared at different pH (3 and 3.5), and analyzed for their physico-chemical and sensory properties.

2. Materials and methods

2.1. Materials

2.1.1. Origin of raw materials

Our study was conducted on two abundant by-products in Tunisia: second grade dates and lemon by-products.

Second grade dates (*Phoenix dactylifera* L.) of the Deglet Nour variety were provided by the National Institute of Arid Zones (Degach, Tunisia). They were collected at the “Tamr” stage (full ripeness). Dates were pitted, washed in running tap water and dried for 12 h in a drying oven at 45 °C. The collected pulp was milled to obtain date paste.

Lemon by-product (*Citrus limon* L.) was supplied by a fruit beverage industry (Zina, Sfax, Tunisia). It was composed of mixed varieties from the Nabeul region (Tunisia). Pips were removed and the remaining material (pulp and peel) was lyophilized, milled and sieved (60-mesh size screen). The powder obtained was stored at 20 °C for further analysis.

2.1.2. Date juice preparation

The date juice was prepared by adding water to date paste at a ratio of 3:1 (v/w) as described earlier [22]. The preparation was cooled at room temperature and then stored at –20 °C until use.

2.1.3. Date juice enrichment

Date juice has been reported to have a low content of pectin (~ 0.17% on a fresh weight basis) [23]. As a solution, a pectin extraction

¹ FAOSTAT, Agricultural data, Agricultural production indices, <http://www.faostat.fao.org/>, 2008.

process from lemon by-products was attempted, using acidified date juice as an extraction solution [23]. The extraction was carried out at 84.34 °C, pH 2.8 for 3 h 34 min. These conditions were found to be suitable for the recovery of an optimal amount of pectin [23]. The obtained pectin is a mixture of date and lemon pectins. This pectin mixture was analyzed and found to be low methoxyl (LM) pectin (unpublished observations). The obtained slurry was filtered through cheesecloth and centrifuged at 20 °C for 30 min at 7 000 rpm to remove solid particles. This pectin extraction process was repeated several times. The obtained supernatants were collected, homogenized and stored at -20 °C until use. Because of the extraction process, the extraction juice was enriched with lemon's pectin and flavors. This juice could be used for the preparation of date-lemon jellies.

2.1.4. Determination of pectin and sugar contents of the extraction juice

Pectin content of the extraction juice was determined by the colorimetric method as described by Englyst *et al.* [24]. Total sugar content was determined using the dinitrosalicylic acid (DNS) method [25] after hydrolysis at 100 °C [26].

2.2. Preparation of jellies

Four jelly formulations were prepared at pH 3 (J₁, J₃) or 3.5 (J₂, J₄) and with 45% (J₁, J₂) or 55% (J₃, J₄) of sugar (fresh basis).

Taking into account the initial content of sugar of the extraction juice, the required quantity was added to this juice to have the fixed final concentrations of sugar (45% or 55%). First, sugar was added to the extraction juice and dissolved at room temperature. Then, pH was adjusted to the desired value using sodium citrate solution (30%). The mixture was heated. When the temperature reached 80 °C, calcium chloride solution (2%) was added, giving a concentration of 0.1 g of calcium·g⁻¹ of pectin. The mixture was boiled for 5 min. Finally, hot jelly was poured into glass jars with screw caps and stored at 4 °C until use.

2.3. Analysis of jelly

Dry matter [27], water activity and total soluble solids were determined using standard methods.

2.3.1. Color study

The CIELAB coordinates (L^* , a^* , b^*) of the jellies were read with a spectrophotometer MS/Y-2500 (HunterLab, In., Reston, VA, USA), calibrated with a white tile. In this coordinate system, the L^* value measures lightness, ranging from 0 (black) to +100 (white). The a^* value ranges from -100 (green) to +100 (red), and the b^* value ranges from -100 (blue) to +100 (yellow). The hue angle (h^*_{ab}) and chroma or intensity (C^*) were calculated according to the following equations: $h^*_{ab} = \tan^{-1}(b^*/a^*)$ and $C^* = [(a^{*2} + b^{*2})^{-2}]$.

2.3.2. Textural analysis

Texture properties of jellies were determined by a texture profile analysis (TPA) test. A texture analyzer (LLOYD instruments, Fareham, UK) was used to measure the force-time curve for a two-cycle compression. All measurements were carried out at 25 °C. Jellies were poured into plastic food containers to a height of 25 mm. Samples were compressed using a cylindrical probe (25 mm in diameter) at 50% deformation with a displacement speed of 25 mm·min⁻¹ and a trigger detection force of 0.005 kg strength. Then, the probe was returned to its original position, followed by a second "down and up" cycle on the same sample. Texture parameters were calculated by the texture "Nexygen plot" software connected to the instrument. Tests were performed in three replicates.

2.3.3. Sensory evaluation

Jellies were served in four glass containers, coded with random three-digit numbers. Hedonic evaluation was carried out by an untrained panel of 36 subjects (21 females and 15 males). These panelists were students and staff members of the National School of Engineering (Sfax, Tunisia). They were asked to evaluate the jellies for color, transparency, brightness, odor, taste, firmness and springiness. Samples were evaluated

Table I.

Physico-chemical characteristics of date-lemon jellies processed from date (*Phoenix dactylifera* L.) and lemon (*Citrus limon* L.) by-products (Tunisia).

Treatment	Jelly formulation		Dry matter (%)	Water activity	Total soluble solids (°Brix)
	pH	Sugar (%)			
J ₁	3.0	45	46.12 ± 0.37 a	0.792 ± 0.01 a	37.50 ± 0.00 a
J ₂	3.5	45	45.06 ± 0.51 a	0.804 ± 0.01 b	36.66 ± 0.11 b
J ₃	3.0	55	52.41 ± 0.26 b	0.767 ± 0.01 c	42.75 ± 0.25 c
J ₄	3.5	55	51.77 ± 0.42 b	0.772 ± 0.01 d	41.75 ± 0.00 d

Means followed by the same letter within a column are not significantly different ($\alpha = 0.05$).

based on a seven-point hedonic scale, from 1 (very disliked) to 7 (very liked). Sample presentation order was determined following a randomized design. Panelists were instructed to evaluate from left to right, and to rinse their mouths twice with mineral water between samples to minimize residual flavor effects. "Global appreciation" was calculated as the mean values for all the sensory characters.

2.4. Statistical analysis

All the analyses were performed in three replicates. Values of different parameters were expressed as the mean ± standard deviation. Statistical analysis was performed using the Statistical Package for the Social Sciences, "SPSS" (version 11). The Duncan test was performed to evaluate the significance of differences between mean values at the level of $P < 0.05$, except for sensory results which were analyzed at a level of $P < 0.01$.

3. Results and discussion

3.1. Pectin and sugar contents of the extraction juice

Chemical analysis showed that the extraction juice contained ~ 0.71% (± 0.06) of pectin (fresh basis). This result indicated that the extraction juice was enriched in pectin (increased from 0.17% to 0.71%, fresh basis).

Total sugar content was ~ 14.77% (± 0.83), on a fresh basis. Thus, sucrose

must be added to the extraction juice to reach the desired level (45% or 55 %).

3.2. Compositional and functional characteristics of the prepared jellies

3.2.1. Physico-chemical characteristics

The results regarding the physico-chemical attributes of the prepared jellies revealed that total soluble solids ranged from 36.66 °Brix to 42.75 °Brix, respectively, for J₂ and J₃ (table I). These data situated between 35 °Brix and 55 °Brix indicate that the elaborated jellies could be considered as reduced sugar jellies [13, 14].

Water activities varied from 0.767 to 0.804, respectively, for J₃ and J₂. Based on these values, the jelly samples can be classified as intermediate moisture products. Since their water activities are within the safe level (< 0.86), they will be safe from development of the majority of bacteria [28]. This could be explained by the presence of sugar and pectin. In fact, these ingredients are able to bind water, making it unavailable for microbial growth [29].

In addition, water activity was lower for jellies with higher content of sugar. Similar results have been reported on date jam samples by Besbes *et al.* [3].

3.2.2. Color of the jellies

Color and appearance constitute the first attribute considered by the consumer to judge the acceptability and overall quality of food [30, 31].

Table II.

CieLab coordinates (L^* , a^* , b^* , h^*_{ab} , C^*_{ab}) of date-lemon jellies processed from date (*Phoenix dactylifera* L.) and lemon (*Citrus limon* L.) by-products (Tunisia) ($n = 3$).

Treatment	Jelly formulation		CieLab coordinates				
	pH	Sugar (%)	L^*	a^*	b^*	h^*_{ab}	C^*_{ab}
J ₁	3.0	45	51.62 ± 0.05 a	4.00 ± 0.08 a	30.05 ± 0.03 a	82.40 ± 0.15 a	30.31 ± 0.04 a
J ₂	3.5	45	49.29 ± 0.03 b	6.83 ± 0.01 b	36.60 ± 0.07 b	79.41 ± 0.04 b	32.25 ± 0.07 b
J ₃	3.0	55	49.80 ± 0.20 c	6.43 ± 0.07 c	32.60 ± 0.36 c	78.84 ± 0.00 c	33.22 ± 0.36 c
J ₄	3.5	55	49.67 ± 0.06 bc	7.04 ± 0.06 b	31.48 ± 0.20 d	77.38 ± 0.03 d	37.23 ± 0.21 d

Means followed by the same letter within a column are not significantly different ($\alpha = 0.05$).

Table III.

Texture properties of date-lemon jellies processed from date (*Phoenix dactylifera* L.) and lemon (*Citrus limon* L.) by-products (Tunisia).

Treatment	Jelly formulation		Firmness (N)	Adhesiveness (N)	Chewiness (N·mm)	Cohesiveness	Springiness (mm)
	pH	Sugar (%)					
J ₁	3.0	45	2.96 ± 0.17 a	0.77 ± 0.03 a	9.12 ± 0.33 a	0.25 ± 0.00 a	11.81 ± 0.17 a
J ₂	3.5	45	2.51 ± 0.21 b	0.71 ± 0.04 a	8.38 ± 0.55 a	0.28 ± 0.00 b	11.77 ± 0.11 a
J ₃	3.0	55	1.65 ± 0.04 c	0.50 ± 0.00 b	5.80 ± 0.25 b	0.29 ± 0.01 b	11.57 ± 0.37 a
J ₄	3.5	55	1.48 ± 0.04 c	0.44 ± 0.03 b	5.20 ± 0.56 b	0.29 ± 0.01 b	11.71 ± 0.36 a

Means followed by the same letter within a column are not significantly different ($\alpha = 0.05$).

The CieLab coordinates measured for the four jellies prepared (*table II*) show that samples had hue angles (h^*_{ab}) ranging between 77.38 and 82.40 for J₄ and J₁, respectively. These jellies had a yellowish brown color, indicating the presence of pigments from both date juice and lemon by-products. The J₂, J₃ and J₄ jelly formulations were darker in color than J₁, as shown by lower L^* . The lightness of J₁ indicates that this sample was much brighter in color than the other jellies. This could be attributed to enzymatic or non-enzymatic browning (Maillard reactions) [32]. In fact, the presence of a higher amount of reducing sugars after inversion of sucrose during cooking, and/or higher pH, could contribute to these browning reactions.

3.2.3. Texture profile analysis of the jellies

The instrument used to analyze the texture profile of the prepared jellies provides two upward positive and two downward negative curves for the samples (*table III*). This test can be considered as an imitation of the mastication operation and may be used to predict the behavior of a semi-solid food in the mouth [3]. The peak force of the first compression is considered as firmness.

The firmness values ranged between (1.48 and 2.96) N, respectively, for J₄ and J₁. At low pH, in the case of gels prepared with low methoxyl pectins, sugar is known to increase gel rigidity by promoting

polymer-polymer interactions rather than polymer-water interactions [33].

However, in our study, firmness was negatively affected by sugar concentration. This could be explained by the difference in the effective pectin concentration for the prepared jellies: samples with lower sugar concentration (J_1 and J_2) contained higher effective pectin content. In fact, we used a higher quantity of extraction juice to prepare these samples. Consequently, because of the high water binding capacity, the increase in pectin concentration in the formulation may contribute to the increased firmness of the finished products. Royer *et al.* reported that both sugar and pectin influenced the textural properties of prepared jellies [34].

On the other hand, pH significantly influenced the firmness of only jellies prepared with 45% sugar. Firmness increased with decreasing pH from 3.5 to 3, with 2.51 N and 2.96 N for J_2 and J_1 , respectively. In fact, in low methoxyl pectin gels, when pH is below 3.5, there is a predominance of non-dissociated carboxyl groups, which leads to more rigid gels [35].

The cohesiveness of products represents how well they resist a second deformation relative to the first one [3]. The J_1 jelly formulation had the lowest cohesiveness (0.25), compared with the other jellies (~ 0.29). This result shows that this product could be more able to exude water during storage.

Adhesiveness is an important parameter for food products. It measures the work necessary to overcome the attractive forces between the surface of the probe and the sample. Consequently, this parameter could predict the degree of adhesion of food on the teeth. The results showed that the jellies with the lowest content of sugar exhibited more adhesiveness (0.77 N and 0.49 N, respectively, for J_1 and J_3), whereas pH did not affect the adhesiveness of jellies.

Chewing properties are very important for the consumers' acceptance of the product. The results were similar to those relative to adhesiveness, with higher values for jellies prepared at the lowest sugar content (9.12 N·mm and 8.37 N·mm for J_1 and J_2 ,

respectively, versus 5.8 N·mm and 5.2 N·mm, respectively, for J_3 and J_4). This could be attributed to their higher pectin content.

All products showed statistically the same springiness, with values around 11.7 mm.

3.2.4. Sensory quality

Sensory tests are still the best method to get an overall impression of a product. Texture is the overall image of the sensory feeling of a product. In our case, it was represented by two parameters: firmness and springiness. Jellies with less total soluble solids (TSS) content were more appreciated by consumers for firmness. Panelists gave a mean score of 5.47 for both the J_1 and J_2 jelly formulations (*table IV*). This score was rated between "quite good" and "good". This result was correlated with that observed in the texture property analysis test (*table III*). In fact, a higher firmness was related to a better appreciation by consumers. However, while the J_4 jelly was found to be less appreciated by panelists, probably because of its soft texture, it was judged as acceptable (firmness score of 4.17).

For springiness, mean scores were statistically the same, with mean springiness scores of 4.64 to 5.33. This result was also in agreement with that found for the texture property analysis test (*table III*).

The results regarding the taste attribute showed that the J_2 jelly was the most appreciated by consumers, followed by the J_1 formulation, with mean scores of 5.67 and 5.42, respectively (*table IV*). These values could be considered as being near the "good" rate. The J_2 jelly had the lowest TSS (36.66 °Brix), and lower acid taste (\sim pH 3.5).

Higher contents of sugar produced sweeter jellies but, apparently, sweet taste was less liked by the consumers, who preferred reduced sugar content jellies.

For color, transparency and brightness, which are visual attributes, panelists' choices seemed to be significantly not affected by differences revealed by color measurement results. The same result was found for odor. Consumers judged the four jellies as having acceptable odor (odor scores of 4.64 to 4.92).

Table IV.

Sensory properties¹ of date-lemon jellies processed from date (*Phoenix dactylifera* L.) and lemon (*Citrus limon* L.) by-products (Tunisia).

Treatment	Jelly formulation		Color	Transparency	Brightness	Odor	Taste	Firmness	Springiness	Global appreciation ²
	pH	Sugar (%)								
J ₁	3.0	45	5.42 ± 1.08 a	5.64 ± 1.15 a	5.56 ± 0.95 a	4.67 ± 1.20 a	5.42 ± 1.32 ab	5.47 ± 1.04 a	5.33 ± 1.31 a	5.35 ± 0.49 a
J ₂	3.5	45	5.33 ± 1.24 a	4.86 ± 1.27 a	5.42 ± 1.18 a	4.92 ± 1.34 a	5.67 ± 1.05 a	5.47 ± 1.30 a	5.14 ± 1.56 a	5.25 ± 0.69 ab
J ₃	3.0	55	5.31 ± 1.07 a	5.22 ± 1.29 a	4.97 ± 1.21 a	4.64 ± 1.29 a	4.78 ± 1.45 b	5.11 ± 1.24 a	4.64 ± 1.99 a	4.81 ± 0.62 c
J ₄	3.5	55	5.33 ± 1.24 a	5.06 ± 1.18 a	5.19 ± 1.22 a	4.72 ± 1.07 a	4.59 ± 1.32 b	4.17 ± 1.64 b	4.97 ± 1.44 a	5.01 ± 0.49 bc

Means followed by the same letter within a column are not significantly different ($\alpha = 0.05$).

¹ Samples were evaluated based on a seven-point hedonic scale, from 1 (very disliked) to 7 (very liked).

² Global appreciation was calculated as the mean values for all the sensory characters.

Thus, the J₁ and J₂ jellies could be considered as the most appreciated by the panelists, which was shown by the “global appreciation” parameter (5.35 and 5.25, respectively, for J₁ and J₂).

4. Conclusion

Our work showed that it was possible to use lemon by-products and date juice for the production of low calorie jellies, using different sugar concentrations and pH.

Physico-chemical analysis revealed that the four jellies prepared had water activities within the safe level (< 0.86), allowing them to be safe from the development of the majority of bacteria. Textural analyses showed that decreasing sugar concentrations led to firmer gels. Adhesiveness, chewiness and cohesiveness were affected only by sugar content. Sensory evaluation revealed that jellies with less quantity of sugar (J₁ and J₂) were the most appreciated by consumers, especially for taste and firmness attributes. These panelists rated them as “quite good” products.

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Preparación y caracterización de jaleas con reducido contenido de azúcar, obtenidas a partir de subproductos de dátil (*Phoenix dactylifera* L.) y de limón (*Citrus limon* L.).

Resumen — Introducción. El aumento de casos de diabetes y de obesidad elevó la demanda para productos pobres en azúcar en las mermeladas y jaleas. Se prepararon cuatro fórmulas de jalea mediante el empleo de jugo de dátil enriquecido en pectina de limón. **Material y métodos.** Al jugo estudiado, ajustado a diferentes pH (3 y 3.5), se le añadieron unas reducidas cantidades de azúcares (45 % y 55 %). Se evaluaron las propiedades físico-químicas y sensoriales de las jaleas preparadas. **Resultados y discusión.** Los valores de actividad del agua de las jaleas variaron entre 0.767 y 0.804, lo que impidió que desarrollaran la mayoría de las bacterias. Tanto la adición de azúcar en menor cantidad como la disminución del pH tuvieron como consecuencia la obtención de jaleas sensiblemente más consistentes, con una adherencia, una masticabilidad y una cohesión más elevadas. En un estudio hedónico de aceptación del consumidor, evaluado mediante una escala de 7 puntos, la evaluación sensorial mostró que las jaleas preparadas obtenían, respectivamente, una media entre 4.17 y 5.47; y, entre 4.59 y 5.67 en cuanto a las características del sabor y de la firmeza. Las jaleas más apreciadas fueron las del menor contenido de azúcar, con una ligera preferencia para la del pH de 3.5. No se encontraron diferencias significativas entre los resultados de las otras características sensoriales (color, transparencia, tinte, olor y elasticidad).

Túnez / *Phoenix dactylifera* / *Citrus limon* / subproductos / productos derivados de las frutas / mermeladas / contenido de carbohidratos / azúcares reductores / calidad

