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Additional Information

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4 **PHYSICOCHEMICAL AND SENSORIAL PROPERTIES OF GRAPEFRUIT JAMS AS**
5 **AFFECTED BY PROCESSING**
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21
22 **Abstract.** Jam is an effective and tasty way of preserving fruit. Jam processing procedures
23 as well as storage conditions and duration are important factors for jam quality. Traditional
24 jam processing involves the application of severe thermal treatments that imply
25 undesirable changes in the product quality characteristics such as colour, texture, flavour
26 and nutritional and functional value. In this work, osmotic dehydration (OD) and/or
27 microwave energy (MW) were proved as adequate to obtain jam with the typical
28 characteristics of water content, °Brix, pH and water activity of jam obtained by
29 conventional thermal heating. The sensory evaluation carried out to compare the product
30 showed that samples submitted to the more intense heating treatments (conventional or
31 MW) were significantly higher scored in colour saturation, brightness, grapefruit taste and
32 extensibility than OD or OD+MW ones. As deduced from the obtained results, OD
33 treatment prevents from grapefruit colour changes and mild MW heating contributes to
34 increase the consistency and decrease of the extensibility of the obtained jam. In this way,
35 OD+MW jam was the preferred by assessors, mainly due to its higher consistency.
36 Sample obtained by this procedure was stable during storage.
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41 **Keywords:** grapefruit, jam, osmotic dehydration, microwaves, consistency, colour,
42 sensory analysis.
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54 **Running Title:** physicochemical and sensorial properties of grapefruit jams
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4 **1. Introduction**

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6 There is an increasing demand by consumers for citrus products as consumption has been
7 recognised as an important factor in reducing the risk of several chronic diseases such as
8 cancer (Poulose et al., 2005; Vanamala et al., 2006), osteoporosis (Deyhim et al., 2006)
9 and cerebrocardiovascular diseases (Sanchez-Moreno et al., 2003; Yu et al., 2005).
10 Grapefruit juices and jams are highly appreciated due to their specific taste, flavour and
11 nutritional value.
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16 Historically, jams and jellies were originated as an early effort to preserve fruit for
17 consumption in the off-season (Baker et al., 2005). In traditional jam manufacture, all the
18 ingredients are mixed in adequate proportions and the mix is concentrated by applying a
19 thermal treatment to reach the required final soluble solids content. Nevertheless, this
20 process also implies an undesirable impact in colour, nutritional value and flavour
21 properties due to the high temperature reached in the cooking process. An alternative for
22 jam formulation is to use dehydrated fruit. In this sense, osmotic dehydration enables us to
23 produce fruit products that have good flavour and nutritional content (Shi et al., 1996;
24 García-Martínez et al., 2002). On the other hand, microwave energy has been proposed
25 as an alternative to traditional heat processing in order to better preserve the natural
26 organoleptic characteristics and essential thermolabile nutrients of fresh fruit (Nikdel et al.,
27 1993; Cañumir et al., 2002; Igual et al., 2010a).
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37 Currently, consumers demand the maximum preservation of the endogenous sensory,
38 nutritional and health-related quality of fruit products. An attractive colour is one of the
39 most important characteristics for the jam processing industry, in addition to a typical fruit
40 flavour and convenient jam consistency. The recipe, processing procedures, storage
41 conditions and stability are important factors for jam quality (Wicklund et al., 2005). During
42 processing or storage the characteristic fruit pigments can suffer degradation reactions
43 that result in browning or loss of typical fruit colour. Colour stability of fruit products may be
44 affected by temperature, pH, oxygen, sugar content, ascorbic acid and metals (Dervisi et
45 al., 2001).
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52 Traditionally, jam glass or plastic jars are stored at room temperature before they are
53 opened. Low temperature is generally not regarded as necessary to prevent degradation,
54 as during processing, in addition to thermal treatment, the jam is added both preservatives
55 and sugar and the pH of the product is usually low. The shelf life of jam is normally 6–12
56 months (Wicklund et al., 2005). However, in the case of jams produced without heat
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4 treatment, refrigeration storage is necessary to avoid the development of fermentation
5 reactions (García-Martínez et al., 2002).

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7 The objective of this study was to evaluate how physicochemical (pH, soluble solids,
8 colour and consistency) and sensorial properties of grapefruit jam are influenced by
9 processing (osmotic dehydration, microwave energy and conventional heating) and
10 storage.
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15 **2. Materials and methods**

16 17 18 19 *2.1. Raw materials*

20 21 *2.1.1. Fruit*

22 Grapefruit (*Citrus paradise* var. Star Ruby) from Murcia in Spain was purchased from a
23 local supermarket. Fruit pieces were peeled and cut perpendicularly along the fruit axis,
24 into 10 mm thick half slices.
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28 29 *2.1.2. Sucrose and osmotic solution*

30 Food grade commercial sucrose was used to prepare conventional and microwave (MW)
31 jams. In the case of jam obtained by osmotic dehydration (OD), an osmotic solution (OS)
32 was prepared by mixing an amount of sucrose with distilled water until it was completely
33 dissolved and forming a 65 °Brix syrup.
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38 39 *2.1.3. Gelling agent*

40 Citrus peel pectin (60% degree of esterification, Fluka Biochemika, Switzerland) was used
41 as a gelling agent.
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45 46 *2.2. Jam preparation procedures*

47 48 49 *2.2.1. Conventional process*

50 Conventional jam was obtained by mixing fresh fruit and sugar in a 67:33 ratio. Half slices
51 of peeled grapefruit (500 g) were precooked at 85 °C for 10 min. After the addition of sugar
52 (250 g) and 100 ppm of potassium sorbate (Panreac, Barcelona, Spain), the mixture was
53 cooked for a further 20 min at 95-100 °C to reach a 40-60 °Brix product as described in the
54 Spanish quality norm for fruit jam approved by RD 670/1990 (BOE N° 130, 1990). The
55 process was carried out in an electrical food processor (Thermomix TM 21, Vorwerk,
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Spain). The jam was placed in sterile glass jars and stored at room temperature for 24 h until analysis.

2.2.2. Microwave process (MW)

A household microwave (Moulinex 5141 AFW2, Spain) was used to produce the MW jam, mixing fresh fruit and sugar in a 67:33 ratio. Half slices of peeled grapefruit (500 g) were precooked at 900 W for 5 min. After the addition of sugar (250 g) and 100 ppm potassium sorbate, the mixture was cooked at 900 W for a further 10 min to reach 40-60 °Brix (BOE Nº 130, 1990). The jam was placed in glass jars and stored at room temperature for 24 h until first analysis.

2.2.3. Osmotic process (OD)

According to a previous kinetic study (Iguar et al., 2010b), half-slices of grapefruit were osmo-dehydrated in the OS at 40 °C for 3 h reaching ≈30 °Brix. Osmo-dehydrated samples were ground together with part of the OS to obtain jam with 60 g fresh fruit/100 g jam, 100 ppm potassium sorbate, and 1% of pectin as a gelling agent. Jams were placed in glass jars and stored at room temperature for 24 h until first analysis.

2.2.4. Combined osmotic-microwave process (OD+MW)

Jams obtained from osmo-dehydrated grapefruit, as described in Section 2.2.3, were cooked at 900 W for 5 min. New jams were placed in glass jars and stored at room temperature for 24 h until first analysis.

2.3 Storage conditions

The jams were stored for 3 months at room temperature, except the OD one which was stored at 4 °C, according to previous studies (García-Martínez et al., 2002). Analyses were carried out after 1, 7, 15, 30, 45, 60, 75 and 90 days of storage.

2.4. Analysis

2.4.1. Physicochemical properties

Moisture content (x_w , g water/g product), °Brix (g soluble solids/100 g liquid phase) and water activity (a_w) were determined for fresh grapefruit and all the formulated jams. The x_w was determined by drying the sample to a constant weight at 60 °C in a vacuum oven

(AOAC method 934.06, 2000). °Brix were measured in previously homogenised samples with a refractometer at 20 °C (Zeiss, ATAGO model NAR-3T refractometer, Japan). A dew point hygrometer (FA-st Lab, GBX, France) was used to analyze a_w and pH was measured with a CRISON pH-meter. Each analysis was carried out in triplicate.

2.4.2. Colour measurement

Colour values were obtained from the reflection spectrum (Minolta, CM 3600D, Tokyo, Japan). CIE-L*a*b* uniform colour space was selected to calculate colour coordinates, where L* indicates lightness, a* indicates chromaticity on a green (-) to red (+) axis and b* chromaticity on a blue (-) to yellow (+) axis. Colour coordinates were obtained from a 10° observer and D65 illuminant. The colour coordinates were then used to calculate the hue angle (Equation 1), chrome (Equation 2) and total colour differences (Equation 3) with respect to the fresh grapefruit sample or conventional jam.

$$h_{ab}^* = \arctan \frac{b^*}{a^*} \quad (1)$$

$$C_{ab}^* = \sqrt{a^{*2} + b^{*2}} \quad (2)$$

$$\Delta E = \sqrt{\Delta L^{*2} + \Delta a^{*2} + \Delta b^{*2}} \quad (3)$$

2.4.3. Consistency

The flow distance of a controlled sample weight for a constant time was measured using a Bostwick consistometer. This device consists of a level stainless-steel trough divided into two compartments. The first compartment initially containing the sample (5x5x3.8 cm) is separated from the second by means of a spring-loaded gate. The second compartment is a trough 5 cm wide, 24 cm long, and about 2.5 cm high and has a series of parallel lines drawn across the floor at 0.5 cm intervals. Once the gate is opened, the distance the sample flows in 30 s was measured (Bourne, 1982). The parameter used to characterise the consistency of the samples was the distance advanced by the samples in the consistometer related to the weight of the samples (mm/g).

2.5. Sensory evaluation

A panel of 50 tasters carried out a sensory analysis of the jams. The age of the panellists ranged from 20 to 50 years. This analysis consisted of two sessions, the first session was a differentiation test by pairs and the second session was a preference test by pairs (UNE-

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4 EN ISO 5495). The attributes evaluated at the first session were colour saturation,
5 luminosity, brightness, smell, body or consistency, product coverage in mouth, extensibility
6 and grapefruit flavour. During the test sessions, panellists worked in individual booths.
7 Samples were served at room temperature in transparent plastic glasses coded with
8 random three digit numbers. Panellist tasted approximately the same amount of each
9 sample and mineral water was provided to the assessors to rinse their mouths.
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15 16 *2.6. Statistical analysis*

17 Analysis of variance (ANOVA), with a confidence level of 95% ($p < 0.05$), using Statgraphics
18 Plus 5.1 Software (Statistical Graphics Corporation, USA), was applied to evaluate the
19 differences among treatments. Friedman analysis for the pairwise ranking test was
20 undertaken on the data of each taster to know in which attribute the samples showed
21 significant differences (Meilgaard et al., 1999). The significance of these differences was
22 determined by applying Tukey's HSD (Honestly Significance Difference) as a multiple
23 comparison procedure (Meilgaard et al., 1999). Correspondence analysis (CA) was
24 applied to the sensorial results using SPSS program version 16.0. Correlations between
25 sensory and instrumental data were determined using the Pearson correlation coefficient
26 (r).
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36 **3. Results and discussion**

37 38 39 *3.1. Effect of treatment on the physicochemical properties of the obtained jams*

40 Table 1 shows the physicochemical parameters of fresh grapefruit and Conventional, OD,
41 OD+MW and MW jams. Fresh grapefruit presented the characteristic physicochemical
42 parameters shown in the bibliography for grapefruit (Moraga et al., 2009) and grapefruit
43 juice (Iguar et al., 2010). The range of °Brix of formulated jams was between 46 and 48.5,
44 with the lowest values for jams obtained by OD and OD+MW. These jams (OD, OD+MW)
45 showed significantly higher pH values than the other samples. The lowest values of a_w and
46 x_w were found in the conventional jam, 0.922 ± 0.003 and 0.526 ± 0.002 , respectively.
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51 The colour coordinates, h_{ab}^* and C_{ab}^* of the jams appear in Table 2. The colour differences
52 of jam in relation to the fresh fruit sample and to conventional jam also appear in this table.
53 There were significant ($p < 0.05$) differences between fresh fruit and the jams. The values of
54 the coordinates L^* , a^* , b^* , hue angle and chrome were greater in the case of fresh fruit. MW
55 jam presented values closer to the raw material than the others and the lowest values of a^*
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4 and b* were those of the OD jams. The hue angle of OD+MW and MW showed no
5 significant ($p>0.05$) differences. Neither were there significant ($p>0.05$) differences in the
6 hue of conventional and OD samples. In addition, jams that were subjected to heat
7 treatment by conventional or microwave treatment showed the lowest L* values, as
8 observed by Igual et al., (2010b). The OD samples showed the lowest chrome values. OD
9 jam showed the smallest total colour differences in comparison to fresh grapefruit, while
10 MW and OD+MW showed the smallest colour difference when compared to the
11 conventional samples. Nevertheless, as ΔE among jams was \leq than 3 units in all the
12 cases, differences no noticeable to the human eye exist among them (Bodart et al., 2008).
13 The values of flow distance related to the sample weight (mm/g) appear in Table 2. The
14 conventional sample was the least consistent (highest distance advanced). Jams obtained
15 by the application of microwaves showed higher ($p<0.05$) consistency than conventional
16 jam, probably as a consequence of the higher pectin solubilisation occurred with this
17 process (Contreras et al., 2008). OD showed the same consistency as MW. In this case,
18 this consistency was achieved due to added pectin. The thickest ($p<0.05$) jam was
19 obtained when combined OD+MW was applied.
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32 33 *3.2. Sensory analysis*

34 Figure 1 shows the scores sum of each jam for each evaluated attribute. Regarding the
35 Friedman's T test results, colour saturation, brightness, body or consistency, extensibility
36 and grapefruit taste were the attributes that showed statistical significant differences
37 ($\alpha=0.05$) in the studied samples. Friedman's T value for these attributes were 110.5, 81.2,
38 51.8, 9.2 and 32.1, respectively, being 7.81 the theoretical T value ($\alpha=0.05$). The HSD
39 method was used to perform a multiple comparison among the treatments. The calculated
40 Tukey's HSD value according to assay conditions was 25.67. Table 3 shows the
41 differences between the sum of ranks for each pair of samples for the attributes that
42 showed significant differences among samples as deduced from Friedman's T test. OD
43 was the jam with the lowest values of colour saturation followed by OD+MW. The score of
44 this attribute was greatest for MW and conventional jams, with no differences between
45 them. OD and OD+MW were perceived with lower brightness and grapefruit taste than
46 MW and conventional jams. As regard to the textural attributes, only OD and conventional
47 samples presented significant differences in extensibility, the last one being scored as
48 more extensible. MW and conventional jams were scored with lower consistency than
49 MDO and MDO+MW. The added pectin in these samples could justify these results. No
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4 significant differences were detected among samples in the rest of the attributes
5 evaluated.
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8 As regards to the results of the preference test, score obtained by OD+MW jam was 108,
9 OD was 79, MW 63 and the conventional one 50. These differences were statistically
10 significant (Friedman's $T = 37.9$). As it can be observed in Table 3, the jam OD+MW was
11 the preferred by the assessors among any of the others. It was also significant the
12 preference of OD jam when compared to conventional one.
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16 To relate the jams obtained by the different treatments with the attributes evaluated and
17 the preferences of the assessors, a component analysis was carried out. From this
18 analysis, two dimensions that explain 97.2% of the variability of results were obtained. The
19 first dimension explained 89.3% of the variability, while the second explained 7.9%. Table
20 4 shows that both, the jams and the attributes, were well represented along the first two
21 dimensions, since high values were obtained for the sum of the relative contributions in all
22 cases. Figure 2 shows the projection in the plane of the jams and attributes. According to
23 the distribution of attributes and samples in the plane, there were four groups that
24 characterised the jam as follows: MW was identified with higher colour saturation attribute;
25 conventional jam with grapefruit taste, smell, brightness and extensibility attributes; OD
26 with luminosity and product coverage in mouth; and finally, OD + MW was identified with
27 consistency attribute and was also the preferred jam. Furthermore, it can be observed that
28 body or consistency was closely related to the preference of the judges, since they were
29 very near in representation. This shows that consistency has considerable influence on
30 jam acceptance by assessors.
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42 *3.3. Relationship between instrumental and sensory data*

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44 Pearson analysis was used to establish correlations between colour and textural data
45 obtained by instrumental and sensory methods (Table 5). As expected, the results showed
46 a significant positive correlation between sensory luminosity and parameter L^* . However,
47 no significant correlation was found between sensory colour saturation and C^*_{ab} . This has
48 been also observed in other study referred to a vanilla dairy dessert (Tárrega & Costell,
49 2007) and reflects the difficulties for judges in identify the meaning of colour saturation.
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51 With respect to flow distance (mm/g) of studied jams, the only textural attribute that
52 showed a significant correlation was the product coverage in mouth, the greater the flow
53 distance, the lower the product coverage in mouth.
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3.4. Evolution of colour and consistency of obtained jams during storage

Figure 3 shows the colour parameters (L^* , a^* , b^* , C^*_{ab} and h^*_{ab}) of jams stored for three months. In general, in all studied jams, L^* values were maintained during storage. Moreover, a^* , b^* and C^*_{ab} were stable in OD and OD+MW samples, whilst these parameters in MW and Conventional jams decreased until 60 days and then remained stable. When the hue angle results were analysed, OD and OD+MW samples showed no significant changes during storage, while MW and Conventional jams showed a significant increase in the first month, related to a change to a less red more orange colour that remains then stable over time. If the global colour changes (ΔE) occurred at the end of storage as related to the newly processed jams are calculated, using the conventional equation proposed to this end ($\Delta E = (\Delta L^* + \Delta a^* + \Delta b^*)^{1/2}$), the obtained values are 2,3 units for MW, 2 for OD, 1 for OD+MW and 0,5 for MW samples. All this values are lower than 3 units, the limit proposed by Bodart et al., (2008) below which colour difference between samples is not noticeable to the human eye.

The evolution of the flow distance corrected by the sample weight of jams stored for three months is shown in Figure 4. The storage time in the OD jam did not show a significant effect. However, in the other samples, the consistency increased (flow distance decreased) significantly ($p < 0.05$) from day 75 in OD+MW and Conventional samples and during the first week in MW jam. By the end of the storage period, MW sample was the most consistent (flow distance 0.16 ± 0.02 mm/g) and the one that showed the greatest increase in the consistency, with an increase in this parameter when compared to sample newly processed of 76 %.

4. Conclusion

The different processes applied to obtain jams can produce products with similar values of water content, °Brix, pH and water activity. In general, when any of the samples submitted to the more intense heating treatments (conventional or MW) were compared with OD or OD+MW ones, significant sensory differences were observed in colour saturation, brightness, grapefruit taste and extensibility, all of them being higher scored in the former. OD+MW sample was the preferred one, mainly due to its higher consistency and the considerable influence of this attribute on jam acceptance by assessors. Sample obtained by this procedure was stable during storage. From the results obtained in this study, intense thermal treatments in jam processing should be recommended to be avoided,

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4 although mild heating is recommendable as it contributes to a desired increase in the
5 consistency and decrease of the extensibility of the product.
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8 9 **Acknowledgment**

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TABLE CAPTIONS

Table 1. Mean values (and standard deviation) of °Brix, water activity (a_w), moisture content (x_w) and pH of formulated jams and fresh grapefruit.

Table 2. Mean values (and standard deviation) of colour parameters and flow distance of formulated jams and fresh grapefruit.

Table 3. Differences between the sum of ranks for each pair of samples for statistically significant attributes

Table 4. Contribution of dimension to inertia of attributes and jams.

Table 5. Pearson's correlation coefficients between instrumental and sensory parameters.

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FIGURE CAPTIONS

Figure 1. Score of the different sensory attributes evaluated in grapefruit jams.

Figure 2. Correspondence analysis. Representation of attributes and samples tested in plain with two dimensions.

Figure 3. Evolution of colour parameters (CIE L*a*b* coordinates, chrome, C*_{ab}, and hue angle, h*_{ab}) of jams stored for three months.

Figure 4. Evolution of the flow distance of jams stored for three months, measured in Bostwick consistometer and corrected for the sample weight.

Figure 1

[Click here to download Figure: Figure 1.doc](#)

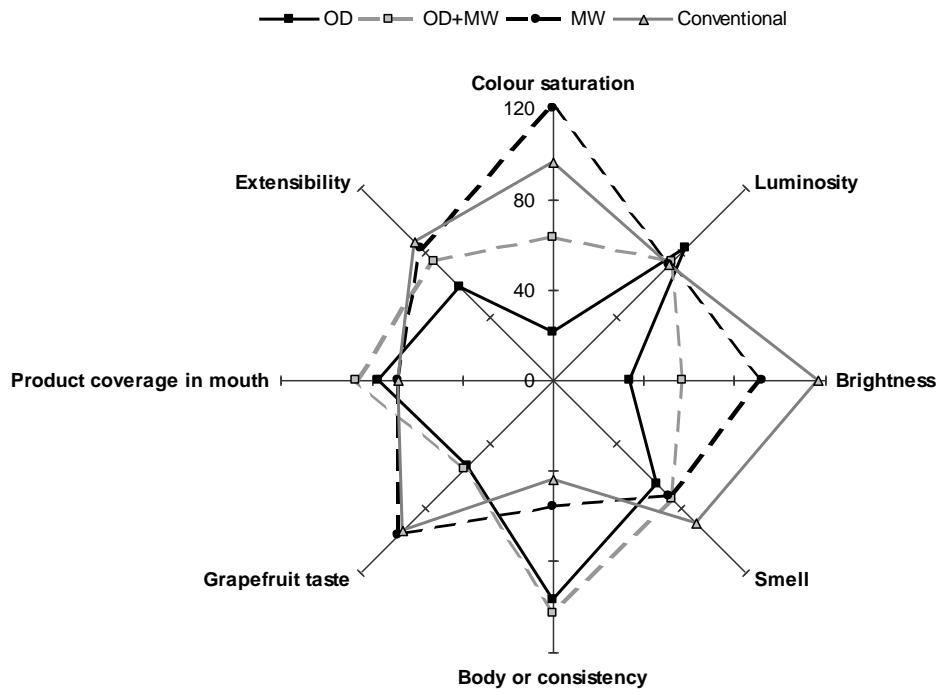


Figure 1.

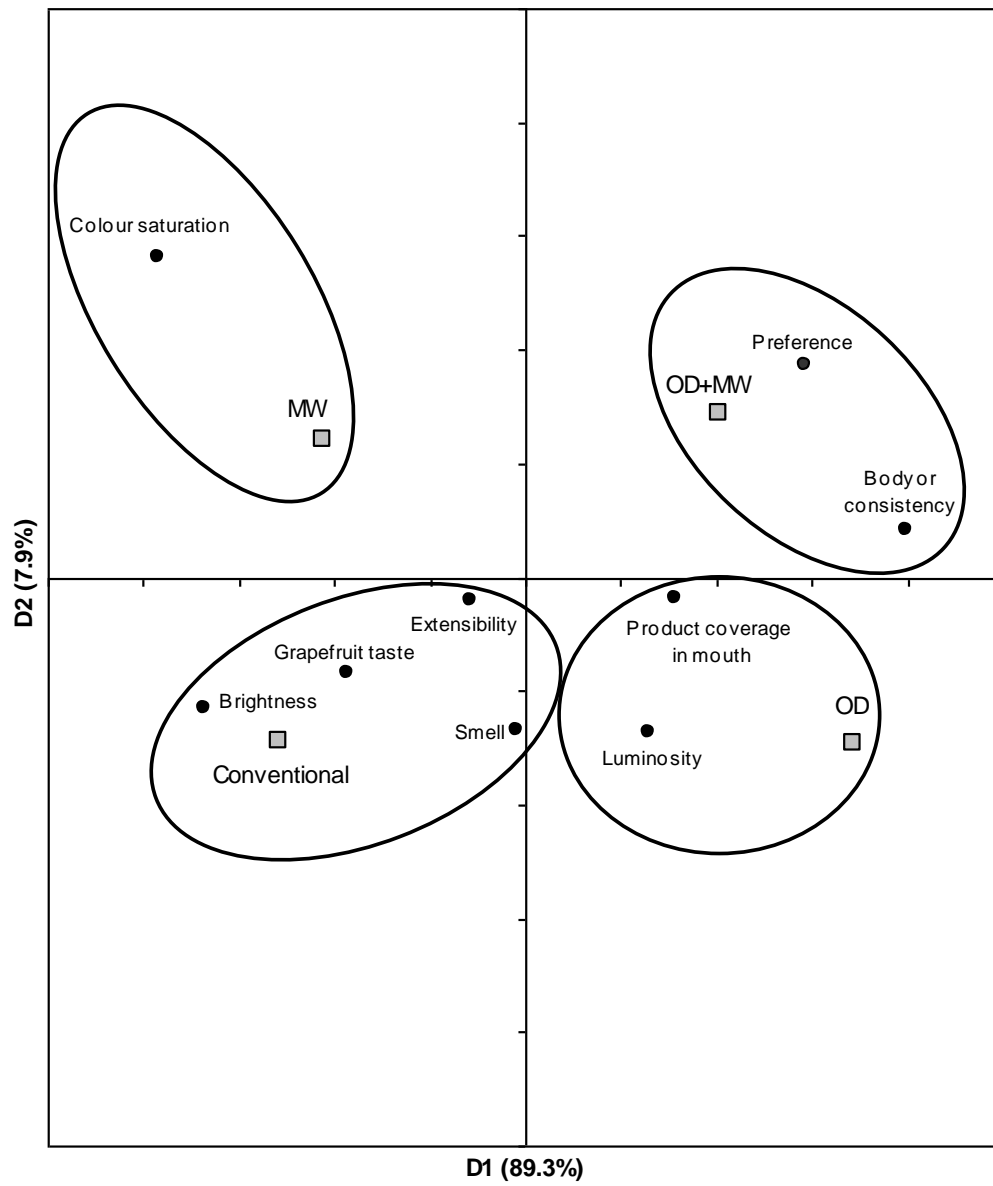


Figure 2.

Figure 3
[Click here to download Figure: Figure 3.doc](#)

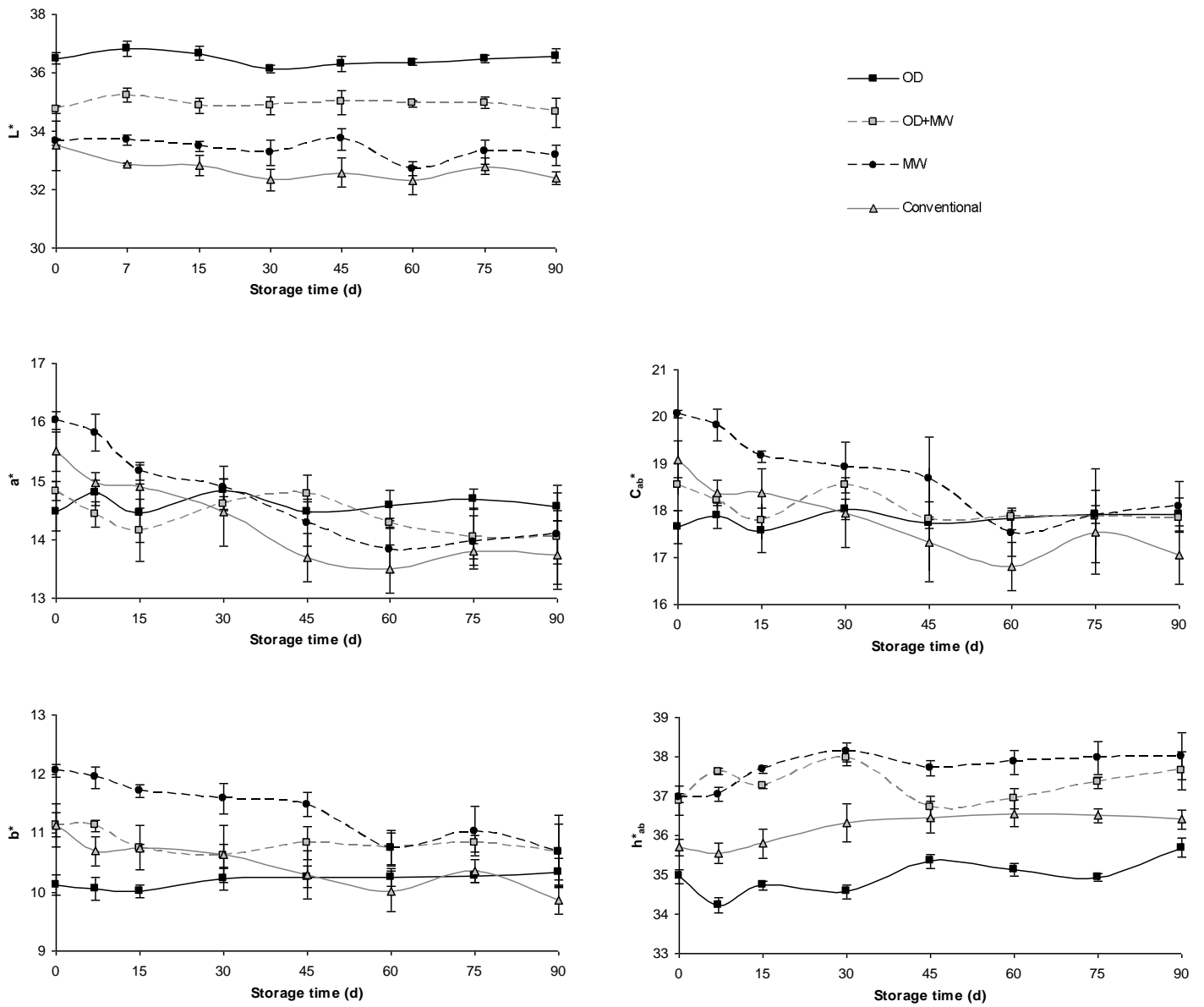


Figure 3.

Figure 4

[Click here to download Figure: Figure 4.doc](#)

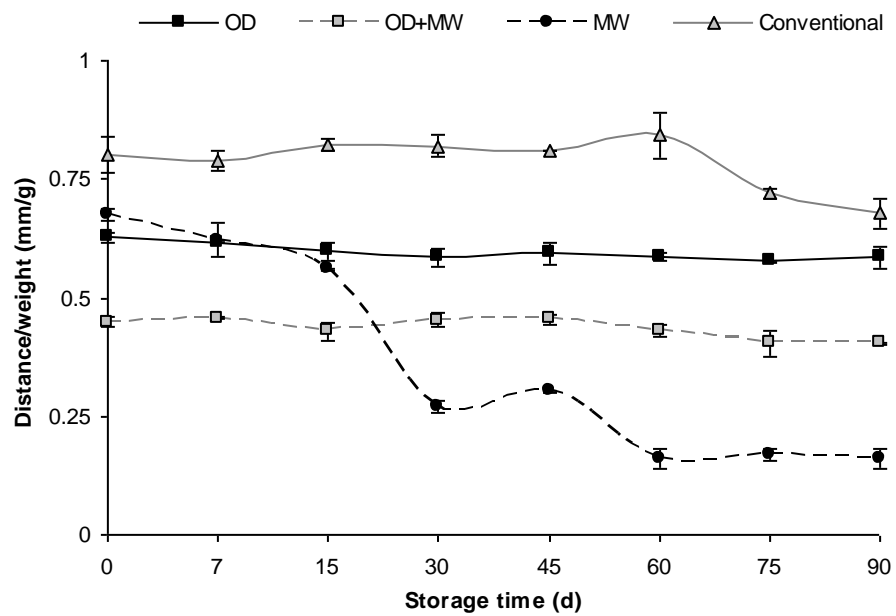


Figure 4.

Table 1[Click here to download Table: Table 1.doc](#)**Table 1.**

Samples	°Brix	a_w	x_w (g w/g product)	pH
Fresh-fruit	12.0 (0.2) ^a	0.989 (0.003) ^a	0.882 (0.002) ^a	3.27 (0.02) ^b
Conventional	48.5 (0.2) ^d	0.922 (0.003) ^e	0.526 (0.002) ^e	3.25 (0.02) ^a
OD	46.1 (0.2) ^b	0.945 (0.003) ^b	0.541 (0.002) ^b	3.39 (0.02) ^c
OD+MW	46.0 (0.2) ^b	0.942 (0.003) ^c	0.537 (0.002) ^c	3.40 (0.02) ^c
MW	47.7 (0.2) ^c	0.924 (0.003) ^d	0.529 (0.002) ^d	3.27 (0.02) ^b

The same letter in superscript within columns indicates homogeneous groups established by the ANOVA ($p < 0.05$).

Table 2[Click here to download Table: Table 2.doc](#)**Table 2.**

	Fresh-fruit	Conventional	OD	OD+MW	MW
L*	49.6 (0.3) ^a	33.5 (0.8) ^d	36.5 (0.2) ^b	34.7 (0.2) ^c	33.6 (0.2) ^d
a*	20,6 (0,2) ^a	15,5 (0,3) ^c	14,5 (0,3) ^e	14,8 (0,2) ^d	16,02 (0,16) ^b
b*	17,76 (0,02) ^a	11,1 (0,2) ^c	10,1 (0,2) ^d	11,1 (0,4) ^c	12,06 (0,12) ^b
C*_{ab}	27.2 (0.2) ^a	19.1 (0.4) ^c	17.7 (0.4) ^e	18.5 (0.2) ^d	20.1 (0.2) ^b
h*_{ab}	40.8 (0.3) ^a	35.7 (0.3) ^c	35.0 (0.4) ^c	37 (2) ^b	37.0 (0.5) ^b
ΔE₁	-	18.2 (0.7) ^c	16.4 (0.2) ^a	17.3 (0.2) ^b	17.6 (0.3) ^{bc}
ΔE₂	-	-	3 (2) ^b	1.5 (0.8) ^a	1.2 (0.5) ^a
Distance/weight (mm/g)	-	0.80 (0.04) ^c	0.63 (0.02) ^b	0.45 (0.02) ^a	0.68 (0.02) ^b

The same letter in superscript within files indicates homogeneous groups established by the ANOVA ($p < 0.05$).
 Colour differences of jam product as compared to fresh fruit (ΔE_1) or conventional jam (ΔE_2).

Table 3[Click here to download Table: Table 3.doc](#)**Table 3.**

	Colour saturation	Brightness	Body or consistency	Grapefruit taste	Extensibility	Preference
OD - OD+MW	42*	23	6	2	16	28*
OD - MW	99*	58*	41*	42*	24	18
OD - Conventional	75*	83*	53*	40*	28*	32*
OD+MW - MW	57*	35*	47*	40*	8	46*
OD+MW - Conventional	33*	60*	59*	38*	12	60*
MW - Conventional	24	25	12	2	4	14

*significant differences at the 0.05 level according Tukey HSD

Table 4[Click here to download Table: Table 4.doc](#)**Table 4.**

Attribute/Jam	D1	D2	Total
Colour saturation	0.860	0.135	0.995
Luminosity	0.637	0.210	0.847
Brightness	0.927	0.033	0.959
Smell	0.014	0.773	0.787
Body or consistency	0.995	0.003	0.999
Grapefruit taste	0.837	0.050	0.887
Product coverage in mouth	0.994	0.004	0.998
Extensibility	0.916	0.031	0.947
Preference	0.874	0.104	0.978
OD	0.936	0.05	0.987
OD+MW	0.829	0.123	0.952
MW	0.867	0.082	0.948
Conventional	0.897	0.084	0.981

Table 5[Click here to download Table: Table 5.doc](#)**Table 5.**

	L*	a*	b*	C*_{ab}	h*_{ab}	Distance/weight (mm/g)
Colour saturation	-0.9745*	0.837	0.7311	0.8055	0.4184	
Luminosity	0.9764*	-0.8179	-0.8741	-0.8618	-0.7334	
Brightness	-0.9144	0.9984*	0.9227	0.985*	0.5791	
Body or consistency						-0.9057
Product coverage in mouth						-0.9552*
Extensibility						0.4886

*significant differences at the 0.05 level