

# Variation in physicochemical and sensory quality of sour orange (*Citrus aurantium* L.) marmalade from the Cap Bon region in North-East Tunisia

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## Variation in physicochemical and sensory quality of sour orange (*Citrus aurantium* L.) marmalade from the Cap Bon region in North-East Tunisia.

**Abstract — Introduction.** *Citrus aurantium* L. sour oranges are poorly exploited in Tunisia, although the Tunisian Cap Bon is one of the major areas of production of sour orange trees. Our study aimed at determining the effect of the geographic origin of the raw material on quality of orange marmalades and consumer acceptance. **Materials and methods.** Fresh sour oranges were collected from five areas in the Cap Bon region in the North-East of Tunisia for producing marmalades. Physicochemical characterisation of marmalades from the five different origins included the determination of soluble solids, total and volatile acidity, the measurement of reducing sugars, and measurement of colour. Sensory panels from Tunisia, Algeria and Europe revealed the marmalades' sensory profiles. The sensory panels then attributed preference and acceptance ranks for the five marmalades. **Results and discussion.** The analyses highlighted an effect of the geographic origin of the raw material on the physicochemical quality. The most suitable geographic conditions seem to be climatic and edaphic criteria. Assessors from various nationalities revealed different sensory profiles depending on the consumer's nationality and fruit production region. In addition, Tunisian consumers are less likely to be marmalade buyers than European and Algerian consumers.

**Tunisia / *Citrus aurantium* / fruits / provenance trials / jams / proximate composition / organoleptic analysis / consumers**

## Variation des propriétés physico-chimiques et de la qualité sensorielle de marmelades d'oranges amères (*Citrus aurantium* L.) originaires de la région du Cap Bon, au nord-est de la Tunisie.

**Résumé — Introduction.** Les oranges amères (*Citrus aurantium* L.) sont peu exploitées en Tunisie bien que le Cap Bon tunisien soit l'une des principales zones de production du bigaradier. Notre étude a cherché à déterminer l'effet de l'origine géographique de la matière première sur la qualité de marmelades d'oranges amères et sur leur acceptation par les consommateurs. **Matériel et méthodes.** Des oranges amères fraîches ont été récoltées dans cinq zones du Cap Bon au nord-est de la Tunisie afin d'en faire de la marmelade. La caractérisation physico-chimique de ces marmelades de cinq origines différentes a porté sur la détermination des solides solubles, des acidités totales et volatiles, du taux de sucres réducteurs et sur la mesure des paramètres de la couleur. Un panel de dégustateurs originaires de Tunisie, d'Algérie et d'Europe a permis de dresser les profils sensoriels des marmelades. Ensuite ces dégustateurs ont attribué à chacune des cinq marmelades un rang de préférence et d'acceptation. **Résultats et discussion.** Les analyses effectuées ont mis en évidence un effet de l'origine géographique des matières premières sur la qualité physico-chimique des différentes marmelades. Les conditions géographiques les plus appropriées semblent être liées à des critères climatiques et édaphiques. Les dégustateurs de différentes nationalités ont révélé différents profils sensoriels en fonction de leur nationalité et de la région de production des fruits. En outre, il est apparu que les consommateurs tunisiens seraient moins susceptibles d'être des acheteurs de marmelade que les consommateurs européens et algériens.

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**Tunisie / *Citrus aurantium* / fruits / essai de provenances / confiture / composition globale / analyse organoleptique / consommateur**

## 1. Introduction

Sour orange *Citrus aurantium* L. belongs to the Rutaceae family. It is a tree that grows all over Tunisia; it is used mainly as a rootstock and also for its flowers. Neroli oil is produced from sour orange flowers using hydrodistillation. Uncollected flowers are fertilised and, after maturation, they form fruits called bitter or sour oranges. These bitter oranges can be transformed into marmalades. The jam and marmalade characteristics (nutritional, technological, hygienic and sensory) depend on various factors, such as the manufacturing technology and essential raw material physicochemical characteristics. Physicochemical characteristics of fruits depend on numerous factors such as geographic, climatic and agronomic criteria [1]. These factors are being studied to search for product authenticity.

The sour orange is propagated in Tunisia in warm regions shielded from intense winds, notably on the Tunisian coastal belt and mainly in the Cap Bon region [2]. This region is characterised by variable climatic and edaphic parameters (nature of the soil, the texture, the depth and the hillside) (*table I*) [2]. Variations in environmental conditions from one region to another can influence the development of *Citrus* and crops (flowers, fruits) [2, 3] and, consequently, can constitute potential sources of differences in the quality of collected products after post-harvest treatments [4, 5].

In our work, the effects of the geographic origins of sour orange *Citrus aurantium* L. on physicochemical and organoleptic characteristics of sour orange marmalade were studied.

## 2. Materials and methods

Fresh sour oranges (*Citrus aurantium* L.) were collected from five regions (M' Hadheba, Somaa, Béni Khair, Tazarka and Diar Ben Salem) in Cap Bon (Nabeul, North-East of Tunisia) in December 2005. The regions of the study have different edapho-climatic characteristics although they are very close to one another (*table I, II*). Various analyses were performed in order to determine fruit

raw material characteristics: pH, °Brix, acidity, total sugars, pulp dry matter and peel dry matter. Marmalades were produced from sour oranges collected from these various regions. The transformation process was performed in the TYPIK factory (Tunis, Tunisia) according to a traditional method. This marmalade was prepared from a mixture of sour orange juice, pulp and peel. Briefly, the transformation steps were: washing, cleaning, cutting into very small slices, addition of sugar, concentration, packaging in glass jars and sterilisation. The prepared marmalades were stored at room temperature for a maximum period of 2 months until analysis was performed.

Microbiological analysis included enumeration of yeasts and moulds according to NP 3277-1 (1987)<sup>1</sup>. Physicochemical characterisation included the determination of soluble solids by measuring °Brix at 20 °C (Abbe Atago 89553 refractometer) according to NF-V-05-105 (1970)<sup>2</sup>; the determination of total and volatile acidity (expressed as g citric acid/100 g<sup>-1</sup> sample) by titration according to NF-V-05-101 (1974)<sup>3</sup> and NF-V05-118 (1974)<sup>4</sup>, respectively; and the measurement of reducing sugars according to Bertrand's method [6]. Colour values were obtained by measuring the reflected spectrum (MINPLOT CR-6300). 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 illuminant D65. The hue angle ( $h^*_{ab}$ ) and chroma or intensity ( $C^*_{ab}$ ) were calculated according to the following equations:  $h^*_{ab} = \tan^{-1}(b^*/a^*)$  and  $C^*_{ab} = [(a^{*2} + b^{*2})^{-1/2}]$ .

<sup>1</sup> Portuguese standard 3277-1: Dénombrement des levures et moisissures des confitures.

<sup>2</sup> French standard – V 05-105: Détermination du résidu sec total (produits dérivés des fruits et légumes), AFNOR, Paris, France.

<sup>3</sup> French standard – V 05-101: Détermination de l'acidité titrable (produits dérivés des fruits et légumes), AFNOR, Paris, France.

<sup>4</sup> French standard – V 05-118: Détermination de la teneur en acidité volatile (fruits légumes et produits dérivés), AFNOR, Paris, France.

**Table I.**

Edaphic characteristics of five regions in Cap Bon (Nabeul, North-East of Tunisia) where sour oranges (*Citrus aurantium* L.) were collected to study marmalade quality.

Region	Bedrock	Ground nature	Ground depth	Slope (%)
M' Hadheba	Mane and limestone	Fersialitic land	A little deep	0–3
Somaa	Mane and gypseous encrusting	Not evaluated land	Very deep	10–15
Béni Khiair	Calcareous crust	Fersialitic land	Slightly deep	3–5
Tazarka	Calcareous crust	Fersialitic land	Slightly deep	3–5
Diar Ben Salem	Calcareous crust and alluvia sand	Fersialitic and hydromorphic land	A little deep	3–5

Slightly deep: 0–0.5 m; a little deep: 0.5–1 m; very deep > 1 m.

**Table II.**

Mean values of maximum temperatures and rainfall of five regions in Cap Bon (Nabeul, North-East of Tunisia) where sour oranges (*Citrus aurantium* L.) were collected to study marmalade quality (2005).

(a) Maximum temperatures (°C)

Region	Jan.	Feb.	Mar.	Apr.	May	June	July	Aug.	Sept.	Oct.	Nov.	Dec.
M'hadheba	15.3	16.1	22.8	23.7	30.1	30.5	34.6	33.9	29.7	27.5	21.3	17.0
Somaa	15.1	16.4	18.8	21.8	25.8	29.5	32.9	32.8	29.4	27.2	21.5	17.4
Béni Khiair	14.7	16.2	19.6	22.1	26.9	31.6	34.1	33.9	30.2	28.2	21.6	16.6
Tazarka	14.9	15.8	18.1	21.1	25.2	29.4	32.6	32.3	28.8	26.9	21.0	13.8
Diar Ben Salem	14.7	16.2	19.6	22.1	26.9	31.6	34.1	33.9	30.2	28.2	21.6	16.6

(b) Rainfall (mm)

Region	Jan.	Feb.	Mar.	Apr.	May	June	July	Aug.	Sept.	Oct.	Nov.	Dec.
M'hadheba	36.2	51.0	20.5	16.0	2.7	5.3	1.3	14.2	43.0	7.0	15.0	50.0
Somaa	51.7	82.0	15.5	38.0	10.0	17.5	1.5	49.0	23.8	28.5	75.5	101.2
Béni Khiair	24.5	33.0	21.2	18.5	5.5	39.3	1.8	14.5	32.5	11.0	8.4	64.0
Tazarka	91.3	42.8	20.9	43.6	7.0	8.0	2.3	45.7	35.8	47.7	91.7	81.3
Diar Ben Salem	24.5	33.0	21.2	18.5	5.5	39.3	1.8	14.5	32.5	11.0	8.4	64.0

The chemical aromatic composition of marmalade required a solid-liquid (20 g / 10 mL) extraction using hexane as a solvent, with magnetic stirring at high speed for 40 min [7]. Then the supernatant was removed and concentrated under a gentle stream of nitrogen to 50  $\mu$ L. This fraction was analysed by Gas Chromatography (GC). GC analysis was carried out on a HP 6890 gas chromatographer with a polar column (30 m  $\times$  0.25 mm; film thickness 0.25  $\mu$ m). The carrier gas was nitrogen with

a flow rate of 1.6 mL $\cdot$ min<sup>-1</sup>. The oven temperature was kept at 35 °C for the first 10 min and then increased at a rate of 3 °C $\cdot$ min<sup>-1</sup> until reaching 205 °C at which it was kept constant for 10 min. Injector temperature was set at 250 °C and the detector temperature at 300 °C. Quantification was guaranteed by adding 10  $\mu$ L of a standard, 6-methyl 5-heptane 2-one (Sigma-Aldrich), before injection. Confirmation of peak identity was carried out by time of retention and calculation of the Kovats index.

**Table III.**

Variation in physicochemical parameter mean values (standard deviation) of marmalade made with sour oranges collected in five different geographic regions in the North-East of Tunisia.

Geographic region	°Brix	Total acidity (g citric acid·100 g <sup>-1</sup> )	Volatile acidity (g citric acid·100 g <sup>-1</sup> )	Reducing sugars (g sugar·100 g <sup>-1</sup> )
M' Hadheba	55.83 (1.00) a	0.060 (0.000) a	0.015 (0.000) ab	3.94 (0.10) a
Somaa	53.45 (0.90) a	0.120 (0.000) b	0.014 (0.000) a	3.47 (0.00) b
Béni Khiar	50.00 (0.80) a	0.096 (0.000) c	0.021 (0.000) b	3.06 (0.10) c
Tazarka	44.65 (3.30) a	0.033 (0.000) d	0.012 (0.000) a	2.30 (0.00) d
Diar Ben Salem	49.60 (3.70) a	0.096 (0.000) c	0.021 (0.000) b	2.55 (0.00) e

Means in the same column with different letters are significantly different ( $p < 0.05$ ).

For sensory analysis, three juries of Tunisian (30), Algerian (30) and European (30) assessors, with slight training (just before tasting) to detect acid (lemon), bitter (coffee) and sugar notes, tasted marmalades originating from the five regions of the study (NF-ISO 8586-1, 1993)<sup>5</sup>. Sensory panels classified samples from the five marmalades according to colour, odour, texture, sugar taste, acid taste and bitter taste. They then attributed preference and acceptance ranks for the five marmalades. The acceptance test was accomplished by ranking 1 the marmalade that the panellist would buy first and 5 the one that he would buy last.

All measurements for physicochemical tests were carried out in triplicate. The statistical analyses of physicochemical characteristics were done by a one-way ANOVA using GraphPad Prism version 4.00 (2003), and those of sensory characteristics were accomplished using the CATMOD procedure in SAS (1989). Pearson's correlation parameters were calculated using SAS (1989).

### 3. Results and discussion

Fruit quality is an important parameter for marmalade quality [8]. The oranges used in our study had physicochemical characteris-

tics (°Brix, acidity, sugars) which conformed to those seen in Garcia-Martinez *et al.*'s study [8] and thus can be used as raw material for marmalade processing. Variation in the physicochemical quality of marmalade made with sour oranges of the five geographic origins (M' Hadheba, Somaa, Béni Khiar, Tazarka and Diar Ben Salem) was summarised (*table III*).

Sugars and acidity seemed to vary ( $p < 0.05$ ) with the oranges' origin. Samples from M' Hadheba, Somaa, Béni Khiar and Diar Ben Salem had a Brix superior to 49%, in agreement with the value recommended for orange jams [9]. The final marmalade °Brix values found for all the samples (*table III*) are in agreement with Codex-Stan 80 [10]. We also noticed that marmalade from M' Hadheba had the highest amount of reducing sugars, and the highest amounts of total sugars (*table IV*). Furthermore, reducing sugars are favourably produced in the acid matrix by heating [11]. Total and volatile acidity amounts were different ( $p < 0.05$ ) from one marmalade batch to another. Marmalade samples from Béni Khiar and Diar Ben Salem regions had similar acidity amounts. This is an expected result since these regions have similarities among their climatic and edaphic criteria (*tables I, II*). The Somaa region marmalade was characterised by the highest total acidity (*table III*) and, in our study, the Somaa region has the deepest soil. This deepness suggests a more significant nutrient uptake than that present in the other regions [11]. The nutrients are transported to productive parts, especially

<sup>5</sup> French standard – ISO 8586-1: Analyse sensorielle Guide général pour la sélection, l'entraînement et le contrôle des sujets.

**Table IV.**

Mean values (standard deviation) of physicochemical characteristics of sour oranges collected in five different geographic regions in the North-East of Tunisia to study marmalade quality.

Geographic region	pH	Acidity (g citric acid·100 g <sup>-1</sup> )	°Brix	Total sugars (g·L <sup>-1</sup> )	Pulp dry matter (g·100 g <sup>-1</sup> )	Peel dry matter (g·100 g <sup>-1</sup> )	Maturity index
M' Hadheba	2.6 (0.0) a	0.542 (0.005) a	11.5 (0.00) a	114.00 (3.50) a	22.80 (1.90) a	24.93 (0.20) a	0.212 (0.020) a
Somaa	2.3 (0.0) a	0.552 (0.005) ab	11.25 (0.30) a	113.30 (3.50) a	21.20 (0.90) a	22.60 (0.80) ab	0.204 (0.010) b
Béni Khiar	2.4 (0.0) a	0.569 (0.003) c	9.75 (0.30) bc	99.30 (3.70) b	20.97 (0.90) a	26.03 (2.40) a	0.170 (0.005) c
Tazarka	2.5 (0.0) a	0.502 (0.001) d	10.25 (0.30) ab	102.05 (3.50) a	22.60 (0.80) a	26.53 (0.20) a	0.203 (0.015) b
Diar Ben Salem	2.4 (0.0) a	0.450 (0.008) e	9.50 (1.40) bc	96.10 (14.30) c	20.10 (1.40) a	22.63 (0.20) ab	0.236 (0.010) a

Means in the same column with different letters are significantly different ( $p < 0.05$ )

fruits, via the sap. Finally, the nutrients are transformed into storage forms: sugars and organic acids [9, 11]. Organic acid transfer from fruit particles to the matrix is ruled by cellular membrane rigidity in fruits, which depends on the presence of Ca<sup>2+</sup> [12]. For guava fruits, for example, when Ca<sup>2+</sup> is present, the rigidity is less and the sugar transfer is easier [12]. All soils in the regions of the study, except for the Somaa region, have calcareous material rich in Ca<sup>2+</sup>. The Béni Khiar and Diar Ben Salem regions also have hydromorphic soils and marmalades made of oranges from these two regions had the highest amounts of volatile acidity. Physicochemical analyses of sour orange marmalade suggest a raw material geographic impact on physicochemical quality. Fruit quality variation could be caused by regional edaphic parameters [3]. These results confirm the findings of previous studies on essential oil quality, which revealed the role of geographic origin and climatic factors (temperature, humidity) in chemical composition of some plants' (*Echinacea purpurea*, *Satureja montana* L., *Satureja cuneifolia*, *Artemisia vulgaris*) essential oils [13, 14].

The results of the sour orange marmalades' microbiological analyses (performed 2 months after sealing) indicated a number of yeasts and mould less than 10 CFU·g<sup>-1</sup> (Colony-Forming Units) in accordance with the expected levels for this parameter [10]. These results indicate the effectiveness of the sterilisation treatment and hermetic sealing of jars, thus preventing air exchanges.

Colour is considered as one of the first sensory criteria for food acceptance [1, 15]. Marmalades made from oranges from different regions showed differences ( $p < 0.05$ ) in luminosity and colour (*table V*). Both the marmalades originating from M' Hadheba and Diar Ben Salem sour oranges had the highest  $C_{ab}^*$  values, indicating that they have a browner colour than the other samples. These differences seem to vary with a combination of multiple factors such as fruit pigmentation and amounts of total and reducing sugars (that determine light deviation by sugar molecules) and they can be attributed to enzymatic or non-enzymatic browning (Maillard reactions) [16, 17]. In order to evaluate chemical aroma compounds, the chromatograms from different marmalades were drawn and their chemical aroma compounds were summarised (*table VI*). It seems that amounts of total identified components vary with the maturity index (*tables IV, VI*). Marmalades made from oranges from different origins had different chemical composition. These differences are mostly clear for limonene,  $\alpha$ -pinene,  $\beta$ -cymene and  $\gamma$ -terpinene. The M' Hadheba and Béni Khiar region marmalade chromatograms showed high resemblances for many compounds (such as limonene, linalyl acetate,  $\alpha$ -terpenyl acetate, etc.) which may be explained by pedoclimatic similarities between these two regions.

The Diar Ben Salem region marmalade had a chromatographic profile rich in oxygenated monoterpenes: nerol, geraniol and eugenol, compared with the other profiles.

**Table V.**

Mean values (standard deviation) of colour parameters from sour oranges from five regions in the North-East of Tunisia.

Geographic region	$L^*$	$a^*$	$b^*$	$h^*_{ab}$	$C^*_{ab}$
M' Hadheba	7.74 (0.70) a	3.06 (0.20) a	5.22 (0.40) a	1.04 (0.08) a	6.05 (0.20) a
Somaa	6.05 (0.20) ab	2.75 (0.00) a	4.02 (0.10) ab	0.97 (0.03) a	4.87 (0.50) b
Béni Khiair	5.62 (0.00) b	2.33 (0.00) a	3.24 (0.90) bc	0.94 (0.04) a	3.99 (0.50) b
Tazarka	6.24 (0.60) ab	1.69 (0.00) b	4.16 (0.50) ab	1.18 (0.09) a	4.49 (0.60) b
Diar Ben Salem	4.80 (0.00) c	5.17 (0.00) c	3.21 (0.00) c	0.55 (0.05) b	6.08 (0.09) a

$L^*$ : Luminosity (100 for white and 0 for black),  $a^*$ : colour varying from (– 60) (green) to (+ 60) (red),  $b^*$ : colour varying from (– 60) (blue) to (+ 60) (yellow).

Means in the same column with different letters are significantly different ( $p < 0.05$ ).

**Table VI.**

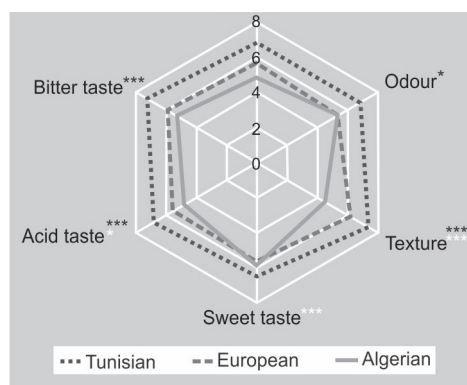
Average aromatic composition (standard deviation) (%) of marmalades made from sour oranges from five regions in the North-East of Tunisia.

Geographic region	$\alpha$ -pinene	Camphene	$\beta$ -pinene	Limonene	$\gamma$ -terpinene	$\beta$ -cymene	Terpinolene	<i>cis</i> -linalool oxide	<i>trans</i> -linalool oxide
M' Hadheba	0.20 (0.06)	0.00 (0.00)	0.00 (0.00)	50.0 (0.9)	0.00 (0.00)	0.00 (0.00)	0.00 (0.00)	0.10 (0.01)	0.10 (0.01)
Somaa	0.40 (0.04)	0.04 (0.01)	0.10 (0.01)	76.7 (1.1)	0.08 (0.00)	1.00 (0.01)	0.40 (0.03)	0.40 (0.02)	0.30 (0.01)
Béni Khiair	0.60 (0.03)	0.02 (0.00)	0.10 (0.00)	54.9 (1.1)	0.10 (0.01)	0.40 (0.01)	0.10 (0.02)	0.10 (0.03)	0.10 (0.01)
Tazarka	1.50 (0.40)	0.00 (0.00)	0.08 (0.01)	63.4 (1.2)	0.00 (0.00)	0.00 (0.00)	0.00 (0.00)	0.00 (0.00)	0.50 (0.01)
Diar Ben Salem	3.30 (0.80)	0.04 (0.01)	0.06 (0.10)	42.8 (1.4)	0.06 (0.01)	0.70 (0.09)	0.30 (0.04)	0.33 (0.04)	0.16 (0.05)
Kovats index	1020	1071	1113	1196	1257	1270	1286	1436	1480

Geographic region	Linalool	Linalyl acetate	$\beta$ -caryophyllene	Terpinene-4-ol	$\alpha$ -terpenyl acetate	$\alpha$ -terpineol	Geranyl acetate	Nerol	Geraniol	Eugenol
M' Hadheba	0.02 (0.00)	0.08 (0.00)	0.20 (0.01)	0.0 (0.00)	0.02 (0.00)	1.30 (0.05)	0.30 (0.01)	0.06 (0.01)	0.07 (0.01)	0.04 (0.01)
Somaa	0.05 (0.02)	0.05 (0.01)	0.20 (0.04)	0.08 (0.02)	0.08 (0.01)	3.10 (0.80)	0.20 (0.02)	0.02 (0.00)	0.04 (0.00)	0.00 (0.00)
Béni Khiair	0.20 (0.05)	0.05 (0.01)	0.30 (0.01)	0.10 (0.00)	0.04 (0.00)	1.70 (0.50)	0.10 (0.00)	0.03 (0.00)	0.05 (0.01)	0.00 (0.00)
Tazarka	0.30 (0.02)	0.06 (0.01)	0.50 (0.03)	0.07 (0.00)	0.03 (0.01)	0.10 (0.00)	0.15 (0.01)	0.00 (0.00)	0.10 (0.01)	0.00 (0.00)
Diar Ben Salem	0.10 (0.01)	0.04 (0.00)	0.30 (0.01)	0.08 (0.01)	0.07 (0.00)	3.30 (0.09)	0.10 (0.00)	0.04 (0.00)	0.10 (0.01)	0.60 (0.06)
Kovats index	1548	1554	1595	1600	1680	1693	1750	1838	1843	1900

According to Stahi Biskup and Laesko [18], during hydrodistillation, linalyl acetate may undergo a rearrangement, leading to nerol and geraniol production. This rearrangement may be caused by cooking (boiling) marmalades rich in aqueous medium at high temperature. In addition, this aromatic profile is characterised by low amounts of limonene and linalool, and a significant compound of  $\alpha$ -terpineol. Limonene and linalool could be  $\alpha$ -terpineol precursors, as indicated by Stahi Biskup and Laesko [18]. The Diar Ben Salem region soil is the only one in this study to have alluvia sand bedrock. Another parameter that could have led to the distinguished aromatic composition of Diar Ben Salem region marmalade is the maturity index of its sour oranges (table IV). All chromatograms revealed the presence of  $\alpha$ -pinene in varying amounts: the highest ones were for the Tazarka and Diar Ben Salem region marmalades. These two regions registered very low temperatures during October and November 2005, compared with the other regions (table II). These observations suggest a combined effect of climatic (temperature, rainfall) and edaphic criteria (bedrock, soil nature) which create a specific microclimate for each production region and thus lead to specific physicochemical characteristics for marmalade produced from various geographic regions. These findings confirm those reported in studies [13, 14, 19, 20] where climate and edaphic conditions were found to lead to qualitative differences.

The marmalade perception varied with the nationality of the consumer and the origin of sour orange fruits (figure 1). In fact, different assessors from Algeria, Tunisia and Europe noticed differences between the five marmalades (from M' Hadheba, Soma, Béni Khiar, Tazarka and Diar Ben Salem). Algerian perceptions seemed to be similar to those of Europeans for colour, odour, sugar and acid taste of the marmalades, Algerians and Europeans observed bitterness in the same way. These similarities and differences in perceptions can be attributed to consumers' food habits. In fact, Tunisians are more accustomed to hot and spicy foods when compared with their Algerian and European counterparts [21]. Tunisia is endowed with a high production of *Citrus*



**Figure 1.** Variation in average perception of marmalades made from sour oranges collected from five regions in the North-East of Tunisia, by Tunisian, Algerian and European consumers (Statistical difference due to the panellist's nationality: \*, \*\*\* in grey:  $p < 0.05$ , and  $p < 0.001$ , respectively; Statistical difference due to the product: \*, \*\*\* in white:  $p < 0.05$ , and  $p < 0.001$ , respectively). 0 to 8: scale of notification.

fruits. Therefore, Tunisians are accustomed to *Citrus* aroma flavours, which may then lead to a low perception threshold compared with Algerians and Europeans. According to our results, colour and texture of marmalades seemed to be the most distinguishing criteria ( $p < 0.0001$ ) for all consumers (figure 1). Indeed, the proportions of variations observed and explained by the consumer nationality and raw material origin were 48% for colour and 23% for texture, respectively. The rest of these variations may be caused by factors related to assessors such as sex, age, socio-economic class, the character appreciation and its expression and, evidently, the residual error (different tasting times for different assessors) [22, 23].

The acid taste and odour had low  $R^2$  using the country of assessor and raw material as explanatory factors; they were up to 12% (0.033–0.12 g·100 g<sup>-1</sup> in total acidity and 0.012–0.021 g·100 g<sup>-1</sup> in volatile acidity (table III). Scores attributed to bitter and sweet tastes did not seem to vary simultaneously with the nationality of consumer and sour orange origin. The consumers from various countries gave similar notes to sugar taste ( $p = 0.11$ ) for various marmalades, but not for bitter taste. In fact, for different assessors, the bitterness did not seem to vary from one marmalade to another. Since the same formulation and concentration time was used for the five batches, the observed differences could be caused by raw material differences (origins). Pearson's correlation coefficients ( $r$ ) between assessors' criteria perceptions and marmalade physicochemical parameters were calculated (table VII). The highest Pearson's correlation coefficients ( $r$ ) were



**Table VII.**Pearson's  $r$  between consumers' perceptions and marmalades' physicochemical parameters.

Consumers' perception	Brix	Total acidity	Volatile acidity	Reducing sugars	Colour		
					$L^*$	$a^*$	$b^*$
Colour	0.53 ***	-0.11 *	0.06	0.52 ***	0.46 ***	0.20 ***	0.45 ***
Odour	0.02	0.02	0.09	0.003	-0.02	0.08	-0.02
Texture	-0.38 ***	0.24 ***	0.07	-0.33 ***	-0.22 ***	-0.19 ***	-0.27 ***
Sugar taste	0.10	-0.09	-0.03	0.09	0.12*	0.04	0.14 *
Acid taste	0.12 *	-0.01	0.11 *	0.07	-0.46	0.17 **	-0.02
Bitter taste	0.05	0.03	0.07	0.03	-0.04	0.06	-0.04

\*, \*\*, \*\*\*: Correlation significant at the level 0.05, 0.005 and 0.001, respectively.

**Table VIII.**Percentage of consumers with the same nationality ( $n = 30$  assessors for each nationality) that attributed the same rank to the same marmalade sample after performing a preference test from marmalades made from sour oranges from five regions in the North-East of Tunisia (preference rank: 1: most preferred, 5: least preferred).

Assessor's nationality	Geographic region	Preference rank				
		1	2	3	4	5
Tunisian	M' Hadheba	32	8	12	12	36
	Somaa	14.5	32.3	17.7	21.3	14.2
	Béni Khiar	4	7	40.8	22.2	26
	Tazarka	40	18	14	14	14
	Diar Ben Salem	14	42	18	22	4
Algerian	M' Hadheba	16	25	16	8	25
	Somaa	8	8	16	25	43
	Béni Khiar	16	10	43	18	13
	Tazarka	25	40	15	15	5
	Diar Ben Salem	33	8	16	25	8
European	M' Hadheba	16	16	9	9	50
	Somaa	4	21	33	29	13
	Béni Khiar	13	25	6	28	28
	Tazarka	21	25	29	17	8
	Diar Ben Salem	50	16	18	16	0

observed between assessors' colour perceptions and °Brix (0.53), reducing sugars (0.52) and colour parameters [ $L^*$  (0.46),  $a^*$  (0.20) and  $b^*$  (0.45)] ( $p < 0.001$ ). These values show how chemical parameters interfere in visual perceptions.

To each sample, every consumer attributed a rank of marmalade preference ranging from 1 (the most liked) to 5 (the least liked) (table VIII). For instance, for Tunisian

panellists, the probability of ranking the M'Hadheba marmalade first out of the five studied samples was 32% and the probability of it being ranked last was 36%. In addition, the probability of the M'Hadheba marmalade being ranked last was high, independent of the nationality (respectively, 32% for Tunisians, 25% for Algerians and 50% for European panellists). These ranks did not vary significantly with the consumer



nationality ( $p = 0.9995$ ). Therefore, all assessors gave different ranks to marmalades made from sour oranges collected in the different regions. Our results suggested that sour orange origin was the most important parameter determining consumers' preferences for marmalades.

Preference probabilities of marmalades by consumers (*table IX*) enabled us to observe that marmalade ranks significantly depended ( $p = 0.0001$ ) on origin of sour oranges. From these data, it was possible to establish a preference classification by all assessors for these marmalades. The highest probability value (0.36) for M'Hadheba marmalade was observed for the rank 5. Similarly, the highest probability for Somaa marmalade was associated with a rank 4, for Beni Khiair marmalade, a rank 3, for Tazarka marmalade, a rank 2, and, for the Diar Ben Salem product, a rank 1. Based on this ranking, we believe that the consumers perceived differences between the five marmalades and they tended to "not like" the marmalade from M' Hadheba region but to "like" the one from Diar Ben Salem region.

Physicochemical analyses revealed that the Diar Ben Salem region sample had an

**Table IX.**

Variation in preferences of marmalades made from sour oranges from five regions in the North-East of Tunisia. Displayed data are the sum of percentages of preference rank for each marmalade sample for each consumer's nationality divided by 3 (30 consumers of each nationality / 90 total number of consumers). Preference rank: 1: most preferred, 5: least preferred.

Geographic region	Preference rank				
	1	2	3	4	5
M' Hadheba	0.24	0.18	0.12	0.10	0.36
Somaa	0.10	0.20	0.22	0.25	0.23
Béni Khiair	0.12	0.13	0.31	0.23	0.21
Tazarka	0.26	0.28	0.19	0.17	0.10
Diar Ben Salem	0.33	0.21	0.18	0.22	0.07

average total acidity (second out of five) and a high volatile acidity (first out of five) (*table III*). In addition, the Diar Ben Salem region marmalade chromatogram showed high levels of oxygenated monoterpenes and  $\alpha$ -terpineol. This is probably why consumers classified the Diar Ben Salem region marmalade differently according to acid taste and odour [24] and considered the acid taste and flavour as determinant factors for

**Table X.**

Variation in acceptance (to buy or not to buy marmalades in the market) of marmalades made from sour oranges from five regions in the North-East of Tunisia by Tunisian, Algerian and European consumers. Data represent the percentage of consumers of the same nationality who attributed the same rank of acceptance to the same sample when taking the acceptance test. Acceptance rank: 1: most accepted to buy, 5: least accepted to buy.

Consumer's nationality	Geographic region	Acceptance rank					Total
		1	2	3	4	5	
Tunisian	M' Hadheba	22	7	4	0	4	37
	Somaa	18	15	8	0	4	45
	Béni Khiair	11	4	4	0	0	19
	Tazarka	11	4	4	4	4	27
	Diar Ben Salem	8	19	4	8	0	39
Algerian	M' Hadheba	16	25	0	0	16	57
	Somaa	9	0	25	0	18	52
	Béni Khiair	25	25	8	16	0	74
	Tazarka	33	17	0	8	0	58
	Diar Ben Salem	17	9	25	9	0	60
European	M' Hadheba	29	4	4	4	18	51
	Somaa	4	12	8	12	12	48
	Béni Khiair	12	20	4	8	0	44
	Tazarka	20	25	20	8	0	73
	Diar Ben Salem	33	8	20	12	0	73

consumers' preferences. The same consumers gave different ranks of acceptance (to buy or not to buy marmalades if they are in the market) of the five marmalades (table X). The order of acceptance (willing to buy) depended significantly on consumer nationality ( $p = 0.0012$ ) and raw material origin ( $p = 0.0128$ ). According to these results, European and Algerian consumers, who had an average of total acceptance ranks of 57.8% and 60.2%, respectively, are more likely to be marmalade buyers than Tunisian consumers, who had only an average of total acceptance ranks of 33.4%. This is probably due to Tunisian food habits, especially their predilection for sugary taste [21].

Our results revealed that marmalades made of sour oranges from different areas in a restricted geographic region are endowed with different physicochemical and sensory properties. The choice of raw material production areas is crucial to guarantee to consumers the best product quality. Because of a low acceptance of these products by Tunisian consumers comparatively with European or Algerian consumers, further studies are needed to elaborate a formula adapted to Tunisian food habits.

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### Variación de las propiedades físico-químicas y de la calidad sensorial de mermeladas de naranjas amargas (*Citrus aurantium* L.) originarias de la región del Cap Bon, al noreste de Túnez.

**Resumen — Introducción.** Las naranjas amargas (*Citrus aurantium* L.) están poco explotadas en Túnez, a pesar de que el Cap Bon tunecino sea una de las zonas principales de producción del naranjo amargo. Nuestro estudio pretendió determinar el efecto que tiene el origen geográfico de la materia prima en la calidad de las mermeladas de naranja amarga y en la aceptación por los consumidores. **Material y métodos.** Se cosecharon naranjas amargas frescas en cinco zonas del Cap Bon, al noreste de Túnez, con el fin de convertirlas en mermelada. La caracterización físico-química de estas mermeladas, de cinco orígenes diferentes, fijó la determinación de sólidos solubles, de acidez total y volátil, del índice de azúcares reductores y la medida de los parámetros del color. Un panel de catadores originarios de Túnez, de Argelia y de Europa permitió diferenciar los perfiles sensoriales de las mermeladas. A continuación, dichos catadores atribuyeron a cada una de las cinco mermeladas un rango de predilección y de aceptación. **Resultados y discusión.** Los análisis efectuados pusieron de manifiesto el efecto del origen geográfico de las materias primas en la calidad físico-química de las diferentes mermeladas. Las condiciones geográficas más apropiadas parecen estar relacionadas a criterios climáticos y edáficos. Los catadores de diferentes nacionalidades revelaron diferentes perfiles sensoriales, en función de su nacionalidad y de la región de producción de los frutos. Por otra parte, resultó que los consumidores tunecinos, aparentemente, son menos propensos a comprar mermelada, que los consumidores europeos y argelinos.

**Túnez / *Citrus aurantium* / frutas / ensayos de procedencias / mermeladas / composición aproximada / análisis organoléptico / consumidores**