

## Central composite rotatable design approach to optimize 'Italia' raisin drying conditions

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### Abstract

Considering its high demand and limited production, raisin represents an important alternative to diversify grape processed products around the world. The aim of this study was to determine the best combination between drying temperature and time required to reach the highest consumer acceptance of 'Italia' raisin produced in the semi-arid climate in Brazil. The drying conditions were combinations between drying temperatures of 50, 56, 70, 84 and 90°C and drying times of 16, 22, 35, 48 and 54 hours, following a central composite rotational design (CCRD). The best combination between drying temperature and time was estimated to be 70°C for 35 hours (h), based on overall consumer acceptance. According to the statistical analysis used, drying at 70 °C for 35h and at 59 °C for 28h results in equal overall consumer acceptance, being the second option the more economical. Taste and texture, raisin chroma values and pH were more positively correlated to overall acceptance. The results indicate that drying at 59°C for 28 h is the most efficient drying condition for raisins from 'Italia' grapes grown in Brazilian semi-arid condition. This study uses a new approach based on the central composite rotatable design to determine the most efficient drying temperature and time for 'Italia' raisins.

**Keywords:** consumer, sensory quality, temperature, time, *Vitis vinifera*

### Uso do delineamento completo central rotacional para otimizar condições de secagem de passas 'Italia'

### Resumo

Dada a sua alta demanda e produção limitada, uvas passas representam uma importante alternativa para diversificar produtos processados de uva em todo o mundo. O objetivo deste estudo foi determinar a melhor combinação entre a temperatura de secagem e tempo necessário para atingir a maior aceitação do consumidor de passas de uva 'Italia', produzida no clima semiárido do Brasil. As condições de secagem foram combinações entre temperaturas de 50, 56, 70, 84, 90 °C e tempo de 16, 22, 35, 48, 54 h, seguindo um delineamento completo central rotacional (DCCR). A melhor combinação entre a temperatura e tempo foi a 70 °C durante 35h com base na aceitação global do consumidor. Pela análise estatística utilizada, a secagem a 70 °C durante 35h e a 59 °C por 28h resulta em aceitação global do consumidor semelhante, demonstrando que a segunda opção poderia ser mais econômica. O sabor e a textura, os valores de croma e o pH foram mais positivamente correlacionados à aceitação global. Os resultados indicam que a secagem a 59 °C por 28 h é a condição de secagem mais eficiente para passas de uva 'Itália' produzida no clima semiárido do Brasil. Este estudo utiliza uma nova abordagem baseada no delineamento completo central rotacional para determinar a temperatura e tempo de secagem mais eficientes para produção de passas 'Itália'.

**Palavras chave:** consumidor, qualidade sensorial, temperatura, tempo, *Vitis vinifera*

Received: 01 Outubro 2014

Accepted: 08 Junho 2015

## Introduction

The world grape production has reached 69 million tons in 2011 with about 2% used for raisin production (FAO, 2015). The largest raisin producing countries are Turkey and United States. Combined, these countries account for about 80% of global raisin production (FAS, 2015). The increasing demand for convenient foods has stimulated raisin production in other grape growing countries. In Brazil, grapes have been produced under different environmental conditions, which determine the typical quality of the fruit and processed products (Pereira et al., 2011). In the Northeast semi-arid region, high light intensity, low relative humidity and high temperatures are environmental factors that can potentially contribute to high raisin quality. However, raisin production under such conditions remains to be investigated for 'Italia' grapes, which is one of the most cultivated grape varieties in the Brazilian semi-arid region.

Dried fruit and vegetables have almost unlimited shelf-life in proper packages and substantially lower transportation, handling and storage costs when compared to products of other preservation methods (Sharma & Adulse, 2007; Michalczyk et al., 2009).

Since the optimal drying temperature and time depends on the chemical composition and moisture contents, the optimal drying conditions have to be determined for each genotype and growing conditions (Esmaili et al., 2007; Bennett et al., 2011; Singh et al., 2012). The drying process is highly energy demanding and attempts to increase drying efficiency and reduce energy consumption require optimization of drying temperature and time (Clary et al., 2007; Margaris & Ghiaus, 2007). During the drying process, water moves from the food core towards its surface, which is followed by evaporation. The highest drying rate mostly occurs during the period for which the rate of evaporated water decreases continuously, and moisture transfer during drying is controlled by internal diffusion (Wang & Brennan, 1992; Doymaz, 2006; Srikiatden & Roberts, 2006). As food loses water, a number of physical and chemical changes take place simultaneously, such as volume, porosity and bulk and particle density, which directly affect

sensory products attributes (Ramos et al., 2003; Rahman et al., 2005). Many thermal methods result in low drying rates (Clary et al., 2005; Zhang et al., 2005; Thakur et al., 2010). The long drying times at relatively high temperatures often lead to undesirable thermal degradation of the finished products (Mousa & Farid, 2002). On the other hand, high drying rates usually require high drying temperatures that can lead to undesirable degradation of physico-chemical components in the dried product (Mwithiga & Olwal, 2005; Jaiswal et al., 2010).

The objective of this study was to determine the best combination between drying temperature and time required to reach the highest consumer acceptance of 'Italia' raisins produced in the semi-arid climate in Brazil, aiming to provide an alternative processing to one of the most produced grapes in this climate.

## Material and Methods

Grape cultivar Italia was harvested from a commercial vineyard located at the São Francisco Valley in Petrolina, PE, Brazil. After harvest, the berries were manually destemmed, randomly mixed and sanitized with 2.5% chlorine for 20 minutes, followed by wash in running deionized water.

The study followed a central composite rotational design (CCRD) with the independent variables drying temperature and time to establish a model and determine the optimal drying conditions (Brito & Narain, 2003; Rodrigues & Iemma, 2009). The assays were accomplished according to a complete factorial design with central points (level 0) and axial points (levels  $\pm 1.41$ ) addition. Values are presented at Tables 2 and 3. Each independent variable was composed by five coded levels. Coded levels and real levels are  $X_1$ : drying temperature (50, 56, 70, 84 and 90°C) and  $X_2$ : drying time (16, 22, 35, 48 and 54h). A total of 11 assays composed by combinations among four factorial, three central and four axial variables were accomplished. Each combination was composed by 1.3 kg of fruit. Grape drying was accomplished in a continuous flow fixed bed dryer (Sulab, Brazil) at a constant air velocity of 3.0 m s<sup>-1</sup> for each combination between drying temperature and drying time. The dryer system

consisted of vertical air flow through trays and was arranged as an open system (Azoubel et al., 2010). Air temperature was controlled by a digital thermostat. The air velocity was monitored using an anemometer (AIRFLOW, model LCS 6000, UK). At harvest, samples were subjected to physico-chemical analysis and after drying, samples were subjected to physico-chemical and sensory analysis.

Consumer's acceptability analysis was carried out by 50 raisin consumers that evaluated each of the following quality parameters: overall acceptance, visual appearance, aroma, flavor and texture of the samples. Consumer's acceptability of the 11 'Italia' raisin samples was accomplished using the traditional nine points hedonic scale where: 1, dislike extremely; 2, dislike very much; 3, dislike moderately; 4, dislike slightly; 5, neither like nor dislike; 6, like slightly; 7, like moderately; 8, like very much; 9, like extremely. In order to avoid sensory fatigue among consumers, the 11 raisin samples were evaluated in two distinct tasting sessions carried out on two successive days. The effects of the presentation order and first-order carry-over of the samples were controlled using the crossover design proposed by Wakeling & MacFie (1995). The best combination between drying temperature and time was calculated as the combination that resulted in the highest overall consumer acceptance. The most efficient combination between drying temperature and time was calculated as the lowest temperature and shortest time that resulted in overall acceptance statistically equal to the best combination between drying temperature and time (Figure 1). The protocol for this research project has been approved by a suitably constituted Ethics Committee and this study was undertaken by the number 0002/220514 CEDEP/UNIVASF.

For physico-chemical analysis, color was determined with a digital colorimeter Colortec PCM (Accuracy Microsensors INC., Pittsford, NY, USA). The readings were recorded and the results presented as lightness ( $L^*$ ) with values ranging from 0 = black to 100 = white, Chroma ( $C^*$ ) representing the color intensity or saturation, and hue angle ( $h^\circ$ ) which is the actual color recognized by the human eye. Firmness measurements were

accomplished at the equator region of each berry with a texture analyzer model TA.XT. Plus (Extralab Brasil, SP, Brazil). The results obtained represent the resistance force required to compress 20% of the berry volume. Soluble solids and pH were analyzed by dissolving 10 g of dried raisin on 100 ml of deionized water at 80°C for 2 h (Feldberg et al., 2008). Soluble solids (SS) content was determined in the dissolved raisin solution with a digital refractometer Abbe Marck II (Reichert Jung, Depew, NY, EUA). The pH was also determined in the dissolved raisin solution with a pH meter (Hanna Instruments, Woonsocket, RI, USA). The SS content and pH were calculated and expressed on the dried raisin weight basis. Total polyphenol content was determined using the Folin & Ciocalteu (1927) method with the modifications described by Larrauri et al. (1997). Berry weight was determined gravimetrically using a semi-analytical balance. Berry length and width were determined with a caliper rule.

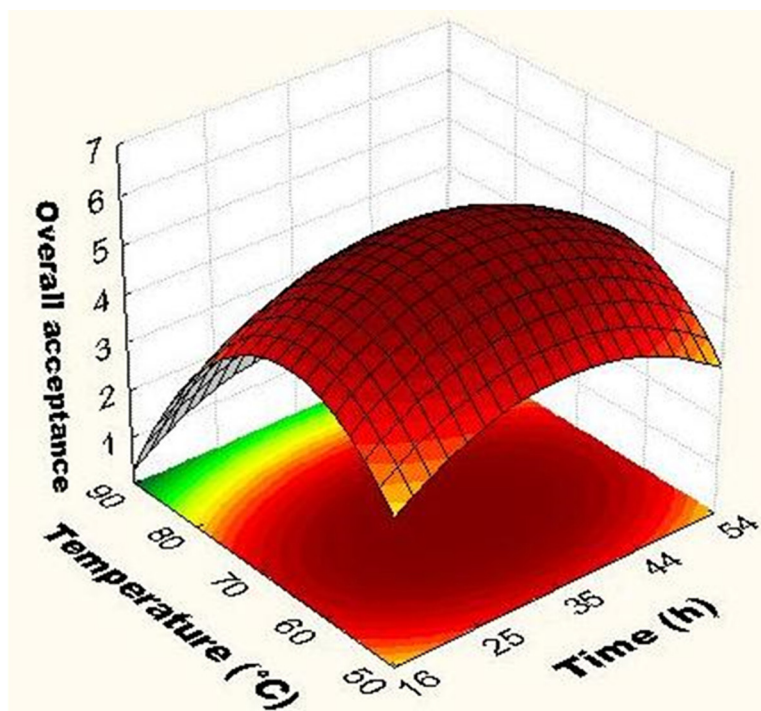
Consumer's overall acceptance was analyzed using the response surface methodology – RSM (Khuri & Cornell, 1996). The polynomial model used was:  $Y = b_0 + b_1X_1 + b_2X_2 + b_3X_1^2 + b_4X_2^2 + b_5X_1X_2$ . Where Y is the consumer's overall acceptance, and  $X_1$  and  $X_2$  represent temperature and time, respectively, which were affected by the regression coefficients ( $b_0$ ,  $b_1$ ,  $b_2$ ,  $b_3$ ,  $b_4$  and  $b_5$ ). The RSM was used to determine the optimal combination between drying temperature and time required to reach the highest consumer's overall acceptance (Brito & Narain, 2003; Rodrigues & Iemma, 2009). The adjusted model and its response surface were determined at  $p \leq 0.05$ . The validity of the model was evaluated as a function of its respective coefficient of determination ( $R^2$ ) as well as by analysis of the lack of fitness. Calculations were performed using the Statistica 7.0 (Statsoft, Tulsa, OK, USA) software package. For comparative evaluation of samples, the results obtained by the physico-chemical analyzes and consumer test were processed using ANOVA and Tukey test ( $p < 0.05$ ).

## Results and Discussion

At harvest, 'Italia' grapes had average color lightness of 29.52, chroma of 5.57 and hue

angle of 99.94, as well as berry firmness of 6.39 N, juice soluble solids content of 12.8%, juice pH of 3.69, total phenolics content of 105.46 mg 100 g<sup>-1</sup>, water activity of 0.98 and average berry weight of 8.85 g. After drying, the best combination between drying temperature and time was determined based on consumer's

overall acceptance results (Figure 1, Tables 3 and 4). According to our results, the highest 'Italia' consumer's overall acceptance was located in the experimental central point with estimated drying temperature of 70°C and drying time of 35h (Figure 1).



**Figure 1.** Consumer's overall acceptance of 'Italia' raisins after drying at different temperatures (50, 56, 70, 84 and 90°C) and times (16, 22, 35, 48 and 54h).

At the central point, increasing or decreasing drying temperature or time decreases overall consumer acceptance. The Analysis of variance (ANOVA) revealed that both drying temperature and time, individually, had a statistically significant quadratic response to overall consumer acceptance with *p*-values lower than 5% (Table 1). The interaction between the drying time x temperature was not statistically significant to overall consumer acceptance.

The highest intensity for all raisin sensory

attributes were obtained only with combinations between drying temperatures and times of 56°C x 22 h, 56°C x 48 h or 70°C x 35 h (Table 2), whereas the lowest intensity of all raisin sensory attributes were obtained only with the combination of 50°C x 35 h.

The color analysis revealed that the combined effect between drying temperatures x times of 84°C x 48 h, 70°C x 54 h, and 90°C x 35 h resulted in the lowest lightness (L) values, indicating the darkest raisin coloration (Table 3).

**Table 1.** Analysis of Variance (ANOVA) for consumer's overall acceptance of 'Italia' raisins dried at different temperatures and times

Factors	Effect	Coefficient	Standard error	<i>p</i> -value
Mean interaction	5.75000	5.75000	0.300249	0.000007**
Time (L) <sup>†</sup>	-0.24762	-0.12381	0.367728	0.530569
Time (Q) <sup>‡</sup>	-1.29000	-0.64500	0.437684	0.031984*
Temperature (L) <sup>†</sup>	-1.95228	-0.97614	0.367728	0.003168**
Temperature (Q) <sup>‡</sup>	-2.80500	-1.40250	0.437684	0.001372**
Time x Temperature	-0.45500	-0.22750	0.520046	0.421647

<sup>†</sup> Linear (L); <sup>‡</sup> quadratic (Q). Statistical significant at *p* ≤ 0.01 (\*\*) or *p* ≤ 0.05 (\*).

The highest L values (whitest coloration) were obtained with the combined effect between drying temperatures and times of 56°C x 22 h, 50°C x 35 h. The highest chroma (C) values, that means highest color intensity or saturation, were observed with the combined effect of drying temperatures and times of 56°C x 22 h, 50°C x 35 h, and 70°C x 35 h, whereas the lowest C values

were observed with drying temperatures and times of 84°C x 22 h, 84°C x 48 h, and 90°C x 35 h. The hue angle ( $h^\circ$  = color definition) was higher with drying temperatures x times of 56°C x 22 h, 56°C x 48 h, and 50° x 35 h, whereas the lowest  $h^\circ$  values were observed with drying temperatures x times of 84°C x 48 h, and 90°C x 35 h.

**Table 2.** Acceptability of 'Italia' raisins dried at different temperatures and times.

Drying conditions		Overall acceptance	Appearance	Aroma	Flavor	Texture
°C	Hours					
56	22	5.6 ab*	6.1 a	5.5 ab	6.1 a	5.7 a
84	22	4.0 bc	2.8 c	4.2 b	4.5 ab	4.2 abc
56	48	5.2 ab	5.2 ab	5.0 ab	5.1 ab	5.0 ab
84	48	2.4 cd	4.9 ab	4.7 ab	2.1 cd	2.4 cd
70	16	4.4 ab	3.4 bc	4.7 ab	4.9 ab	4.6 ab
70	54	4.0 bc	3.5 bc	5.1 ab	3.8 bc	3.3 bcd
50	35	2.3 cd	4.2 bc	4.2 b	2.2 cd	2.5 cd
90	35	2.0 d	3.4 bc	4.4 ab	1.8 d	2.2 d
70	35	5.6 ab	5.7 a	5.1 ab	5.9 a	5.3 a
70	35	5.1 ab	5.2 ab	5.0 ab	5.0 ab	5.2 ab
70	35	6.0 a	6.2 a	5.8 a	6.2 a	5.9 a
CV (%)		43.0	44.3	32.6	46.7	47.4

† Mean intensities based on a nine point hedonic scale where 1 = dislike extremely and 9 = like extremely. \*Different values in the column: means are significantly different according to Tukey's test (p < 0.05). Mean values, n=50 consumers.

**Table 3.** Firmness (Firm), soluble solids (SS), pH, total phenol content (Phen), water activity (aw), berry weight (BW) and color parameters (Lightness (L), Choma (C) and Hue angle ( $h^\circ$ )) of 'Italia' raisins dried at different temperatures and times.

Drying conditions		Firm (N)	SS (%)	pH	Phen (mg/100g <sup>-1</sup> )	aw	BW (g)	(L)	(C)	$(h^\circ)$
°C	h									
56	22	31.5c	30.3e	4.08cde	134.2g	0.85a	3.67b	29.9a	11.7ab	77.1a
84	22	0.8e	78.6a	4.19ab	830.3d	0.41cd	1.82c	23.5c	8.0ef	60.5c
56	48	4.2e	59.6d	4.14abc	273.7f	0.45c	1.68cd	24.6c	9.2de	71.6ab
84	48	25.5cd	67.6c	4.00e	2194.8b	0.34d	1.17e	19.8d	7.0f	36.2e
70	16	0.8e	73.0abc	4.24a	303.0f	0.45c	1.81c	26.2b	10.9bc	67.8bc
70	54	50.2b	76.0ab	3.86f	1021.6c	0.37d	1.43cde	20.0d	10.2cd	46.5d
50	35	68.7a	29.5e	3.67g	117.0g	0.74b	4.57a	30.6a	11.2abc	74.4ab
90	35	68.9a	69.6c	3.78fg	2811.4a	0.40cd	1.37ed	18.8d	7.4f	34.5e
70	35	21.0d	72.6abc	4.05cde	522.4e	0.35d	1.43cde	23.4c	12.3a	52.0d
70	35	25.4d	71.6abc	4.11bcd	438.0e	0.38d	1.73cd	21.6c	11.4abc	50.0d
70	35	29.0cd	72.0abc	4.02de	438.8e	0.38d	1.68cd	19.9c	11.8ab	50.4d
CV (%)		12.8	4.5	1.0	5.5	6.09	8.1	2.37	5.19	5.98

\*Mean values within each column followed by different letters are significantly different according to Tukey's test (p < 0.05).

The highest raisin flesh firmness was obtained with drying temperature x times of 50°C x 35 h, and 90°C x 35 h, whereas the lowest firmness was obtained with 84°C x 22h, 56°C x 48 h, and 70°C x 16 h (Table 3). The highest SS content was observed in response to the combined effect of drying temperatures x time at 84°C x 22 h, 70°C x 16 h, 70°C x 54 h, and 70°C x 35 h, whereas the lowest SS contents were observed

at in response to drying at 56°C x 22 h, and 50°C x 35 h. The highest pH values were observed in response to drying temperatures x times of 84°C x 22 h, 56°C x 48 h, and 70°C x 16 h. The lowest pH values were obtained with drying temperatures x times of 70°C x 54 h, 50°C x 35 h, and 90°C x 35 h. The highest total phenol content was observed with the drying temperature x time of 90°C x 35 h, whereas the lowest total phenol contents were

observed with drying temperatures x times of 56°C x 48 h, and 70°C x 16 h. The highest water activity was observed with drying temperature x time of 56°C x 22 h and the lowest water activity was observed with drying temperatures x times of 84°C x 48h, 70°C x 35 h and 70°C x 54 h. The highest average berry weight was observed with drying temperature x time of 50°C x 35 h, whereas the lowest average berry weights were observed with drying temperatures of 84°C x 48 h, 70°C x 54 h, 90°C x 35 h, 70°C x 35 h.

#### 'Italia' raisin consumer acceptance

The processing quality of raisins is determined by genotype, as well as growing and drying conditions (Angulo et al., 2007; Meng et al., 2011). During the drying process, temperature and time are the most important factors affecting raisin processing quality (Mousa & Farid, 2002, Clary et al., 2005; Zhang et al., 2005). Accordingly, our study shows that both drying temperature and time had statistical significant effect on consumer's overall acceptance of raisins, which was the parameter used to determine the most efficient combination between drying temperature x time. The highest overall acceptance of 'Italia' raisins was located in the experimental central point with drying temperature of 70°C and drying time of 35 hours (Figure 1). At the central point, increasing or decreasing drying temperature and time is increased, the overall acceptance of 'Italia' raisins decreases. The most efficient drying

conditions was determined as described by Rodrigues and lemma (2009), which determines the lowest drying temperature and time required to reach the consumer's overall acceptance similar to the central point. The most efficient drying condition was estimated as 59°C for 28 h. This drying condition represents lower processing cost required to reach the highest consumer's overall acceptance. After estimating the most efficient drying condition, the study was repeated to validate the results.

#### Sensory quality and physico-chemical attributes influencing overall consumer's acceptance

The Pearson's correlation analysis of consumer's overall acceptance and physico-chemical attributes are presented on Table 4. Our results show that consumer overall acceptance was more correlated to consumer flavor acceptability ( $R^2=+0.99$ ) and texture ( $R^2=+0.98$ ) than to appearance ( $R^2=+0.63$ ) and aroma ( $R^2=+0.79$ ). Accordingly, other studies have shown that sensory attributes, such as flavor, have a strong influence on consumer overall acceptance (Chauvin et al., 2009; Oupadissakoon et al., 2010).

The color analysis showed that lightness (L) was poorly correlated to overall acceptance ( $R^2=+0.11$ ). Increasing temperature and time resulted in darker raisin coloration. The low correlation between L and overall acceptance is possibly due to the fact that raisin darkening increased overall acceptance up to an optimal

**Table 4.** Pearson correlation coefficients ( $R^2$ ) between consumer's overall acceptance and sensory or physico-chemical attributes of 'Italia' raisins dried at different temperatures and times.

Sensory attributes	$R^2$	<i>p</i> -value
Appearance	+0.63	0.0800
Aroma	+0.79	0.0040
Flavor	+0.99	0.0001
Texture	+0.98	0.0001
Physico-chemical attributes	$R^2$	<i>p</i> -value
Lightness (L)	+0.11	0.66
Chroma (C)	+0.60	0.17
Hue angle (h°)	+0.35	0.34
Firmness	-0.56	0.05
Soluble solids	+0.10	0.77
pH	+0.63	0.03
Total phenol content	-0.68	0.02
Water activity	+0.02	0.95
Berry weight	-0.10	0.77

darkening. Further increase in drying temperature and time resulted in undesirable darkening, reducing overall acceptance. Accordingly, in the central point (70°C and 35 hours) samples reached the highest overall acceptance and with intermediate L values. Among color parameters, the C values showed the highest correlation ( $R^2=+0.60$ ) to overall acceptance, suggesting that color intensity had an important contribution to the acceptability of raisin visual appearance. Higher C values resulted in higher appearance acceptability and consequently higher overall acceptance. The  $h^{\circ}$  values showed an intermediate correlation ( $R^2=+0.35$ ) to overall consumer acceptance compared to L or C values, suggesting that increasing  $h^{\circ}$  increases overall acceptance, but too high  $h^{\circ}$  values reduced overall consumer acceptance of 'Italia' raisins by consumers.

Flesh firmness of 'Italia' raisins was negatively correlated to consumer's overall acceptance ( $R^2=-0.56$ ), showing that with a flesh firmness increase, the overall acceptance was reduced. Flesh firmness is an important component of texture, which explains its high contribution to the final overall acceptance of 'Italia' raisins. The soluble solids content showed a positive correlation to overall acceptance, which is expected since sugars positively influence consumer likeness of different kind of foods (Crisosto et al., 2004; Crisosto et al., 2005). The pH showed a positive high correlation to overall acceptance, suggesting that the increasing pH resulted in higher consumer acceptance of 'Italia' raisins. Other studies have also showed that increasing pH (decreasing acidity) is associated to higher consumer likeness of fruits and processed products (Harker et al., 2008; Iglesias & Echeverría, 2009). The combined perception of sweetness and sourness (acidity) has also been reported to strongly affect consumer likeness (Crisosto et al., 2004). In addition to sugar and acid contents, phenols can also determine consumer flavor acceptability and overall acceptance (Lesschaeve & Noble, 2005; Chiremba et al., 2009). Our results show that total phenol content is negatively correlated to overall consumer acceptance. Accordingly, bitterness and the mouthfeel sensation of astringency

are elicited primarily by phenols (Lesschaeve & Noble, 2005; Del Llaudy et al., 2008). Therefore, increasing phenolic concentration during the drying process possibly increased bitterness and astringency, decreasing consumer's overall acceptance of 'Italia' raisins.

### Conclusions

'Italia' grapes dried at 59°C for 28 h was the most efficient processing to reach the highest overall consumer acceptance.

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