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Postharvest dehydration of Syrah grapes (*Vitis vinifera* L.) under controlled temperature conditions with real-time monitoring of mass loss

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The partial dehydration of grapes after harvest aimed at winemaking has been shown to be a process that brings increased concentration of sugar and phenolic compounds in the must, which affects the quality of the wines produced. However, the works developed so far have studied the process for temperatures up to a maximum of 25°C and air velocity lower than 1 m.s^{-1}. This study aimed to analyze the physical-chemical changes concentration of total soluble solids (TSS) and phenolic compounds (PC) after partial dehydration of 'Syrah' grapes subjected to two treatments combining two temperatures and one air velocity (T_1= 22.9°C/1.79 m.s^{-1} and T_2 = 37.1°C/1.79 m.s^{-1}) and relative humidity of 40%. The water loss of the grapes was approximately 14% and the drying process lasted between 34 and 68 h for treatments T_2 and T_1, respectively. We experimentally and statistically verified that the treatments promoted significant increase in TSS and PC; however, for PC at the temperature of 37.1°C, the increase accounted for approximately 27%, whereas, for the temperature of 22.9°C, it was only 12%. For TSS, the increase was on average 12.47±0.9% between both treatments. The results demonstrate that it is possible to moderately dry grapes, which consequently results in improvements in their chemical composition and can improve the quality of wine.

Key words: Wine, polyphenols, soluble solids, drying, instrumentation.

INTRODUCTION

In the last decade, studies began emerging confirming that controlled postharvest dehydration could enable not only cost savings with adjust of grape must, but also provide superior quality wines (Bellincontro et al., 2004; Constantini et al., 2006; Moreno et al., 2008; Barbanti et al., 2008). The literature reports that advanced studies

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have been developed in Europe aiming to identify the effect of postharvest dehydration on specific components of the grapes. De Sanctis et al. (2012) identified wide variations in the composition of the main carotenoids, genistein and diazine of white grapes dehydrated at low temperature with forced air ventilation. In Rizzini et al. (2010), changes in the gene transcription profile of 'Raboso Piave' grapes were identified after dehydration of up to 30% of grapes. Recently, Panceri et al. (2013) highlighted the effects of moderate dehydration in the mineral content, phenolic compounds and antioxidant activity in 'Merlot' and 'Cabernet Sauvignon' grapes.

According to Zoccatelli et al. (2013), physical-chemical changes caused by harvesting reflect specially activated metabolic processes, which not only modify the composition, but also the structure, such as the cell wall polymers of the skin. The understanding on the especially activated metabolic processes and their effects has been reported by Santonico et al. (2010), Cirilli et al. (2012) and Bonghi et al. (2012). Note that the submission of the product to a condition of stress results in the action of defense mechanisms, especially secondary metabolites, which are precursors of important components of the grape for the winemaking process. According to Xi et al. (2013) the stress caused by temperature difference induces the synthesis of phenolic compounds and aromatic substances, Mencarelli and Tonutti (2013) justify the postharvest partial dehydration of grapes in low temperature because of the benefits of the increased phenolic content and concentration of sugars, since important organic components are volatilized at certain temperature levels above the room temperature (Mori et al., 2005).

In this study we sought to analyze the potential of the partial dehydration under controlled temperature conditions to increase the soluble solids and total phenolic content in the grape must of the Syrah cultivar.

MATERIALS AND METHODS

Raw material

Grapes of the Syrah cultivar (*Vitis vinifera* L.) of the June 2013 harvest were collected in the city of Indaiatuba, State of São Paulo. The grapes were stored in cardboard boxes with a capacity of 7 kg and transported to the Laboratory of Thermodynamics and Energy of the School of Agricultural Engineering at the University of Campinas (LTE - UNICAMP). After completion of the pre-cleaning of the grape clusters to remove stems and grapes damaged or compromised by the presence of fungi, we carried out the distribution of samples according to the heat treatment to be applied and the subsequent analyses of the physical-chemical characterization.

Partial dehydration of grapes

For each treatment, the fruits were stored in plastic package with 25% of effective opening area (50 x 30 x 25 cm), containing 7 kg of grapes, being the bunches longitudinally arranged. The package was inserted inside an adapted tunnel (80 x 40 x 80 cm) with a cooling system with forced air (air flow rate of 2,900 $m^3.\ h^{-1}$), which is installed inside a cooling room (cooling capacity of 4,400 $kcal.h^{-1}$ at -10°C) (Figure 1a). The system is instrumented with sensors for temperature, relative humidity of the air and mass measurement (Figure 1b). The system instrumentation was already carried out in previous works of Silva and Teruel (2011).

The temperature sensors are of the Pt 100 type (FM= 0 at 100°C; model TR106; 4 to 20 mA; accuracy = ±0.2%); the ones to measure the relative humidity are of the RHT-WM type with compact electronic module and transmitter of values (FM= 0 at 100%UR; 4 to 20 mA; accuracy = ± 1.5%); and to measure mass, we used a weighing system comprising a load cell, model PW12C3 – IMB (50 N (50 kgf), sensibility of 2±0.1% $mV.V^{-1}$).

The application for the real-time monitoring was developed in the graphical environment of the Labview programming software (National Instruments). The information related to the sensing instruments is integrated into a central processing and data acquisition unit according to the diagram in Figure 2.

The data acquisition of the temperature and relative humidity was carried out by the data acquisition board (PCI-NIDAQ 6229) coupled to the connector block (CB-68LP) both from National Instruments. This board has as inputs the analogical values of temperature and relative humidity expressed between 4-20 mA and as outputs a voltage that acts on the frequency inverter compressor and exhaust fan and can vary between 0 and 10 V depending on the desired cooling efficiency and air velocity. The digital data of the electrical meters and weighing system are transmitted to the microcomputer systems via Modbus protocol through the RS485 serial port, thus enabling data to be read and stored.

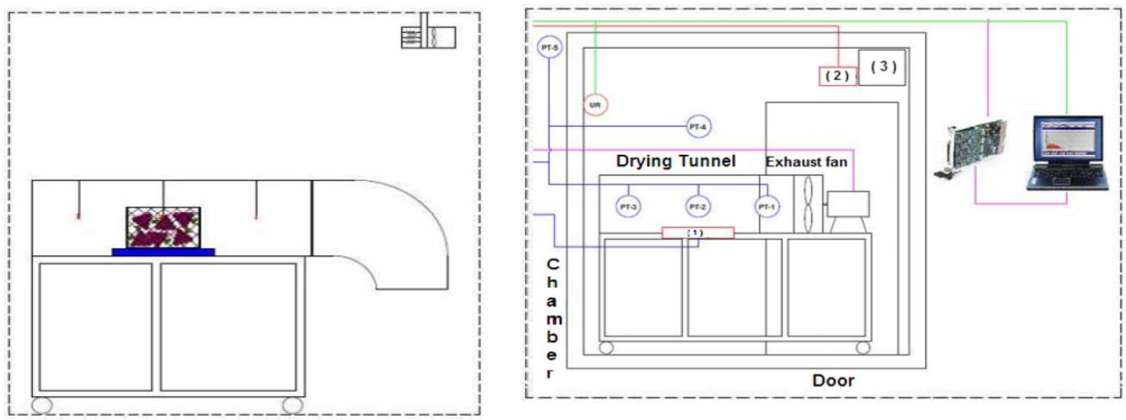
All signals obtained with the instrumentation system, after being processed by the computer, are displayed in real time on the application of supervision and are available as a source of information and support to the decision-making related to changes in the parameters governing the kinetics of the process, such as temperature and velocity of the drying air. The data are structured from sample means at each minute and stored in spreadsheets for further analysis.

Physical-chemical analyses

Before and at the end of each test, samples were selected for the physical-chemical analyses. We proceeded with the random removal of six berries per bunch from a total of 28 bunches by treatment, after which the berries were separated in three repetitions. Remotion of berries was considered in the methodology proposed by Araújo et al. (2009), in which the selected berries must have their representative location for the regions of the base, middle and apex of the bunch. Then, the selected samples are macerated for must preparation followed by the respective analyses. The characterization of the must was based on specific methodologies standardized by the Adolfo Lutz Institute (2005). To analysis of Total Soluble Solids (TSS) in °Brix was used a refractometer model Pocket Pal-1, manufactured by ATAGO.

Moisture content, in dry basis, was determined by drying a sample (100 g) in an oven (model MA035/1, manufacturer Marconi) with forced air circulation at 60°C, until reaching the constant weight of the sample.

The concentration of Total Phenolic Compounds (TPC) was quantified in mg of gallic acid per 100 g of must, according to the methodology described by Obanda and Owuor (1997), in which an extraction solution consisting of 90% ethanol solutions and concentrated HCl is applied in each sample of must content. From the aqueous extract of each sample, 0.5 ml is put in a vial and 4.75 ml of distilled water and 0.3 ml of Folin-Ciocalteu reagent are added. The solution is homogenized and, after 3 min, 0.9 ml of a



(a) (b)

Figure 1. (a) Schematic of the structure of forced-air drying; (b) Schematic of the system instrumentation. Legend: (1) Scales; Resistance (2); Evaporator (3).

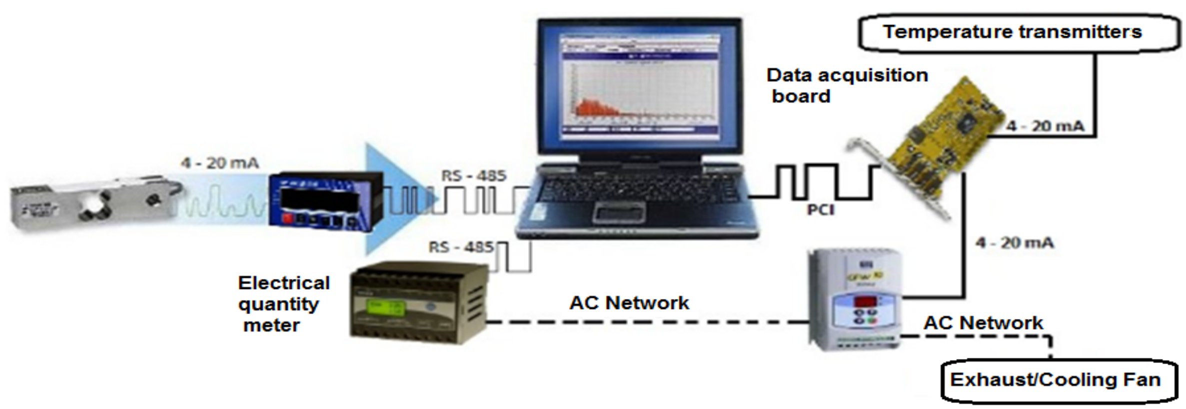


Figure 2. Diagram of the instrumentation.

saturated solution of $NaCO_3$ is added. After resting for one hour at the temperature of 75°C, absorbance readings are performed in triplicate to each must sample in a spectrophotometer at 465 nm. Gallic acid was used as standard, at the concentrations of 100, 150, 250, 500 and 1000 mg.l^{-1} to construct a calibration curve (Figure 3). From the straight line obtained, we carried out the calculation of the total phenolic content, expressed in milligrams of Gallic acid. 100 g^{-1} of grape must (vargas, 2008).

Experimental design

The experimental design was completely randomized with two treatments; the effects of treatments were evaluated in pairs by comparing the values before and after treatment. The treatments were a combination of two temperatures (T_1 = 22.9°C and T_2 = 37.1°C) with an air velocity of 1.79 m s^{-1}. These temperature values were defined based on subsidies obtained from previous works (Santiago et al. 2013) in which a range of temperature between 20 and 50°C were studied, and the best results obtained for the concentration of soluble solids and phenolic compounds with greater weight loss were the temperature values of 22.9 and 37.1°C. All the values of the physical-chemical analyses are the averages of three repetitions of the samples (± SE). The analysis of variance (ANOVA) was performed on the data obtained and the Tukey's test was conducted to identify significant differences of $p < 0.05$ between the samples.

RESULTS AND DISCUSSION

Partial dehydration of grapes

The preliminary characterization of the grapes in the processing units fully favors the decision making for the process, because, according to studies developed by Barnabé and Filho (2006), it is recommended that the comprising of some parameters in a specific range of values for winemaking or juice. Therefore, decisions regarding the need to adequate raw materials, as well as the technique to be used in their suitability that provides a lower cost can be taken more effectively. Seeking the better ways to improve the wine quality, Santiago et al.

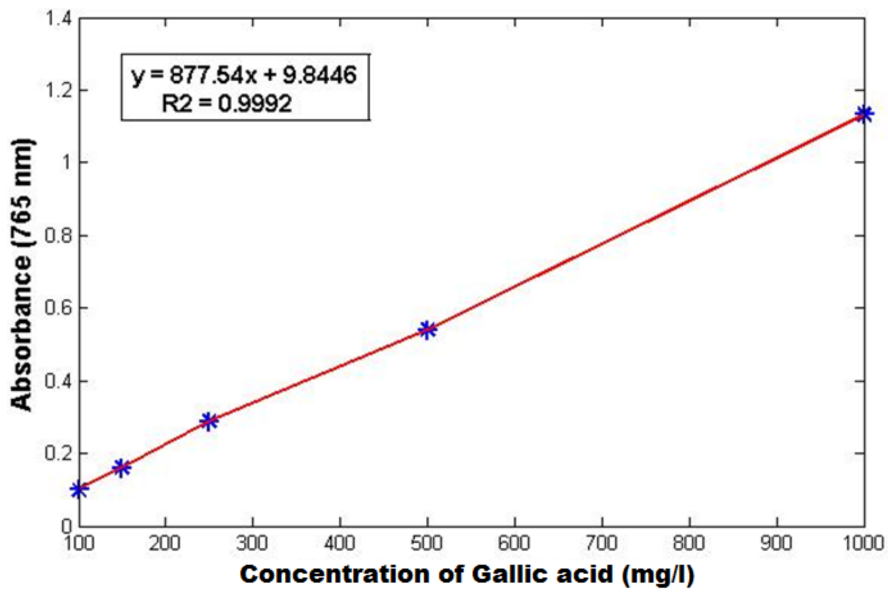


Figure 3. Calibration curve for total polyphenol concentration.

Table 1. Physical-chemical characterization and analysis of variance for the concentration of total soluble solids (TSS) and concentration of phenolic compounds (CPC) in the must.

Test (ºC. m.s^{-1})	TSS (°Brix)		CPC (mgGallicAc.100 g^{-1} must)		TA (mEq. L^{-1})		pH		MC (d.b.)	
	Before	After	Before	After	Before	After	Before	After	Before	After
T_1 (22.9/1.79)	19.93	22.33*	849.33	1080.66*	115.91	133.10*	3.32	3.10*	3.22	2.84*
T_2 (37.1/1.79)	19.93	22.50*	995.33	1118.00*	115.91	122.54*	3.32	3.03*	3.22	2.88*
Mean	19.93	22.42	922.33	1099.33	115.91	127.82	3.32	3.06	3.22	2.86

*Parameters with significant change at 95% confidence level compared to initial value.

(2013) studied the potential of the process of partial dehydration of grapes “Niagara rosada” in improving/adjusting the quality of the grape must to make wine. The authors had high index of changes on phenolic compounds and total soluble solids, but long time to process was necessary. The results of the physical-chemical analyses of the characterization of the grapes before the treatments for the parameters soluble solids, expressed as a percentage of soluble solids concentration, polyphenol content in mg of gallic acid/mg of juice, hydrogen potential (pH), titratable acidity and moisture on wet base are presented in Table 1.

The results confirm one of the premises already investigated to date, which demonstrates that the effects of controlling the thermal and psychometric parameters of the dehydration process provide beneficial changes in the concentration of phenolic compounds and total soluble solids. The acidity in food is the result of the organic acids present in its composition and those occurring after physical-chemical changes in the composition. In the case of grapes, the acidity is also affected by the effect of certain fermentation yeasts that can produce organic acids, as well as the dissolution of minerals and acids released from the skin and pulp (Rizzon and Miele, 2002). The values found were similar to those observed in the literature for the cultivars used in winemaking in Brazil (Rizzon and Miele, 2002; Manfroi et al., 2004).

The mean final of moisture content in dry basis of the samples after the treatments was 2.86%. The results for TSS ranged from 19.93 to 22.50%, while the content of phenolic compounds varied from 849.33 to 1118.00 mg of Gallic acid.100 g^{-1} of grape must, showing statistically significant changes for all parameters evaluated at both temperatures. The significant effect of the treatment at 37.1°C may be associated with the fact that the high temperature can disrupt or break the pectin molecules of the skin, thus allowing the release of the phenolic compounds present there (Vedana et al., 2008).

Although there are advances in the quality obtained using European cultivars (*Vitis Vinifera*) as Syrah grapes,

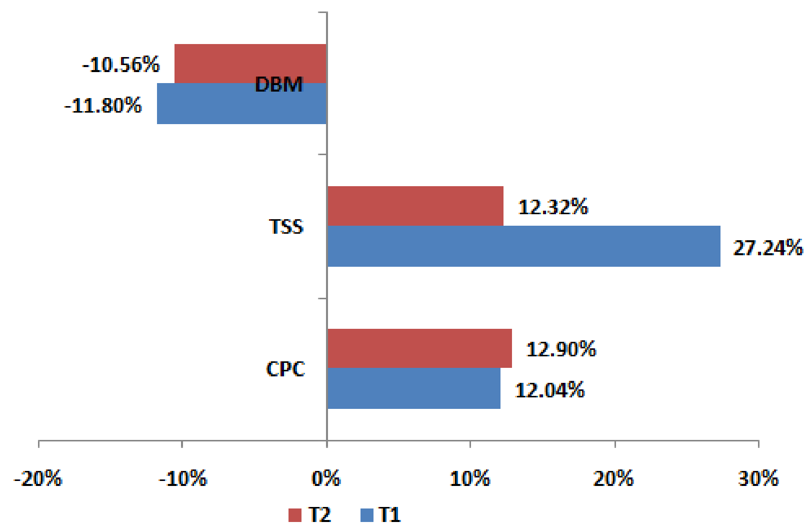


Figure 4. Gain ratio by TSS and CPC through partial dehydration of grapes.

further efforts are needed to ensure that the final product can become superior to the point of being competitive with those imported. Traditionally, the main postharvest process used in brazilian wineries to increase the quality or suitability of the raw material for winemaking has been the chaptalization (Rizzon and Miele, 2005). Chaptalization is the addition of sucrose to the wine must before or during fermentation stage; this technique is normally used for the enrichment of alcohol grading of wine when the must used to make this cannot naturally reach the desired level (Rizzon and Miele, 2005; Pineau et al., 2011). So, the results displayed on Figure 4 indicate that partial dehydration of grapes to winemaking have a great potential to replace the chaptalization technique in brazilian wineries, once which the results from this research agree with previous results descripted on scientific literature (Bellincontro et al., 2004; Barbanti et al., 2008; Mencarelli and Tonutti, 2013).

In the evaluation of the effectiveness of applied heat treatment, it is noted that both conditions are promising and could be applied if carried out under controlled conditions, once the smaller gains for the main physicochemical parameters evaluated were 12.04 and 12.32% to phenolic compounds and total soluble solids, respectively. Furthermore, the identified changes show that temperatures above the ambient and under controlled conditions can lead to increased levels of phenolic compounds without causing volatilization of compounds or damages the final quality of the wines, providing a new analysis of previous work done in Europe, where dehydration has been performed only at temperatures between 10 and 25°C (Bellincontro et al., 2004; Barbanti et al., 2008; Zoccatelli et al., 2013). According to Barbanti et al. (2008), usually the loss of water in grapes at psychrometric ambient conditions and without control may last from 90 to 120 days to reach the optimum vinification, and the grapes may still lose up to 40% of mass, exceeding the 20% limit recommended by the International Code of Oenological Practices (2006).

In a study carried out by Zoccatelli et al. (2013) with three cultivars of *Vitis vinifera* grapes ("Corvina", "Sangiovese" and "Oseleta"), maximum water loss was 30% at temperature ranging from 7 to 16°C. Dehydration lasted up to 100 days (2400 h), for the cultivars "Corvina" and "Sangiovese", and 47 days (1128 h), for the variety "Oselata". Evaluating the results of the parameter total soluble solids in the same dehydration pattern achieved in this work (approximately 11%), we can note that the mean of the values observed herein was 6.4 and 4.1% higher than those for the cultivars "Corvina" and "Oseleta" and 0.6% lower than the value obtained for the cultivar “Sangiovese," respectively. It is important to highlight that while the dehydration of the three cultivars at the approximate level of 11% of water loss was 13 days (312 h), the maximum time required for the samples of this work to reach the same level of water loss was 3 days (72 h), that is, the dehydration method using the forced air system and the temperatures of 22.9 or 37.1°C provides a reduced process time and a statistically significant increase of the quality parameters of the partially dehydrated grape must.

Conclusion

The results obtained so far open a new perspective of application from this technology of partial dehydration of grapes on temperature of 22.9°C for the wine sector, helping with both the standardization of pre-fermentation from must and with the increase of final quality of wines done from Syrah grapes on Brazil. The significant reduction of time spent to remove the amount of water content on the grapes before the winemaking together with the increase of physicochemical properties evaluated would contribute for the advancement of relationship cost-benefit of processes involved inside the wine productive chain.

Conflict of Interest

The authors have not declared any conflict of interest.

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