Encapsulation of phenolic compounds by spray drying of Ancellotta and Aspirant Bouchet wines to produce powders with potential use as natural food colorants

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Abstract

Ancellotta and Aspirant Bouchet, *Vitis vinifera* L., are wines with a high anthocyanin concentration that have been used to increase color in high-quality red wines. This work was focused on the encapsulation of Ancellotta (ANCE) and Aspirant Bouchet (AB) wines by spray drying and obtaining wine powders (WP) with a high concentration of phenolic compounds. Spray drying conditions were optimized and the phenolic compounds in the WP were characterized. All WP were evaluated for moisture content, water activity, color parameters, anthocyanins, and flavan-3-ols composition. There was no significant difference in total anthocyanins content of ANCE and AB WP when inlet air temperature increased from 135 to 145°C with a fixed carrier concentration of 8% (*w*/*w*) but a decrease in the total anthocyanins was observed when carrier concentration increased to 10% (*w*/*w*). Regarding color evaluation, ANCE and AB WP corresponded to the fourth quadrant of color space CIELAB indicating blue - red color (348–357°), characteristic of anthocyanins and wine. In general, the values of hue angle were not affected at higher percentages of carrier agent, consequently, an increase in the ratio carrier-wine did not lead to a dilution of material. These results indicate that inlet air temperature (in the selected range) did not influence the anthocyanin profile in the WP and has allowed for optimization of the conditions to obtain phenolic-rich colored powders with potential use as natural antioxidants and food colorants.

Keywords: Ancellotta – Aspirant Bouchet - anthocyanins – spray drying – encapsulation –– natural colorants.

1. INTRODUCTION

Over the last decades, consumers have become more conscious of food ingredients, boosting the food industry in the search for novel sources and processes to obtain natural ingredients. On the other hand, the wine sector is evolving because of the new trends in wine consumption. Moreover, the arise of the non-alcoholic wine market has generated new demands related to wine-based products (Di Giacomo & Romano, 2021).

Red wine contains a wide range of phenolic compounds that are extracted from red grape skin and seeds during winemaking. This is why red wine consumption has been positively correlated with health benefits due to its composition of phenolic compounds content (Opie & Lecour, 2007; Apostolidou et al., 2015). Nevertheless, the medical community recommends only moderate consumption of red wine, due to the risks linked to excessive alcohol consumption (Weiskirchen & Weiskirchen, 2016). Moreover, part of the population abstains from wine and alcohol consumption for various reasons (religion, culture, health, age, etc.) and are deprived of the advantages of these compounds (Stanco et al., 2020).

The potential health benefits of phenolic compounds are mainly related to their antioxidant activity (Lu et al., 2021). Many research studies have evidenced that an increase of these compounds in the diet could contribute to the reduction of oxidative damage and chronic diseases (Chen et al., 2020; Cianciosi et al., 2020). According to their chemical structure, phenolic compounds can be divided into flavonoids and non-flavonoids. The main flavonoid compounds present in red wine comprise several classes such as flavanols [e.g., (epi)(gallo)catechins, procyanidins and prodelphinidins], flavonols (e.g., myricetin and quercetin) and anthocyanins (e.g., malvidin-3-glucoside), while non-flavonoid compounds present in wine include phenolic acids, phenols, and stilbenes (Fernandes et al., 2017). During winemaking, the phenolic compounds are extracted from grape skin and seeds. In the particular case of anthocyanins, they are extracted since the beginning of fermentation whereas flavan-3-ols require the presence of ethanol for extraction (Maza et al., 2019). Furthermore, chemical reactions of phenolic compounds are particularly important in wine because they are responsible for color and taste changes that occur in wine during winemaking and aging (Fernandes et al., 2017).

In the food industry, color is one of the main sensory parameters that influence consumers' choices. Anthocyanins are water-soluble phenolic compounds responsible for blue, red, or purple color in fruits, flowers, vegetables, and wine (Sinopoli et al., 2019). Therefore, anthocyanins can be used as natural food colorants with health benefits. However, one general disadvantage of anthocyanins, and phenolic compounds, is their lack of long-term stability being susceptible to degradation, oxidation, epimerization, and polymerization during production and storage processes. Light, heat, pH variations, enzymatic activities, humidity, metal ions, and oxygen can modify their color and antioxidant capacity (Galmarini et al., 2013; Turturică et al., 2015).

Encapsulation by spray drying is defined as the entrapment of a bioactive compound or sensitive ingredient (core material) within an immiscible substance (carrier). The carrier provides a physical barrier between the core compounds and the environment (Tyagi et al., 2011; Mahdavee Khazaei et al., 2014). The main advantage of spray-drying compared with other encapsulation technologies is that it is more suitable for industrial scale-up, and heat-sensitive substances and it improves stability and shelf life at a reasonable cost (Tyagi et al., 2011; Ozkan et al., 2019).

Parameters in the spray drying process, such as inlet and outlet air temperatures, carrier concentration, and type of carrier have a direct impact on final product characteristics (Fazaeli et al., 2012). Additionally, material carrier selection for encapsulation plays a crucial role in efficient spray drying (Tolun et al., 2016). Maltodextrin (MD) is extensively used as a carrier material in spray drying to satisfy demand and low cost, non-toxicity, exceptional biocompatibility, high solubility, low viscosity, odorless and tasteless and turned out to be essential in preserving the integrity of anthocyanins during their encapsulation (Fang & Bhandari, 2010; Lu et al., 2021). Nevertheless, previous studies have demonstrated that a combination of MD with gum arabic (MD:GA) has an acceptable level of water solubility, encapsulation efficiency, and stability, and helps to overcome some difficulties, such as stickiness and high hygroscopicity, that could appear during spray drying process when using only MD (Aguiar et al., 2016; Labuschagne, 2018).

The drying/encapsulation of red wine in presence of adequate carbohydrates (e.g., MD) can lead to water and alcohol removal obtaining a glassy amorphous microstructure in which the phenolic compounds of wine are entrapped (Alvarez Gaona et al., 2018a). Moreover, the wine industry can take advantage of using wines with some alterations originating in the vinification process (e.g., volatile acidity, *Brettanomyces* taint), overstock wine, and wine by-products as raw material to encapsulate wine’s phenolic compounds (Alvarez Gaona et al., 2018a,b), thus helping the sustainable development and promoting the circular economy in the wine industry.

Aspirant Bouchet (AB) is a teinturier grape (*Vitis vinifera* L.) that has red anthocyanin pigments in the pulp and skin of the berry. It is used as a color enhancer in high-quality red wines, and it is cultivated worldwide for this purpose. Furthermore, AB teinturier varieties typically contain monoglycosylated anthocyanins, malvidin-3-glucoside is the most important pigment in this grape variety (Muñoz et al., 2009). Therefore, AB as teinturier wine can be used to produce natural color powders rich in bioactive compounds such as anthocyanins. On the other hand, Ancellotta (ANCE) is not a teinturier grape variety, but the wine obtained from it has high anthocyanin content as compared with other wine varieties namely Malbec, Merlot, or Pinot Noir. The differences between wines in the present work were reflected in their dry extract values of 2.57 ± 0.06 % *w*/*w* and 3.22 ± 0.06 % *w*/*w* for ANCE and AB wine, respectively. In Argentina in the last two decades, the cultivated surface of AB has increased by +3.178 acres and by +1.983 acres for the ANCE variety (INV, 2021).

In a previous study Alvarez Gaona et al., (2018a) reported that the threshold inlet air temperatures required for successful spray drying of Cabernet Sauvignon wine were comprised between 135 and 170°C, whilst carrier amount could be lower than 13.5% (*w/w*). However, carrier type and percentage addition lower than 13.5%, were not studied. Alvarez Gaona et al., (2019) used ANCE wine to obtain a WP by spray drying and its potential use as a natural colorant in the food industry was explored. Therefore, the inlet air temperature threshold and the carrier type and percentage were selected as starting points for this study. So far there is no published data related to detailed phenolic compounds from Argentinean AB wine and there are no studies related to spray drying of AB wine to be used as a natural powder colorant.

Hence, the objectives of this research were to characterize, by HPLC-DAD-MS, the anthocyanin and flavanol composition of Argentine ANCE and AB wines and to evaluate spray drying conditions, namely inlet air temperature (135°C and 145°C), carrier percentage (8% and 10 % *w*/*w*) and type of carrier material (MD_{DE10} and MD:GA) to obtain encapsulated WP. The study was conducted in both wine varieties to evaluate the performance of a teinturier wine (AB) and a non-teinturier wine (ANCE).

2. MATERIALS AND METHODS

2.1. Reagents

The carrier materials used were maltodextrin DE10 Dextrose Equivalent 10 (MD_{DE10}) from Ingredion S.A (Buenos Aires, Argentina) and gum arabic (GA) from Gelfix (Buenos Aires, Argentina).

All solvents were HPLC grade, and all chemicals were analytical reagent grade. Acetonitrile (Fisher Scientific, Waltham, MA, USA/ Labkem, Barcelona, Spain), trifluoroacetic acid (TFA) (Riedel-de Haen, Hanover, Germany), and formic acid (PROLABO, Fontenay-sous-Bois, France) were used as solvents in HPLC analysis.

Anthocyanin standards were purchased from the Polyphenol Laboratory (Sandnes, Norway).

Chlorogenic acid was purchased from Sigma-Aldrich (St. Louis, MO, USA) and added as an internal standard in a final concentration of 0.025 mg mL^{-1} for the flavan-3-ols analysis.

Ultrapure water was obtained from a Milli-Q Element water purification system (Millipore, Milford, Mass., U.S.A.).

2.2. Red wines

Monovarietal dry red wines, namely, *Vitis vinifera* L. cv ANCE and AB from the 2021 vintage were produced, provided, and analyzed by Experimental Agricultural Station Mendoza, National Institute of Agricultural Technology (INTA) after fermentation and stabilization.

The values reported by INTA for ANCE and AB wine compositions were as follow: alcohol % (*v/v*) 12.07 and 12.05; total acidity 5.59 g and 5.15 g as tartaric acid/L; dry extract 2.57 ± 0.06 % *w/w* and 3.22 ± 0.06 % *w/w*; pH 3.80 ± 0.01 and 3.30 ± 0.01 for ANCE and AB, respectively.

2.3. Experimental design and spray-drying

Spray drying experiments were performed according to experimental design with 2-factor levels (+1 high level and –1 low level), with a total of 8 treatments (C1 to C8; **Table 1**). Inlet drying air temperature (135°C and 145°C), type (MD_{DE10} and MD:GA), and percentage (8% and 10 % *w/w*) of the carrier were selected as independent variables. Moisture content, water activity, anthocyanin, flavan-3-ols content, and CIELAB parameters were the variables selected as responses.

Encapsulation was performed in a mini spray dryer Buchi model B-290 (Büchi Laboratoriums Technik, Switzerland), under operating conditions previously used by Alvarez Gaona et al., (2019): feed flow rate 600 g/h; flow meter spraying air (rotameter) 30 mm; 0.23 bar pressure drop; and 439 L/h actual volume flow (at standard temperature and pressure).

2.4. Water activity

Water activity (a_w) was measured using a dew-point hygrometer Aqualab Series 3 (Decagon Devices, USA), previously calibrated against standard saturated salt solutions.

2.5. Dry Extract

Ten (10) g of each wine (liquid) sample were weighed in tared glass containers and dried in a constant temperature convection oven for 2 h at 105°C. Then cooled for 1 h in a glass desiccator and re-weighted to calculate the dry extract content.

2.6. Yield of Encapsulation Process

The yield of the encapsulation process was calculated (in percentage, %), as the ratio of the total dry weight of powder obtained (W_2) and the dry weight of material in its feed mixture (W_1).

$$\text{Yield (\%)} = (W_2 / W_1) \times 100$$

2.7. HPLC-DAD-MS analysis of anthocyanins

ANCE and AB wines were analyzed in triplicate utilizing HPLC-DAD-MS after dilution (1:3) in acidified water (HCl 0.1 N) and filtration using a Millex® syringe-driven Filter unit (0.45 mm) (Millipore Corporation, Bedford, MA, USA).

Samples of WP of each treatment were prepared by dissolving 0.06 ± 0.002 g to a final volume of 2 mL with acidified water (HCl 0.1 N). Afterward, it was diluted (1:1) with acidified water (HCl 0.1 N), filtered using a Millex® syringe-driven Filter unit (0.45 mm) (Millipore Corporation, Bedford, MA, USA), and analyzed by HPLC-DAD-MS.

HPLC–DAD analysis was performed in a Hewlett-Packard 1100 series liquid chromatography. An AQUA C18 reverse-phase, 5µm, 150 mm × 4.6 mm column (Phenomenex®, Torrance, CA, USA) thermostatted at 35°C, was used. The solvents used were: (A) an aqueous solution (0.1%) of trifluoroacetic acid (TFA) and (B) 100% HPLC-grade acetonitrile. The gradient was: isocratic 10% B for 5 min, from 10 to 15% B for 15 min, isocratic 15% B for 5 min, from 15 to 18% B for 5 min, and from 18 to 35% B for 20 min, at a flow rate of 0.5 mL min^{-1}. Detection was carried out at 520 nm as the preferred wavelength and spectra were recorded from 220 to 600 nm (Alcalde-Eon et al., 2007). The mass analyses were carried out in positive mode (ESI+) (Alcalde-Eon et al., 2019). Three types of mass experiments were performed: a full mass analysis (EMS mode, collision energy (CE) 10 V), where all ions were detected, and an MS^2 analysis (EPI mode, CE 30 V), where major ion of full mass analysis was fragmented and an MS^3 analysis (CE 30 V, excitation energy (AF2) 80 V), where major fragment ion of the MS^2 analysis was, in turn, fragmented. Spectra were recorded between *m/z* 150 and 1400.

The identification of anthocyanins was carried out using the chromatographic, spectral, and mass spectrometric data and by comparison to literature. Calibration curves prepared from anthocyanin standards (delphinidin, cyanidin, petunidin, peonidin, and malvidin 3-O-glucosides) were used for quantification (Alcalde-Eon et al., 2007).

The total anthocyanin content (TAC) was calculated from the sum of individual concentrations obtained for each compound and was expressed in mg L^{-1} of wine or mg per 100 grams of WP.

2.8. HPLC-DAD-MS analysis of flavan-3-ols

ANCE and AB wines were diluted with acidified water (HCl 0.1 N), in a 1:1 radio (in volume). WP of each treatment was prepared by dissolving 0.06 ± 0.002 g of powder to a final volume of 2 mL with acidified water (HCl 0.1 N). Before HPLC-DAD-MS analysis´ wines and WP samples were fractionated following the procedure reported by Alcalde-Eon et al., (2014). Briefly, 2 mL of diluted samples were loaded on an Oasis MCX cartridge (Waters Corporation, Milford, MA, USA) previously conditioned. After washing with water (4 mL), flavonols were eluted with 8 mL of methanol, afterwards, the eluate was concentrated under reduced pressure and re-dissolved in 0.5 mL of water. Then, chlorogenic acid was added as an internal standard at a final concentration of 1.0 mg min^{-1}.

The HPLC–MS analysis was performed in a Hewlett-Packard 1200 series liquid chromatograph (Agilent Technologies, Waldbronn, Germany). The stationary phase was an Agilent Poroshell 120 EC-C18 column (150 mm × 4.6 mm, 2.7 µm) thermostatted at 25°C. The mobile phase was composed of solvent A, 0.1% (*v*/*v*) formic acid aqueous solution, and solvent B, HPLC grade acetonitrile. The following elution profile was used at a flow rate of 0.5 mL min^{-1}: from 100% to 90% A for 3 min, from 90% to 85.5% A for 34 min, from 85.5% to 80% A for 3 min, from 80% to 65% A for 15 min, from 65% to 40% A for 5 min, and a final isocratic gradient of 40% A for 3 min (García-Estévez et al., 2017). The mass spectrometer was connected to the HPLC system via the DAD cell outlet. MS detection was performed in a 3200 Qtrap (Applied Biosystems, Darmstadt, Germany) equipped with an ESI source and a triple-quadrupole linear ion trap mass analyzer and controlled by Analyst 5.1 software. Multiple reaction monitoring analysis (MRM mode) was employed to detect transitions (each parent ion − daughter ion pair) corresponding to each kind of flavanol and the internal standard following the procedure described by García-Estévez et al., (2017).

The total content of each kind of flavanol was expressed in mg L^{-1} of wine or mg per 100 grams of WP.

2.9. Color

Color parameters of WP were determined using a MINOLTA CM-600d colorimeter (Konica-Minolta INC., USA), with the illuminant D65 and an observation angle of 2°. The measurement was made by placing 1.5 g of each WP in a plastic container with a white background. The values of L* to b* (CIELAB) were recorded directly from the equipment. All measurements were made in triplicate. Chroma (*C**), hue (*H°*), and whiteness index (WI) values were calculated using equations (1) to (3):

$$C^* = \sqrt{a^{*2} + b^{*2}} \quad (1)$$

$$H^\circ = \arctan\left(\frac{b^*}{a^*}\right) \quad (2)$$

$$WI = 100 - [(100 - L)^2 + a^2 + b^2]^{1/2} \quad (3)$$

Color difference (ΔE^*_{ab}) was calculated between treatments of ANCE and AB WP, under the same conditions, as the euclidean distance between two points (1 and 2) in three-dimensional (L*, a*, b*) space (equation 4).

$$\Delta E^*_{ab}\,(L^*_1, a^*_1, b^*_1; L^*_2, a^*_2, b^*_2) = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2\,]^{1/2} \quad (4)$$

where $\Delta L^* = L^*_1 - L^*_2$, $\Delta a^* = a^*_1 - a^*_2$, and $\Delta b^* = b^*_1 - b^*_2$.

2.10. Statistical analyses

All measurements were performed in triplicate. Results were expressed as means ± standard deviation (SD). Data were analyzed by one-way analysis of variance (ANOVA) and was followed by a Tukey's honestly significant difference (HSD) test.

Total anthocyanin and flavan-3-ols contents of ANCE and AB WP were analyzed by two-way analysis of variance (ANOVA) and regression analysis to examine the statistical significance (at $p<0.05$) of the experimental response of inlet air temperature, type, and percentage of the carrier.

All analyses were done using InfoStat software.

3. RESULTS AND DISCUSSION

3.1 Water activity, moisture content, and yield in WP

A_w is an important factor controlling processability, handling properties, and stability of spray-dried powders. Previous works showed that WP with a_w values lower than 0.30 can be stored for 90 days under controlled conditions, with minimal risk of stickiness, caking, and anthocyanins degradation (Alvarez Gaona et al., 2018a,b; 2019).

Table 1 summarizes drying conditions and physicochemical characteristics of ANCE and AB WP. The average a_w value for the eight treatments of ANCE WP was 0.17 ± 0.002 and, the lowest a_w value was obtained with treatment C4 (145°C and 8% MD_{DE10}). The moisture content was, on average, 4.31 ± 0.58% and the lowest value was 3.66 ± 0.06% (also obtained with treatment C4), whereas the highest moisture content was 5.29 ± 0.24% under conditions of treatment C5 (135°C and 10% MD:GA). The moisture contents of ANCE WP obtained were lower than 5.5%, similar to those values previously reported for ANCE WP with MD_{DE10} (Alvarez Gaona et al., 2019), to those from jaboticaba peel extracts spray-dried with MD (Silva et al., 2013) and those from by-products of Bordo grapes using MD as agent carrier (de Souza et al., 2015).

AB WP´s treatments had the same average a_w value as ANCE WP (0.17 ± 0.002). However, the lowest a_w value was obtained with treatment C2 (145°C and 8% MD:GA). With regards to moisture content, the average value was 3.96 ± 0.46%, with the lowest moisture content determined for treatment C6 (145°C and 10% MD:GA) and the highest one for treatment C1 (135°C and 8% MD:GA).

The a_w values in all treatments were below or equal to 0.25, low enough for WP stability (Alvarez Gaona et al., 2018a,b). As for moisture results, the highest content was related to inlet air temperature of 135°C and MD:GA as a carrier, and the lowest outlet air temperature was 67°C (ANCE) and 66°C (AB). Prinn, Constantino, & Tracy, (2002) published that such a low outlet temperature is undesirable because it leads to elevated residual moisture or high-level of a_w in the spray-dried powders. Tonon et al., (2008) indicated that anthocyanins retention would inevitably be cut back an outlet air temperature above 90°C. The WP with 10% of carrier and minor moisture content was due to the increase in total solids before spray drying which reduced the amount of water for evaporation.

Nonetheless, there was not a significant correlation between a_w and outlet air temperature. This may be attributed to the fact that spray drying may be considered a sort of random process during atomization. The breakup of liquid feed into a large number of droplets of different sizes reduces internal resistances to moisture transfer influencing drying time and water content.

Low a_w and moisture values are necessary to avoid caking phenomena during storage and losses of bioactive compounds (Alvarez Gaona et al., 2018a,b; 2019).

--- Table 1 insert here ---

The yield, expressed as a percentage, was calculated as a ratio between the solid amount obtained after the spray drying process and the solid amount in the feed solution (dry base) (**Table 1**). The values were similar to those obtained in previous works made with ANCE and Cabernet Sauvignon WP (Alvarez Gaona et al., 2018a,b; 2019). The highest yield in ANCE WP and AB WP (69% and 67%, respectively) was obtained in treatment C8 (145°C and 10% MD_{DE10}). It should be noted that with both wines the best yield was obtained in treatments with MD_{DE10} (≈ 64%). These values were higher than those determined for spray drying powders obtained for jussara pulp, which ranged between 33.88% and 76.55% (Santana et al., 2016), and for beetroot juice, which ranged between 41.31% and 54.63% (Bazaria & Kumar, 2016).

Fazaeli et al., (2012) and Daza et al., (2016) reported that the temperature process influences the amount of powder recovered. When drying temperature and carrier agents increase leads to an increased yield, which can be attributed to better water removal and the protective effect of the carrier at higher concentrations, avoiding stickiness of material in the dry chamber wall. These results indicate a limit carrier percentage to obtain the highest yield.

3.2. Anthocyanin profile

Table 1 indicates the total anthocyanin retention values for all WP. The percentage retention was calculated considering total anthocyanins content in WP (mg /100 g WP) after the spray drying process and total anthocyanins content in wine (mg /100 g wine). High anthocyanin retention was observed, ranging from 90 to 100% and 86 to 100% for ANCE and AB WP, respectively. Retention of close to 100% suggests that anthocyanins were not affected by temperatures of 135 and 145°C used in the spray drying process. These results agree with previous studies done with Cabernet Sauvignon and ANCE wines (Alvarez Gaona et al., 2018a,b; 2019). Also, similar anthocyanin retention values were reported for Bordo grape skin extract microencapsulated by spray drying using GA (de Souza et al., 2015), partially hydrolyzed guar gum, and polydextrose as encapsulating agents (Kuck & Noreña, 2016). de Souza et al., (2015) found a range of anthocyanins retention among 88.3 – 93.8% when 10% MD was used for encapsulated grape skin aqueous extract.

The highest anthocyanin retention for ANCE WP was found in treatments C3 and C4 with 8% MD_{DE10} as a carrier at both temperatures evaluated (135 and 145°C). Moreover, no effect was shown in retention percentage when increasing inlet air temperature, with the same percentage of MD_{DE10} and MD:GA. In AB WP, the highest retention was observed for treatment C2 (145°C and 8% MD:GA), showing a slight increase in anthocyanin retention when the inlet air temperature was increased. An increase (+10°C) in inlet air temperature used during the spray drying process resulted in minimal effect on anthocyanin content, which is in agreement with results previously reported by Alvarez Gaona et al., (2018a) for spray drying of Cabernet Sauvignon.

As can be observed in **Table 1**, a carrier increase (as %) has a negative effect on retention which can be explained due to a dilution effect. In specific treatments (C8 ANCE; C2, C4, and C6 AB) an increase in inlet air temperature revealed greater anthocyanin retention which could be due to fast evaporation in the spray drying process and to better protection achieved by carrier agent (similar performance with MD_{DE10} and MD:GA). Tonon et al., (2008) observed a decrease in anthocyanin retention in acai juice with increased drying temperatures. Ersus & Yurdagel, (2007) also found loss of anthocyanin with higher spray drying temperatures in black carrots using three types of MD. However, these last authors found a slight increase in anthocyanin content in samples dried at 180°C, compared with those at 160°C, when MD_{DE10} was used, and similar performance was observed in the present work for WP encapsulated MD_{DE10}.

Table 2 summarizes the detailed profile obtained by HPLC-DAD-MS of ANCE and AB wines (mg L^{-1} liquid wine) that were subsequently used in the spray drying process to obtain WP. Results show that the total anthocyanin content was superior in AB than in ANCE wine. Even though it is known that anthocyanin content in AB is higher than in typical wine varieties, the profile adds new information about their composition and distribution.

AB wine, which is a teinturier variety, shows a concentration of anthocyanins almost two-fold higher than ANCE wine (**Table 2**). But, in both wines, the ratio between non-acylated and acylated anthocyanins was very similar, being non-acylated group the main type of anthocyanins, followed by acetylated and *p*-coumaroyl and caffeoyl derivates. Malvidin-3-glucoside was the main anthocyanin in non-acylated and derivates compounds. However, some differences were found in the pigment profile. The main difference is related to a higher content of peonidin and cyanidin derivatives (mainly glucosides) in AB wines than in ANCE. In fact, the ratio between di-substituted anthocyanidins (peonidin and cyanidin) and tri-substituted anthocyanidins (delphinidin, petunidin, and malvidin) in AB wines is 1:3 whereas in ANCE wines is 1:15. Also, the proportion of derivative pigments in AB is lower than in ANCE, mainly the proportion of A-type visitins. This is the first time that AB pigment composition has been reported while the ANCE results coincide with those previously published for ANCE wine (Alvarez Gaona et al., 2019).

--- Table 2 insert here ---

Anthocyanin profile was also determined for ANCE and AB WP after spray drying in order to evaluate the impact of the process on pigment composition. **Figure 1 (a)** and **(b)** show the content of non-acylated, acetylated, *p*-coumaroyl, and caffeoyl, direct condensation products, flavanol-anthocyanin adducts, vitisin vinylphenol pyranoanthocyanins, A and B-type vitisins in ANCE and AB WP spray-dried under different conditions. As was observed in wines, non-acylated derivates were the most abundant group of anthocyanins pigments in WP (71% ANCE and 75% AB) as compared to acylated and *p*-coumaroyl and caffeoyl forms.

--- Figure 1 insert here ---

It should be noted that total anthocyanins content decreased significantly ($p<0.05$) when the carrier was scaled up to 10% (*w/w*) (treatments C5 to C8), independently of carrier type (MD_{DE10} or MD:GA) and wine variety, owing to a dilution effect as mentioned before and suggesting a better encapsulation at 8%. The observed behavior for individual anthocyanin compounds in dried WP may be also attributed to the protection afforded by 8% MD_{DE10} and MD:GA, mainly because the ratio of the carrier: wine dry extract is high (2.5:1 ANCE and 3:1 AB) (Gibbs et al., 1999). In ANCE and AB WP, the decrease of anthocyanin content was not significant in treatments (C5 through C8) with the same carrier %, even though a different carrier type and inlet air temperature were used during the spray drying process. ANCE WP obtained by treatment C3 had the highest total anthocyanins content (1014mg/100 g WP), followed by C4 (995 mg/100 g WP) and C1 (966 mg/100 g WP), all with 8% of the carrier. In AB WP, the greatest content was found for treatment C2 (1539 mg/100 g WP) also with 8% of the carrier.

Results reported by Mahdavee Khazaei et al., (2014) in microencapsulated saffron petal anthocyanin with GA and MD indicated a decrease in anthocyanin content raising MD level. The same phenomenon was reported by Tonon et al., (2010) using different carrier agents including MD for spray drying of acai juice; Jafari et al., (2017) in the anthocyanin content of pomegranate juice powder spray dried with three MD levels, and by Murali et al., (2015) for encapsulation of black carrot juice using spray and freeze-drying using MD_{DE20}. Bednarska & Janiszewska-Turak, (2020) studied the relationship between inlet air temperature (160 and 200°C), carrier type (MD, GA, and MD:GA), and contents of total anthocyanins in chokeberry juice powders obtained after spray drying, and statistical analysis showed that carrier type had no significant impact on anthocyanin content. Similar results were observed in the present work for total anthocyanins and derivates. Therefore, carrier percentage affected the total anthocyanins compounds in the dried WP, but the anthocyanin derivates proportion was maintained (**Figure 1 (a)** and **(b)**).

Overall, considering all spray drying conditions, the most convenient treatments to get an elevated anthocyanin concentration to seem to be: C3 (135°C and 8% MD_{DE10}) for ANCE and C2 (145°C and 8% MD:GA) for AB.

Table 3 shows the detailed anthocyanins profile (mg anthocyanin/100 g WP) of the treatments with the maximum anthocyanin concentration (C3 for ANCE and C2 for AB).

--- Table 3 insert here ---

As expected, malvidin-3-glucoside was the predominant anthocyanin (in wines and WP), mostly followed by malvidin-3-acetylglucoside and malvidin-3-*p*-coumaroylglucoside. The cyanidin derivatives showed the lowest proportion, confirming the behavior observed in previous work (Alvarez Gaona et al., 2019). In terms of anthocyanin distribution according to the type of anthocyanidin, in ANCE WP the second most abundant anthocyanidin was delphinidin and in AB WP it was peonidin, like it was observed in wines. Also, the ratio between di-substituted and tri-substituted anthocyanins was the same in WP as in wine, which confirms that spray-drying does not change the pigment profile of wines.

The achieved results of total anthocyanin retention and profile validate the optimal conditions for ANCE WP were achieved in treatment C3 (135°C and 8% MD_{DE10}), and for AB WP, C2 (145°C and MD:GA) had the highest anthocyanin content. The combination of MD:GA had been previously studied (Akhavan Mahdavi et al., 2016; Mazuco et al., 2018; Papillo et al., 2018) as the most common combination and it has shown better encapsulation properties than MD due to better emulsification. However, in the present study, no significant differences were found in the anthocyanin profile between MD_{DE10} and MD:GA for ANCE and AB WP, which points out that, in the case of wine, this mixture of carrier agents works as MD by itself.

3.3. Flavan-3-ols profile

The flavan-3-ols profiles of ANCE and AB WP (mg/ 100 g WP) were determined by HPLC-MS (García-Estévez et al., 2017). To simplify the discussion of results about flavan-3-ols composition, these compounds were grouped according to the type of flavan-3-ol [catechins and procyanidins (PC) and gallocatechins and prodelphinidins (PD)], and their polymerization degree (monomers, dimers, trimers, tetramers, and pentamers). A detailed profile obtained of ANCE and AB wines vintage 2021 (mg L^{-1}), is shown in the appendix section (**Table A.1**). The identity of each compound was assessed through its chromatographic retention time. The results obtained show that the total flavan-3-ols content was superior in ANCE (339 ± 22 mg L^{-1}) than in AB (267 ± 27 mg L^{-1}) wine, and catechins and procyanidins (PC) were the major groups in both wines varieties. However, the profile shows that the total value of gallocatechins and prodelphinidins (PD) is higher in AB (13 ± 3 mg L^{-1}) wine than in ANCE (8 ± 1 mg L^{-1}).

Figure 2 (a) and **(b)** show the concentration of each group of flavan-3-ol determined for the different treatments assayed in this work.

--- Figure 2 insert here ---

The total flavan-3-ols content in the different treatments of ANCE WP ranged from 424 to 872 mg/100g with a mean content of 634 mg/100 g. Regarding AB WP, the total flavan-3-ols content ranged from 284 to 375 mg/100g with a mean content of 338 mg/100g. As expected, the total flavan-3-ol in AB WP was roughly 50% lower than ANCE WP, bearing in mind that AB is a teinturier variety and that the flavan-3-ol content in wines was higher in ANCE than AB.

In AB WP, the total catechins and procyanidins (PC) derivates percentages were higher than gallocatechins and prodelphinidins derivates (PD), the distribution was found in a range among 93 to 94% for PC and 5.5 to 7.0% for PD, in the eight AB treatments. In terms of distribution, monomer derivates represented 37% of total PD derivates and dimers derivates around 60% of PC derivates.

ANCE WP also showed an elevated percentage of PC and a low percentage of PD derivates, 97%, and ≈ 3%, correspondingly. The monomers derivates corresponded to 40% of total flavan-3-ol and dimers stand out among the total PC derivates with a 60% value. Moreover, in ANCE WP observed a decrease in PC derivates when carriers scale up to 10%. However, this effect was not observed in AB WP.

Finally, for AB WP the highest content was found in treatment C3 (135°C and 8% MD_{DE10}) for ANCE the highest content was obtained in treatment C4 (145°C and 8% MD_{DE10}). **Table A.2** in the appendix section shows a detailed profile composition.

The flavan-3-ol profiles of ANCE and AB WP are useful for assessing the best spray drying conditions to obtain the highest content, taking into account that the flavan-3-ol compounds can improve the anthocyanin chemical stability through copigmentation. Moreover, the interactions between flavan-3-ols and anthocyanins can also increase the health-promoting properties of these natural colorants through additive or synergistic effects (Gordillo et al., 2018).

3.4. Color in WP

To have a better overview of the influence of spray drying conditions on color, ANCE and AB WP were studied regarding their tristimulus parameters (**Table 4**).

--- Table 4 insert here ---

Table 4 shows that the highest values of L* and WI were obtained in treatments with an inlet air temperature of 135°C and 10% of the carrier (C5 and C7 for ANCE; and C5 for AB). The increase in carrier percentage resulted in a rise of L* and WI due to the dilution effect owing to carriers, bearing in mind that MD_{DE10} and GA are white powders (Ferrari et al., 2013; Pereira et al., 2020). Similar results were observed by Caliskan & Dirim, (2016) for sumac extract powders obtained by spray drying with a significant increase in brightness values with MD raising concentration. Nogueira et al., (2020) reported that blackberry powders with lighter colors were obtained when prepared with high encapsulating agent concentration. Moreover, statistical differences were found in L* and WI values when inlet temperature was increased to 145°C using the same percentage and type of carrier, except for C3 and C4 treatments in AB WP. These results agree with Quek et al., (2007), who reported that an increase in inlet air temperature caused a reduction in L* values of spray-dried watermelon powders.

The highest value for a* parameter was observed for treatment C3 (a*= 25.3), and this can be related to the aforementioned high anthocyanins content in this sample. On the other hand, the lowest values were obtained for ANCE WP with the carrier of 10% and 145°C, independently of carrier type. These results point out that WP shows lesser redness colors when a high-ranking carrier percentage was used, which is in good agreement with results previously reported by Jafari et al., (2017). With regards to AB WP treatments, the maximum a* value was 29.4 for treatment C5 (135°C and 10% MD:GA) with no significant differences with treatment C2 (treatment with the highest anthocyanin content). Amongst inlet air temperature, carrier type, and percentage no significant differences were obtained to a* values.

The hue (*H*°) values determined for ANCE and AB WP obtained by eight treatments (C1 to C8) corresponded to the fourth quadrant of color CIELAB space regardless of inlet air temperature, carrier percentage, and carrier type used in spray drying. This indicates blue-red color in powders (348–357°), characteristic of anthocyanins (**Table 4**). A similar result was found for Bordo grape skin aqueous extract encapsulated with 10% MD by spray drying (de Souza et al., 2015) and grape (*Vitis labrusca* var. Bordo) skin phenolic extract encapsulated with GA, polydextrose, and partially hydrolyzed guar gum as carriers (Kuck & Noreña, 2016). AB WP showed the highest hue (*H*°) highest values and closest to the red hues (hue = 360°) and also higher chroma values, which means that these WP have elevated saturation or color purity. To be precise, a mean of 28.6 chroma values for AB WP and 24.6 chroma values for ANCE WP were determined, which are desirable characteristics in WP to be used as a natural colorant. In general, the hue values were not affected at a high level of carrier percentage, consequently, an increase in the ratio carrier-wine seems not to lead to a dilution of material in hue terms. As mentioned before in section 3.2. and as shown in **Figure 1**, anthocyanin profiles of ANCE and AB WP (mg/100 g powder) were not significantly different in total anthocyanin concentration between treatments with the same carrier percentage, [(treatments C1 to C4 with 8%) and (treatments C5 to C10 with 10%)]. Therefore, no effect of carrier type was observed.

Furthermore, the color differences (ΔE^*_{ab}) were calculated among ANCE and AB WP treatments (**Figure 3**). According to Martínez et al., (2001), a $\Delta E^*_{ab}>3$ units indicate that color differences are detectable by the human eye (as an average observer).

--- Figure 3 insert here ---

As **Figure 3** depicts, detectable differences were found among treatments (same spray drying conditions) when ANCE and AB WP were compared. However, Lightness (ΔL), Chroma (ΔC), and Hue (ΔH) contributions to those differences were dependent on conditions. The greatest contribution values of ΔL in WP color differences were found in treatments using MD_{DE10} (C3, C4, C7, and C8), due to the white color of MD_{DE10}, and a scale-up of 8% to 10% produced a rise in the contribution of ΔL (C7 and C8). The results related to ΔC, highlight that the maximum contribution of this color parameter to color differences was found for treatments with 8% of MD:GA, specifically, for treatment C2, its high-level ΔC contribution in ΔE^*_{ab} can be related to that AB WP obtained with 8% and 145°C had the highest anthocyanin retention. According to Silva et al., (2013), the use of GA as a carrier increases the ΔE^*_{ab} value of powder relative to MD carrier. Nevertheless, in ANCE and AB WP results of color differences were mainly attributed to carrier percentage instead of carrier type. Mirhojati et al., (2017) reported in encapsulation of anthocyanins from chokeberry that color differences of powders depend on the type of coating material used and that; there is a relationship between the percentage of coating material and the brightness of powder.

Moreover, the ΔE^*_{ab} was calculated to compare C3 ANCE and C2 AB WP, treatments with the high-ranking anthocyanin content. As seen in **Figure 3**, ΔE*ab = 5.1 units were obtained, which indicates that a visual color difference exists between ANCE and AB WP and revealed that the main difference contribution was due to ΔC. As expected, owing to the highest anthocyanin content in AB WP, a ΔC difference is reasonable considering that C* is related to color saturation. The results here are presented to strengthen the idea that AB WP has the potential to be used as a natural food colorant.

Conclusions

All the conditions evaluated in this investigation allowed to obtain wine powders (WP) with an a_w <0.25 which is known to be adequate for storage stability of WP. In the case of ANCE wine, the optimum spray drying conditions were those used in treatment C3 (145°C and 8% MD_{DE10}), whereas for AB wine, treatment C2 (145°C and 8% MD:GA) was one providing the best results. In both cases, these conditions led to greater anthocyanin concentration in the final WP. The observed behavior of individual anthocyanin compounds may also be attributed to the protection afforded by MD_{DE10} and MD:GA. It is to be noted that the ratio of the carrier: wine dry extract is also detrimental to the final concentration of anthocyanin in WP due to a dilution effect, for this reason, an adequate balance must be selected. The carrier percentage was the most important variable for total anthocyanin encapsulation whereas for flavan-3-ols this parameter was observed with a lesser effect. These results point out that the spray-dried ANCE and AB WP could potentially be used as functional food ingredients providing red-blue colors and a high concentration of antioxidant compounds, as long as the a_w and pH values of foods are adequate.

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TABLES

Table 1. Drying conditions and physicochemical characteristics of ANCE and AB WP.

Table 2. Anthocyanin profile by HPLC-DAD-MS of ANCE and AB wine (mg L^{-1}).

Table 3. Anthocyanin profile by HPLC-DAD-MS of treatments C3 ANCE and C2 AB WP (mg/100 g WP).

Table 4. Tristimulus parameters were determined for ANCE and AB WP.

FIGURES

Figure 1. Anthocyanin composition in ANCE **(a)** and AB **(b)** WP for spray-dried treatments (C1 to C8). Mean ± SD (mg/100 g WP, n=3). Different letters indicate significant differences between treatments for total content (Tukey HSD test, $p<0.05$).

Figure 2. Content of catechins and procyanidins (PC) and gallocatechins and prodelphinidins (PD) in (a) ANCE and (b) AB WP for the eight spray drying treatments assayed (C1 to C8). Results are shown as mean ± SD (mg/100 g WP, n=3). Different letters within each wine variety indicate significant differences between treatments (Tukey HSD test, $p<0.05$).

Figure 3. Color differences (ΔE^*_{ab}) between ANCE and AB WP for each treatment under the same conditions (inlet air temperature, carrier type, and % carrier) and color differences among ANCE C3 vs AB C2. The graphic is also showing the contribution of Lightness, Chroma, and Hue (ΔL, ΔC, ΔH) to those differences.