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#### The impact of high voltage electrical discharge plasma on the chromatic characteristics and phenolic composition of red and white wines

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#### ABSTRACT

The cold plasma is an emerging electrotechnology for the improvement of food safety without loss of physicochemical or sensory properties. The purpose of this study was to evaluate the effects of plasma treatments on the chromatic characteristics and phenolic composition of red and white wines. The red wine Cabernet Sauvignon and white wine Graševina were treated with high voltage electrical discharge plasma considering the variations in frequency (60, 90 and 120 Hz) and processing time (3, 5 and 10 min). Total phenolics, total anthocyanins, total tannins and chromatic characteristics were analyzed by spectrophotometry while free anthocyanins, phenolic acids and flavan-3-ols by the HPLC-UV/Vis. Obtained results illustrated that plasma treatments have influenced the stability of phenolic compounds in wines without major changes in color parameters. Also, among two different processing parameters, the duration time was the most significant factor inducing changes on wines.

*Industrial relevance*: High voltage electrical discharge plasma has been shown to affect the stability of wine phenols without any significant change in the color. An increase in the concentration of certain phenolic compounds in white wine suggest that this technique could be used in the wine industry as an alternative technique for enhancing the oxidative stability of wine and consequently the wine quality during the aging process.

#### 1. Introduction

The cold plasma, as new processing technology, has been already widely investigated in terms of microbial inactivation and food safety improvement (Misra & Jo, 2017; Moreau, Orange, & Feuilloley, 2008; Shi et al., 2011; Vukušić et al., 2016; Ziuzina, Patil, Cullen, Keener, & Bourke, 2013). Recently, the focus has begun to shift towards the use of cold plasma for food properties modification (Segat, Misra, Cullen, & Innocente, 2015; Zhu, 2017), enzyme inactivation (Pankaj, Misra, & Cullen, 2013; Surowsky, Fischer, Schlueter, & Knorr, 2013; Tappi et al., 2016) and bioactivity enhancement (Elez Garofulić et al., 2015; Herceg et al., 2016). Generally, the plasma is described as partially or completely ionized gas with characteristic electrical, chemical and physical properties, which can be generated by many methods such as electrical discharges (corona, spark, glow, arc, microwave discharge, plasma jets and radio frequency plasma) and shocks (electrically, magnetically and chemically driven) (Petitpas et al., 2007). The most important physical effects of electrical discharges are a high electric field, intense UV radiation and overpressure shock waves (Zhang, Chen, & Li, 2009), while major chemical effect is manifested through the generation of various reactive species, namely hydrogen peroxide, hydroxyl, oxygen and hydrogen radicals (Locke, Sato, Sunka, Hoffmann, & Chang, 2006). Recent studies showed that the types of cold plasma systems and their applications are numeorous, including the variety of methods used to generate cold plasma, size of the reactor, distance between the electrodes, working gas and sample type (Almeida et al., 2015; Grzegorzewski, Ehlbeck, Schlüter, Kroh, & Rohn, 2011; Misra & Jo, 2017; Shi et al., 2011; Surowsky, Fröhling, Gottschalk, Schlüter, & Knorr, 2014). In addition, each of these processing parameters can significantly affect the final outcome and therefore it is difficult to make general conclusions on plasma efficiency. Furthermore, the cold plasma is not yet entirely employed in the food industry primarily due to the largely unexplored effects on different food components. Therefore, it is important to understand the basic interactions of bioactive compounds with plasma-generated reactive species, in order to avoid nutritional degradation and any other undesired effects of plasma applications. Recently, the wine industry has focused on the possible application of innovative electrotechnologies in different stages of winemaking

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process, primarily in terms of wine microbial stabilization, but also in improving oxidative stability of wine and consequently the wine quality. In the case of application of cold plasma in wine production, to the best of our knowledge, no studies have investigated the influence of this technology on the quality characteristics of wine, such as phenolic composition and color.

Generally, the composition of wine is very complex and continuosly changes during aging. Phenols are a large and complex group of compounds that significantly affect the quality of the wine and play important role in distinguishing red and white wines (Ribéreau-Gayon, Glories, Maujean, & Dubourdier, 2000). These compounds are important in determining the color of wine as well as taste and flavor (Toshihiko, 2007). Furthermore, phenolic compounds in wines are primary substrates for oxidation (Oliveira, Ferreira, De Freitas, & Silva, 2011). They are known as natural antioxidants, which protect cells against the damaging effects of free radicals (Lopéz-Vélez, Martínez-Martínez, & Valle-Ribes, 2003). Due to the disadventages of the standard aging technologies, such as long time needed and high costs, innovative aging technologies have been developed. The available literature reports about the wine quality improvement using physical methods, such as ultrasound (Ferraretto & Celotti, 2016; Martín & Sun, 2013), electric fields (Zeng, Yu, Zhang, & Chen, 2008) and high hydrostatic pressure (Chen et al., 2012; Santos et al., 2016). Considering plasma as the new electrotechnology, some studies have shown that its application on fruit juices resulted in numerous physical and chemical changes of phenolic compounds, with retention or even improvement of overall quality. Application of high voltage electrical discharge plasmas on fruit juices (apple juice and Marasca sour cherry nectar) and its influence on the physicochemical and organoleptic properties were described in a recent dissertation (Vukušić, 2016). Furthermore, an increase in total phenolic (Herceg et al., 2016) and anthocyanin content (Bursać Kovačević, Putnik et al., 2016) in pomegranate juice after argon plasma treatment has been reported, as well as the increase in anthocyanin and phenolic acid contents in sour cherry Marasca juice (Elez Garofulić et al., 2015). Also, the plasma treatment is mentioned in the context of improvement the extraction of phenolic compounds in pomegranate juice (Bursać Kovačević, Putnik et al., 2016).

Based on previously stated facts and possibilities of cold plasma, this technique has a great potential as an alternative to the current available aging technologies used in wine industry. But, firstly, the influence of plasma processing parameters on the overall quality of wine should be examined in more detail. Therefore, the aim of this study is to investigate the impact of high voltage electrical discharge plasma treatments on the chromatic characterictics and phenolic compounds of red and white wines.

#### 2. Material and methods

#### 2.1. Wine

The wines used for the present study were young red wine Cabernet Sauvignon (*Vitis vinifera* L.) and white wine Graševina (*Vitis vinifera* L.), harvest 2016, obtained from winery Erdutski vinogradi d.o.o., Erdut, Croatia. The physicochemical characteristics of the treated wines are presented in Table 1.

#### 2.2. Chemicals

Sodium carbonate anhydrous (99%) and formic acid (98–100%) were purchased from T.T.T. (Sveta Nedjelja, Croatia). Folin-Ciocalteu reagent was obtained from Kemika (Zagreb, Croatia), sodium bisulfite from Acros Organics (Geel, Belgium), hydrochloric acid (37%) from Carlo Erba (Val del Reuil, France) and ethanol (96%) from Gram-Mol (Zagreb, Croatia). Malvidin-3-O-glucoside-chloride ( $\geq$  95%), gallic acid (97.5–102.5%), protocatechuic acid ( $\geq$  97%), p-hydroxybenzoic acid ( $\geq$  99%), vanillic acid ( $\geq$  97%), syringic acid ( $\geq$  95%), caftaric acid

Table 1
Oenological parameters of the red (Cabernet Sauvignon) and white (Graševina) wines.

Parameters	Cabernet Sauvignon	Graševina
Alcohol level (vol%)	13.1	11.4
Total acidity (g/L, expressed as tartaric acid)	5.30	5.10
Volatile acidity (g/L, expressed as acetic acid)	0.61	0.31
Reducing sugars (g/L)	4.10	2.80
pH	3.46	3.37
Malic acid (g/L)	0.10	1.20
Lactic acid (g/L)	1.30	0.30
Reducing sugars (g/L) pH Malic acid (g/L)	4.10 3.46 0.10	2.80 3.37 1.20

(≥ 98%), chlorogenic acid (≥ 95%), caffeic acid (≥ 95%), *p*-coumaric acid (≥ 98%), ferulic acid (≥ 99%), (+)-catechin (≥ 99%), (-)-epicatechin (≥ 98%), procyanidin B1 (≥ 90%) and procyanidin B2 (≥ 90%) were obtained from Sigma-Aldrich (St. Louis, USA). HPLC-grade methanol and acetonitrile were purchased from J.T. Baker (Deventer, Netherlands).

#### 2.3. The plasma treatments of wine

The plasma treatments were conducted in a 1000 mL glass vessel with a point to point electrode configuration in a so called hybrid reactor with discharges in and above the liquid (Fig. 1). The plasma was generated by high-voltage (HV) pulsed power supply (Spellman, UK), by charging a load capacitor of 1.13 nF to up to 30 kV and then discharging the stored charge into the plasma reactor via a rotating spark gap (Fig. 1a). The voltage in the plasma reactor was measured and recorded using a Tektronix P6015A high voltage probe connected to a Hantek DS05202BM oscilloscope (data not shown). The experiments were performed at positive polarity and argon (purity 99.99%; Messer Croatia, Zagreb, Croatia) was bubbled trough steinless steel needle (Microllance TM 3.81 cm) at the gas flow of 4 L/min. The influences of two main factors, namely applied frequency and duration of plasma treatment on wine quality parameters were taken into account. 300 mL of wine was treated with plasma running at the combination of following processing parameters: frequency at 60, 90 and 120 Hz and treatment duration of 3, 5 and 10 min. The temperature of samples before and after the plasma treatment was monitored using a InfraRed Thermometer PCE-777 (PCE Instruments, Germany). Before treatment all samples were at the room temperatures of 21  $\pm$  1 °C, while after the plasma exposure temperature rised up to 6 °C, depending on the duration time and applied frequency. After treatments, wine samples were subjected to chemical analysis.

#### 2.4. Color measurement

The chromatic characteristics measurements were carried out using the CIELab space (Method OIV-MA-AS2-11, 2006). The spectra were registered directly on the wine, using a 10 mm optical path glass cell and a Specord 50 Plus AnalytikJena spectrophotometer (Jena, Germany) set to measure in the visible spectra ( $\lambda=380\text{--}770~\text{nm}$ ) at constant intervals ( $\Delta\lambda=5~\text{nm}$ ) and integrated using the software WinASPECT PLUS (Jena, Germany). Color was expressed as CIE coordinates of L\* (lightness), a\* (redness/greenness) and b\* (yellowness/blueness) with illuminant  $D_{65}$  and observer  $10^\circ$  standardisation. From the CIELab space, other parameters were also defined, such as chroma (C\*) and hue angle (H\*). Three replicate measurements were performed and the results are showed as average measure with standard deviation.

#### 2.5. Phenolic composition by spectrophotometric methods

The determinations of the main phenolic families were performed by a Specord 50 Plus spectrophotometer (AnalytikJena, Jena, Germany), with all the analyses conducted in triplicate.

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