

Contents lists available at ScienceDirect

Food Chemistry

journal homepage: www.elsevier.com/locate/foodchem



Changes in analytical and volatile compositions of red wines induced by pre-fermentation heat treatment of grapes



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ARTICLE INFO

Article history: Received 22 January 2015 Received in revised form 20 April 2015 Accepted 21 April 2015 Available online 23 April 2015

Chemical compounds studied in this article: β-Damascenone (PubChem CID: 5374527) β-Ionone (PubChem CID: 638014) β-Citronellol (PubChem CID: 8842) α-Terpineol (PubChem CID: 17100) Linalool (PubChem CID: 6549) 4-Mercapto-4-methyl-2-pentanone (PubChem CID: 88290) 3-Mercaptohexanol (PubChem CID: 521348) 2-Furfurylthiol (PubChem CID: 7363) 3-Isobutyl-2-methoxypyrazine (PubChem CID: 32594) Guaiacol (PubChem CID: 460)

Keywords:
Pre-fermentation heat treatment
Thermovinification
Conventional enological parameters
Aroma compounds
Nitrogen
Amino acids

ABSTRACT

Experiments were carried out on Grenache, Carignan and Fer grapes in order to characterize the changes in nitrogen content of the musts, conventional enological parameters and aroma compounds of the wines induced by pre-fermentation heating of the grapes followed by alcoholic fermentation in liquid phase or in solid phase. In comparison to a standard vinification, we showed that a two-hour heat treatment at 70 °C induced a significant loss in several grape-derived aroma compounds (terpenols, norisoprenoids and some phenols) associated with an increase in α -terpineol, guaiacol and 2,6-dimethoxyphenol, which suggests thermal degradation. A significant increase in most of the ethyl esters, in acetates and in fatty acids were observed in wines fermented in liquid phase, together with a decrease in fusel alcohols. The substantial modification in the amino acid composition of the must seems to be a crucial element for the understanding of these changes.

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1. Introduction

There is an increasing interest in the world wine market in red wines with fruity characteristics. As winemakers face a lack of new references and techniques to produce wine that can fulfill these expectations, old techniques developed some decades ago in a completely different context of production, such as pre-fermentation heat treatment or thermovinification, are becoming more widespread.

The first works on heat treatment of musts were conducted more than 60 years ago in California, both in the laboratory and

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in an artisanal manner (Berg, 1950). The development of industrial heating systems in the seventies and the large number of associated research papers published in the same decade (Lowe, Oey, & Turner, 1976; Marteau & Olivieri, 1970; Poux, 1974; Rankine, 1973) reflect the strong interest of the wine industry in this technology at that time.

The volume of wine elaborated in France with this technique was estimated some years ago at 500 million liters (Escudier, Mikolajczak, & Bes, 2008). Pre-fermentation heat treatment is spreading quickly through French vineyards and is becoming a must-use in the production of wines either with or without Protected Geographical Indication (PGI). This pre-fermentation heat treatment consists of heating grapes between 70 and 75 °C

for a length of time varying from 30 min to 24 h. When heating is limited to a short time period (<1 h), this technique is known as "thermovinification." If the heating is extended over a longer period, up to 24 h, it is known as pre-fermentation heat treatment (Escudier et al., 2008). Heating allows the extraction of phenolic compounds in aqueous phase, mainly anthocyanins and, to a lesser extent, tannins (Girard, Kopt, & Reynolds, 1997). In practice, this weak tannin/anthocyanin ratio in thermovinified wines can often lead to color instability, especially when the heating is not maintained for enough time. Heating also assists in the extraction of grape polysaccharides, responsible for roundness in wine (Doco, Williams, & Cheynier, 2007). Maceration heat treatment is often associated with straight pressing at hot temperatures, clarification and fermentation in liquid phase at low temperatures around 18 °C. It was originally used on botrytized grapes in order to destroy laccase activity. Another advantage of the maceration heat treatment is that it quickly eliminates pomace and therefore decreases the need for fermentation vessels in the cellar. The recent rapid development of this technique can be explained by the availability of more advanced technologies to clarify heated must, which has a high solids content. The rotary-vacuum drum and the latest generations of cross-flow filters, centrifugation and flotation are commonly used to clarify must after heating. The treatment can be combined with specific technologies such as flash-détente or thermo-détente that induce a rapid vacuum or a high pressure. The must is then brought back to atmospheric pressure and the variation of pressure that occurs on weakened heated skins provokes the lysis of skin cells and allows significant tannin extraction (Escudier et al., 2008). A variant of the maceration heat treatment, used to obtain a higher extraction of polyphenolic compounds, consists of fermenting grapes after heating with pomace as a standard vinification. This alternative was found to increase phenolic compounds in wine from 25% to 45% (Cottereau & Desseigne, 2007).

Most recent research on this maceration technique has been focused on phenolic extraction and antioxidant potential (Atanackovic et al., 2012: Fretté, Hansen, Raasthøi, Broe, & Christensen, 2012) while just a few works on aroma chemical composition have been carried out to date. Thermovinification or prefermentation heat treatment is known to produce wines with a standardized sensory profile often described as "banana yogurt" by winemakers. Research works on volatile composition showed that maceration heat treatment allowed the elimination of a high amount of 3-isobutyl-2-methoxypyrazine (Roujou de Boubée, 2000) and that fermentation conditions of thermovinified wines particularly enhanced ester formation (Cottereau & Desseigne, 2007; Fischer, Strasser, & Gutzler, 2000; Girard et al., 1997), while the thermal inactivation of lipoxygenase enzyme system contributed to reducing C6-alcohols and their subsequent esters. The purpose of the present work is to investigate the changes in the analytical and volatile composition of red wines induced by the pre-fermentation heat treatment of grapes. In 2009 and 2010, pre-fermentation heat treatments followed by fermentation with and without pomace were compared to a standard vinification of Grenache, Carignan and Fer grapes, three cultivars which are known to produce wines with different sensory profiles. In 2011, an additional trial was carried out to study the changes in nitrogen content of the must induced by the heating of the grapes.

2. Material and methods

2.1. Grape varieties and vineyard locations

Carignan and Grenache grapes used for this study were obtained from two commercial vineyards located in Cariñena

(Aragon, Spain). The Grenache vineyard (lat. $41^{\circ}20'35'''$ N; long. $01^{\circ}15'35'''$ W), non-irrigated and goblet trained with $2.50 \text{ m} \times 2.50 \text{ m}$ vine spacing, was selected as representative of an area with vines older than 50 years of age with a low production level (4–6 t/ha). The Carignan grapes were sampled from a young and productive (15–20 t/ha) espalier trained vineyard (lat. $41^{\circ}20'31'''$ N; long. $01^{\circ}14'22'''$ W) with $2.20 \text{ m} \times 1 \text{ m}$ vine spacing, equipped with an underground irrigation system. The soil of the two Spanish vineyards was managed by mechanical weeding. The Fer grapes were collected from a hillside dryland vineyard (lat. $43^{\circ}53'26'''$ N; long. $01^{\circ}46'50'''$ E) with $2.20 \text{ m} \times 1 \text{ m}$ vine spacing located in the South West of France, in the heart of the Gaillac Protected Designation of Origin (PDO) area with moderate crop yields (10–15 t/ha). The soil was managed by chemical weed control under the vines and by grass cover in every inter-row.

The pre-fermentation heat treatments tested on the three varieties were replicated twice in 2009 and 2010. The harvest dates were 14 Sept 2009 and 16 Sept 2010 for the Grenache grapes; 25 Sept 2009 and 8 Oct 2010 for the Carignan grapes; and 24 Sept 2009 and 1 Oct 2010 for the Fer grapes. In both years, grapes of each variety were hand harvested in 18 cases of 20 kg each. Six homogenous lots of 60 kg were constituted in our experimental winery by randomly collecting bunches from each case. To ensure the homogeneity of each lot, standard analyses such as Potential Alcohol, pH, TA, tartaric and malic acids, potassium and fermentable nitrogen were performed on the grape must just after crushing. These analyses confirmed the good homogeneity between the lots. The grapes were stored overnight at 4 °C before being processed the following day.

2.2. Winemaking and maceration techniques

Fermentation took place in our experimental cellar (Lisle Sur Tarn, France). In 2009 and 2010, three maceration treatments were investigated in duplicate for each variety: control vinification (CTRL), pre-fermentation heat treatment with pomace (PHTS) and pre-fermentation heat treatment without pomace (PHTL).

Vinification operations were carried out using the standard procedures validated in our experimental cellar. Due to the excellent sanitary conditions of the harvests in 2009 and 2010, sulfur dioxide addition was limited to 40 mg/L using a 10% bisulfite liquid solution. Standard vinification (CTRL) was carried out at 25 °C using selected dry yeasts chosen for their ability to express and optimize the aromatic potential of each variety. Strains ICV D21® (ICV, Lattes, France), ICV GRE® (ICV, Lattes, France), and Vitilevure MT® (Martin Vialatte, Epernay, France) were applied at a rate of 200 mg/L to Grenache, Carignan, and Fer, respectively. Maceration treatments were conducted after destemming and crushing. Destemming was carried out with modern vibrating equipment (Socma, Narbonne, France).

The grapes subjected to the control treatment (CTRL) were fermented with the skin for 8 days until the volumetric mass reached 994 g/L. The volumetric mass was measured during alcoholic fermentation at a fixed time every day with a mustimeter (Dujardin-Salleron, Paris, France). A single punch down per day was performed with a stainless steel manual plunger for exactly 15 s until the volumetric mass of the musts reached 1000 g/L. No extraction operations were carried out after this period.

The pre-fermentation heat treatment was performed using a water bath system. The stainless steel tank fermenter containing the crushed and destemmed grapes was submerged into heated water for 3 h. The tank was closed by a lid during the heat treatment to avoid any water evaporation. The temperature was carefully monitored during this period and the grapes were mixed every 30 min using a manual plunger to homogenize their temperature. Thanks to the elongated shape of the tank (height = 75 cm;

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