

Evolution of flavoured compounds during maturation of Chardonnay grapes

Cristian Codreşi^{1*}, Gabriela Râpeanu², Petru Alexe²

¹ S.C. Sodinal SRL, Timisoara str 100., Bucharest, Romania

² "Dunarea de Jos" University of Galaţi, Faculty of Food Science and Engineering, Domneasca Str 111,
800201 Galati, Romania

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Abstract

The total terpenic and C₁₃ norisoprenoid compounds of the Chardonnay grape variety from Murfatlar vineyard were quantified in order to establish the grapes full maturity. During the grapes ripening the amount of compounds related to wine aroma increased from veraison to full maturity stage and after this a slight decrease was registered. Also, an increase in total polyphenols content of grapes after the grape veraison in years with favorable climatic conditions was noticed and this increase was extended until the grape harvest. The quantity of aromatic compounds enhances the possibility to obtain the very good flavoured wines from Chardonnay grapes.

Keywords: grapes, terpenes, norisoprenoids, flavored wines

1. Introduction

Wine aroma is due to a lot of volatile compounds with different chemical natures and origins, found at a wide range of concentrations. Nowadays there is an increasing demand for young white wines with a fresh and fruity aroma, this being a major factor determining wine character and quality [1].

Different terpenic and norisoprenoid compounds were identified in *Vitis vinifera* grapes and wine [1]. These compounds contribute significantly to the flavor characteristic of grapes and are generally present only at low levels in the floral grape varieties. In grapes, aromatic compounds are present in free-odor form and more abundantly as non-volatile glycosides [2].

Glycosidically bound compounds contribute significantly to aroma by hydrolysis [3].

To obtain the maximum intensity of floral characteristic aromas in grapes and related aromatic varieties, several factors can be taken into consideration: period of grapes harvesting when the

total terpenes and norisoprenoids levels are at highest content [4]; obtaining the maximum available aroma components by using extended skin contact [5] and the hydrolysis of bound terpenes and norisoprenoids by using exogenous enzymatic preparation addition [6].

The concentration of compounds responsible of wine aroma may increase during grape maturation, while sugar concentration changes only slightly.

The concentration of these compounds in the berry may continue to increase even no increase in sugar concentration in the berry is noticed.

The extraction of flavoured and phenolic compounds from grape skin and their transfer into must is an essential part of flavoured wine processing.

The aim of this study was to assess the flavoured and phenolic compounds extracted from grapes skins depending on different stages of grape maturation.

* Corresponding author: e-mail: c.codresi@sodinal.com

2. Materials and Method

2.1. Grape samples

Grapes Chardonnay variety were obtained from private vineyards in Murfatlar region during the period 2008-2011. A quantity of 5 kg grapes has been harvested periodically, at time intervals of 5 days during the ripeness stage, full maturity and respectively, technological maturity. All grapes were manually destemmed and randomly grouped and used immediately for standard and phenolic maturity measurements.

For ripening characterisation the physico-chemical analysis of main composition characteristics of grapes (sugar content, titrable acidity, the weight of 100 berries, terpenes and norisoprenoid content and total polyphenols content) was carried out.

Two hundred grape berries were weighted and used for determining the sugar content, the titratable acidity according with the analytical methods recommended by the OIV. Sugar concentration was measured using a refractometer. The titratable acidity was measured by titrimetry using NaOH 0.1 N and Bromothymol blue as indicator. The total phenol content in grape skins samples was determined spectrophotometrically according to the Folin - Ciocalteu colorimetric method [7] using gallic acid as a standard polyphenol: 0.1 ml of grape skin extract was mixed with 7.9 ml distilled water and 0.5 ml of Folin-Ciocalteu reagent. After 1 min, 1.5 ml of 20% Na₂CO₃ was added. The absorbance was measured after 120 min at 760 nm. The concentration of the total phenolic compounds was expressed as gallic acid equivalents (g/kg).

2.2. Terpenes and norisoprenoids separation and quantification

Terpenes separation and quantification was done by using a method described by Armada *et al.* [8] with small modifications. A volume of 100 ml must/wine was applied to a preconditioned 500 mg RP C18 SPE column. Preconditioning was performed by purging at 3 ml/min the column with 25 ml portions of methanol and water. After loading a sample onto the column it was washed with 150 ml of water. Non-polar fraction (NPF) was eluted using 25 ml of a mixture of pentane/dichloromethane (2/1, v/v). Sub-sequently, polar fraction (PF) was eluted using 25 ml of methanol and subjected to hydrolysis. Non-polar fraction was evaporated to approximately

500 µl firstly heating it at 30 °C water bath without mixing or stirring for 30 min, then in a delicate stream of nitrogen and 1 µl of it was introduced in a splitless mode into GC MS system. The terpenes and C₁₃ norisoprenoids from wines were separated and measured using a Varian GC-MS with flame ionization detector (FID) and equipped with a capillary column (30 m × 0.32 mm i.d.; film thickness 0.5 µm). A volume of 2 µl sample of the extract was injected in splitless mode (30 s). Temperature program: 1 min hold at 45 °C, ramping at 3 °C/minute to 230 °C, and isotherm during 25 min. Helium was used as the carrying gas (18°psi). Temperature of the injector and detector was 230 °C.

Compounds identifications were performed by comparing linear retention index and electronic mass spectra with published data or authentic samples.

All determinations were done in triplicate and the relative SD's were less than ±1%.

3. Results and Discussion

Climatic conditions in autumn of the 2011 year led to a low contamination with gray mold favorable for making high quality wines.

During the period (01.04 - 30.09.2011) the presence of rainfall in the vineyard Murfatlar caused a forced grapes ripening and full maturity was reached at 30.08-2011.

For white grape Chardonnay variety full maturity was reached in 05.09.2011 at a sugar content of 183 g/l, acidity level of 6.30 g/l H₂SO₄, and weight of the 100 berries 170 g. The grape harvest coincided with technological maturity and was carried out from 20 to 30 09.2011.

The Chardonnay grapes variety at harvest (30.09.2011) presented a sugar content of 236 g/l, the acidity was 4.30 g/l H₂SO₄ and weight of the 100 berries was 161.

At harvest during the period 2008-2011, the grapes presented a sugar content of between 198-236 g/l, the highest value being reached in 2010 (Figure 1). As shown in Figure 1 sugar accumulation was achieved gradually during the grapes ripening. This evolution of the sugar content was observed by other researchers too [9,10,11].

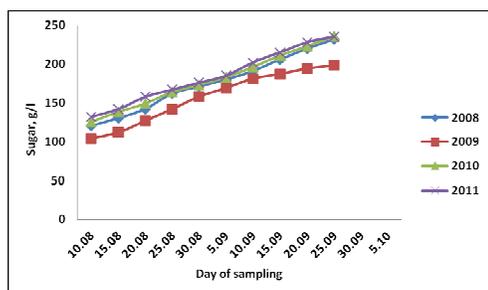


Figure 1. Evolution of sugar content during grapes maturation (2008-2011)

Acidity of the grapes at harvest ranged from 3.9 to 5.8 g/l H₂SO₄. The lowest acidity was recorded in 2009 and the highest value was obtained in 2011 (Figure 2).

Grape acidity depends on grape variety, soil and climatic conditions and its value is directly correlated with the ripeness of the grapes [12].

The decrease of acidity during maturation is due combustion phenomena occurring and because of the compounds dilution. Some researchers have been observed the degradation suffered by some acidic forms due to temperature [13].

The weight of the 100 berries showed an ascendant trend until full maturity of the grapes, and between full maturity and technological maturity of the grapes the index of 100 berries decreased gradually.

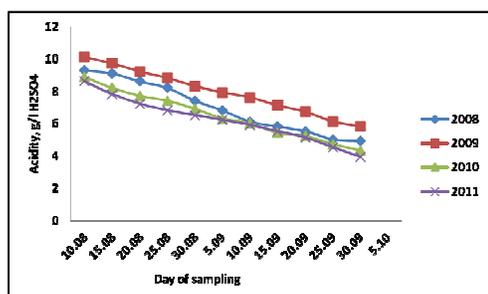


Figure 2. Evolution of acidity during grapes maturation (2008-2011)

These results are in accordance with Esteban *et al.*, [11] who indicate that the large mass of 100 berries

is directly correlated with the grapes ability to absorb water and is a specific of each variety individually. Dimensions of the grapes will affect the extraction of free and bound flavored compounds in wine by its impact on cellular proportions of skins and juice.

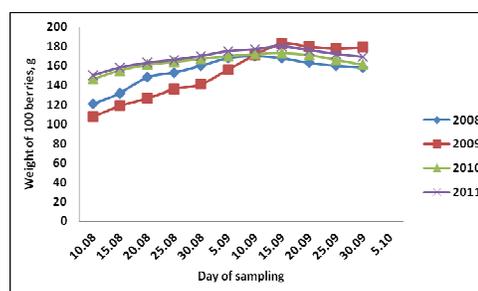


Figure 3. The weight of 100 berries evolution during grapes maturation (2008-2011)

Change in weight of 100 berries is shown in Figure 3. During harvest the weight of 100 berries ranged from 161 g to 179 g, the lowest value was recorded in 2008 and the highest was recorded in 2009.

Dynamic evolution of total polyphenolic content is presented in Figure 4. At harvesting total polyphenols content was between 2.35 g/kg and 2.69 g/kg, the highest value being obtained for harvest in the year 2010, namely 2.69 g/kg gallic acid.

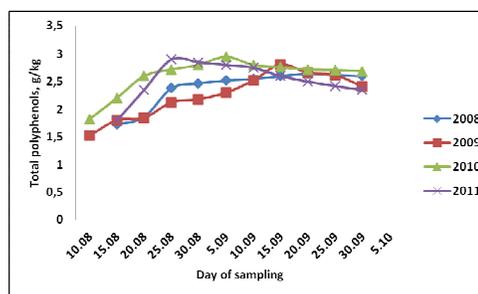


Figure 4. The total polyphenols content evolution during grapes maturation (2008-2011)

As shown in Figure 5, free terpenes content showed

an ascendant trend. Since the period of veraison to the harvest period the terpenes content ranged from 0.47 to 5.85 µg/l.

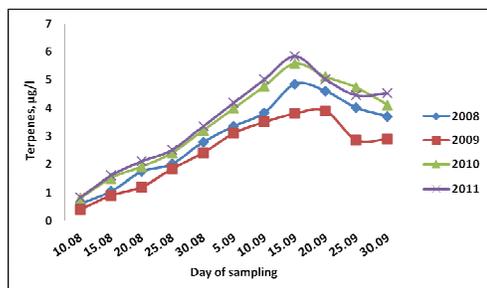


Figure 5. Terpenes content evolution during grapes maturation (2008-2011)

The highest value was recorded for the year 2011. During technological maturity, the terpenes content decreased slightly, especially in the case of grapes at over maturation. This was also observed by Fenoll *et al.* [14] for the Hamburg Muscat grapes. When a maximum of terpenes accumulation is desired in grape berries, they should be harvested before their over ripening.

In Figure 6 the total norisoprenoids content is depicted. The highest value of total norisoprenoids content was observed for the year 2011.

It is difficult to explain evolution of terpenes and norisoprenoids content during grapes ripening. There are many factors affecting these values. First is important to note that the location of these compounds is in the skins.

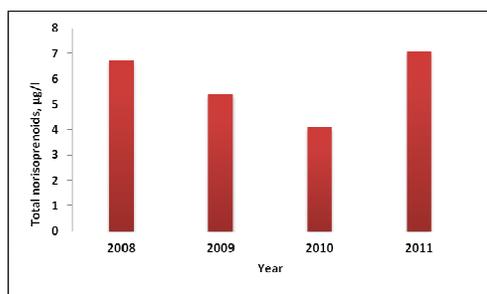


Figure 6. Norisoprenoids content of Chardonnay grapes at harvesting (2008-2011)

In the early stages of maturation the proportion of the total skin from berry is much higher. As the grapes are mature, berry weight increases and decreases the proportion of skins. Therefore at the full maturity of grapes the terpenes and norisoprenoids content doesn't have maximum values.

Grape production achieved in this period (2008-2011) varies from year to year ranging from 8.3 t/ha in 2011 and 9.5 t/ha in 2009 (Figure 7). The highest production of Chardonnay was recorded in 2009 (9.5 t/ha).

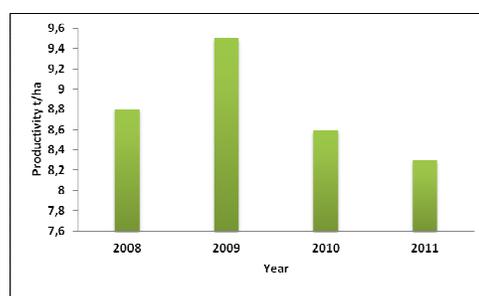


Figure 7. Productivity of Chardonnay grapes during 2008-2011

4. Conclusion

The full maturity of the Chardonnay grapes in the Murfatlar vineyard during 2008-2011 was carried out from September 5th to 20th, exception was the 2011 year when the full maturity of the grapes was forced (25.08) because of the dry period.

The sugar content of the Chardonnay grapes at full maturity ranged from 198-236 g. The total acidity of the Chardonnay grapes at full maturity recorded values between 3.9 to 5.8 g/l H₂SO₄. The weight of 100 berries at full maturity of grapes gives values ranging from 158-179 g. The polyphenolic potential of the grapes at full maturity ranged between 2.35 to 2.60 g/kg.

During the grapes ripening the amount of total polyphenols increased from veraison to full maturity and after a slight decrease was registered.

An increased in terpenes and norisoprenoids content of the grapes after grapes veraison in years with favorable climatic conditions was noticed and this increase is extended until the harvest.

Climatic conditions influence the amount of flavoured compounds accumulation in grapes. Presence of rain, cold and wet weather are leading to slow the accumulation of these compounds in grape skins.

The grape vintage has to be made when the quantity of flavoured compounds reached the highest values before the contamination with gray mold which can induce the flavor degradation.

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