

RECOVERY OF WINE VOLATILE COMPOUNDS DURING THE VINIFICATION

Encarna GÓMEZ*, A. MARTÍNEZ* and J. LAENCINA**

*Centro Regional de Investigaciones Agrarias, Ctra. La Alberca s/n, 30150 Murcia (Spain)

**Dep. Tecnología de los Alimentos, Facultad de Veterinaria,
Universidad de Murcia, 30009 Murcia (Spain)

INTRODUCTION

The volatile compounds of wines appear mainly during fermentation. Quantitatively, alcohols and esters are the most important volatile compounds, but their impact on the wine aroma is not the same. The alcohols are compounds that individually do not have pleasant odor, but when diluted, reinforce the wine aroma (RIBÉREAU-GAYON, 1978 ; RAPP and MANDERY, 1986). Esters strongly contribute to the fruity aroma of young wines, mainly short-chain fatty acid esters (MONTEDORO and RICCIA, 1987 ; VERNIN *et al.*, 1986).

During the fermentation of grape sugars there is a formation of CO₂. This CO₂ escapes during must fermentation and this effect is most intense during the vigorous phase of fermentation. Together with this CO₂ escape, there is a loss of other volatile compounds being formed during the fermentation. Some of these volatile compounds are important for the aromatic profile of the wine.

To determine the main compounds that are lost together with the CO₂ during must fermentation, we placed an experimental system in a winery. The system consisted in a cold trap connected to the exit of the fermentation tanks. The use of this system also showed the possibility of the recovery and storage of these volatile compounds, that could allow us to reconstitute the wine aroma when its fermentation is finished.

MATERIAL AND METHODS

The experimental system has been tested in two different vinifications of red grapes (*Vitis vinifera* L.) c.v. Monastrell.

One of the vinifications was carried out with prolonged skin contact, the time of skin contact was five days (red wine vinification). The initial density (20°C) of the must was 1110 g/l and

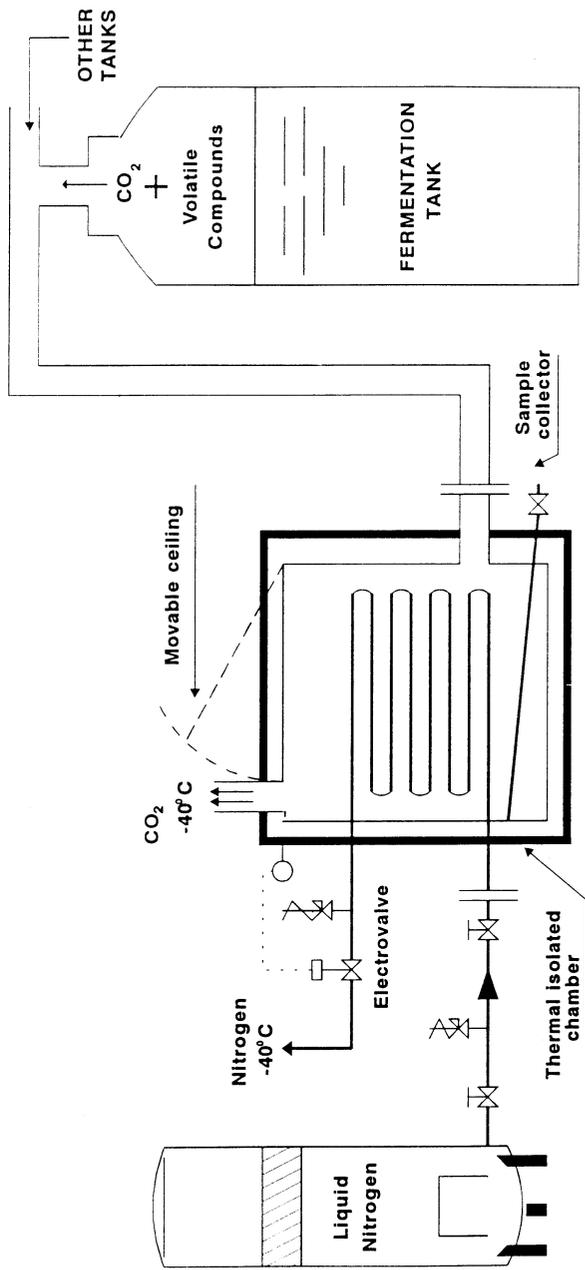


Fig. 1 — Design of the experimental system used for the recovery of volatile compounds during the vinification

the titratable acidity 4.84 expressed as g/l tartaric acid. The fermentation temperature was held at 28°C. When fermentation was finished, the wine density was 990 g/l, the ethanol content 14.16 p. cent (v/v) and the adjusted titratable acidity 5.96 g/l.

The other vinification was performed with short skin contact, the skins were separated after 6 to 8 hours (rose wine vinification). The initial must analysis was the same as in the former vinification. The must was clarified then at 18°C with pectolitic enzymes. The temperature of fermentation was 20.5°C. The final wine had an ethanol content of 13.98 p. cent, the density was 990 g/l and the acidity 4.51 g/l.

The experimental system consisted of a sealed isothermal cold trap with a liquid nitrogen circulation through the interior coil to keep a constant temperature of - 40°C (figure 1). Fermentation tanks were closed except for the exit connected directly to the isothermal cold trap. With exception of CO₂, the volatile compounds escaping from the fermentation tanks were retained in the cold trap. The temperature of the cold trap was not high enough to condense the CO₂. The opening of the ceiling trap was controlled by pressure. When the CO₂ pressure reached a certain point the ceiling opened and CO₂ escaped. The other volatile compounds remained frozen in the trap.

Two samples from each of the vinifications were collected. The first sample consisted of the volatile compounds condensed since the beginning of the fermentation to a must density of 1020 g/l. At this point, due to the large quantity of condensed compounds, the trap was defrosted and the sample collected. The clean trap was then connected again to the system. The second sample consisted of volatile compounds condensed from this point until the end of the fermentation. Once the samples were defrosted, the volatile compounds were in an ethanolic solution because ethanol was the largest compound in the condensed samples.

0.5 µl of each sample was directly injected on a GC HP-5830 (Hewlett Packard, Avondale, Philadelphia), equipped with a flame ionization detector (250°C) and a capillary column (OV-101, 50 m x 0.25 mm i.d.). Hydrogen was used as a carrier gas. The injector temperature was 225°C and the oven temperature was programmed from 40°C to 180°C at 1.5°C/min. 2-Octanol was used as an internal standard. The volatile compounds were identified by gas chromatography-mass spectrometry (Hewlett-Packard 5870) using the same column and programs as described above and by comparing their retention times with those of authentic reference compounds.

RESULTS AND DISCUSSION

Table I shows the volatile compounds that appeared in the different collected samples and their concentration expressed as grams per liter of condensed sample.

The alcohols that appeared at detectable concentrations in the collected samples of both vinifications were ethanol, propanol, 2-methyl-1-propanol, 3-methyl-1-butanol and 2-methyl-1-butanol. Butanol only appeared in the samples of red wine vinification.

Ethanol was the largest component in the condensed samples but its concentration was not measured since it was not considered as an important aromatic compound.

The following esters were also found : ethyl acetate, 3-methyl-1-butyl acetate and hexyl acetate. Propyl acetate was only found in samples of red wine vinification. Four short-chain fatty acid esters were also detected : ethyl butanoate, ethyl hexanoate, ethyl octanoate and ethyl decanoate.

The loss of aromatic compounds during fermentation was related to the concentration of these compounds in the wine. Propanol, butanol, 2-methyl-1-butanol and 3-methyl-1-butanol are the alcohols that appear at largest concentrations in the wine (RAPP and MANDERY, 1986). The identified esters in the samples were also those that appear at highest concentrations in wine. The ester with the highest concentration in the collected samples, ethyl acetate, is also the ester that can be found at the highest concentration in wines (RIBÉREAU-GAYON, 1978).

TABLE I
Concentration of the volatile compounds in the condensed samples (g/l)

Compounds	A		B	
	Red	Rose	Red	Rose
Alcohols				
1-propanol	0.16	10.60	0.20	nd
2-methyl-1-propanol	0.64	0.22	nd	0.32
1-butanol	nd	nd	0.24	nd
3-methyl-1-butanol	2.70	0.43	0.46	1.70
2-methyl-1-butanol	0.63	0.08	0.88	0.40
Esters				
Ethyl acetate	1.38	0.04	nd	0.99
Propyl acetate	nd	nd	0.14	nd
3-methyl-1-butyl acetate	0.23	0.02	0.01	0.62
Hexyl acetate	0.02	0.01	0.01	0.07
Ethyl butanoate	0.01	nd	0.01	0.02
Ethyl hexanoate	0.13	0.01	0.05	0.11
Ethyl octanoate	0.13	0.01	0.08	0.05
Ethyl decanoate	0.02	nd	0.01	0.03

A : Volatile compounds trapped from onset of fermentation until must density reached 1020 g/l ;

B : Volatile compounds trapped from must density of 1020 g/l until end of fermentation ;

Red : Collected volatile compounds from red wine vinification ;

Rose : Collected volatile compounds from rose wine vinification ;

nd : not detected

The presence of the alcohols was quantitatively more important than the esters, mainly in the samples collected at the beginning of the fermentation and especially propanol in the first collected sample of rose wine vinification (10.6 g/l).

The escape of other aromatic compounds in the first part of the fermentation was quantitatively larger in the red wine vinification than in the rose wine vinification. This fact was related to the higher temperature of fermentation in the red wine vinification ; a higher temperature induced a larger volatile compound escape. Only the most volatile alcohol, propanol, appeared at higher concentration in the sample of rose wine vinification.

In the second condensed samples the alcohols were again the quantitatively most important fraction of volatile compounds. Only butanol and 2-methyl-1-butanol had an important increase in their concentrations in the second sample of red wine vinification but in the sample of rose wine vinification, except propanol, the other alcohols increased their concentration. These different evolutions were due to the different behavior of both fermentations. The apparition of volatile compounds in the rose wine vinification was slower than in red wine vinification due to the lower fermentation temperature. That explains that the maximum of the collected alcohols appeared later in the samples of rose wine vinification than in the samples of red wine vinification.

The amount of recovered esters in the first sample of red wine vinification was larger than in rose wine vinification, but their concentration decreased from the first condensed sample to the second, whereas the evolution of the esters in the collected samples of rose wine vinification was different since their concentration increased for all of them in the second condensed sample, that is, at the end of the vinification. Moreover, their concentrations in the second sample of rose wine vinification were bigger than in the sample of red wine vinification, except for propyl acetate. These data agreed with those of CASP et LOPEZ (1989) and PIRACCI (1987) who found that a lower fermentation temperature increases the esters formation in wine. The higher concentration of esters in rose wine and, so therefore, in the vapor phase in equilibrium with it, gave a higher loss of these esters than comparing with the red wine vinification, even with the higher fermentation temperature of the red wine vinification.

CONCLUSIONS

As the results show, there is a loss of volatile compounds during the fermentation process. Some of them are very important to wine quality and they can be recovered in a great extent with the experimental system described.

There are two factors affecting the loss of volatile compounds during wine fermentation: temperature and concentration of these compounds in the wine. A higher temperature promotes a larger loss of volatile compounds, but also, those compounds that are present at the highest concentrations are easily lost.

The recovery of these compounds to reconstruct the wine aroma when the fermentation is finished, could be an interesting enological practice. In this way, CIOLFI and DI STEFANO (1983) demonstrated that the addition of 2-methyl-1-butyl and hexyl acetate as well as the ethyl hexanoate, ethyl octanoate and ethyl decanoate positively reinforced the wine aroma.

The next steps in this ongoing research would be a reinforce of the wine aroma by adding these recovered volatile compounds and a sensorial analysis of these reconstructed wines as well as an economic study of the experimental system.

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