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# Effect of grape pectic enzyme treatment on foaming properties of white musts and wines

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## Abstract

White grapes of three different varieties (Macabeo, Xarel.lo and Parellada), which are used in Spanish sparkling wines production (O.A.C. Cava), were treated with a pectic-enzyme preparation on an industrial scale. Foam capacity (foamability (HM), Bickerman coefficient ( $\Sigma$ ), foam stability time (TS) and surface tension (ST)) of nine musts from pectic enzyme treated grapes and their corresponding wines were compared to their respective control samples. The HM,  $\Sigma$ , and TS values decreased and ST increased in most of the treated samples. This could be the case, because samples from enzyme-treated grapes had lower protein and polysaccharide contents and higher values of polyphenols than untreated samples: HM and  $\Sigma$  were positively correlated with proteins and polysaccharides and negatively with ST; polyphenol contents had a negative relationship with TS values.  $\bigcirc$  1999 Published by Elsevier Science Ltd. All rights reserved.

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# 1. Introduction

High quality wine can only be obtained as a result of a careful elaboration. To obtain quality wines different enological practices are applied (pressing, racking, clarification, filtration...). These practices modify the composition and sensory characteristics such as flavour and colour. However, when the base wines are destined to produce the quality sparkling wines, another characteristic should be considered: their capacity to produce foam. The foam characteristics are related to the chemical composition of wine: ethanol, proteins, polysaccharides, polyphenols and organic acids have an important influence on foam capacity and foam stability time (Andrés-Lacueva, López-Tamames, Lamuela-Raventós, Buxaderas, & Torre-Boronet, 1996; Brissonet & Maujean, 1991; López-Barajas, Viu Marco, López-Tamames, & Buxaderas, 1997; Malvy, Robillard, & Duteurtre, 1994). Some studies have shown that process such as the system of extraction must, racking, and fining affect the composition of wines, and thus the foaming properties of musts and wines (Andrés-Lacueva et al.; Brissonet & Maujean, 1991; Hardy, 1990; López-Barajas, López-Tamames, & Buxaderas, in press; Maujean, Poinsaut, Dantan, Brissonet, & Cossiez, 1990).

Nowadays, many winemakers of Spanish sparkling wines (Cava appellation) use pectic enzymes in the pressing and clarification of grape juice. The use of these enzymes gives very important advantages: increase in juice yield, faster flow of juice, rapid settling and greater clarity. However, for base wines, i.e. the substrate for a second fermentation in bottle (Method champenoise), it is necessary to assess how pectic enzyme treatment affects to foaming ability.

There are several studies (Brown & Ough, 1981; Canal Llauberes, 1989, 1990; Lao, López-Tamames, Buxaderas, & De la Torre-Boronat, 1996, 1997; Liu, Gallander, & Wilker, 1987; Williams, Ough, & Berg, 1978) on pectic enzyme treatment, but they have only compared the organoleptic quality and composition of wines made with or without pectic enzymes. These authors reported that the application of pectic enzyme preparations to the grapes caused changes in the must

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and wine composition (methanol, galacturonic acid, total and acid polysaccharides, total and nonflavonoids phenols) and in their physico-chemical characteristics (turbidity, colour). However, none of these authors studied the influence of pectic enzymes on the foaming properties of musts or wines.

The main aim of the present study was to determine how a commercial pectic-enzyme preparation (Citolase 446<sup>®</sup>, of widespread use in the Penedés region) affects the foam capacity of musts and base wines from Macabeo, Xarel.lo and Parellada grapes. This is of interest to winemakers since the knowledge of the foam characteristics of winemaking products allows them whether these products will be destined to the elaboration of cava or table wine. For this purpose, monovarietal musts and wines were obtained from treated and untreated grapes. We compared the foam capacity of treated must and wines with their respective controls. We determined four foam parameters: foamability (HM), Bickerman coefficient ( $\Sigma$ ), foam stability time (TS) and surface tension (ST), and several compounds which defined foam behaviour. The study was repeated in three consecutive years and at industrial scale, in the same winery of the Cava region.

## 2. Materials and methods

# 2.1. Enzymes

The commercial enzyme used for this experiment was the pectin enzyme preparation Citolase  $446^{\text{(B)}}$  (Biocatalysis Ltd, UK). This was added as a liquid formulation to grapes at the levels recommended by the manufacturer.

## 2.2. Samples

The grapes used were of three white varieties: Macabeo (M), Xarel.lo (X) and Parellada (P) from AOC Cava, harvested in three consecutive years (1, 2 and 3) at the commercial vineyard of l'Arboş del Penedés, Tarragona, Spain. Grapes were received in 20000 kg lots for each cultivar. Each lot was divided into two fractions of 10 000 kg. From one, we obtained the control samples (untreated samples). From the other lot we obtained the treated samples. In this case, the pectic enzyme preparation was added to grapes at 10 g/ 100 kg at 22°C. Both lots of grapes were pressed in a Wilmex<sup>®</sup> horizontal-pneumatic press (pressure was < 0.2 bar; three cycles (4 min) were used) and only free run juices were obtained. Then SO<sub>2</sub> was added at  $75 \text{ mg litre}^{-1}$ . Both musts types were racked by natural settling for 24 h. Although the settling of treated must was completed in 8 h, both lots of fermentations were conducted in parallel in Freixenet winery with Saccharomyces cerevisiae ( $2 \times 10^6$  cells cm<sup>-3</sup>) on 1000 litre deposits. Wine samples were collected before being prepared to make sparkling wines and thus, no fining or filtration was performed. These winemaking processes were repeated in three consecutive years to consider differences in the composition of the substrate, mainly due to climatic factors.

### 2.3. Analytical methods

#### 2.3.1. Measurement of foaming properties

Three foam measurements were carried out by the Mosalux procedure, using the parameters recommended by Gallart, López-Tamames, and Buxaderas (1997): foamability (HM) is the maximum height (mm) reached by the foam after carbon dioxide injection through the glass frit, expressed in mm; Bickerman coefficient  $(\Sigma)$ (Robillard, Delpuech, Viaux, Malvy, Vignes-Alder & Duteurtre, 1993), expressed in seconds, is taken to represent the average bubble lifetime, in steady state, when foam formation and destruction are balanced. Foam stability time (TS), until all bubbles collapse when the CO<sub>2</sub> injection is interrupted, expressed in seconds, is taken to represent the foam stability time, once effervescence has decreased. The Mosalux procedure was performed in quadruplicate. Surface tension (ST), expressed in mN m<sup>-1</sup>, was determined at room temperature  $(22\pm1^{\circ}C)$  with a Krüss GMBH K6 tensiometer (a correction factor (ratio of theoretical (72 mN  $m^{-1}$ )) to experimental surface tension of double-distilled water) was used). The tensiometer used is based in the ring method and the surface tension was measured when the ring is pulled out of the surface.

# 2.3.2. Enological parameters

Conventional enological parameters (ethanol, total and volatile acidities, total and free  $SO_2$ , pH), absorbance at 420, 520 and 280 nm, proteins, polyphenols (orthodiphenols, flavonoids and nonflavonoids), total, acid and neutral polysaccharides, were determined by OIV official methods, as reported in Lao et al. (1996).

## 2.3.3. Statistical procedures

Statgraphics 7.0 program (Rockville, MD) was used to carry out multiple analysis of variance (ANOVA), and simple regression was applied. p < 0.05 was considered as significant. Pectic enzyme treatment, harvest and variety were considered as qualitative variables. Foam properties (HM,  $\Sigma$ , TS and ST) and the other physico-chemical parameters were considered as quantitative variables.

## 3. Results and discussion

The foamability (HM) decreased in many pectic enzyme treated samples, (Fig. 1). Must from treated

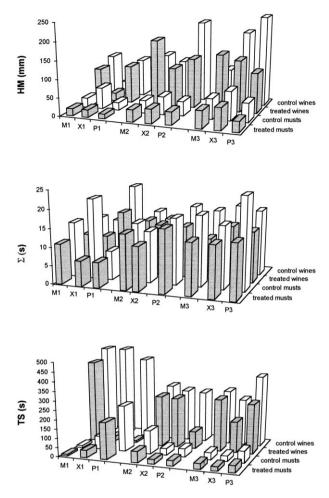


Fig. 1. Foamability (HM), Bickerman coefficient ( $\Sigma$ ), and stability time (TS) values of musts and wines from Macabeo (M), Xarel.lo (X) and Parellada grapes (P), from the three harvests (1, 2 and 3).

grapes had lowest values of HM in all three varieties. Decreases in HM values in treated musts could be attributed to the decrease in colloids in these musts. Some experiments (Andrés-Lacueva et al., 1996; López-Barajas et al., 1997, in press; Pueyo, Martí, & Polo, 1995) have shown the importance of polysaccharides and proteins for foam capacity. These compounds increase the viscosity of the wall of bubbles and decrease surface tension, thus render the bubbles more resistant to coalescence. Pectic enzymes broke and removed colloids, mainly polysaccharides by pectinesterase, polygalacturonase and pectinlyase activities, but also proteins by concomitant protease activity present in the commercial enzyme preparation Citolase 446<sup>®</sup> (Lao et al., 1996). There was a positive relationship between HM and total polysaccharides (r = 0.5463,  $p \le 0.006$ ) (Fig. 2); neutral polysaccharides (r = 0.6497,  $p \le 0.001$ ); and proteins (r = 0.7453,  $p \le 0.001$ ) (Fig. 2). Moreover, surface tension was negatively correlated with HM  $(r = -0.3786, p \leq 0.022).$ 

Wines showed a similar trend to must (Fig. 1). Treatment produced a decrease in HM in Parellada and

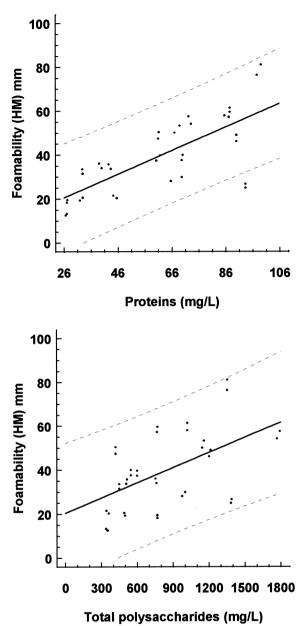


Fig. 2. Regressions between HM and total polysaccharides and proteins of grape juices.

Xarel.lo wines, but not in Macabeo (M<sub>2</sub> and M<sub>3</sub>). Foamability of grape juices conditioned the HM values of wines from these juices; there was a positive relationship between HM of musts and HM of wines (Table 1). In contrast, the extent of HM decrease in treated wines respect to their control wines were not of the same magnitude as those observed in musts (Fig. 1). This may be due to changes in physical and chemical parameters during fermentation and winemaking. New components appear, and the extent of these changes depends on variety and harvest. In wines there was also a positive correlation between HM values and total polysaccharides (r=0.4027,  $p \le 0.014$ ) and neutral polysaccharides (r=0.4625,  $p \le 0.005$ ) and a negative

Table 1 Correlation coefficients (r) and significance (p) between foam properties of musts and foam properties of wines

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	HM wines	$\Sigma$ wines	TS wines
HM musts	$r = -0.4357^{\rm a}$	r = 0.4082	$r = -0.5204^{a}$
	p = 0.0080	p = 0.0134	p = 0.0011
$\Sigma$ musts	$r = -0.5210^{a}$	n.s. <sup>b</sup>	$r = 0.5510^{a}$
	p = 0.0011		p = 0.0005
TS musts	n.s.	n.s.	n.s.

<sup>a</sup> Reciprocal regressions (1/y = a + bx). On this model, the negative sign of the coefficient of regression means a positive relationship. <sup>b</sup> n.s., no significant correlation.

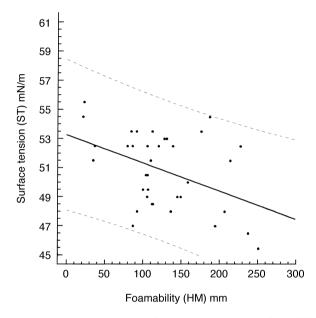


Fig. 3. Regression between surface tension (ST) and foamability (HM) of wines.

correlation with surface tension (r = -0.4305,  $p \le 0.005$ ) (Fig. 3).

Bickerman coefficient ( $\Sigma$ ) values of musts and wines were also affected by enzyme treatment: musts and wines from treated grapes had lower  $\Sigma$  values than untreated ones, except in Xarel.lo wine from second harvest  $(X_2)$ , (Fig. 1). As with HM, the extent of the decreases was different in musts and wines. However, Bickerman coeffients of musts were not correlated to Bickerman coefficient of wines (Table 1). There was a positive correlation between  $\Sigma$  values and total polysaccharides  $(r=0.5404, p \le 0.002 \text{ in musts and})$ r = 0.5059,  $p \le 0.002$  in wines), neutral polysaccharides  $(r=0.5484, p \le 0.001$  in musts and  $r=0.5484, p \le 0.001$ in wines) and proteins (r = 0.5195,  $p \le 0.002$  in musts and r=0.3520,  $p \le 0.035$  in wines). We found also a negative correlation between Bickeman coefficient values of must and wines and surface tension  $(r = -0.5509, p \le 0.001$  in musts, and  $r = \le 0.5704,$  $p \leq 0.001$  in wines). Proteins and polysaccharides could have surfactant properties, which may reduce the sur-

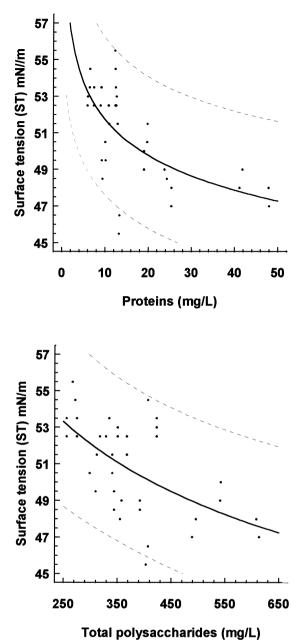


Fig. 4. Regressions between  $\Sigma$  and polysaccharides and proteins of wines

face tension of wines (Fig. 4) and thus increase the  $\Sigma$ values.

Pectic-enzyme treatment conditioned foam stability time values (TS) in musts and wines (Fig. 1). All the samples from treated grapes had lower TS than control. The mean percentage decrease was 33% and the 95% confidence interval was 69-81 s for control wines and 45-57 s for treated wines (Table 1). In contrast to the two previous foam properties, TS was not correlated with colloids or surface tension, but we found a relationship between TS and phenol compounds in wines  $(r = -0.6003, p \le 0.001$  for total polyphenols; r = -0.4871,  $p \le 0.003$  for ortodiphenols; r = -0.6369,

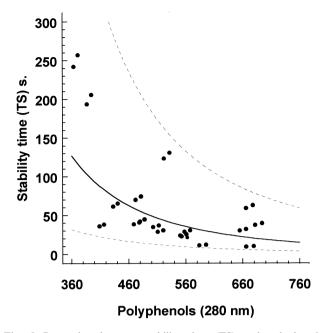


Fig. 5. Regression between stability time (TS) and polyphenols (absorbance at 280 nm) of wines.

 $p \le 0.001$  for flavonoids and r = -0.3346,  $p \le 0.046$  for non flavonoids). In our previous study we reported that treated samples have higher polyphenol contents than untreated ones (Lao et al., 1996). This fact could explain the lower values of TS in treated samples. Likewise, there was a negative correlation between TS and absorbance at 280 nm (r = -0.6281,  $p \le 0.000$ ) (Fig. 5).

In conclusion, the use of pectic enzyme-preparation Citolase 446<sup>®</sup> in the production of base wine destined to Cava are detrimental to foam behaviour.

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