Foam Measurements in Wines: Comparison of Parameters Obtained by Gas Sparging Method

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Two procedures for foam determination, based on the method of gas sparging, were applied in samples with different foam capacities. The foaming parameters measured by Maujean et al. (1990) [maximum height (HM), stability height (HS), and stability time (TS)] and Robillard et al. (1993) [foam expansion (E), foam stability (L_F), and Bikerman coefficient (Σ)] were compared. HM, E, and L_F were positively correlated ($r > 0.9$), so E and L_F do not provide additional information on wine foamability. Furthermore, measurement of E in samples with low foamability is unreliable, and in samples with high foamability E is less able to discriminate foaming properties than HM. TS did not have any predictive relationship with the other parameters, so it could be used as a parameter of another aspect of foam. Σ is preferable to HS, because it is independent of determination conditions. The precisions of HM and TS were low (80%) in some wines, but the coefficients of variation were related with the mean values. The most appropriate parameters to characterize the foam capacity of several wines were HM, Σ, and TS.

Keywords: *Foam parameters; gas sparging method; Mosalux; wine*

INTRODUCTION

Foam behavior is studied to characterize sparkling wines. The foaming properties of sparkling wines are related to the base wines (Maujean et al., 1990), which depend, in turn, on the grape variety (Andrés-Lacueva et al., 1996a,b). There has thus been increasing interest in the effects of chemical composition on foaming properties (Brissonnet and Maujean, 1991, 1993; Dussaud et al., 1994; Malvy et al., 1994; Pueyo et al., 1995; Andrés-Lacueva et al., 1996a; López-Barajas et al., 1997) and the influence of treatments during winemaking (Hardy, 1990; Robillard et al., 1993; Viaux et al., 1994; Brissonnet et al., 1995; Andrés-Lacueva et al., 1996a,b). To this end a need arose to develop standard parameters for the measurement of foam properties of several winemaking products. Furthermore, most of the methods used to measure foam properties were developed for beer and are unsatisfactory for wine (Edwards et al., 1982). In sparkling base wines, the usual method is based on that proposed by Bikerman (1938), which produces foam by gas sparging (air, N_2, or CO_2) through wine (Edwards et al., 1982; Maujean et al., 1990; Robillard et al., 1993; Pueyo et al., 1995). One of the foam parameters used (Σ) was already established by Bikerman (1938) as a coefficient that is independent of the apparatus and procedure employed. The first equipment to be automated and computerized, based on gas sparging, was the Mosalux, commercialized by Maujean et al. (1990). They proposed the following parameters: the maximum height of foam (HM), the foam stability height (HS), and the foam stability time (TS), until all bubbles collapse, when CO_2 injection is interrupted. According to Robillard et al. (1993), the procedure proposed by Maujean et al. (1990) lacks reproducibility and artifacts sometimes appear. These artifacts consist in the formation of a foam ring above the main foam column during the sparging, which could alter the values of HM. To avoid these problems, but using the same equipment (Mosalux), Robillard et al. (1993) propose changing the experimental procedure: to increase the CO_2 pressure and flow and to saturate the glass cylinder with CO_2 by sparging through the glass frit (at least 2 min). Moreover, the gas flow is interrupted at 80 s, before foam height reaches the maximum. With this procedure, they obtained other foam parameters: the foam expansion (E) 80 s after CO_2 was injected and the foam stability (L_F). Moreover, under different CO_2 pressure, flow, and time of gas sparging, they measure the average bubble lifetime or Bikerman coefficient (Σ). Therefore, for foam characterization with the parameters of Robillard et al. (1993) two Mosalux procedures, under different conditions, were required for each sample. In recent studies, the Mosalux or an equivalent nonautomated system (Pueyo et al., 1995), was used. Therefore, this equipment can be considered suitable for foam determination. However, the parameters used for foam characterization were different: researchers use either the parameters of Robillard et al. (1993) (E, L_F, and Σ) (Dussaud et al., 1994) or those of Maujean et al. (1990) (HM, HS, and TS) (Hardy, 1990; Poinsaut, 1991; Brissonnet and Maujean, 1991; Marchal et al., 1993; Malvy et al., 1994; Brissonnet et al., 1995; Andrés-Lacueva et al., 1996a,b) or a combination of the parameters of Maujean et al. (1990) and Bikerman (1938) (Pueyo et al., 1995; López-Barajas et al., 1997).

The aim of this study was to determine whether the different foam parameters (HM, HS, TS, E, L_F, and Σ) obtained by the gas sparging method, with the two procedures described (Maujean et al., 1990; Robillard et al., 1993), are comparable. Moreover, the advantages and disadvantages of these parameters were studied. All of the parameters described above were determined on nine samples of different wines, which were presumed to have different foam capacities. Statistical tests were used to evaluate the relationships between the foam parameters.

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MATERIALS AND METHODS

Samples. Nine samples of wine (W1, W2, W3, W4, W5, W6) and sparkling wine *cava* (C1, C2, C3) with different foam behaviors were used. They proceeded from different grape varieties [Macabeo, Xarel.lo (W3), Parellada (W5), Chardonnay (C3), or their blending (W1, W2, W4, W6, C1, C2)], different harvests [1993 (C1), 1994 (C3), 1995 (C2), or 1996 (W1, W2, W3, W4, W5, W6)], and different wineries [A (W1, W3), B (W2), C (C1, C2, C3), or D (W4, W5, W6)].

Equipment. The equipment used was a gas CO_2 carboy, with a pressure regulator (from 0 to 800 kPa), and the automated system Mosalux (Maujean et al., 1990). The Mosalux has the following elements: glass cylinder (40 mm i.d. $\times$ 230 mm) placed on a glass frit, gas flow regulator, infrared emission source, photoelectric receiver, and IBM-PC to process and store results.

Cleaning Procedure. To avoid interference in foam measurements, the impurities in the glass frit or on the glass cylinder internal walls were removed with the cleaning procedure described by Poinsaut (1991). Before each experiment, all glassware was rinsed in distilled water, ethanol, and a large volume of distilled water to eliminate ethanol. A small volume of sample to be assayed (20 mL) was then poured down the sides of the glass cylinder, to eliminate water, and then discarded. When a large number of samples were analyzed ($n > 20$), all glassware was treated with a sulfochromic mixture overnight. It was then rinsed with a large volume of distilled water, and finally CO_2 was injected through the glass frit to eliminate water.

Sample Preparation. The sparkling base wines were previously degassed by magnetic stirring (3000 rpm) for 15 min, and all wines were centrifuged at 4000 rpm for 20 min.

Analytical Procedures for Measurement of Foaming Properties of Maujean et al. (1990). The glass cylinder placed on a glass frit was filled with 100 mL of the sample to be analyzed. CO_2 was injected into the glass cylinder through the glass frit at a rate of 7 L/h under a pressure of 100 kPa for 7 min. The values of foam height over time were represented in a curve plot: foam increase to a maximum height, HM, expressed in millimeters. This is taken to represent the wine's ability to foam. Thereafter, it decreases to a stable, constant height, HS, expressed in millimeters. This is taken to represent the foam collar. At 7 min, gas injection was stopped, and the time until all bubbles collapse was measured, obtaining TS, expressed in seconds. This is taken to represent the foam stability time. The Bikerman coefficient, Σ, was measured following the method of Maujean et al. (1990). Expressed in seconds, this is taken to represent the average bubble lifetime, under steady state, when foam formation and destruction are balanced. It is the ratio of HS expressed in volume (milliliters) to the gas flow at constant pressure (milliliters per second).

Analytical Procedures for Measurement of Foaming Properties of Robillard et al. (1993). Gas flow and pressure were 20 L/h and 300 kPa, respectively, and the time of gas injection was 80 s. Two parameters (E and L_F) were obtained with the plot curve: foam expansion to 80 s (E), expressed in millimeters per milliliter, is the ratio of the foam height at 80 s (H_0), before maximum height was accomplished, to the sample volume (100 mL). L_F or foam stability, in seconds, is the average lifetime of the foam after gas injection is stopped. It was calculated as the ratio between the area under the curve after 80 seconds and the foam height at 80 s (H_0).

The foam parameters were calculated in triplicate for each sample and for each procedure.

Statistical Analysis. STATGRAPHICS 7.0 was used to calculate the relationships between the foam parameters. Linear ($y = a + bx$), multiplicative ($y = ax^b$), exponential ($y = e^{(a + bx)}$), and reciprocal ($1/y = a + bx$) regression models were considered.

RESULTS AND DISCUSSION

The foam parameters of samples W2 and C2 following the Robillard et al. (1993) procedure presented the maximum height before 80 s (Figure 1A). For these samples the CO_2 sparging is stopped after the maximum foam height, and so H_0 does not correspond to the maximum. Due to these phenomena the E value is not really accurate. This could be due to the fact that these two samples (W2 and C2) have the lowest values of E and HM (Table 1). Robillard et al. (1993) analyzed only two wines, subjected to various filtrations, and the values of the foam expansion (E) obtained by those authors range between 0.87 and 2.09 mm/mL (Figure 2). They chose 80 s of CO_2 without considering samples with lowest E values. In the current study, the E values obtained for the nine samples ranged from 0.44 to 2.57, outside the interval studied by Robillard et al. Therefore, before this E parameter is applied to several samples, it may be advisable to choose another time at which to stop sparging or another CO_2 flow rate.

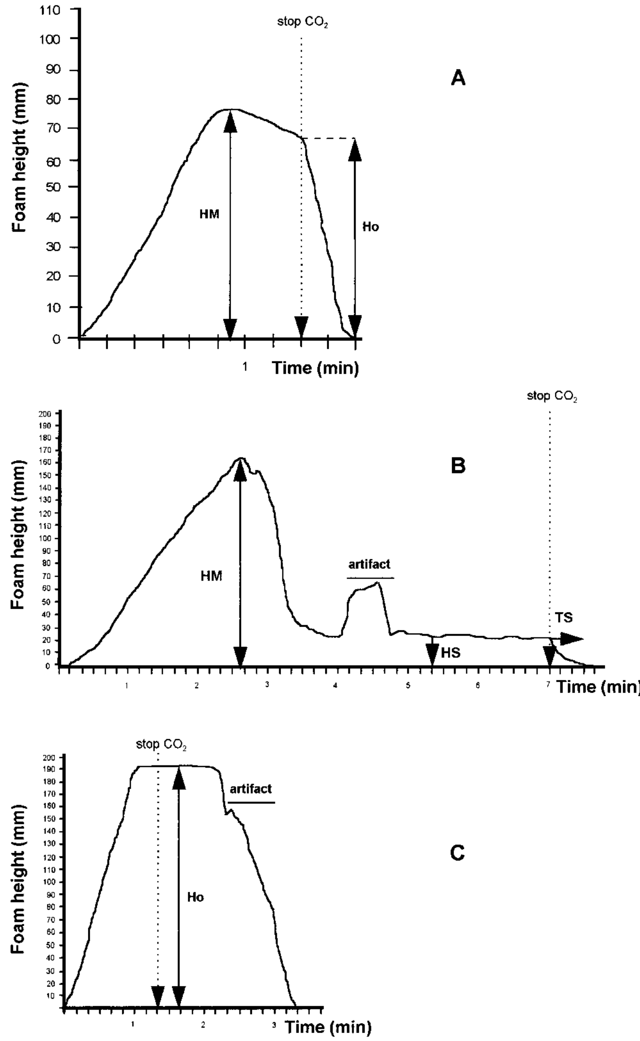

Figure 1. (A) Foam evolution in sample C2 to apply Robillard et al. (1993) procedure; (B) artifact formation in sample W3 to apply Maujean et al. (1990) procedure; and (C) artifact formation in sample W3 to apply Robillard et al. (1993) procedure.

When the procedure of Maujean et al. (1990) was applied to samples W3 and W6, the formation of artifacts was observed. In the plot (Figure 1B), a discontinuity appeared when the foam ring was separated from the main column. These artifacts could alter the values of maximum height of foam (HM). However, the ring rises above the upper limit detection of Mosalux (>192 mm) and, thus, does not interfere with the measurement of foam height (HM). Moreover, the

Table 1. Values of Foam Parameters Obtained by Method of Gas Sparging ($n = 3$)

	Maujean et al. (1990) parameters			Robillard et al. (1993) parameters		
sample	HM (mm) $x \pm$ Sn-1	HS (mm) $x \pm$ Sn-1	TS (s) $x \pm$ Sn-1	E (mm/mL) $x \pm$ Sn-1	Σ (s) $x \pm$ Sn-1	L_f (s) $x \pm$ Sn-1
W1	143 ± 9	24 ± 2	43 ± 2	2.25 ± 0.17	16 ± 1	36 ± 2
CV[a] (%)	6	8	5	8	6	6
W2	17 ± 3	15 ± 1	23 ± 5	0.44 ± 0.02	10 ± 1	6 ± 1
CV (%)	18	7	22	5	10	17
W3	160 ± 9	22 ± 1	37 ± 6	2.55 ± 0.03	14 ± 1	68 ± 7
CV (%)	6	5	16	1	7	10
W4	225 ± 5	32 ± 1	47 ± 6	2.57 ± 0.10	21 ± 1	98 ± 16
CV (%)	2	3	13	4	5	16
W5	183 ± 6	26 ± 2	45 ± 5	2.53 ± 0.03	17 ± 1	65 ± 6
CV (%)	3	8	11	1	6	9
W6	137 ± 1	24 ± 1	37 ± 8	2.18 ± 0.10	16 ± 1	50 ± 8
CV (%)	1	4	21	5	6	16
C1	39 ± 2	27 ± 1	297 ± 18	1.09 ± 0.01	18 ± 1	26 ± 3
CV (%)	5	4	6	1	5	12
C2	30± 4	23 ± 2	243 ± 18	0.72 ± 0.04	15 ± 1	14 ± 1
CV (%)	13	9	7	5	7	7
C3	58 ± 4	39 ± 2	443 ± 29	1.37 ± 0.01	25 ± 1	47 ± 8
CV (%)	7	5	7	1	4	17

[a] CV, coefficient of variation.

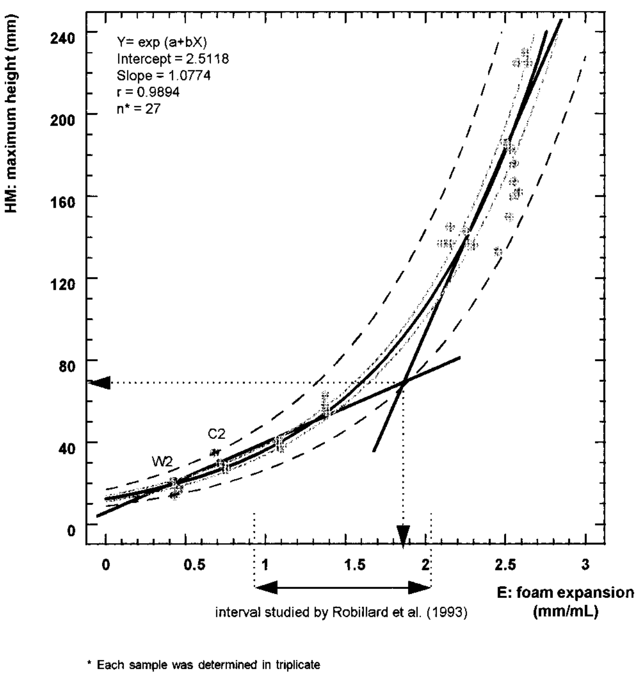

Figure 2. Exponential relation between the foamability (HM) and the foam expansion (E).

repeatability of HM values in these samples was satisfactory (Table 1). Presaturating the column with CO_2 and increasing the flow and pressure, according to the recommendations of Robillard et al. (1993), did not casue the artifacts to disappear. When the Robillard et al. (1993) procedure was applied to the same samples, artifacts were also formed (Figure 1C). In this case, the artifacts altered the height values used in area calculation and therefore often the foam stability values (L_F). Our hypothesis is that the artifact formation is due to the sample composition and does not depend on the cleaning procedure or experimental conditions. These artifacts are probably due to the nature of the wine biosurfactants and their different migration capabilities.

The repeatability, expressed as variation coefficients, of foam parameters obtained by the two procedures is shown in Table 1. The coefficients of variation of HM were <8%, except for the samples W2 and C2 with low values of HM (≤30 mm). That is why an inverse linear regression (Table 2) was observed between mean HM and the coefficients of variation ($r = -0.75$, $p < 0.05$).

Table 2. Correlation Coefficients (r) and Significance Levels (p) of Linear Relationships between Foam Parameters

	$n = 27$	HM	HS	TS	ϵ	E	L_f
HM	r	1.0000					
	p	0.0000					
HS	r	0.2028	1.0000				
	p	0.3103	0.0000				
TS	r	**−0.5702**	**0.6464**	1.0000			
	p	**0.0019**	**0.0003**	0.0000			
ϵ	r	0.2030	**1.0000**	**0.6463**	1.0000		
	p	0.3100	**0.0000**	**0.0003**	0.0000		
E	r	**0.9521**	0.2268	**−0.4788**	0.2270	1.0000	
	p	**0.0000**	0.2553	**0.0115**	0.2550	0.0000	
L_f	r	**0.8926**	**0.4485**	−0.2796	**0.4486**	**0.8582**	1.0000
	p	**0.0000**	**0.0190**	0.1578	**0.0189**	**0.0000**	0.0000

The E measurements also showed coefficients of variation <8% (Table 1), and no significant differences between the coefficients of variation of HM and E were observed. The stability height (HS) and the Bikerman coefficient, Σ, had coefficients of variation <10%. The time of stability (TS) was the parameter that had the lowest repeatability, mainly in samples with low foam stability (W2, W3, W5, and W6) with values of up to 22%. An inverse multiplicative regression was observed between average TS values and their coefficients of variation ($r = -0.78$, $p < 0.05$). This fact could explain the low precision found for some samples. L_F or foam stability was obtained with a variation <17% (Table 1). There were no significant differences between the coefficients of variation of TS and L_F.

Figure 2 shows the curve of the relationship between HM and E. In samples with HM and E values >80 mm and >1.7 mm/mL, respectively, the theoretical slope >1 shows that the HM parameter was more sensitive than E (a low E increase corresponded to a higher HM difference between samples). In samples with HM and E values <80 mm and <1.7 mm/mL, respectively, the theoretical slope <1 shows the opposite: the HM is less sensitive and E could be the best parameter to discriminate different foamabilities in wines. For four of nine samples E would be more sensitive than HM. However, for two of these four samples (C2 and W2) E is not really accurate (Figure 1A).

The strong correlation between E and HM (Figure 2) allows us to obtain theoretical values for E, from experimental HM values, with a high reliability ($r = 0.99$, $p < 0.0001$). The foam stability parameters, L_F

and TS, both expressed in seconds, were not correlated (Table 2). Nevertheless, L_F is related to E and HM (r = 0.86 and 0.89, respectively). Consequently, the parameters L_F and E, defined by Robillard et al. (1993), do not provide additional information. The relationships between TS and the other parameters have no predictive value ($r < 0.65$); thus, TS could be used to describe a different foam characteristic.

CONCLUSIONS

The formation of artifacts was not avoided by either of the procedures described (Maujean et al., 1990; Robillard et al., 1993), and artifacts appear to be a sample characteristic. To obtain the parameters proposed by Robillard et al. (1993) (E, L_F, and Σ), two Mosalux procedures were necessary for every sample, while to obtain the Maujean et al. (1990) parameters (HM, HS, and TS) a single procedure was sufficient. Moreover, less information about foam capacity was obtained with E and L_F, since these parameters were strongly correlated with each other: HM, E, or L_F could describe the same foamability. In samples with low foamability, E had better precision and discrimination than HM, but stopping the flow of CO_2 at 80 s was frequently unsuitable for the determination of H_0 at the maximum height, and thus the E value was not really accurate. This last error affected also the L_f value. TS has no predictive relationship with any of the other parameters; therefore, it could describe a different foam characteristic. The values of HM and TS were related with their coefficients of variation, and therefore the precision of the method was established. The Bikerman coefficient (Σ) was HS normalized. Σ was preferable to HS since it allows comparison of results with those of other research groups. We conclude that the best parameters to characterize the foam capacities of several wines were HM, Σ, and TS.

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