Effect of Juice Clarification by Flotation on the Quality of White Wine and Orange Juice and Drink – Short Communication

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Abstract

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White wines of Debina cultivar were made from musts clarified by flotation using nitrogen as foaming agent. Flotation using air as the foaming agent (must hyperoxidation), without SO_2 addition, was also applied. Turbidity and suspended solids were lower in must clarified by flotation using nitrogen than in that clarified by sedimentation (control). Flotation with hyperoxidation led to a reduction of must phenolics. All experimental wines exhibited similar gross compositions (alcohol, reducing sugars, total acidity, and volatile acidity). Wines made from musts clarified by flotation using nitrogen had similar total phenolic content, browning capacity, and organoleptic quality as the control wines. Wines made from musts clarified by flotation using air had lower total phenolic contents and browning capacity than were those in control wines. These wines were of well acceptable quality but exhibited a slightly oxidised aftertaste. The results indicate that flotation using nitrogen can be effective in the production of typical Debina wine, while flotation using air may be useful in that of table wine without SO_2 addition. Orange juice was clarified by flotation using nitrogen or air as the foaming agent. Orange juice clarified by flotation using nitrogen as the foaming agent exhibited lower turbidity and a similar pulp content to that clarified by centrifugal separator (control). It had an acceptable taste and aroma. Juice clarified by flotation using air as the foaming agent, along with pectolytic enzyme treatment, exhibited much lower turbidity and pulp content compared to control. The clear juice had an acceptable taste but no aroma. The fermented clear juice was averagely rated, exhibiting a pleasant aroma and only a slightly bitter taste. The results indicate that flotation using nitrogen can be effective in the production of natural orange juice, while flotation using air may be useful in the production of orange drink.

Keywords: must; wine; orange juice; orange drink; flotation; hyperoxidation; pectolytic enzyme

The technique of flotation is used to clarify grape musts and fruit juices before they undergo fermentation or concentration (Ferrarini *et al.* 1992, 1995; Otto *et al.* 1985; Wucherpfenning & Otto 1991).

With flotation, the solids are separated from the must rapidly thus improving the aroma of the future wine by ensuring that it ferments in a state of absolute purity. In winemaking, hyperoxidation occurs when the white juice is oxidised to the ex-

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tent that the phenols polymerise and precipitate out of the solution. This results in white wine with more stable colour. Sensory benefits and deficits can vary greatly from variety to variety (Cheynier *et al.* 1991; Vaimakis & Roussis 1993; Schneider 1998).

Flotation can also be beneficially applied to the technology of fruit juice production, particularly of apple juice. In orange juice technology, juice clarification is usually performed by centrifugal separators. In the Mediterranean countries, orange wine may be produced. In orange wine production, only strained juice is used. The white pith and the segment partitions impart an unpleasantly bitter taste to the wine.

The aim of the present work was to assess the efficiency of flotation used for the must clarification on the quality of Debina white wine. Flotation coupled with hyperoxidation was also assessed. Moreover, we assessed the efficiency of flotation used for orange juice clarification on the quality of orange juice. Flotation coupled with hyperoxidation was also assessed in the production of clear orange juice and drink.

MATERIAL AND METHODS

Wine production. Grapes used were of the cv. Debina which is late-ripening and easily oxidisable. Vines from Debina bear wine of golden yellow colour, delicate appley aromas, a low sugar content and fresh acidity; it is best enjoyed in its youth. Debina wine is protected by Appellation of Origin (Zitsa, Epirus, Greece).

Must preparation was performed by means of a typical industrial process (winery Zitsa, Epirus, Greece) using a batch press. Must portions were treated as follows:

(1) Addition of SO_2 (gas) at 60 mg/l. Addition of bentonite at 0.04% and agitation for 10 minutes. Sedimentation at 5°C for 24 h in plastic (polyethylene) barrels. The supernatant must was removed from the lees by decantation. The must thus prepared was the control, since must clarification by sedimentation is a common procedure.

(2) Addition of SO_2 (gas) at 60 mg/l. Addition of bentonite-fining agent at 0.04% and agitation for 10 minutes. Application of the technique of flotation, using nitrogen gas as the foaming agent.

(3) Addition of SO_2 (gas) at 60 mg/l. Addition of pectolytic enzyme (Lafazym CL, Lafort), 4 g/100 l, and a rest for 2 hours. Addition of bentonite-fining agent at 0.04% and agitation for 10 minutes. Application of the technique of flotation, using nitrogen gas as the foaming agent.

(4) Addition of bentonite-fining agent at 0.04% and agitation for 10 minutes. Application of the technique of flotation, using air as the foaming agent. Pectolytic enzyme was not used in order to avoid a long contact of must with air, since cv. Debina is an easily oxidisable cultivar.

Bentonite used in the must, as well as in the orange juice, treatment had the following composition (in %): SiO_2 – 71.02, Al_2O_3 – 13.58, TiO_2 – 0.24, Fe_2O_3 – 1.89, P_2O_5 – 0.05, MnO – 0.04, MgO – 2.20, CaO – 1.94, K_2O – 2.53, Na_2O – 2.50.

A pilot scale batch flotation system was used. The must – 190 l – was put in a pressurisation tank along with the fining agent. The foaming agent was used at 4 bars, and the must was agitated for 10 min using an industrial agitator (1500 rpm/min). Afterwards, the must was slowly and gently transferred to an open vessel through the bottom inlet, this process lasting for about 10 minutes. This was followed by the process of flotation which lasted for 15 minutes. The clarified must was removed from the bottom of the vessel.

In all cases, the clarified musts were fermented in plastic (polyethylene) barrels of 35 l capacity at 18°C, using *Saccharomyces cerevisiae* Zymaflore VL1 (Lafort), 10 g/100 l. At the end of fermentation, young wines were standardised at 100 mg/l SO_2, and at 5.5 g/l total acidity (using $CaCO_3$). The wines were then put into plastic barrels and stored at 10–13°C. Six months after the start of fermentation wines were filtered and bottled in green glass bottles (0.75 l).

Orange juice and drink production. The Valencia orange, the most popular cultivar used for juicing, coming from the region of Arta (Epirus, Greece), was used. Juice extraction and finishing were performed using a typical industrial process (juice factory Esperis, Arta, Greece). Juice extraction was carried out using FMS extractor followed by passing through a finisher, while clarification was done using a centrifugal separator. The juice prepared by this treatment was the control, since orange juice clarification using separator is the typical one. The technique of flotation was used instead of the separator.

The pilot scale batch flotation system employed in the must clarification was also used. The extracted juice – 190 l – was put in a pressurisation tank along with bentonite-fining agent at 0.04% (w/v).

Nitrogen gas or air was used as the foaming agent at 4 bars. When nitrogen was used as the foaming agent, the juice was agitated for 5 min using an industrial agitator (1550 rpm/min). Afterwards, the juice was slowly and gently transferred into an open vessel through the bottom inlet; this process lasted for about 10 minutes. This was followed by the process of flotation which lasted for another 10 minutes. The clarified juice was removed from the bottom of the vessel. When air was used as the foaming agent, the treatment with pectolytic enzyme was employed. Pectolytic enzyme (Lafazym CL, Lafort), 6 g/100 l, was added to the juice and allowed to stand for 90 min prior to processing. In this case, the juice was agitated for 20 min and the flotation lasted for 20 minutes. In the case of flotation using air, pectolytic enzyme (90 min treatment), and longer agitation (20 min) and flotation (20 min) time were used in order to obtain orange juice of high clearness.

Orange juice was fermented using *Saccharomyces cerevisiae* Zymaflore VL1 (Lafort), 10 g/100 l. The fermentation was carried out at 20°C. At the end of fermentation, the fermented juice was centrifuged at 3000 rpm/min for 10 min, and examined.

Analyses. Gross composition of the must and wine samples was determined by classical methods (Ough & Amerine 1988). Reducing sugars were determined by the Lane-Eynon method and total acidity by volumetric analysis. Alcohol was determined pycnometrically, volatile acidity by the steam distillation method, and total sulphite SO_2 by the Ripper method.

Must turbidity was determined using a nephelometer (Hach, model 2100AN). Suspended solids were determined by drying the sediment obtained after centrifugation of samples at 3000 rpm for 10 min to constant weight at 100°C.

Total phenolics of the must and wine samples were determined by the Folin-Ciocalteu method (Singleton & Rossi 1965) using gallic acid as a standard. The browning capacity of wine samples was determined as described previously (Singleton & Kramling 1976), except that bentonite treatment was not used.

The turbidity of orange juice was determined as described for must, and brix using a refractometer. The pulp content of orange juice was determined by measuring sample sediment after centrifugation at 1500 rpm for 10 minutes.

Total acidity of orange juice and drink was determined by volumetric analysis using 0.1N NaOH and phenolphthalein as indicator, and pH using a pH-meter.

Alcohol, volatile acidity and the content of reducing sugars in orange drink were determined as described above for wine.

In all cases, a five-member panel, who expressed their appraisal during a table discussion, carried out organoleptic evaluation of the samples.

All experiments were run in triplicates and the results reported here are the means of the three runs.

RESULTS AND DISCUSSION

Must clarification and wine production

The characteristics of the clarified musts are given in Table 1. The contents of reducing sugars and acidity were similar in all experimental musts. Turbidity and suspended solids of the musts clarified by flotation using nitrogen with or without the enzyme treatment were lower than in that clarified by sedimentation (control). Especially, the contents of suspended solids were low in the musts clarified using pectolytic enzyme. However, the musts clarified by flotation using nitrogen or sedimentation (control) exhibited similar contents of total phenolics.

The must clarified by flotation using air exhibited turbidity and suspended solid values similar to that clarified by sedimentation (control). These musts exhibited much lower contents of total phenolics than control musts. This could be attributed to a greater degree of oxidative polymerisation of phenolic compounds and the consequent precipitation (removal) of the oxidation products. The

Table 1. Characteristics of musts after flotation

Characteristic (must)	1	2	3	4
Reducing sugars (g/l)	150	156	149	141
Total acidity (g/l tartaric acid)	9.0	8.6	8.7	7.5
Turbidity (NTU)	481	144	131	320
Suspended solids (g/l)	0.27	0.19	0.10	0.30
Total phenolics (mg/l gallic acid)	490	460	450	170

1 – must clarified by sedimentation; 2 – must clarified by flotation using nitrogen; 3 – must clarified by flotation using nitrogen along with pectolytic enzyme treatment; 4 – must clarified by flotation using air

Table 2. Characteristics of experimental wines at the end of fermentation

Characteristic (wine)	1	2	3	4
Alcohol (ml per 100 ml)	10.1	9.8	10.2	8.7
Total acidity (g/l tartaric acid)	8.6	8.6	8.7	7.2
Volatile acidity (g/l acetic acid)	0.22	0.26	0.24	0.39
Total SO_2 (mg/l)	51	45	47	0
Reducing sugars (g/l)	1.0	1.2	1.1	1.4

1 – wine from must clarified by sedimentation; 2 – wine from must clarified by flotation using nitrogen; 3 – wine from must clarified by flotation using nitrogen along with pectolytic enzyme treatment; 4 – must clarified by flotation using air

reduction of the phenolics content during must hyperoxidation has also been observed by others (Vaimakis & Roussis 1993).

The characteristics of experimental Debina wines are given in Table 2. All wines were dry, and no differences among them in the gross composition were observed. Hence, it could be said that the must clarification by flotation did not affect the gross composition of white wines Debina.

Table 3 shows the total phenolics, A 420 nm, and browning capacities of the experimental wines. Wines made from the musts clarified by sedimentation (control) and flotation using nitrogen – with or without the enzyme treatment – exhibited similar values in the above parameters. On the other hand, wines made from the must clarified by flotation using air exhibited lower values in total phenolics, A420 nm, and browning capacities than the others. The low contents of total phenolics of these wines indicate that they had been subjected to oxidation (browning) to a greater extent, since the oxidation products are precipitated during winemaking. Moreover, the low values of these wines in A420 nm and browning capacities indicate that they were stable against browning through the time.

Table 3. Total phenolics, A 420 nm and browning capacities of experimental wines

Characteristic (wine)	1	2	3	4
Total phenolics (mg/l gallic acid)	320	310	290	160
A 420 nm	0.120	0.100	0.120	0.060
Browning capacity	0.483	0.400	0.420	0.141

1 – wine from must clarified by sedimentation; 2 – wine from must clarified by flotation using nitrogen; 3 – wine from must clarified by flotation using nitrogen along with pectolytic enzyme treatment; 4 – wine from must clarified by flotation using air

Total phenolics and A 420 nm of 6-month and browning capacities of 8-month experimental wines were determined

The wines produced by must sedimentation (control) were typical of the grape variety used. They exhibited a golden yellow colour, aroma of matured fruits, balanced taste and aromatic aftertaste. Wines produced by must flotation using nitrogen were more or less of organoleptic properties similar to those of control wines. All of these wines were of very good organoleptic quality, typical of the grape variety used. On the other hand, the wines produced by must flotation using air revealed some different properties. They exhibited a pale yellow colour with a green hue, a complex aroma of aged wine with only slight freshness, not so well balanced taste, and a slightly oxidised aftertaste. These wines were of good organoleptic quality but not typical of cv. Debina.

All the above indicate that the technique of flotation using nitrogen as the foaming agent can be effective in the production of typical Debina wine. Similarly, successful must clarification by flotation in making other wines has been reported by others (Ferrarini *et al.* 1995; Marchal *et al.* 2003). The must clarification by flotation using air (hyperoxidation), without SO_2 addition, may be useful in making table wine using cv. Debina. The combination of flotation and hyperoxidation has also been reported by others (Ferrarini *et al.* 1995).

Orange juice clarification and drink production

The characteristics of crude and clarified experimental orange juice are given in Table 4. The crude juice had a pulp content of 12% and high turbidity. It exhibited a pleasant taste and aroma, with the organoleptic properties being typical of the orange variety used.

Juice clarified by flotation using nitrogen exhibited lower turbidity and a similar pulp content as that clarified by the separator (control). It is possible that flotation using nitrogen removes particles other that pulp which contribute to the turbidity. The juice clarified by flotation using ni-

Table 4. Characteristics of experimental orange juices

Characteristic	Crude juice	Juice clarified by separator	Juice clarified by flotation/nitrogen	Juice clarified by flotation/air
Turbidity (NTU)	8208	4226	1966	219
Pulp content (%)	12.0	4.8	4.2	1.0
Brix (°)	10.9	10.9	10.8	10.8
Total acidity (% citric acid)	0.92	0.92	0.93	0.96
pH	3.70	3.65	3.62	3.70

Table 5. Characteristics of experimental orange drinks

Characteristic	Drink from juice clarified by separator	Drink from juice clarified by flotation/nitrogen	Drink from juice clarified by flotation/air
Alcohol (ml/100 ml)	4.4	4.4	4.6
Total acidity (% citric acid)	0.88	0.81	0.81
Volatile acidity (g/l acetic acid)	0.44	0.17	0.23
pH	3.62	3.67	3.73
Reducing sugars (g/l)	1.1	1.0	1.0

trogen had an acceptable flavour. However, it was slightly worse than that clarified by the separator (control) which exhibited the pleasant organoleptic characteristics of the crude juice. These two juices did not show any difference in brix, acidity, and pH values, which were similar to those of the initial juice.

The juice clarified by flotation using air along with pectolytic enzyme treatment exhibited very low turbidity and pulp content. It is likely that the use of air as the foaming agent may lead-via hyperoxidation and removal of the products- to less turbid juice. Moreover, the treatment with enzymes may have an impact. It has been reported that enzymic degradation of pectins leads to an improvement of the juice clarity and to spontaneous (natural) flotation and clarification effect (Ferrarini *et al*. 1995; Le Quére *et al*. 1988; Wucherpfenning *et al*. 1986). The juice clarified by flotation using air had an acceptable taste, while, at the same times, it was characterised by the absence of aroma. This juice also had brix, acidity and pH values similar to that of the initial juice.

The characteristics of the experimental orange drinks are given in Table 5. All experimental drinks were dry (about 1 g/l reducing sugars) and soft (about 4.5% alcohol), and exhibited similar values of acidity, volatile acidity, and pH.

Drinks made from juice clarified by the separator had orange colour, a clear bitter taste, and the aroma of overmature orange. They were unacceptable drinks. Similarly, drinks made from juice clarified by flotation using nitrogen exhibited yellow colour, a bitter taste and an aroma like sour drinks. They were not rated as acceptable drinks by the panelists. Drinks made from juice clarified by flotation using air were transparent, exhibited a pale yellow colour, a pleasant aroma and only slightly bitter taste. It seems that the absence of aroma in the experimental clear juice permits the development of aroma during fermentation. These drinks were much better than the others, even though not fully acceptable.

The present results indicate that the technique of flotation using nitrogen gas as the foaming agent can be effective in the production of natural orange juice. On the other hand, flotation using air as the foaming agent along with the treatment with pectolytic enzymes may be useful in the production of clear orange juice and drink. Of course, the disadvantage of the possible oxidation should be overcome, e.g by using a natural reducer as it has been reported previously (Vaimakis & Roussis 1996). It should be noticed that the technique of flotation possibly permits the use of both the foamed and residual fractions, as it was proposed for the isolation of a carotenoid-rich fraction by others (Pinnola & Oberto 1975).

CONCLUSIONS

The present results indicate that the technique of flotation using nitrogen as the foaming agent can be successfully used in winemaking of Debina and possibly similar grape cultivars yielding typical wines.

Must clarification by flotation using air, without SO_2 addition, can be successfully used in winemaking of cv. Debina and similar grape cultivars yielding table wines of very good and fast colour with good organoleptic quality.

The present results also indicate that the technique of flotation may be also useful in orange juice and drink technology. Flotation using nitrogen as the foaming agent may lead to natural orange juice of good quality. On the other hand, flotation using air as the foaming agent along with pectolytic enzymes is promising for the production of clear orange juice and drink of acceptable quality. The standardisation of the technological parameters may further improve the quality of these products.

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