# Float or sink?
## *Comparing the impacts of flotation and cold settling on the non-volatile composition, taste and mouthfeel of white wines*

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**Flotation is a high-through-put continuous process for white juice clarification that is more efficient than traditional cold settling/racking. While the efficacy of flotation is well understood, its impact on white wine composition and sensory properties has been largely unexplored. This article presents results from a study comparing the effects of cold setting and flotation on the characteristics of Chardonnay and Frontignac wines.**

### INTRODUCTION

When white grapes are crushed, the resultant juice contains pulp and skin cell wall fragments called 'grape solids' (solids). Most solids are removed from juice prior to fermentation as their presence can lead to lower fruit expression either from enzymatic oxidation, decreased yeast esterase activity, or from the production of higher alcohols (Riberéau-Gayon *et al*. 1975). In commercial practice solids removal is often achieved by cold settling where the must is chilled, the solids settle to the bottom of the tank, and the clarified juice is racked into a second tank for fermentation. This process is effective but costly due to its high energy requirements and inefficient due to the necessity of using more than a single tank.

In the late 1990s winemakers began using flotation to clarify white juices, finding it more efficient and cost effective than cold settling due to its high flow and continuous nature (Falkenberg 1997). Flotation involves super-saturating white juices containing solids with either nitrogen or air, which upon depressurisation increases the buoyancy of the solid particles as they stick to the gas microbubbles and rise to the surface where they are removed by skimming. In practice, solids are floated off faster when pectolytic enzymes and a flocculating agent such as bentonite are added. The enzymes reduce juice viscosity and the flocculating agent increases microbubble formation and adherence to the solids (Marchall *et al*. 2003).

The economic efficiency of flotation has been compared with cold settling (Falkenberg 1997), as has its efficacy when using different flocculating agents, floating gases, temperatures and pressures (Davin and Sahraoui 1993). But how does juice clarification by flotation affect the composition and the resultant taste and texture of white wine when compared with wines made from cold settled and full solids juices?

### METHODS

#### Juice preparation and winemaking

Chardonnay and Frontignac grapes from the Murray Valley region were processed into juice by different commercial wineries using similar protocols. Pectolytic enzymes were added at crush and the juice drained off skins into a pre-flotation storage tank. Two 20-litre juice samples of each treatment were collected for fermentation. These treatments (summarised in Table 1, see page 18) were:

- high solids (HS) taken from an upper racking valve of the storage tank to preclude gross solids
- low solids by settling (LS-SE) where HS juice was settled at 0°C before being racked off fine solids
- low solids by flotation (LS-FL) produced by dosing in-line with bentonite before floating off solids using nitrogen gas at a discharge rate of 30kL/h.

The juice samples were chilled and adjusted to pH3.4 by tartaric acid addition and to a free $SO_2$ of 10-25mg/L. They were inoculated at the same time using *S. cerevisiae* strain EC1118 at 15-16°C. The resultant wines were racked and 60mg/L of $SO_2$ added; they were then cold stabilised at 0˚C using 4g/L KHT, pad and membrane filtered, adjusted to 35mg/L free $SO_2$ and bottled in 375mL units under screw cap.

#### Wine and juice analysis

The solids content of the high solids juices was measured as % wet weight, while the solids content of the floated and settled juices were determined by nephelometry (Table 2, page 18). Ethanol, organic acids ▸

> ## IN BRIEF
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> - Chardonnay and Frontignac juices were clarified by cold settling and by flotation using nitrogen gas prior to winemaking under commercial conditions, with wines produced from unclarified juice as controls.
> - The phenolic and polysaccharide profiles of wines produced using flotation were similar to those clarified by cold settling/racking, which was reflected in their similar mouthfeel and taste properties.
> - The wines produced by flotation were slightly more viscous than the wines made from either the settled or unclarified juice. The differences in perceived viscosity were best correlated with pH and total phenolic content.

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and glycerol were determined by HPLC, and other compounds given in Table 3 were determined by NIR spectroscopy. Total phenolic concentrations were determined using the Folin-Ciocalteu method. A targeted HPLC analysis of phenolic compounds representative of the major phenolic classes found in white wines was conducted by reverse phase C18 HPLC. Total wine polysaccharides were quantified by the phenol-sulfuric method, and the polysaccharide molecular weight distribution was determined by size exclusion chromatography.

### Sensory methods

Nine assessors experienced in descriptive analysis rated the relevant taste and mouthfeel attributes (viscosity, astringency, hotness and bitterness) using an unstructured line scale. Samples of 30mL of wine were presented in an order and timing determined to minimise taste carry-over effects using ISO standard wine glasses at 22-24°C in isolated booths under daytime lighting.

## RESULTS AND DISCUSSION

The solids content of the low-solids treatments produced by settling and flotation were similar (Table 2) and the wine compositional parameters were within acceptable ranges (Table 3).

### Total phenolics in juice and wine

The floated Frontignac juice had significantly lower total phenolic concentration than the settled juice (Figure 1A). A similar but non-significant trend was observed in the Chardonnay juices. The lower total phenolics in floated juices compared with settled juices is consistent with previous studies (Ferrarini *et al*. 1995, Sindou *et al*. 2008) and may be explained by the shorter contact time between grape lees particles and juice experienced during flotation compared with settling (Table 1). The total phenolic concentrations of the high-solids juices were not significantly different from the cold-settled juice for both varieties, which is expected as the grape solids were in contact with the juice for the same duration. However, these explanations are speculative as the phenolic content of white grape solids and their extractability into juice pre-fermentation has yet to be determined. Another possible explanation is that some phenolics may have been removed from the floated juices by the bentonite added to improve the efficacy of flotation. Lastly, it should be noted that the differences in total phenolic concentration between treatments were relatively small compared with the average concentrations, suggesting that the majority of total phenolics were extracted into the juices prior to clarification (i.e. during crushing, draining and the settling and removal of coarse solids).

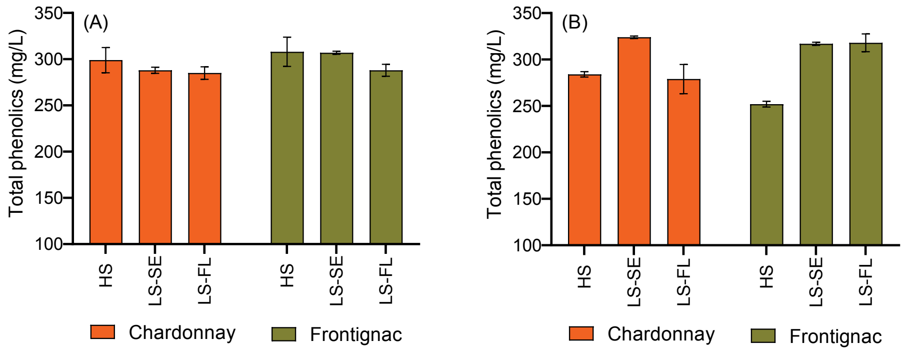


**Figure 1. Total phenolics (A) juice and (B) wine expressed in gallic acid equivalents. HS – high solids, LS-SE – low solids via settling, LS-FL – low solids via flotation. Error bars represent two standard errors.**

The effect of juice clarification on total phenolic concentrations in the bottled wines varied between the varieties. The total phenolic concentrations of the high-solids Frontignac wines were lower than those clarified by settling and flotation (Figure 1B), but the low-solids settled Chardonnay wines were higher in total phenolics than the high-solids and floated wines. Other researchers have also not observed a relationship between juice solids content and total wine phenolic concentration (Singleton *et al*. 1975, Sindou *et al*. 2008). However, here, the significant decreases in total phenolics seen after fermentation on high solids suggests that the solids may be acting as a phenolic 'fining agent', possibly related to non-covalent interactions with polysaccharides, which were in higher concentrations in these wines (Figure 3, see page 20).

### Wine phenolic profile

Wines made from settled and floated low-solids juices showed similar phenolic profiles, different from those produced from high-solids juices (Figure 2). Specifically, the wines made from low-solids juices had significantly higher concentrations of caftaric acid and generally lower concentrations of its derivative grape reaction product (GRP) than the high-solids wines. The ratio of caftaric acid to GRP can be influenced by $SO_2$, which may explain the differences between the two varieties as the grapes were processed in different wineries with different $SO_2$ regimes.

**Table 1. Summary of winemaking procedures and contact of grape solids with juice and fermenting wine.**

| Treatment | High solids | Low solids-settled | Low solids -floated |
|---|---|---|---|
| Enzyme at crusher | Yes | Yes | Yes |
| Contact with gross lees | Yes | Yes | Yes |
| Bentonite addition to juice | No | No | Yes |
| N2 addition to juice | No | No | Yes |
| Contact time with grape solids (from racking off gross lees to yeast inoculation) | 3.5 days | 3.5 days | <0.1 hrs |
| Fermentation on high grape solids | Yes | No | No |

**Table 2. Solids content of winemaking treatments (n=2)**

| | High solids (% wt) | Low solids-settled (NTU) | Low solids-flotation (NTU) |
|---|---|---|---|
| **Chardonnay** | 3.75% | 100 | 97 |
| **Frontignac** | 2.75% | 95 | 90 |

**Table 3. Basic wine analysis (mean, n=2).**

| Treatment | Ethanol % (v/v) | Glucose + fructose g/L | pH | Titratable acidity g/L | Free SO2 mg/L | Total SO2 mg/L | Malic acid g/L | Volatile acidity g/L Acetic | Succinic acid g/L | Lactic acid g/L | Glycerol g/L |
|---|---|---|---|---|---|---|---|---|---|---|---|
| **Chardonnay** | | | | | | | | | | | |
| **HS** | 13.9 | 0.95 | 3.45 | 5.6 | 35 | 155 | 2.9 | 0.25 | 3.3 | 0.5 | 7.2 |
| **LS-SE** | 14.0 | 1.00 | 3.48 | 5.9 | 32 | 202 | 3.3 | 0.37 | 3.3 | 0.5 | 7.6 |
| **LS-FL** | 13.8 | 0.90 | 3.45 | 6.2 | 32 | 163 | 2.8 | 0.32 | 3.3 | 0.5 | 7.1 |
| **Frontignac** | | | | | | | | | | | |
| **HS** | 11.0 | 0.55 | 3.51 | 4.4 | 30 | 160 | 0.7 | <0.1 | 1.7 | 1.0 | 6.3 |
| **LS-SE** | 11.4 | 0.40 | 3.46 | 5.2 | 30 | 180 | 2.2 | <0.1 | 1.8 | 0.5 | 6.0 |
| **LS-FL** | 11.1 | 0.55 | 3.56 | 4.3 | 32 | 167 | 0.5 | <0.1 | 1.8 | 0.5 | 6.0 |

The combined concentrations of the monomeric flavan-3-ols, catechin and epicatechin were significantly higher in the settled wines than in the floated and high-solids wines (Figure 2). Ferrarini *et al.* (1995) also found that cold settling resulted in higher catechin concentrations in white Picpoul wine compared to flotation. The tyrosol concentrations of the settled and floated wines were similar, but the effect of high solids was variety dependent (Figure 2). Konitz *et al.* (2003) reported higher tyrosol concentrations in Riesling wines made from juice floated using air compared with those made from settled juices, a result which may be explained by a healthier yeast fermentation due to the must oxygenation.

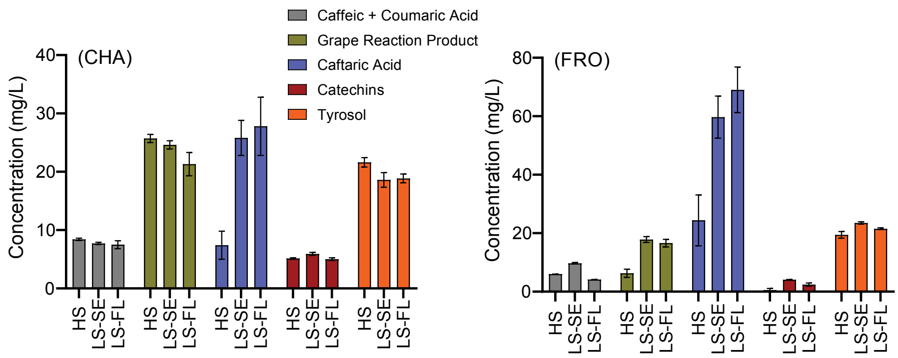


**Figure 2. Mean concentration of phenolic compounds in wine. (CHA) Chardonnay, (FRO) Frontignac. Error bars represent two standard errors.**

High-solids juices produced wines with a higher total polysaccharide concentration (Figure 3) and a higher proportion of high molecular weight polysaccharides (Figure 4, see page 20) compared with the wines produced from low solids juice using flotation. Previous work at the AWRI has shown that white wine polysaccharides greater than 93kDa consist mostly of mannoproteins derived from yeast during fermentation and yeast lees contact. This result is consistent with the knowledge that yeast-assimilable sterols adsorbed on grape solids help improve cell viability and maximum populations of yeast cells, particularly at the end of fermentation, which could promote the production and release of mannoproteins by yeast autolysis.

Wines made from low-solids juices clarified by cold settling and by flotation did not differ in total polysaccharide concentration (Figure 3) or their molecular weight profile (Figure 4), suggesting that the longer clarification time involved in cold settling compared with flotation did not result in significantly greater extraction of polysaccharides from the grape solids prior to fermentation.

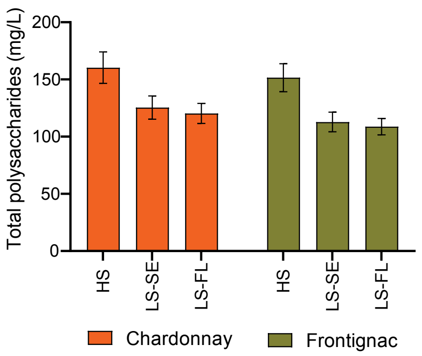


**Figure 3. Concentration of total polysaccharides in wine as glucose equivalents. HS – high solids, LS-SE – low solids via settling, LS-FL – low solids via flotation. Error bars represent two standard errors.**

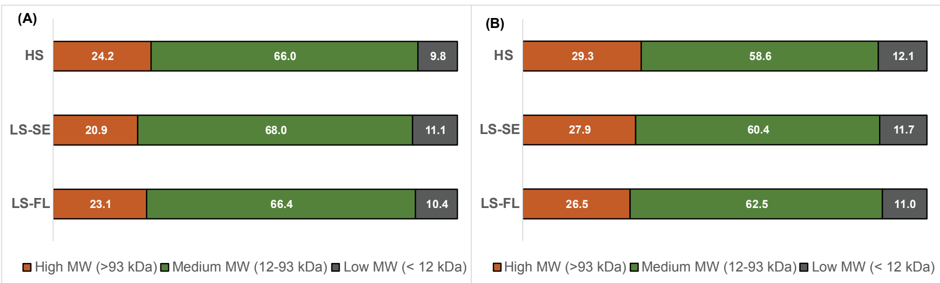


**Figure 4. Polysaccharide molecular weight profile (A) Chardonnay, (B) Frontignac. Numbers inside bars represent percentages based on HPLC peak area. HS – high solids, LS-SE – low solids via settling, LS-FL – low solids via flotation**

### Sensory effects of clarification and relationship to composition

There was consistent evidence across both varieties that the wines made from floated juice were slightly more viscous and, in the case of Frontignac, the settled wines were significantly less bitter than the high-solids and floated wines (P<0.1). The hotness and astringency of the wines were not affected by the treatments applied.

Perceived viscosity correlated with total phenolics (Gawel *et al*. 2013) and specifically with caftaric acid concentration (P<0.05) (Gawel *et al*. 2014) but was most strongly correlated with pH (P<0.01); that is, greater perceived viscosity was positively associated with increasing pH (Runnebaum *et. al*. 2011). Higher total polysaccharide concentration was associated with lower perceived viscosity (P<0.1) (Figure 5), a result contradictory to that of some other studies which found higher concentrations of neutral polysaccharides in white and red wine increased their perceived viscosity (Vidal *et al*. 2004, Gawel *et al*. 2016).

The cold-settled Frontignac wines were less bitter than the high-solids and low-solids floated wines (P=0.05). The differences in bitterness cannot be attributed to total wine phenolics (Figure 1B) nor to any of the quantified phenolic compounds (Figure 3) including catechin and epicatechin, which elicit bitterness albeit at higher concentrations than found in these wines. This result, together with the lack of influence of ethanol (which also elicits bitterness) (Figure 5) suggests that the compounds responsible for the greater bitterness in the settled wines were not captured in this study.

| | Total Polysaccharides | High MW PS | Med MW PS | Low MW PS | Glycerol | Glucose+Fructose | Ethanol |
|---|---|---|---|---|---|---|---|
| Viscosity | -0.595 | -0.143 | 0.062 | -0.045 | 0.032 | 0.159 | 0.036 |
| Acidity | 0.135 | -0.102 | 0.359 | 0.358 | 0.061 | -0.080 | -0.008 |
| Hotness | 0.445 | 0.049 | -0.275 | 0.040 | -0.029 | -0.102 | 0.012 |
| Astringency | -0.149 | -0.294 | -0.062 | -0.072 | -0.023 | -0.011 | 0.003 |
| Bitterness | 0.023 | -0.229 | 0.054 | 0.061 | 0.008 | 0.019 | 0.009 |

| | Total phenolics | GRP | Caftaric | Catechins | Caffeic+Coumaric | Tyrosol |
|---|---|---|---|---|---|---|
| Viscosity | 0.691 | 0.249 | 0.574 | 0.317 | -0.094 | -0.105 |
| Acidity | 0.007 | -0.037 | -0.161 | -0.081 | -0.222 | -0.192 |
| Hotness | -0.422 | -0.150 | -0.319 | -0.079 | 0.111 | -0.052 |
| Astringency | 0.137 | 0.052 | 0.079 | 0.130 | -0.183 | -0.157 |
| Bitterness | 0.063 | 0.009 | -0.026 | 0.054 | -0.242 | -0.200 |

| | pH | Total acidity | Tartaric acid | Malic acid | Lactic acid | Succinic acid |
|---|---|---|---|---|---|---|
| Viscosity | 0.735 | -0.130 | -0.252 | 0.049 | -0.118 | 0.013 |
| Acidity | -0.079 | -0.007 | 0.111 | -0.200 | -0.227 | -0.015 |
| Hotness | -0.371 | 0.082 | 0.181 | -0.034 | 0.127 | -0.032 |
| Astringency | 0.361 | -0.124 | -0.075 | -0.036 | -0.015 | 0.007 |
| Bitterness | 0.279 | -0.139 | 0.001 | -0.132 | 0.016 | -0.005 |

**Figure 5. Heat map showing the correlation (r) between mouthfeel and taste characters and analytical parameters. Green indicates positive correlation and red negative correlation. Density of colour represents strength of correlation. Correlations in boxes were statistically significant (P<0.05).**

**SUMMARY**

The composition and sensory qualities of Chardonnay and Frontignac wines made from juices clarified using flotation were compared with wines made using the less efficient method of cold settling. The non-volatile composition including total phenolics, total polysaccharides, phenolic profile and polysaccharide molecular weight profile of the wines produced by flotation were similar to those made using cold-settled juices, which was reflected in the wines having similar mouthfeel and taste properties. Both the Chardonnay and Frontignac wines produced by flotation by two different wineries were perceived to be slightly more viscous than the wines made from either the settled or unclarified juice. Perceived viscosity was best correlated with higher pH and higher total phenolic content.

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