NTU vs wine filterability index – what does it mean for you?

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Introduction

THE BLOCKAGE OF sterile filtration media, either rapidly or slowly via an exponential decline, can occur during wine bottling, even though the wine meets a pre-bottling turbidity specification suitable for the chosen filtration media. This article will explore the relationship between turbidity and the filterability of wine.

Background

Turbidity is used as a means of assessing the particulate level in a wine (visual clarity) and therefore its suitability for bottling. There are many potential suspended components in a liquid, such as silt, yeast, bacteria, amorphous and crystalline materials that cause turbidity. A commonly used threshold for bottling is ≤1 nephelometric turbidity units (NTU). If a wine has an NTU ≤1, it is deemed suitable for bottling in terms of how it will present in the bottle and its low likelihood of fouling filtration media, specifically sterile membranes. If the pre-bottling NTU >1, and the wine is to be sterile filled, then it is recommended that the wine receives extra filtration. This may be crossflow filtration in the cellar or depth filtration on line, depending on the severity of the problem and the cost to the owner of the wine.

A nephelometer (turbidity meter) measures the extent that light is scattered by any suspended particulate in the sample. This method of analysis is used when bottling wine as a means of estimating how the suspended material may block filtration media, but does have some limitations. For example, two wines may have similar turbidity values but the nature of the suspended material is different. The first wine may contain very fine particles which rapidly block and create a film on the filtration media, rendering it unable to complete the filtration task. The other wine may have larger particles, which create a film on the filtration media, but do not totally block the media. Therefore, filtration is able to continue, albeit at a reduced efficiency.

There is surprisingly little in the way of published research literature that covers the relationship between filtration fouling and turbidity in wine. Roger Boulton published an article in 2001 in which he states specifically that "fouling of wines on membrane filters is not related to their clarity" (Alarcon-Mendez and Boulton 2001). Since clarity is typically expressed as turbidity (NTU), this is an important statement. He also points out the strong influence of temperature on fouling in an earlier publication, that is cooler temperatures will typically produce reduced filterability (La Garza and Boulton 1984). Czekaj, López and Güell (2000) indicate that membrane fouling is mainly caused by colloidal components of the wine. When they analysed two wines with similar colloidal (macromolecular) content but different turbidity, they found that the wine with the higher turbidity caused greater membrane fouling. After further filtration treatment of the wine with the higher turbidity, to reduce its turbidity to the same as that of the lower turbidity wine, it was still found that this treated wine had a much greater membrane fouling potential. It was stated that this may be due to a difference in polyphenol concentration between the two wines.

El Rayess *et al.* (2011) states that macromolecular compounds such as polysaccharides, phenolics and proteins are major causes of membrane fouling. Given the variation in polysaccharide structures (*viz.* pectins, mannoproteins and glucans), this is of particular relevance to wine producers who do not use pectolytic enzymes, add mannoprotein to their wine or perform lees ageing, as these practices will contribute to higher levels of pectins and mannoproteins and potentially higher fouling rates of filtration media. When Botrytis is a problem during vintage, such as that experienced during the 2011 vintage in South Australia, there is an increased risk of increased glucan levels being produced. This knowledge made it possible to predict that red wines from certain areas in 2011 would very likely present filtration difficulties. Blue H2O Filtration advised its customers of this increased risk. Many filtration problems were circumvented through preventative filterability analysis. Perhaps the most relevant statement comes from a 2003 paper by Vernhet, Cartalade, and Moutounet, where the authors maintain that membrane fouling will correlate more consistently with colloidal size rather than turbidity, and that this explains variations in wine filterability.

Filterability index (FI) as a measure of filtration media fouling

Given that the major concern is the ability of a wine to pass through a sterile membrane (typically defined as 0.45 microns in Australia), it is important to use a test that demonstrates how the wine will block the filtration media over time, such as a filterability index test. This test is relatively simple to perform, yet it is a rare practice in Australian wineries and third party contract bottling companies. The equipment required is simple and inexpensive (Figure 1a), yet this process is somewhat labour-intensive, requiring an operator to be present throughout the testing protocol. This approach is not suitable in high throughput facilities. Fully automated equipment is available, yet this can be cumbersome to use and can require a large volume of wine for measurement, for example, the Begerow BECO Liqui-Control filterability unit requires 3L of wine for a filterability assessment and our experience has shown that it is difficult to drain when not all of the wine is used for the test. It also poses a challenge to keep clean. A good compromise is semi-automated equipment, such as the Tecnoelettric Filterability Tester available through Blue H2O Filtration (Figure 1b). This combines the robustness of the manual method with the ease of computerised monitoring of the

Figure 1. Different types of apparatus for measuring FI: (a) manual equipment; (b) semi-automated equipment.

analysis and recording of results, achieved through the use of a balance and computer data collection. In essence, once the test has commenced, the operator can walk away and the results are recorded, plotted and archived in real time.

Filterability index (FI), as defined by Laurenty in 1972 (as referenced in Table 7.6, Boulton, *et al.* (1999), is tested by passing wine through a 0.45 µm membrane filtration media, at constant pressure, and timing how long the membrane takes to filter two volumes, typically 200mL (T_{200}) and 400mL (T_{400}) in seconds. Thus, FI = $T_{400} - 2 \times T_{200}$. (Note: the Tecnoelettric Filterability Tester measures the time taken to filter 200g and 400g of wine, ignoring the density of the wine). If the wine is perfect in terms of filterability the ratio of T_{400} to T_{200} will be 2:1, and so FI = 0. Since a wine will usually have some fouling components, T_{400} will typically be more than double T_{200}. In this case an index is generated by the calculation, and this can be used as a de facto measure of wine filterability. Sometimes this value is multiplied by a factor of 1.66, but since this is a constant it is of no great significance in calculating the final result. By setting some nominal FI thresholds, filterability measured in this way can then be used to determine whether a wine requires some form of pre-filtration, either in the cellar, prior to being sent to the bottling line, or in line, during the bottling process. Laurenty recommended that if the FI <20, then the wine would be considered to be filterable. Importantly, the measurement of filterability must use the same membrane as is being used on the bottling line for meaningful results. For example, filterability analyses made using nylon membranes cannot be relied upon if polyethersulfone membranes are being used at bottling. Also the membrane porosities need to be comparable between the test apparatus and the media that will be used during the bottling process.

Table 1. NTU and FI data for a 2010 Barossa Shiraz with different Becodisc depth filtration applied.

2010 Barossa Shiraz		
Becodisc grade	NTU	FI
Unfiltered	25	Not achievable
550	0.97	84
450	0.58	18
550 then 220	0.54	13
450 then 220	0.45	6.1

It is also possible to use the filterability measurement to test the efficiency of different grades of depth (pad) filtration media. As a comparative exercise, a wine was examined for both turbidity and filterability, and then subjected to various grades of depth filtration, on a lab scale, to determine the effectiveness of the depth filtration to remove wine particulates. The results are given in Table 1 and Figure 2.

The control wine prior to any filtration had a turbidity of 25 NTU and failed filterability testing. When passed through coarse grade pad material (Becopad 550), NTU was reduced to 0.97, which is within bottling specification, yet the FI was 84, which is far greater than the FI bottling threshold of 20. When the control wine was subjected to slightly tighter filtration (Becopad 450), turbidity dropped to 0.58 NTU and FI to 18, both of which indicate suitability for bottling. When a Becopad 220 was used to undertake a second filtration post the Becopad 550 filtration, turbidity dropped further to 0.54 NTU and FI to 13. Becopad 450 onto Becopad 220 yielded turbidity of 0.45 NTU and FI 6.1.

These results demonstrate the importance of the filterability measurement. If the wine had been put through coarse filtration in the cellar, it would have been able to meet the sterile membrane bottling specification of turbidity ≤1 NTU, but it would have caused a lower filtration rate due to fouling of the filtration media. This is due to the wine's filterability being well above the bottling FI threshold of 20. The likely result would be premature exhaustion of any depth medium in place, but also potentially blocking of the final membrane or membrane pre-filters. This would prove costly in terms of both filtration media and downtime on the bottling line. The implementation of a filterability protocol would have identified the poor filterability of this wine in time to avoid bottling difficulties.

Vinpac International, in collaboration with Blue H2O Filtration, has recently developed a filterability analysis protocol that is based on the filterability appartus manufactured by Tecnoelettric. This analysis will become a core component of Vinpac's laboratory activities, specifically where sterile filtration has been requested by the customer. This allows a measurement of filtration fouling ability to be determined, rather than relying on turbidity alone. This is important as some of the substances that can cause filtration fouling, such as glucans, are soluble in wine and do not contribute to a measurement of filterability based on turbidity. In developing this filterability protocol, some interesting comparisons between turbidity (NTU) and filterability (FI), as presented in Table 2, have come to light and warrant further discussion.

Wines 11-16 in Table 2 exhibit an interesting conundrum: turbidity is above the bottling specification, but filterability is acceptable. These wines would currently not be deemed acceptable for

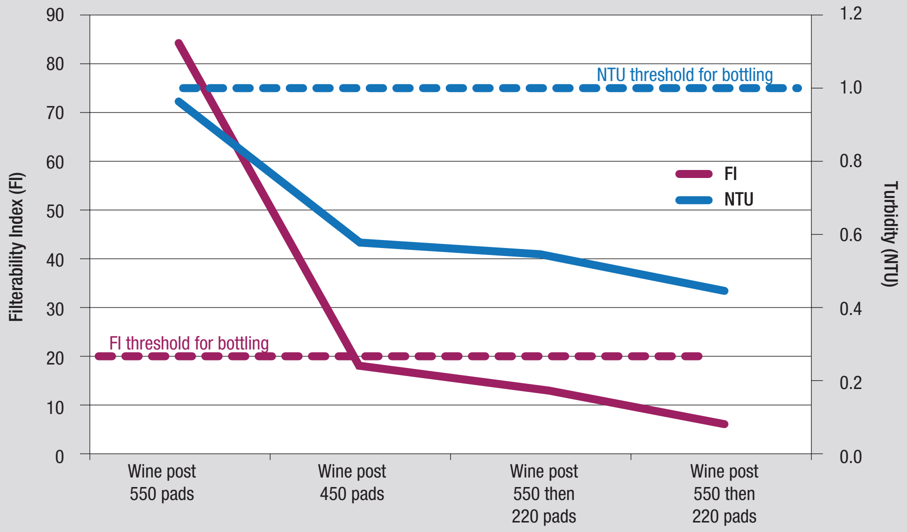

Figure 2: Wine NTU and FI for a 2010 Barossa Shiraz after filtering through different grades of pad.

Table 2. NTU and FI data for a series of wines analysed by Vinpac International.

	Vintage	Wine Type	Filterability (FI)	Turbidity (NTU)
1	2012	White	22	0.35
2	2011	Red	66	0.42
3	2011	Red	25	0.59
4	2011	White	25	0.62
5	2012	White	32	0.64
6	2011	White	68	0.71
7	2012	White	681	0.85
8	2011	White	24	0.88
9	2012	Red	Fail	0.89
10	2012	White	21	0.96
11	2011	Red	9	1.17
12	2012	Red	16	1.17
13	2011	Red	8	1.22
14	2010	Red	6	1.26
15	2012	White	7	1.32
16	2011	Red	15	1.49
17	2012	Red	Fail	1.45

Key

(purple)	Outside turbidity specification (>1 NTU)
(green)	Outside filterability specification (FI >20)
(blue)	Outside both turbidity and filterability specification

bottling even though they would cause no significant problems in regards to membrane fouling. Conversely wines 1-10 would have been deemed suitable for bottling as they have turbidity values of ≤1 NTU, but filterability is not acceptable. This is clearly shown by wine 7, where the turbidity is within specification, but the FI is extremely high. It could be expected that this wine would foul the membranes quite rapidly. Identifying and preventing wines like those marked in green in Table 2, from being bottled in their present state, provides benefits to both the bottling company and the producer. The primary reason why Vinpac International is implementing filterability measurement as part of its quality assurance programme is to ensure that the filtration operation runs smoothly during bottling, reducing the risk of product degradation.

Wine additive influence on FI

In addition to known indigenous fouling components that can be found in wines as a result of natural occurrences and standard winemaking practices, such as polysaccharides and polyphenols; certain exogenous additives are known to cause membrane fouling to greater or lesser extents. Examples of such additives include gum arabic, tannins, mannoproteins and, more recently, carboxymethylcellulose (CMC). CMC (Bowyer *et al.* 2010), which is a potassium bitartrate crystallisation inhibitor that was recently added to the Australian standard for wine production, can contribute to a reduction in the filterability of a wine. This is illustrated in Figure 3, where a highly filterable wine (Control FI = 0.7) was dosed with different rates of CMC, being 50ppm, 100ppm (the standard legal CMC dosage) and 300ppm. The wine's filterability index increased due to the increased colloidal loading from the CMC additions. Therefore, if a wine has a borderline filterability result (eg FI = 19) adding CMC to provide cold stability protection may well generate filtration problems, by pushing that wine over the bottling FI threshold of 20. This is more likely to be the situation if the manufacturer's recommendations for the use of their CMC product have not been followed. That is, manufacturers recommend that additions of CMC must be made to a heat stable wine at a minimum of 48 hours through to 5 days prior to final filtration and bottling. If this practice is not adhered to, CMC could be removed during filtration causing clogging of the filtration membranes. This raises another question for the winemaker: how much of the added CMC remains and will this be enough to stabilise the tartrate in the wine? Based on this information, it would be important to assess the CMC product being used, with regard to its behaviour on wine filterability as measured by FI, prior to making an addition to the wine.

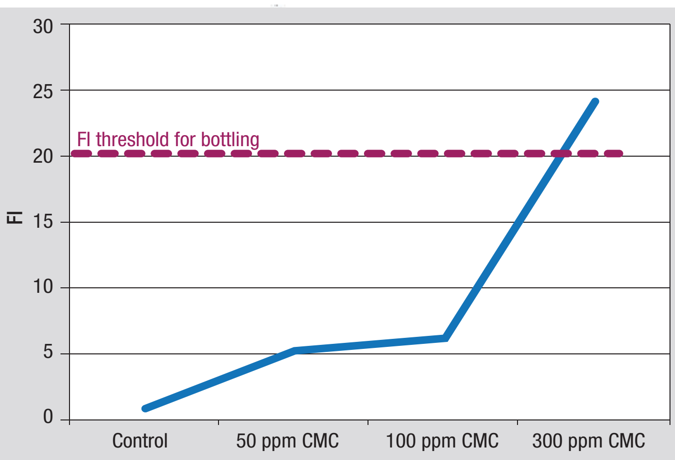

Figure 3. Wine FI as a function of CMC content in a highly-filterable wine.

Summary

Turbidity, as a measure of wine filterability, does not provide a complete determination of how the wine will interact with the filtration media during the bottling process. A far superior method of determining wine filterability is the measurement of a wine filterability index. This must be done using membrane discs of identical material to that which the wine will be subjected to on the bottling line. This technique can also be used to optimise cellar filtration choices, to reduce a wine below the filterability threshold of FI <20. Filterability measurement is also a useful technique to evaluate the filterability impact of wine additives, such as gum arabic, tannin, mannoprotein and CMC. In implementing a wine filterability measurement rather than relying on turbidity as an indicator of a wine's filterability, Vinpac International is ensuring that filtration processes during bottling will operate more efficiently, requiring less downtime due to filtration blockage.

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