Turbidity versus filterability as a means of evaluating wine impact on filtration media

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Introduction

Turbidity is used as a means of assessing the particulate level in a wine (visual clarity), and from this its suitability for bottling is determined. 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 sterile bottling is < 1 nephelometric turbidity unit (NTU): If a wine has an NTU < 1, it is deemed suitable for sterile bottling in terms of how it will present in the bottle and its likelihood of fouling filtration media, specifically "sterile" membranes and membrane pre-filters. If the pre-bottling wine NTU > 1, and the wine is to be "sterile" filled, then it is recommended that the wine receives extra prior filtration. This may be depth or cross flow 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.

NTU

A nephelometer (turbidity meter) measures the extent to which 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 it also has some severe 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 second wine may have larger suspended particles, which can create a film on the filtration media that does not totally block the media. Different suspended solids also have differing levels of reflectivity (e.g. yeast and tartrate micro-crystals).

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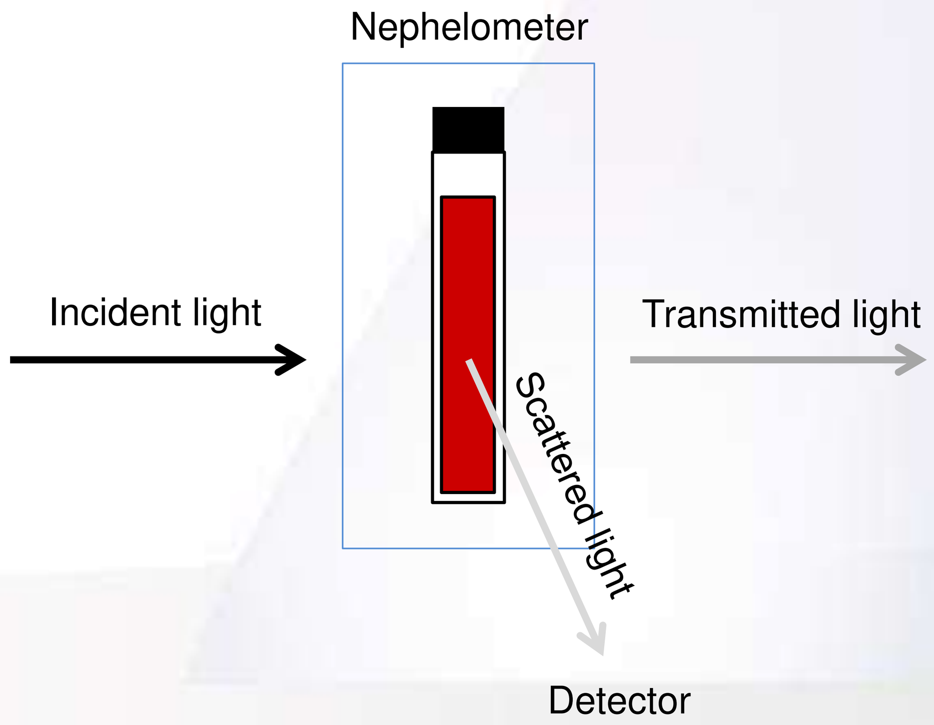
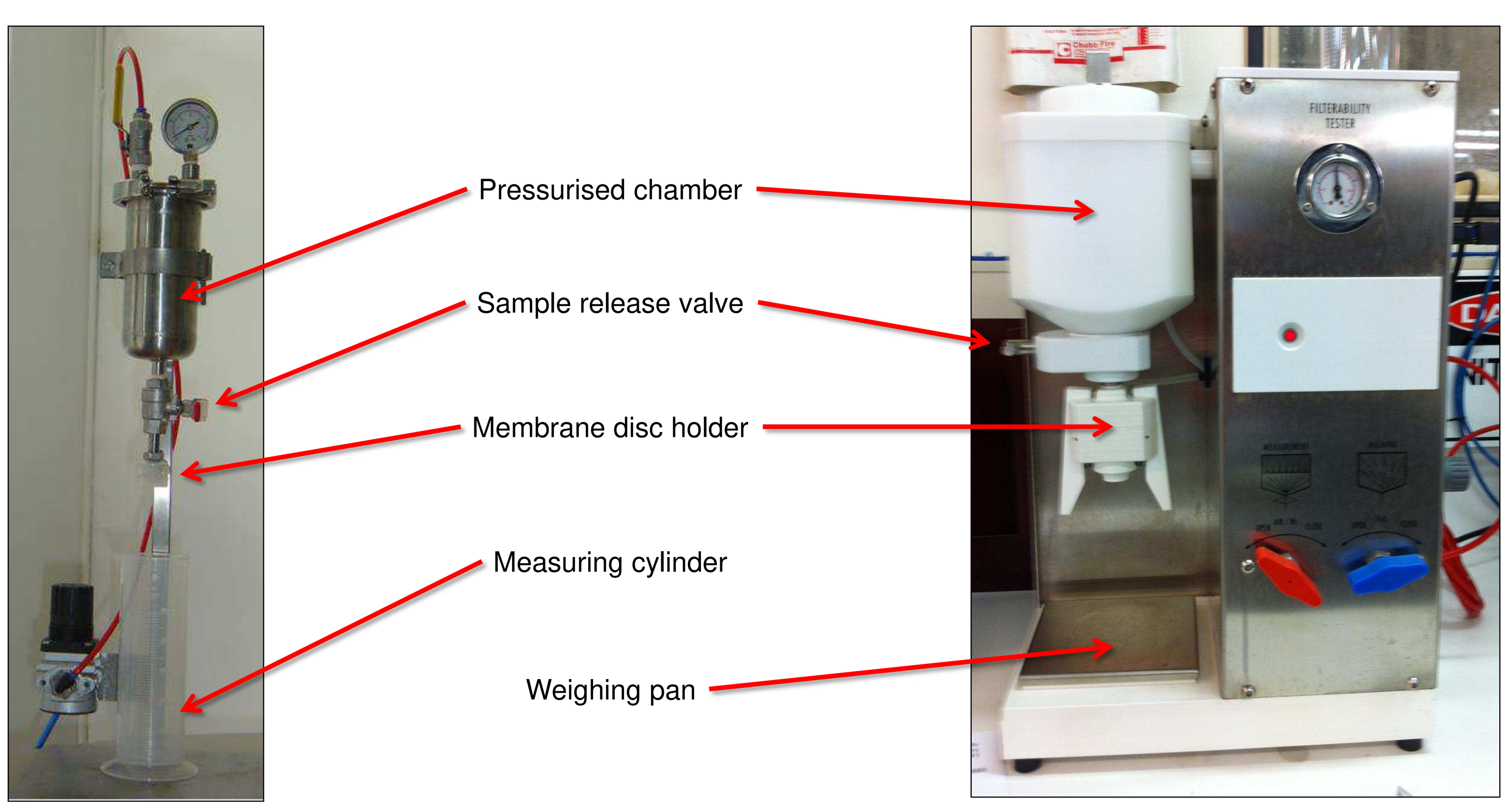


There is surprisingly little in the way of published research literature that covers the relationship between filtration fouling and turbidity in wine. Roger Boulton published an article in 2001 in which he states specifically that "fouling of wines on membrane filters is not related to their clarity" (Alarcon-Mendez & 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 (de la Garza & Boulton, 1984). Czekaj, López & Güell (2000) indicate that membrane fouling is mainly caused by colloidal components in 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. Perhaps the most relevant statement comes from a 2003 paper by Vernhet, Cartalade, & 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. For a recent discussion of this topic, see Bowyer, Edwards and Eyre (2012).

El Rayess et al (2011) state that macromolecular compounds such as polysaccharides, phenolics and proteins are the 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, perform lees ageing or when *Botrytis* infection is evident, as these practices may contribute to higher levels of polysaccharides 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 vastly increased risk of glucan membrane fouling. This knowledge made it possible to predict that red wines from certain areas in 2011 would very likely present filtration difficulties. Many filtration problems were circumvented through preventative filterability analysis.

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 0.45 µm in Australia), it is important to use a test that demonstrates how the wine will impact 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 many contract bottling facilities. The equipment required is simple and inexpensive (see below left), yet this process can be 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. A good compromise is semi-automated equipment, such as the filterability tester (below right). This combines the robustness of the manual method with the ease of computerised monitoring of the 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.



A manual filterability index unit

A semi-automated filterability index unit

Measuring FI

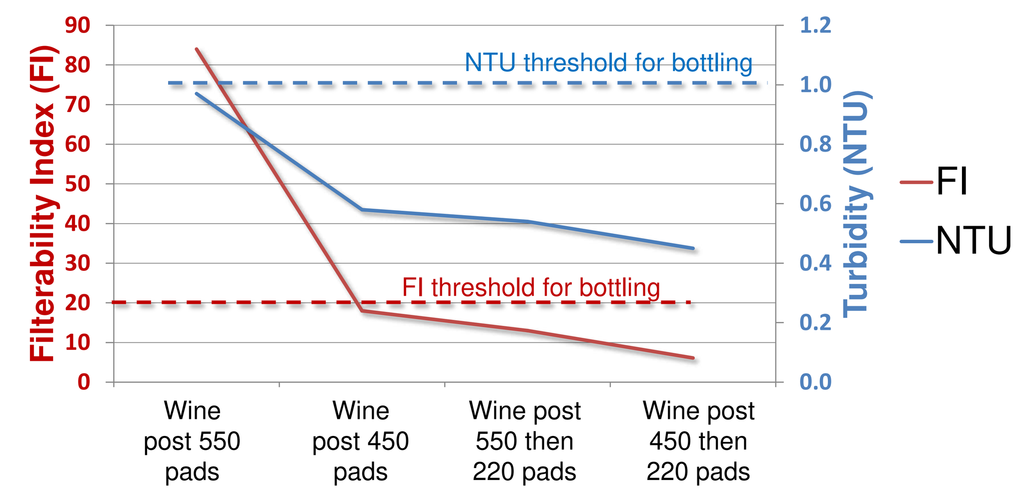
Filterability index (FI) is tested by passing wine through a 0.45 µm membrane disc, at constant pressure (2 bar), and timing how long the membrane takes to filter two volumes, typically 200 mL (T_{200}) and 400 mL (T_{400}) in seconds. Thus, FI = $T_{400} - 2 \times T_{200}$. If the wine is perfect in terms of filterability the ratio of T_{400} to T_{200} will be 2:1, and so FI = 0 when the formula is applied. 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. By setting some FI thresholds determined by extensive testing, 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. If 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 PES membranes are being used at bottling. Also, the membrane porosities need to be equivalent between the test apparatus and the media that will be used during the bottling process.

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 below.

2010 Barossa Shiraz

Becopad grade	NTU	FI
Control (unfiltered)	25	> 500
550	0.97	84
450	0.58	18
550 then 220	0.54	13
450 then 220	0.45	6.1

This wine would be passed by NTU but fails by FI, and would cause membrane fouling



Is there a correlation between NTU and FI?

From the chart above it would appear that there may be a correlation between NTU and FI, at least below 0.6 NTU. To test this postulate, a database was created, a selection of which appears below:

Sample #	Vintage	Type	NTU	NTU pass?	FI	FI pass?
1	2012	Sauvignon blanc	0.35	Y	21.6	N
2	2011	Shiraz	0.38	Y	29.1	N
3	2008	Shiraz	0.44	Y	69.6	N
4	2012	Sauvignon blanc	0.63	Y	82.3	N
5	2012	Rosé	0.64	Y	FAIL	N
6	2010	Botrytis	0.66	Y	623	N
7	2012	Pinot grigio	0.69	Y	450	N
8	2011	Sauvignon blanc	0.71	Y	FAIL	N
9	2013	Chenin blanc	0.77	Y	174	N
10	2010	Cabernet	0.79	Y	898	N
11	2012	Riesling	0.80	Y	625	N
12	2012	Cabernet	0.84	Y	691	N
13	2010	Shiraz	0.84	Y	FAIL	N
14	2012	Sauvignon blanc	0.85	Y	681	N
15	2012	Chardonnay	0.86	Y	1720	N
16	2013	Sauvignon blanc	1.00	Borderline	1420	N
17	2011	Shiraz	1.04	N	8.7	Y
18	2012	White blend	1.07	N	15.4	Y
19	2011	Shiraz/Grenache	1.17	N	8.7	Y
20	2012	Shiraz	1.17	N	16.0	Y
21	2011	Shiraz	1.20	N	5.7	Y
22	2011	Shiraz	1.22	N	7.5	Y
23	2010	Shiraz	1.26	N	6.4	Y
24	2011	Cabernet	1.30	N	9.0	Y
25	2012	Semillon/SB	1.32	N	6.9	Y
26	2009	Cabernet	1.47	N	17.8	Y
27	2013	Riesling	1.76	N	6.2	Y
28	2013	Pinot Gris	2.04	N	9.0	Y
29	2010	CSM	6.11	N	8.1	Y
30	2011	Pinot noir	15.5	N	11.8	Y

FAIL = incomplete test due to membrane blockage. A value of 1000 was used in calculating the coefficient of determination.

Wines that pass by NTU but will foul a membrane. These wines would have been sent to bottle but would have fouled the media causing increased cost to both bottler and winemaker.

$r^2 = 0.045$

Wines that fail by NTU but will not strongly foul a membrane. These wines would incur unnecessary cost to the winemaker through extra filtration that is not needed.

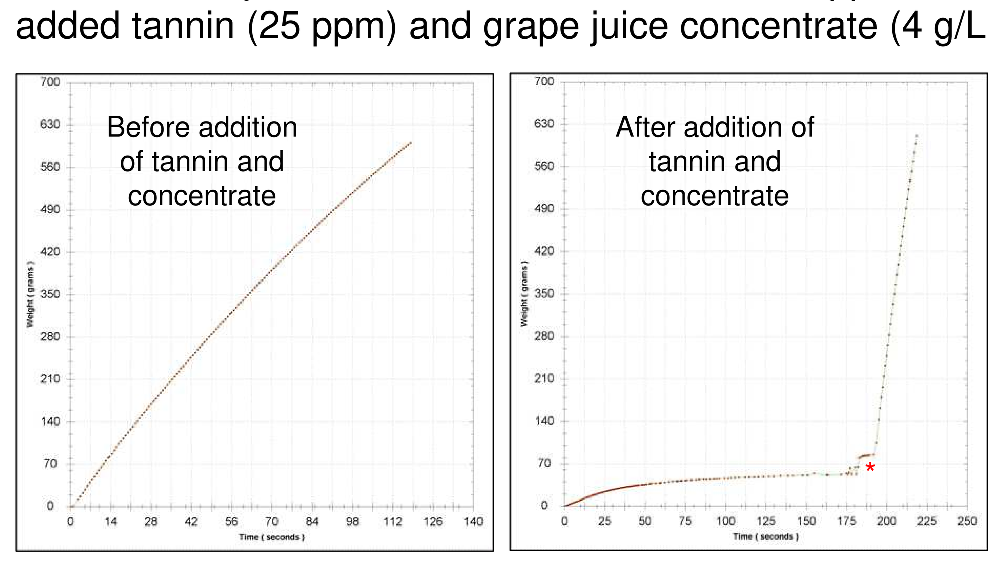
A correlation coefficient of **r^2 =0.045** was calculated from these data, which indicates no correlation at all. Using only NTU as a guide, wines 1-16 would be approved for bottling but would cause fouling problems to various degrees. For example, wine 1 might simply cause premature blockage of a membrane, but wine 13 would cause serious problems. Conversely, wines 17-30 would not be approved for bottling by NTU alone, yet their filterability is quite good. This is most likely due to the presence of more reflective particulates like tartrate microcrystals that cause stronger light scattering, yet such particulates do not present a serious filtration problem. Clearly, there is no correlation between NTU and FI, nor is there any strong correlation with vintage or varietal.

Can wine additives influence FI?

The filterability measures below are for a wine approved for bottling (NTU = 0.56, FI = 7.6) to which was added tannin (25 ppm) and grape juice concentrate (4 g/L residual sugar). The wine's NTU rose to 1.1 but it failed filterability (FI > 1000). Additives that can increase FI are:

- Tannins
- Yeast extracts
- Gum Arabic
- Carboxymethylcellulose (CMC)
- Grape juice concentrate

Some increases may be temporary and some may be permanent, so FI testing is advised when using these additives.



* *Measurement arrested due to membrane blockage*

Conclusion

There is no correlation between NTU and FI. Reliance upon NTU alone will eventually lead to increased membrane fouling during sterile filtration and even unnecessary filtration. The use of both measures can help avoid filtration difficulties and reduce costs for both bottler and winemaker. FI is a simple measurement to perform which should become standard practice in the wine industry.

References

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