Filterability measurement for Process Optimisation in Wine Packaging

Dr Paul Bowyer
paul@blueh2o.com.au
www.bhftechnologies.com.au
Greg Edwards (Vinpac International)

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AUS & NZ

WINEENG 2018

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Napier

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Napier

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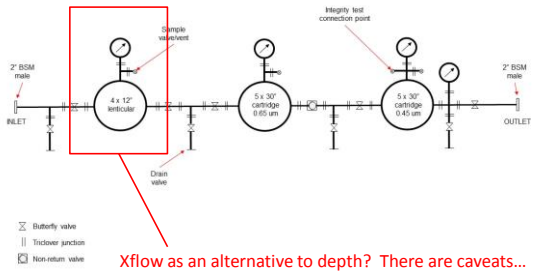
Typical bottling filtration setup



Xflow as an alternative to depth? There are caveats…

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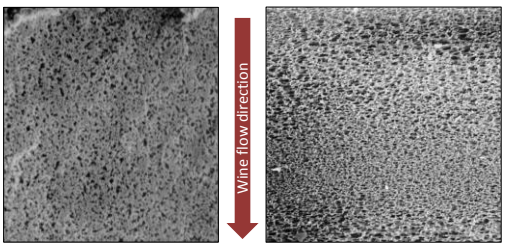
Membranes

- Not a sheet with defined holes in it – it is cast polymer



Nylon, symmetrical, 0.45 μm, profile

PES, asymmetric, 0.45 μm, profile

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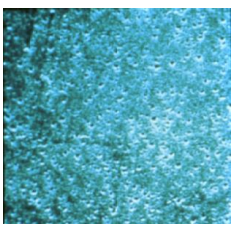
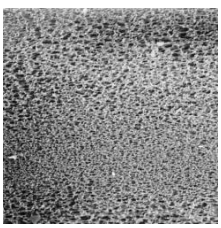
Membranes

- Not a sheet with defined holes in it – it is cast polymer



PES, asymmetric, 0.45 μm, top view



PES, asymmetric, 0.45 μm, profile

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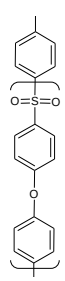
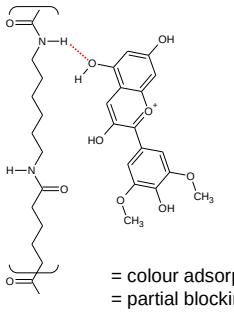
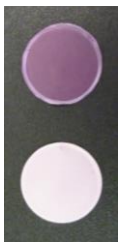
Membranes







= colour adsorption
= partial blocking

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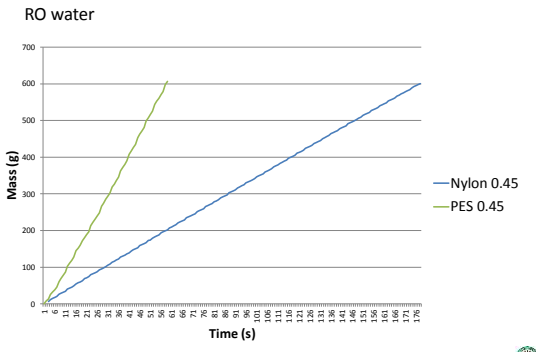
Membranes



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NTU & Filterability index (FI)

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NTU

- Wine < 1 NTU = "OK" = "this wine is clean and it is suitable to sterile filter". Really?
- A measure of light scattering orthogonal to the incident beam.
- Gives an indication of suspended particulates.
- Does not account for reflectance of particulate (e.g. tartrate crystals reflect more than microorganisms).
- Provides no indication of dissolved material. What about the things we can't see?
- NTU does *not* indicate colloidal status of wine (e.g. glucans, mannoproteins, tannins, protein, gum Arabic, CMC).

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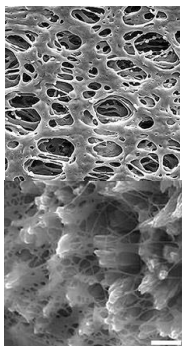
Glucans

Clean PES membrane

PES membrane fouled with glucans from *Botrytis*



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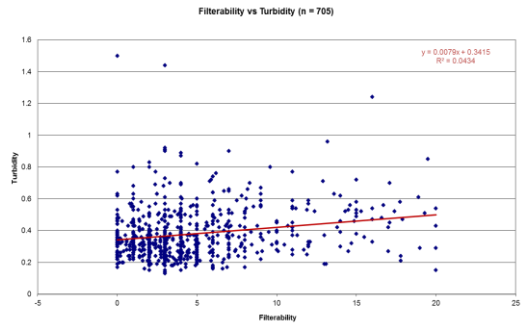
NTU vs. FI: Correlation? No.



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- See article, The Australian and New Zealand Grapegrower & Winemaker: Paul Bowyer[1], Greg Edwards[2] and Amelia Eyre[2] (2012) NTU vs. wine filterability index – what does it mean for you? *The Australian and New Zealand Grapegrower & Winemaker*, Issue 585 (October), 76-80.

[1] Blue H_2O Filtration
[2] Vinpac International

winemaking

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

Paul Bowyer, Greg Edwards and Amelia Eyre

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

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

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-Morales 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

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Filterability measurement

- Many winemakers have little awareness of FI.
- Several methods in use to measure "FI".
- All require a technician to monitor the analysis from start to finish – a big disadvantage, especially if the wine sample being run is not greatly filterable (ie high FI = long analysis).
- Some labs use 0.45 um, some 0.65 um, regardless of final wine membranes (porosity & medium) being used in bottling.
- Often cellulose acetate (lab cell counting) membranes are used to measure FI. Cellulose nitrate often used in NZ.
- Vast performance differences between membrane types and therefore data obtained.
- FI measurement is not a replication of bottling filtration. It is a method for flagging potentially problematic wines. In reality bottling depth and 0.65 prefiltration *should* be doing little work.

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Filterability measurement

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Usual NZ filterability method

Measures **volume** for given times. Rankine method.

1. 25 mm 0.45 um cellulose nitrate membrane (but bottling using 0.45 um PES)
2. Sample @ 2 bar
3. Record volumes for 30-90 s (T1) and 120-180s (T2).
4. **FI = T2/T1**
5. FI ≥ 1.20 = fail.

Note:

- The division dilutes the data. Small numbers are problematic.
- Difficult to interpret results – is there much value in such a narrow data range?
- Is it valid to call 1.19 a pass but 1.20 a fail? A narrow line.
- Is the method reproducible and accurate?
- Does not accommodate modern membranes with high flux.

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BHF filterability measurement

Measures **time** for a given volume.

- 25 mm 0.45 um disc identical to bottling membrane.
- Push wine through at 2 bar constant pressure.
- Record time to pass 200 mL & 400 mL.
- Index:

$$FI = T_{400} - 2T_{200}$$

- **Bottling: FI ≤ 20**

Note:

- Useful as a guide to bottling performance.
- Reflects wine colloidal and particulate loading.
- Reliable data + works with any membrane test disc.
- Can never run out of wine.
- Results must be evaluated in conjunction with other data – e.g. wine volume, condition of filters etc.

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So how do we make FI measurement easy enough to be routine?

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Semi-automated BHF filterability measurement

- Partial automation improves user-friendliness
- Easy to use & clean.
- Fast between samples.
- Does not require monitoring once started.
- Live data plotting.
- Can measure FI at different temperatures by chilling sample.
- Same method as manual, so data are transposable.
- Robust unit with a small footprint (~A4).

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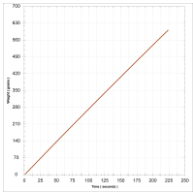
BHF Technologies
Dr Paul Bowyer
paul@blueh2o.com.au
0401 446 119
bhftechnologies.com.au

TYPE : Step Rd 802 Boronia
STOCK :
BASIN :
MEASURE DATE : 6/17/2014 1:33:55 PM

	WEIGHT (gr)	TIME (sec)
START POINT	10	4.540
POINT NR. 1	200	73.446
POINT NR. 2	400	147.952
POINT NR. 3	600	223.768

FILTERABILITY INDEX	IF = T2 – 2 * T1	5.599
MODIFIED FILT.INDEX	IFm = (T3 – T1) – 2 * (T2 – T1)	1.310
MAX VOLUME	Vmax = 400 + ((400 * T1) / IF)	522.399



NOTES :
NTU 0.52

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FI alone can be misleading! FI vs FI_m

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FI vs FI_m

$$FI = T_{400} - 2T_{200}$$

$$FI_m = (T_{600} - T_{200}) - 2(T_{400} - T_{200})$$

- FI_m covers a broader measurement range (to 600 g) and so should be more accurate.
- Could better illustrate membrane loading effects.
- Both FI and FI_m are useful when analysing a wine, along with line shape and the test duration (flux).
- Both FI and FI_m can be measured using a standard manual apparatus. No extra equipment required!

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Some filtration suppliers contend that the membrane disc type makes no difference to the FI analysis result.

Let's examine that.

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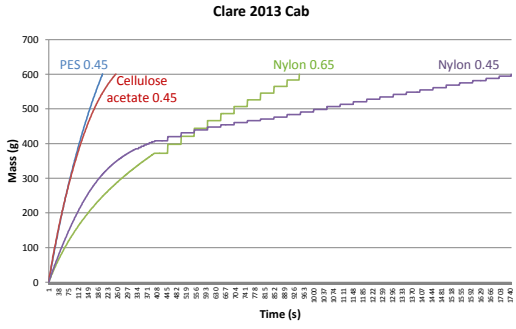
Membrane comparison 0.45 um



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FI values

2013 Clare Cabernet Sauvignon

Disc type	FI	FIm
PES 0.45 um	17	23
Cellulose ester 0.45 um	23	61
Nylon 0.65 um (manufacturer A)	162	186
Nylon 0.45 um (manufacturer B)	161	1100

In this example:
- Compare FI and FIm values…
- PES membranes - will work well
- Nylon membranes - not so much…

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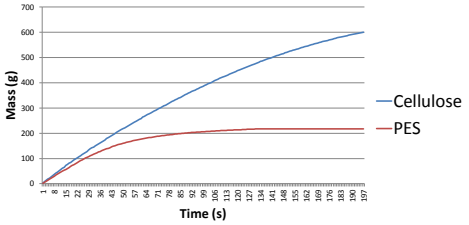
Membrane comparison 0.45 um

2014 Merlot NTU 0.5



	FI	FIm
PES 0.45 um	Fail	Fail
Cellulose 0.45 um	15	36

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Concentrate influence on FI

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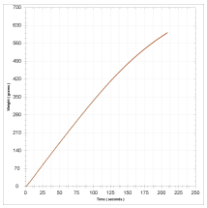
Concentrate

- 2014 Riesling tank sample



FI	FIm
8.1	24

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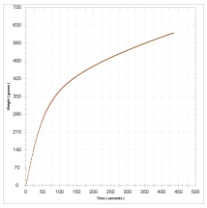
Concentrate

- 2014 Riesling + 5 g/L concentrate



FI	FIm
53	228

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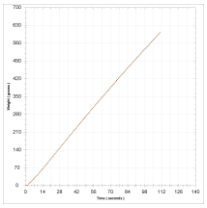
Concentrate

- 2014 Riesling + 5 g/L conc. post 0.1-0.3 um depth (Beco 170)



FI	FIm
1.9	1.6

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Xflow influence on FI

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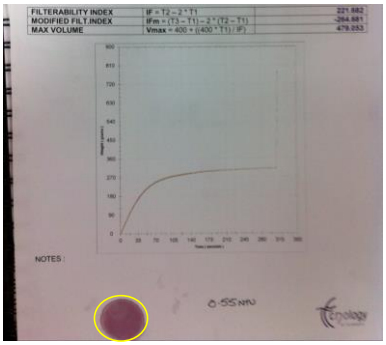
Xflow influence on FI



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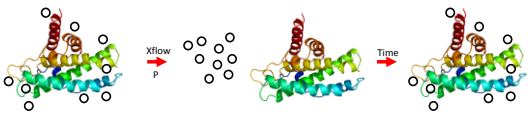
Xflow influence on FI

Colloids
- Macromolecular (eg mannoprotein, CMC, gum Arabic)
- Association (eg protein-tannin)



High FI Low FI High FI

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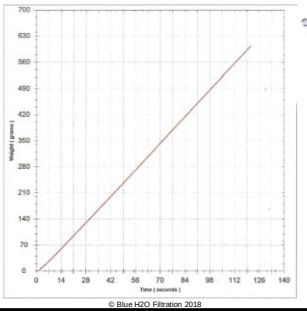
Believe it or not, what BHF wants to see is:

FILTERABILITY INDEX	IF = T2 – 2 * T1	0.000
MODIFIED FILT.INDEX	IFm = (T3 – T1) – 2 * (T2 – T1)	0.686
MAX VOLUME	Vmax = 400 + ((400 * T1) / IF)	Infinity



And beyond !!

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2016 winners - packaging

WINE INDUSTRY IMPACT AWARDS

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Thank you.

Paul Bowyer (BHF)
Greg Edwards (Vinpac International)

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WEA, 17-18/10/2018

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