

Whatman[™] filter papers for use in the beer industry

Quality control (QC) methods used by the brewing industry are published by a number of national and international bodies such as the European Brewery Convention (EBC), Mitteleuropäische Brautechnische Analysenkommission (MEBAK), and the American Society of Brewing Chemists (ASBC). Many of these QC tests involve a filtration step for sample preparation. In this study, three grades of Whatman filter paper (Grades 2V, 597½, 2555½) were evaluated in analyses representing different steps in the brewing process. These tests, from ASBC methods, were 1) removal of turbidity from wort, 2) removal of CO₂ from finished beer, and 3) removal of yeast cells after fermentation. The data presented here demonstrate that all three filter grades are suitable for use in the filtration steps that these methods require.

Introduction

Growing competitiveness in the global market for beer has led brewers and standard-setting agencies for regional varietals to increase focus on product consistency for characteristics such as color, alcoholic strength, flavor, and clarity.

Several national and international bodies, notably the EBC, MEBAK, and ASBC, publish analytical methods to guide beer quality testing. Many of these require filtration to prepare the sample for testing. Some methods rely on the traditional concept of filtration, wherein the filter's sole purpose is to remove particulate matter from a liquid sample prior to analysis of the clarified liquid (i.e., the filtrate). Other methods require testing of the retained solids (i.e., the retentate). See Table 1 for examples of these tests.

This application note shows how Whatman filters of three grades performed in supporting three ASBC methods of importance to the overall QC process. These tests were 1) removal of turbidity from wort (Malt-4), 2) removal of CO₂ from finished beer for quality control purposes (Beer-1,D), and 3) removal of yeast cells after fermentation (Beer-8).

Table 1. MEBAK analytical methods that use filtration. Numbers refer to the designation within the MEBAK handbook $^{\rm 1}$

1.4.3.1 Soluble extract in wet spent grains obtained by pressing (rapid method)
1.4.3.2 Soluble extract in wet and dry spent grains obtained by rinsing (EBC)
1.4.4.2 Available residual extract
1.4.5 lodine value of brewery spent grains
1.6.1 Solids in wort (Labor veritas method)
1.6.2 Solids or trub material (Field method)
1.6.3 Cold trub
2.6.2 Coaguble nitrogen (thermal coagulation of protein)
2.6.3.1 Nitrogen fractionation (precipitation with magnesium sulfate)
2.6.3.2 Nitrogen fractionation (precipitation with phosphomolybdic acid)
2.8.1 Limit of attenuation in wort (fermentation tube method)
2.8.2 Limit of attenuation in wort (reference method – EBC)
2.8.3 Limit of attenuation in wort (rapid method – EBC)
2.9.1 Degassing a sample (EBC)
2.10.3.2.1 Total glucose – hydrolysis method
2.12.2 Spectrophotometric color (EBC)
2.14.2.2 Alcohol chill haze test, CHAPON (cold sensitivity)
2.16.3 Tannoids
2.17.3 Determination of hop bitter substances in wort and beer (EBC)
2.20.1 Membrane filterability test of beer
2.21.3.3 4-vinyl guaiacol and 4-vinyl phenol detection
2.21.8.3 Detection of SO_2 with continuous flow rate
2.22.1 Chloride, sulfate, nitrate and phosphate in beer (EBC)
2.22.5 Sulfate ions

Wort filtration

The quality of a wort batch is dependent on the quality of the malt used to produce it. Measurement of wort filtration speed and turbidity allows a brewer to qualify whether the malt used to produce it is leading to a desirable end product and to quantify a tolerance range for quality control of end product consistency. A slower filtration speed reflects a lower wort solubility. Measuring the filtration speed also helps the brewer understand the malt's contribution to fermentable extract, pH, color, viscosity, and nitrogen content². A wort sample with low turbidity is required to support later photometric testing. Gravity filtration using an appropriate filter paper supports all of these objectives.

CO₂ removal prior to further testing

During fermentation, CO_2 is produced and dissolves into the solution. When a QC lab prepares a sample of this solution for analysis, this dissolved CO_2 may lead to inaccurate results in tests such as total acid determination. Therefore, the CO_2 content needs to be minimized prior to testing. One method for achieving this is to pass the beer sample through an appropriate paper filter using gravity filtration.

Removal of yeast cells after fermentation

Measuring the total acidity of the final brewed product requires that the remaining yeast cells are removed. This simple separation of particulate matter from liquid sample can be achieved through gravity filtration.

Methods

In this study Whatman Grades 2V, 597½, and 2555½ from GE Healthcare's Life Sciences business were evaluated for their suitability in the three tests described below. All filters were 320 mm in diameter and prepleated with 16 pleats (Fig 1).



Fig 1. A prepleated Whatman filter paper.

All tests were performed according to the ASBC methods presented in Table 2. All testing was performed by the Biotechnology School at Jiangnan University, No 1800 Lihu Avenue, Wuxi, Jiangsu, 214122, China.

Table 2. Study design

Whatman filter	Product code	Lot number	Tests (ASBC Method)	
Grade 2V	1202-320	G8605164	Wort filtration (Malt-4)	
Grade 597½	10311853	G7471143	CO ₂ removal (Beer-1,D) Yeast cell removal	
Grade 2555½	10313953	G5137158	(Beer-8)	

Removal of turbidity from wort

Two batches of malt were prepared to support testing of the filtration step for method ASBC Malt-4. The malt was prefiltered prior to further testing in order to isolate the wort. Spent grain was removed by prefiltering the malt using a coarse filter bag. The wort in the filtrate was adjusted to a turbidity level of 10 prior to measuring the base turbidity. A 150 ml sample of wort was filtered using gravity through Whatman filter paper Grade 2V, 597½, or 2555½. The turbidity pre- and post-filtration was measured and the percent reduction calculated. This process was repeated for a total of three samples. The filtration times were also recorded.

CO, removal from bottled beer

The amount of CO_2 in beer is expressed as mg/ml and is derived from the following formula:

$$\frac{\mathsf{C} \times (\mathsf{V}\text{-}\mathsf{V}_0) \times 44}{10} \times \frac{\mathsf{V}_1 + 1}{\mathsf{V}_1}$$

Where:

C = concentration (M) of HCl standard

 V_0 = volume of HCl (ml) required to adjust pH in a water blank to 3.9 V_1 = sample volume (ml)

V = volume of HCl (ml) required to adjust pH of the sample to 3.9

Per ASBC method Beer-1,D a 4 ml sample of beer chilled to 4°C was filtered using gravity through Whatman Grade 2V, 597¹/₂, or 2555¹/₂ and transferred to a conical flask. NaOH (1 ml of a 10 M stock) was added to the beer and mixed thoroughly. A 10 ml sample of this mixture was transferred to a fresh beaker and 20 ml of distilled water added. The mixture was then titrated against 0.5 M HCl, with the volume required to reduce the pH to 3.9 recorded. The measurement was repeated with 50 ml of unfiltered beer and then with 50 ml of water (as a blank control).

The amount of CO_2 in unfiltered and filtered beer samples was measured, and the amount and percentage of CO_2 removed was calculated. This process was repeated for a total of three samples.

Removal of yeast cells after fermentation

A typical fermentation broth containing yeast cells suspended in solution was prepared. In order to test such a broth for acidity per ASBC method Beer-8 such a broth would first have yeast cell content removed. The initial number of suspended yeast cells was estimated using a cell counting chamber. Aliquots of 150 ml of this suspension were then filtered by gravity through one of the three filter paper grades. The filtrate was subjected to serial dilution. The resulting samples were plated on YPD agar plates to determine the number of viable yeast cells not retained by the filter. The difference between the two counts was used to determine the efficiency of filtration. This process was repeated for a total of three samples.

Results and discussion

Removal of turbidity from wort

The results for the two prefiltered wort batches are provided in Table 3.

Table 3. Turbidity reduction for wort batches. Results are the average of triplicate measurements

Whatman filter	Initial turbidity		Turbidity after filtration		Turbidity reduction (%)	
grade	Batch 1	Batch 2	Batch 1	Batch 2	Batch 1	Batch 2
2V	10.6	30.1	6.54	6.39	38.3	78.8
597½			8.02	7.25	24.3	75.9
2555½			7.24	8.96	31.7	70.2

Although Grade 2V was superior for turbidity removal, filtration time was much longer than with the other two papers (up to 413 s for a 120 ml sample compared to 83 s and 52 s for Grades 597½ and 2555½, respectively). The 597½ grade paper showed better performance characteristics with higher turbidity starting material, and Grade 2555½ performed better when initial turbidity was low. Filter selection ultimately depends on the time allotted to perform the test and the required level of sample clarity. Table 3 and the filtration times should allow for an informed choice with respect to these parameters.

CO, removal from bottled beer

Results for CO_2 removal are given in Table 4. The Grade 2V paper showed the highest efficiency in removing CO_2 from the beer, indicating its particular suitability for this analysis.

Table 4. CO_2 removal from bottled beer by Whatman paper type. Results are the average of triplicate measurements

Filter	CO ₂ (m	g/ml)	CO ₂ removed		
grade	Unfiltered	Filtered	(mg/ml)	% removal	
2V		1.06	3.69	77.68	
597½	4.75	1.94	2.81	59.16	
2555½		2.07	2.68	56.42	

Removal of yeast cells after fermentation

Table 5 provides results for yeast cell removal from fermentation broth. All three papers performed well in the test, removing greater than 99.5% of suspended yeast cells from the broth. Whatman Grade 2V filter paper achieved 99.99% removal.

 Table 5. Yeast cell removal from fermented beer. Results are the average of triplicate measurements

	Yeast cells (
Filter grade	Before filtration	After filtration	% removal
2V		1.9×10^{3}	99.991
597½	2×10^{7}	4.3×10^{4}	99.783
2555½		2.5×10^{4}	99.874

Conclusions

GE produces a wide range of Whatman filter papers suitable for use in the beer industry. A sample of this range, Whatman Grades 2V, 597½, and 2555½, were tested in support of three representative quality control procedures from ASBC. All three grades of paper performed well in removing turbidity, CO_2 , and yeast cells. Whatman Grade 2V in particular stands out as providing the highest rate of turbidity reduction and CO_2 and yeast cell removal from samples. However, use of Grade 2V comes at a cost of a longer time to filter. The data presented in this application note will be useful to brewers deciding what filtration papers to use given their resource, time, and quality constraints.

References

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- Kühbeck *et al.* Influence of Lauter turbidity on wort composition, fermentation performance and beer quality – a review. *J Inst. Brew.* **112**, 215–221 (2006).

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