Gusmer Enterprises, Inc.

Technical Service Report

4/15/15

Re: TSR# 15-035 Boston Sake Fining & Filtration Study

Bench scale fining & filtration studies were completed to determine the best method of achieving either visual clarity or sterility of a sample from Boston Sake. Results suggest that at a minimum the customer should chill & decant the sake prior to filtration and filter life can be improved further by fining with 10# /1000gal of Vitiben bentonite.

If visual clarity is desired, 10 CSF-XC 40cm filter sheets are recommended to clarify decanted sake while less than 4 CSF-XC 40cm filter sheets are recommended to clarify fined & decanted sake. Following CSF-XC filtration, the sample appears visually clear but is not suitable for membrane filtration.

If sterile membrane filtration is desired, 20 CSF-SF 40cm filter sheets are recommended to clarify decanted sake while only 7 CSF-SF 40cm filter sheets are recommended to clarify fined & decanted sake. Following CSF-SF filtration, clogging index results suggest the sake is ready for efficient 0.45µ membrane filtration.

OBJECTIVE

The purpose of this study is to provide recommended fining/filtration options for clarifying sake to achieve visual clarity or sterility.

PROCEDURES

1 gallon of unclarified sake was submitted by Boston Sake for bench scale fining and filtration trials. The customer currently chills the sake to 2-4^{o}C and is interested in filtering it through either a 5µ nominal filter for visual clarity or a 0.45µ membrane for sterility using their current 40cm filter press.

Unclarified sake was filtered through CSF-XC (5µ nominal) or CSF-SF (0.45µ nominal) in a 2" Posiseal housing at 28gal/hr/ft^2 or 8gal/hr/ft^2 respectively based on manufacturer recommended flow rates. Pressure was monitored throughout the filtration and testing was terminated at 21psid based on pump limitations and/or recommended maximum pressure. These filtrations were repeated using unclarified sake that had been chilled at 2^{o}C for 24hrs and decanted to remove precipitate prior to filtration. Results of these filtrations are displayed in Figures 1-2 and summarized in Table 2.

Fining trials were conducted to determine if fining agents could further clarify the sake before filtration. 200mL of unclarified sake was dosed with either silica hydrogel (Britesorb A100) or Bentonite (Vitiben) and chilled at 2^{o}C for 24hrs before decanting to remove precipitate. Silica hydrogel was dosed at 400, 600 & 800ppm and Bentonite was dosed at 5, 7 & 10lb/1000gal based on manufacturer recommendations & past sake customer experience. The turbidity of decanted samples (including an un-fined control) was measured and displayed in Table 1.

10lb/1000gal bentonite fining was identified as the sample providing the best sake clarity, so a larger (2L) sample of sake was fined, chilled and decanted at this dose rate in preparation for filtration. The sample was filtered through CSF-XC or CSF-SF in a 2" Posiseal housing as previously described, with results displayed in Figure 3 & summarized in Table 2.

To determine if filtration through CSF-XC or CSF-SF adequately prepared the sake for membrane clarification, a clogging index test was conducted to determine membrane filterability. Fined sake pre-filtered through either CSF-XC or CSF-SF was passed through a 0.45µ membrane using 2 bar pressure while recording the time it takes to filter the first 200mL and 400mL of fluid. The clogging index value is calculated using the formula [T_{400} (min) – 2 T_{200} (min)] X 100. A value of 0-12 indicates good filterability; 12-25 provides acceptable filterability, and a value greater than 25 displays poor filterability. Results are included in Table 3.

RESULTS AND DATA

Unclarified sake was filtered through CSF-XC & CSF-SF as received or chilled and decanted prior to filtration through CSF-XC & CSF-SF. Filtration results following each preparation method are displayed in Figures 1-2.

Figure 1: Unclarified Sake Filtration

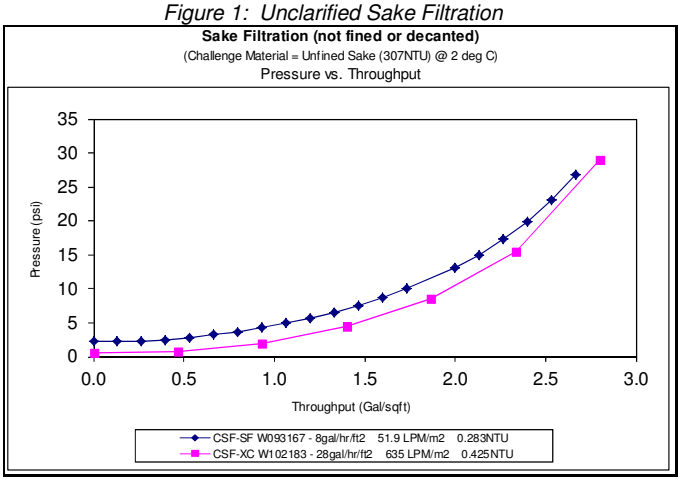
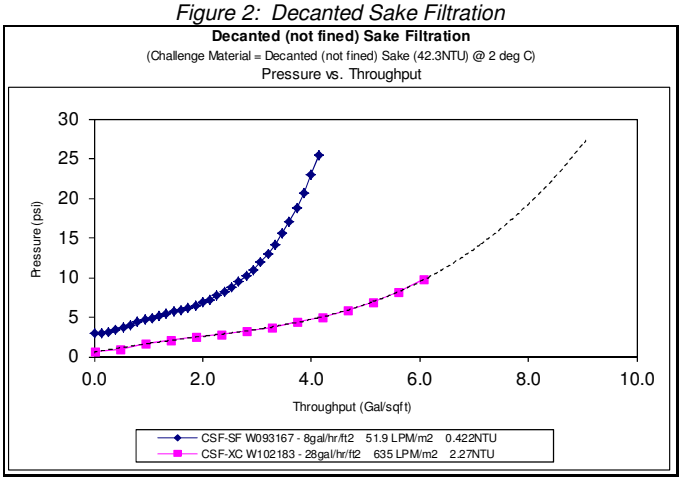


Figure 2: Decanted Sake Filtration



Note: decanted sake reservoir was depleted before reaching terminal CSF-XC pressure – dashed line indicates an extrapolated curve

Unclarified sake plugged both the coarse CSF-XC (5µ) and the fine CSF-SF (0.45µ) filters very quickly. Similar plugging profiles despite the different porosity of these filters suggests that the primary plugging particulate is >5µ, resulting in surface blinding of both filters.

Chilling & decanting sake prior to filtration significantly improved throughput on both filters, though throughput remained lower than desired. Filter life differences between grades also suggests decanting removed some of the large particulate, allowing smaller particulate to pass through the CSF-XC (resulting in longer filter life but slightly higher turbidity).

To further improve the clarity of sake before filtration, fining trials were conducted at various dose rates using silica hydrogel or bentonite. Dose rates and resulting turbidities are displayed below in Table 1.

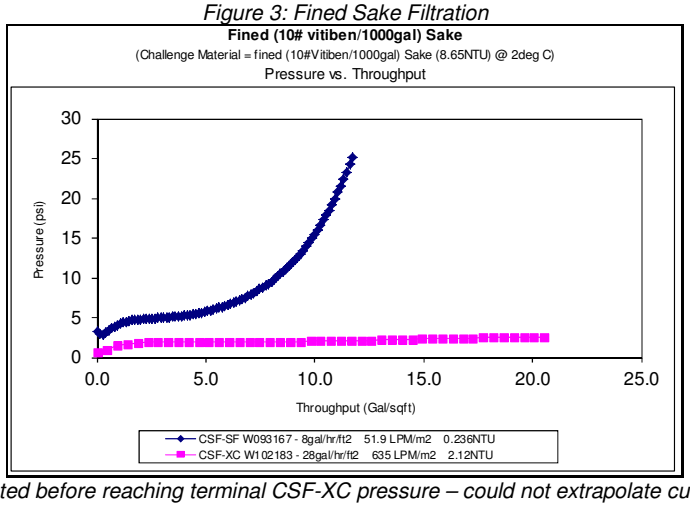
Table 1: Fined Sake Turbidity

Sample	Turbidity (NTU)
Original (not fined or decanted)	309
Decanted (not fined) control	36.5
Silica hydrogel - 400ppm	36.3
Silica hydrogel - 600ppm	33.3
Silica hydrogel - 800ppm	32.8
Bentonite - 5 #/1000gal	13.7
Bentonite - 7 #/1000gal	10.7
Bentonite - 10 #/1000gal	8.56

Results suggest that chilling and decanting sake significantly reduced turbidity (309NTU reduced to 36.5NTU), but addition of silica hydrogel, even up to 800ppm, did very little to reduce turbidity further. However, bentonite successfully reduced turbidity at 5-10 #/1000gal dosing (13.7-8.5NTU).

Since 10#/1000gal of bentonite provided the best sake clarity, additional sake was dosed at this rate before filtering through CSF-XC & CSF-SF. Results of fined sake filtrations are displayed below in Figure 3 and a comparison of all filtrations is summarized in Table 2.

Figure 3: Fined Sake Filtration



Note: fined sake reservoir was depleted before reaching terminal CSF-XC pressure – could not extrapolate curve because plugging didn't occur

Table 2: Filtration Summary

Sample Prep	Filter Media	Filtration Results		Production Needs (assuming 500L batch)	
		Filtrate Turbidity (NTU)	Throughput @ Δ21psi (gal/ft^2)	Media Area (ft^2)	# of 40cm sheet needed (1.6ft^2/sheet)
None	**CSF-XC**	0.4	2.5^	52.8	33
None	**CSF-SF**	0.3	2.5	52.8	33
Chilled/Decanted	**CSF-XC**	0.4	~8.3*^	~15.9	~10
Chilled/Decanted	**CSF-SF**	2.3	4.1	32.2	20
Fined/Chilled/Decanted	**CSF-XC**	0.2	>20.5*^	<6.4	<4
Fined/Chilled/Decanted	**CSF-SF**	2.1	11.6	11.4	7

** Note: chilled/decanted throughput estimated by extrapolating plugging curve, fined/chilled/decanted throughput will be significantly greater than 20.5gal/ft^2 but could not be extrapolated since plugging did not occur*

^Note: CSF-XC samples were terminated at Δ21psi due to pump limitations, but manufacturer recommends max Δ 45psi for this grade making expected throughputs slightly higher than reported

Filter life of both CSF-XC & CSF-SF was significantly improved by fining the sake with bentonite prior to filtration. In the customer's 500L batch production process only <4 or 7 filter sheets would be needed to filter through CSF-XC or CSF-SF respectively after fining.

To determine if the sake was suitable for sterile membrane filtration following CSF-XC or CSF-SF pre-filtration, filtrate was passed through a sterile membrane according to Clogging Index test procedures. Results are displayed below in Table 3.

Table 3: Clogging Index Results

Sample	Clogging Index	Membrane Filterability Rating
Fined & CSF-XC filtered Sake	Plugged prematurely	Extremely Poor filterability
Fined & CSF-SF filtered Sake	**3.4**	**Good Filterability (<12)**

Results confirm that pre-filtration through CSF-XC (normally used for coarse filtrations) does not provide sufficient protection for a downstream sterile membrane, plugging prematurely before the test reached completion. However, pre-filtration through CSF-SF successfully prepared the sake for membrane filtration, providing "good" filterability for efficient use of the sterile membrane.

CONCLUSIONS

Bench scale fining & filtration studies were completed to determine the best method of achieving either visual clarity or sterility of a sample from Boston Sake. Results suggest that at a minimum the customer should chill & decant the sake prior to filtration and filter life can be improved further by fining with 10# /1000gal of Vitiben bentonite.

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If you have any questions, please contact us.