Development of a New Cartridge Filter for Significantly Improved Wine Microbiological Stability

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Abstract

Controlling spoilage microorganisms prior to packaging is critical for wine production. Eliminating these spoilage organisms without affecting the other wine properties is of vital importance to producing a consistent, high quality wine. Installation of final membrane filters into the production process provides the means to control and eliminate spoilage organisms in the finished wine. Of equal importance is attaining long on-stream filter service life to minimize the operating costs associated with installation of filters into the production line.

CUNO initiated a project to develop a robust, highly microorganism retentive final filter that would withstand the rigorous cleaning and sanitation regimens employed by most wine producers. The highly durable membrane and cartridge design withstands repeated exposure to hot water sanitation, steam sterilization and industry common chemical cleaning and sanitizing agents. This filter also delivers the performance required by the wine Industry. The new membrane filter design delivers an increased product flow rate at a lower pressure drop across the filters that results in the installation of smaller filter assemblies, thus lowering the overall operational costs.

This poster reports on the development of a new membrane final filter whose construction is optimized to increase product throughput. This results in a reduction in the number of installed filters, avoiding the need to increase the filtration system footprint size, thereby, reducing the impact of filtration costs. BevASSURE® PES membrane filter design employs a new highly-asymmetric polyether sulfone membrane, a patented pleat technology [1] and patent pending upstream and downstream support design that delivers excellent spoilage organism retention with minimal impact on the organoleptic properties of the finished wine.

Laboratory and actual customer field data are presented from bench-scale filtration studies to the results from production line trials.

Materials of Construction

The filters employed in this study were manufactured by CUNO Incorporated, a 3M company (Meriden, CT). The filter media is a new single-layer, highly-asymmetric polyether sulfone (PES) membrane.

As shown in the membrane cross-section in Figure 1, the upstream pores are very open to provide both pre-filtration and retention. The downstream pores are tighter than the upstream pores and provide retention of the spoilage organisms. The asymmetric design allows larger particles, colloids and spoilage organisms to be trapped by the larger upstream pores, preventing these contaminants from fouling or blinding-off the tighter downstream pores. The finer downstream pore structure then traps finer particulates, cell debris and haze components. In this design, the effective depth of penetration and entrapment of microorganisms and particles is increased (greater contaminant capacity) as compared to a conventional uniform pore size membrane filters, resulting in increased fluid throughput without sacrificing filtrate quality.

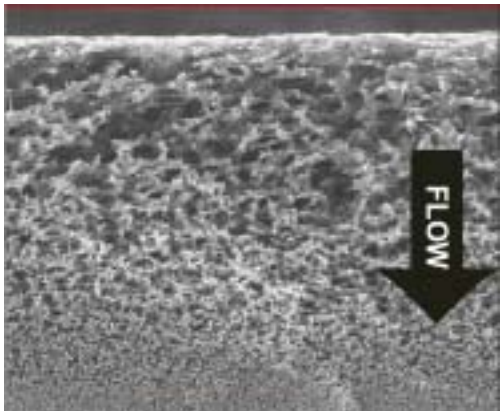


Figure 1. Scanning electron micrograph of PES membrane cross section. Magnification 2500X

PES is highly durable and extremely resistant to typical cleaning and sanitization agents used in the Wine Industry.

BevASSURE PES filter cartridges are constructed by pleating the highly asymmetric PES membrane with polypropylene upstream and downstream support materials. The core, cage and end cap adapters are made of polypropylene. The media, support materials and cartridge components are thermally sealed. No resin or binder compounds are added. Multiple length cartridges with industry standard connection styles are produced to fit the most widely used housing designs.

As shown in Figure 2, the media and support materials are fabricated into the cartridge pleat pack with the CUNO patented Advance Pleat Technology™ (APT) design. This patented design provides greater service life and faster flow rates than other media designs. The variation in pleat height maintains open flow paths between pleats. The design maximizes access by contaminants to the available filtration surface area thus increasing the contaminant holding capacity of each cartridge. The pleats are well separated at the cartridge core, reducing the resistance to fluid flow resulting in a lower pressure drop across the cartridge. The combination of a lower pressure drop across the cartridge

and the maximum available filtration surface area leads to longer service life, faster flow rates and improved filtration economics.

Cartridges are available in both 0.45 μm and 0.65 μm retention ratings and were specifically developed and tested for Food and Beverage applications as final filters to control spoilage organisms.

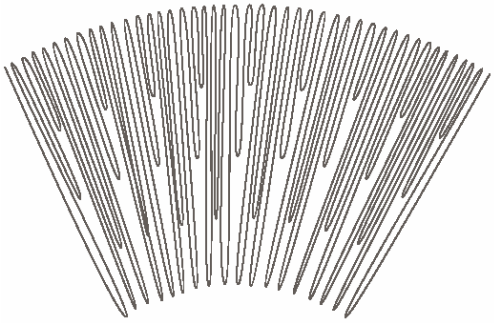


Figure 2. Graphic representation of the Advanced Pleat Technology membrane pleating design.

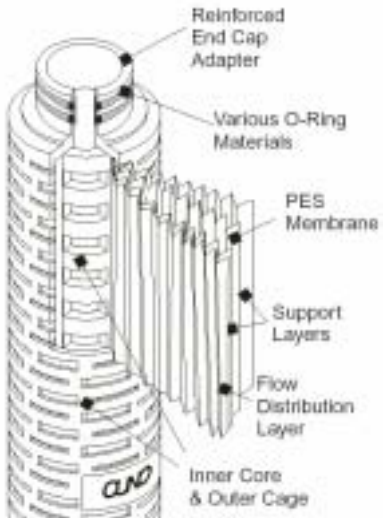


Figure 3. BevASSURE PES filter cartridge components and construction.

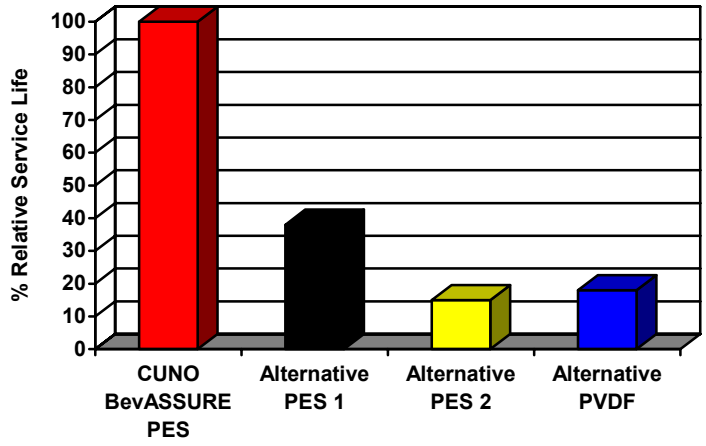
Laboratory Based Product Performance Test Results

Service Life

BevASSURE PES filters and alternative membrane filters used in wine filtration, all rated at 0.45 μm, were flushed with water at 3 gpm for 5 minutes with a 5 psi backpressure and forward flow integrity tested. The cartridges were then challenged at a constant flow rate of 3 gpm with an aqueous suspension of particulates, colloids, and complex carbohydrates common to the Food and Beverage industry. The test is designed to simulate beverage filtration at an accelerated rate. The throughput was measured in gallons until a terminal differential pressure of 15 psid above the clean pressure drop across the cartridge was reached, indicating the filters were plugged.

The alternative filters tested were commercially available membrane filters commonly used in wine filtration. Two of the filters were constructed with alternative polyether sulfone (PES) membranes and one contained a polyvinylidene fluoride (PVDF) membrane.

Chart 1. Relative Service Life of Comparable 0.45 micron Rated Membrane Filters Using Surrogate Plugging Mixture



Results: BevASSURE PES filter cartridges demonstrated greater than twice the service life, as expressed in the number of gallons processed prior to filter plugging, of the nearest alternative PES cartridge.

Microorganism Retention

Basis of tests

The primary purpose of a membrane filter installed in a food or beverage process is to effectively control spoilage microorganisms. Microorganisms specific to the food and beverage industry were selected to demonstrate the retention capabilities of BevASSURE PES filter cartridges.

Procedure

BevASSURE PES 10 inch cartridges rated 0.45 μm and 0.65 μm from three different production lots were flushed with water at 3 gpm for 5 minutes with a 5 psi backpressure then forward flow integrity tested. The cartridges were then challenged with a suspension of the selected organism in an appropriate carrier fluid at concentrations ranging from 1 x 10^6 CFU/mL to 1 x 10^7 CFU/mL. The filtrate from each cartridge was assayed to determine the log reduction ratio value (LRV) for each cartridge. Post-challenge, the cartridges were flushed with deionized water and forward flow integrity tested to confirm they were still integral after the bacteria challenge.

The challenge organisms tested were:

Oenococcus oeni suspended in white wine, *Serratia marcesens* suspended in sterile deionized water, *Lactobacillus brevis* suspended in degassed beer and *Dekkera intermedia (Brettanomyces)* suspended in dilute acetic acid solution at pH 4.0.

Chart 2 - Summary of Microorganism Retention Testing

BevASSURE PES	Microorganism	Typical Log Reduction Value (LRV)
BNA045	*Serratia marcescens*	8
BNA045	*Oenococcus oeni*	9
BNA045	*Lactobacillus brevis*	10
BNA045	*Dekkera intermedia*	9
BNA065	*Lactobacillus brevis*	7
BNA065	*Dekkera intermedia*	9

Log Reduction Values are calculated using the following formula:

$$LRV = \log_{10}\left(\frac{\text{total number of organisms entering the filter}}{\text{total number of organisms exiting the filter}}\right)$$

Exposure to Hot Water and Steam

In order to evaluate the effects of thermal stress, BevASSURE PES 10-inch cartridges from different production lots were subjected to repetitive Hot Water (90°C) and in-situ steam cycles at 135°C. The hot water and in-situ steam cycles were 30 minutes each with cooling between cycles. Test filters were evaluated for integrity by Forward Flow Integrity Testing (FFIT) at a test pressure of 22 psi or 15 psi for 0.45 μm rated and 0.65 μm rated, respectively, initially and at various intervals following the thermal cycles.

Chart 3 - The Effect of Thermal Stress on BevASSURE PES Cartridges

Treatment	Grade	Cartridge Serial Number	Integrity Test Results		
			50 Cycles	100 Cycles	150 Cycles
Hot Water 194°F/90°C 30 minutes	0.45 µm	05N020-0007	Pass	Pass	Pass
		05N020-0020	Pass	Pass	Pass
		05N020-0029	Pass	Pass	Pass
		05N024-0005	Pass	Pass	Pass
		05N024-0012	Pass	Pass	Pass
		05N024-0019	Pass	Pass	Pass
		05N033-0001	Pass	Pass	Pass
		05N033-0009	Pass	Pass	Pass
		05N033-0027	Pass	Pass	Pass
	0.65 µm	05N016-0004	Pass	Pass	Pass
		05N016-0015	Pass	Pass	Pass
		05N016-0041	Pass	Pass	Pass
		05N023-0004	Pass	Pass	Pass
		05N023-0013	Pass	Pass	Pass
		05N023-0020	Pass	Pass	Pass
		05N037-0006	Pass	Pass	Pass
		05N037-0019	Pass	Pass	Pass
		05N037-0034	Pass	Pass	Pass
			75 Cycles	100 Cycles	150 Cycles
Steam 275°F/135°C, 30 minutes	0.45 µm	05N020-0002	Pass	Fail	NA
		05N020-0015	Pass	Pass	Pass
		05N020-0025	Pass	Pass	Pass
		05N024-0021	Pass	Pass	Pass
		05N024-0025	Pass	Pass	Pass
		05N024-0045	Pass	Pass	Pass
		05N033-0003	Pass	Pass	Pass
		05N033-0017	Pass	Pass	Pass
		05N033-0025	Pass	Pass	Pass
	0.65 µm	05N016-0001	Pass	Pass	Pass
		05N016-0008	Pass	Pass	Pass
		05N016-0034	Pass	Pass	Pass
		05N023-0003	Pass	Pass	Pass
		05N023-0012	Pass	Pass	Pass
		05N023-0018	Pass	Pass	Pass
		05N037-0011	Pass	Pass	Pass
		05N037-0029	Pass	Pass	Pass
		05N037-0050	Pass	Pass	Pass

Sanitation Agent Compatibility – Oxonia Active[2]

In order to evaluate the compatibility of BevASSURE PES filter cartridges with peracetic acid/hydrogen peroxide sanitation agents typically used in the Food and Beverage industry, BevASSURE PES 10-inch cartridges from different production lots were subjected to repetitive exposures to a 1% aqueous solution of Oxonia Active at ambient temperature. Initially, and prior to each exposure interval, the cartridges were forward flow integrity tested at a test pressure of 22 psi or 15 psi for 0.45 µm rated and 0.65 µm rated, respectively. After each exposure interval, the cartridges were forward flow integrity tested to confirm the cartridges remained integral after exposure to the Oxonia Active solution. Following the forward flow integrity testing, each cartridge was forward flow bubble point tested to release as much water as possible from the pores of the membrane and then exposed to a fresh 1% aqueous Oxonia Active solution for the next exposure interval.

Cleaning Agent Compatibility – Sodium Hydroxide

In order to evaluate the compatibility of BevASSURE PES filter cartridges with caustic cleaning solutions, BevASSURE PES 10-inch cartridges from different production lots were subjected to repetitive exposures to 1 M aqueous sodium hydroxide (NaOH) solution at 65°C. Initially, and prior to each exposure interval, the cartridges were forward flow integrity tested at a test pressure of 22 psi or 15 psi for 0.45 μm rated and 0.65 μm rated, respectively. After each exposure interval, the cartridges were forward flow integrity tested to confirm the cartridges remained integral after exposure to the 1 M (4% w/v) NaOH solution. Following forward flow integrity testing, each cartridge was forward flow bubble point tested to release as much water as possible from the pores of the membrane and then exposed to a fresh 1 M NaOH solution for the next exposure interval.

Chart 4 - Compatibility of BevASSURE PES Filters to Cleaning and Sanitation Agents

Treatment	Conditions	Total Number of Hours Tested	Results
Oxonia Active	1% aqueous solution, ambient temperature, continuous soak	150	No loss of Integrity
Sodium Hydroxide (NaOH)	1 M (4% w/v) aqeuous solution, 65°C	100	No Loss of Integrity

Beta-Site Testing

CUNO conducts beta-site testing at commercial wineries for all New Products prior to the formal New Product Launch. The purpose of beta-site testing is to collect information from the wineries on whether or not the product as designed meets the winery's performance criteria. Test sites are chosen based on requesting the winery test the New Product in the exact way they would normally use the filter during their production process. Wineries were selected that could meet the following criteria: 1) familiarity with membrane filtration practices and procedures, 2) a stable production schedule and 3) the ability to provide careful monitoring and recording of key filtration data, such as, throughput volume, production flow rate, differential pressure across the filter installation, bacteria retention, integrity test results and the SIP/CIP data (time, temperature, number of cycles, etc.). CUNO's Scientific Applications Support Services personnel, a dedicated group of Scientists and Engineers that provide technical support to our Customers, assist the winery in conducting these beta-site tests. The beta-site results are used to confirm whether or not the new product performs as expected under actual field conditions as compared to the internal laboratory testing results obtained during product development

Chart 5 - Field Beta-Site Testing Results

Test Site/ Wine Filtered	Filter Assembly	Test Site Objectives	Results
California Wine Producer/ Red and White Wines	10 x 30" cartridges 0.45 µm rated	2 million gallons throughput	Previous alternative filters reportedly processed 1 million gallons on average. Final BevASSURE PES cartridges achieved 2.1 million gallons throughput. Integrity was maintained throughout the test.
European Wine Producer/ Sweet White Wine	16 x 30" cartridges 0.65 µm rated	Equal to better throughput compared to current filtration (current throughput ranges from 3000 hL to 10,000 hL depending on wine quality) Equal to better finished wine quality and microbial control. Current filtration scheme is a 2-stage system of lenticular depth pre-filter followed by a 0.65 µm alternative PES filter. Production flow rate = 90 hL/hr with filter change-outs every 2 to 3 months based on differential pressure or an integrity failure.	Successful first trial: 8,000 hL filtered; no microorganisms detected in finished wine. Integrity maintained after each hot water sanitation and citric acid cycle. Met expectations for finished wine quality. Successful second trial: 9,200 hL filtered, no microorganisms detected. Integrity maintained after hot water sanitization. Daily regeneration/sanitation: 20 minutes at 50°-55°C warm water followed by 20 minutes at 85°C hot water.
European Contract Mobile Wine Filtration/ Red and White Wines	5 x 20" cartridges 0.45 µm rated	Finished Wine Quality Eliminate concerns over current nylon membrane affecting the taste of finished wine. Confirm BevASSURE PES membrane does not influence the taste of white wine, especially after steaming new filters after initial installation.	Trial conducted on a mobile filling line mounted on a truck. Finished wine quality unaltered. Aromatic composition of the finished wine unaffected by BevASSURE PES filtration. Throughput target achieved. Filter integrity confirmed.
European Wine Producer/ Red and Rosé Wine	8 x 30" cartridges 0.65 µm rated	Finished wine quality and microbial control.	Trial in-progress. Red and white wines filtered. 4070 hL filtered with ΔP only 0.2 bar.
European Wine Producer/ White Wine	8 x 30" cartridges 0.45 µm rated	Finished wine quality and microbial control Surpass current throughput target of 4,600 hL achieved with alternate filter train.	BevASSURE PES filters provided 12,800 hL throughput as compared to 4,600 hL throughput typically provided by the alternate filter. During the trial, the filters were exposed to 31 warm water (55°C)/hot water (90°C) regenerations plus 7 cold water/0.5% peracetic acid cycles. Turbidity after CUNO depth pre-filter = 1.5 NTU, after BevASSURE PES = 0.4 NTU. Alternative pre-filters to alternative final filters only filtered 4200 hL. Second trial in-progress. Regeneration with NaOH and hot water (90°C) instead of peracetic acid/hot water. Through 15 regeneration cycles, 4600 hL filtered with only a small increase in differential pressure. Test site reported that regeneration step using warm water (50° - 55°C) was very short (1 minute maximum.)

Conclusions

- BevASSURE PES cartridges utilize a robust cartridge design containing a highly asymmetric PES membrane.
- BevASSURE PES filters are compatible with routine cleaning and sanitization procedures used in the Food & Beverage Industry, including multiple hot water and steam sanitization cycles, caustic cleaning agents and peracetic acid/peroxide sanitization agents.
- Laboratory testing demonstrates the membrane design is highly efficient for removal of spoilage organisms, yeast and secondary fermentation bacteria.
- BevASSURE PES filters provide exceptional protection of finished wine, thus increasing shelf life by removal of spoilage organisms.
- The unique BevASSURE PES cartridge design provides significantly longer service life for improved filtration economics.
- The excellent performance of BevASSURE PES filters was proven through beta-site testing at commercial wineries in both the US and Europe.

Footnotes

[1] US Patent 6,315,130 and other patents pending

[2] Oxonia Active is a trademark of EcoLab, Inc

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