Application Note

Filtration of viscous / particle-laden samples using the Samplicity® Filtration System

Introduction

Samples of foods, beverages, pharmaceutical and other consumer products are frequently tested for safety and quality using chromatographic or other analytical separation techniques. Proper filtration of samples, solvents and buffers not only generates higher quality, more consistent results, but it also increases instrument uptime and prolongs chromatographic column life. Syringe filtration has proved to be an efficient means by which these samples can be clarified and made ready for downstream analyses.

However, for hard-to-filter samples (samples containing particulate materials as well as samples that are viscous), syringe filtration is not easy and the particle load can easily clog the syringe filter. Food and beverage samples such as juices, honey, soups, salad dressing and ketchup can be very difficult to filter prior to analysis. Other hard-to-filter samples include many pharmaceutical suspensions, shampoos, conditioners, creams and other household products.

Most of these samples are challenging to filter either because of their high viscosity, which can greatly slow down filtration, or because they contain high levels of particulates, which causes clogging of the filter. If manual syringe filtration is used, these samples can require excessive manual force, greater time per sample, and lower recovery—all reducing laboratory efficiency. Sometimes the particles in these samples clog the syringe filter so quickly that multiple filters are required to obtain necessary sample volume. In some cases the samples need to be diluted excessively to allow for filtration, making it challenging to obtain satisfactory signal-to-noise ratios during downstream analysis. In contrast, the vacuum-driven Samplicity® filtration system filters even highly viscous samples in seconds, with minimal manual force.

The Samplicity® filtration system also delivers high recovery because of the low hold-up volume of its Millex Samplicity® filters, making the system ideal for preparing samples for liquid chromatography, such as high performance liquid chromatography/ultra-high pressure liquid chromatography (HPLC / UHPLC), for which the typical sample volume requirement is not more than 1.5 mL.

EMD Millipore is a division of Merck KGaA, Darmstadt, Germany

Materials and Methods

Optimization of sample concentration

To maintain efficiency in an analytical laboratory, samples should be of an appropriate concentration to enable complete collection of 1 mL filtrate in 1 minute filtration time, while still being sufficiently concentrated such that analytes can be detected in downstream analyses with good signal-to-noise ratios. To determine the optimum concentration for each test substance, we prepared serial dilutions of each substance at 100%, 75%, 50%, 20%, 10%, 5%, 2% and 1% (all w/w) in water. We then filtered the serially diluted samples using 0.45 µm and 0.2 µm Millex Samplicity® hydrophilic PTFE filters (Cat. Nos. SAMPLG001 and SAMPLCR01) and 0.45 µm Millex Samplicity® PVDF filters (Cat No. SAMPHV001).

Filtration time trials

Once the appropriate concentration of each sample was determined, average filtration time at that concentration was reported as average of 4 replicates. Filtration studies were conducted by adding 2 mL of each appropriately diluted sample to the funnels of each Millex Samplicity® filter, placed in the Samplicity® filtration system.

Viscosity measurement

Viscosity measurements on all the samples at appropriate concentrations were made using the Brookfield Viscometer (Model No. LVDV-II+). Spindle # S61 was used in all viscosity measurements and the temperature of measurements was 22 °C. All viscosity measurements were reported as average of 3 measurements. To calibrate the viscometer, Milli-Q® water was used as the viscosity control.

Measurement of manual force required for syringe filtration and Samplicity® filtration

Fifteen users were observed and timed as they filtered 24 samples using both the Samplicity® system and syringe filtration. Samples (1.0 mL volume) of 1% Pepto Bismol® were filtered either through hydrophilic PTFE Millex® syringe filters with a 10 mL syringe, or through 0.45 µm hydrophilic PTFE Millex Samplicity® filters using the Samplicity® filtration system. Approximately half the subjects operated the Samplicity® system first, and the other half used syringe filters first. Users then rated their subjective experience of force required on a scale of 1 to 10.

Results

Efficient filtration of hard-to-filter samples

Compared to syringe filtration of viscous or particulate-laden samples, which required excessive manual force and greater filtration time per sample, the Samplicity® filtration system filtered even highly viscous samples in seconds, with minimal manual force. After using both systems, users, on average, rated the Samplicity® filtration system as requiring 6–7 times less manual force than that required to operate syringe filters (Figure 1).

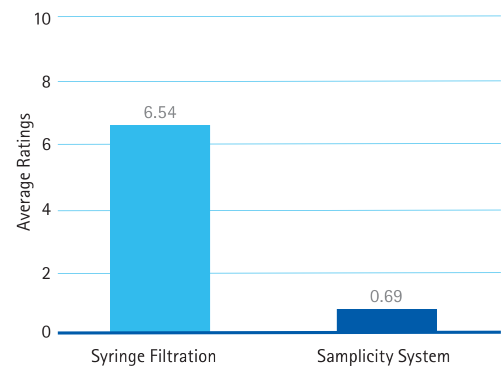


Figure 1. Average ratings of manual force required to operate syringe filters vs. the Samplicity® filtration system

To assess the performance of the Samplicity® filtration system in filtering samples of varying viscosity and particulate load, we chose samples to represent products of various industries and various application areas. The samples were classified into 3 different categories:

a) Food and beverage samples (Tables 1-3)

b) Pharmaceutical samples (Figures 2-4)

c) Industrial and chemical samples (Figures 2-4)

Filtration of food and beverage samples

All three Millex Samplicity® filters were able to successfully filter various food and beverage samples, some of which exhibited very high viscosity (Tables 1–3). Depending on membrane and sample characteristics, each filter performed at differing speeds with differences in the maximum concentration of each sample it could efficiently filter. In general, 0.45 µm filters filtered samples faster than 0.2 µm filters (at the same sample concentration) or were able to filter samples at much higher concentration. This finding was expected, because small pore-sized membranes frequently are associated with lower throughput and/or higher rate of clogging.

Sample	Concentration Filtered, %	Viscosity, Centipoises	Filtration time, seconds	% RSD
Tomato juice	100	18600.0	51.5	2%
Honey	50	8.0	60.5	2%
Yellow mustard	5	4.1	41.8	1%
Protein shake	2	2.1	49.0	2%
Coconut water	100	2.1	4.5	13%
Italian dressing	50	60.0	42.8	2%
Ranch dressing	25	NA	58.8	4%
Chocolate syrup	25	24.7	43.8	5%
Sports drink	100	2.1	2.0	0%
Ketchup	50	1576.0	55.3	1%
Red wine vinegar	100	2.1	2.0	0%
Iced tea with lemon	100	2.1	17	0%
Tomato soup concentrate	2	201.33	52.8	1%
Water	100	2.1	2	0

Table 1. Hydrophilic PTFE Millex Samplicity® filters (0.45 µm) efficiently processed hard-to-filter food and beverage samples in seconds. Filtration times were the average of 4 replicates. Water is shown for reference.

Sample	Concentration Filtered, %	Viscosity, Centipoises	Filtration time, seconds	% RSD
Tomato juice	100	18600.0	37.5	2
Honey	10	2.3	19.0	0
Yellow mustard	5	4.1	55.5	1
Protein shake	1	2.1	10.5	1
Coconut water	100	2.1	8.5	1
Italian dressing	25	12.1	40.8	4
Ranch dressing	10	NA	18.3	1
Chocolate syrup	25	24.7	53.3	1
Sports drink	100	2.1	2.0	0
Ketchup	25	252.3	14.3	1
Red wine vinegar	100	2.1	2.0	0
Iced tea with lemon	100	2.1	17.0	0
Tomato soup concentrate	1	201.3	5.0	0
Water	100	2.1	2	0

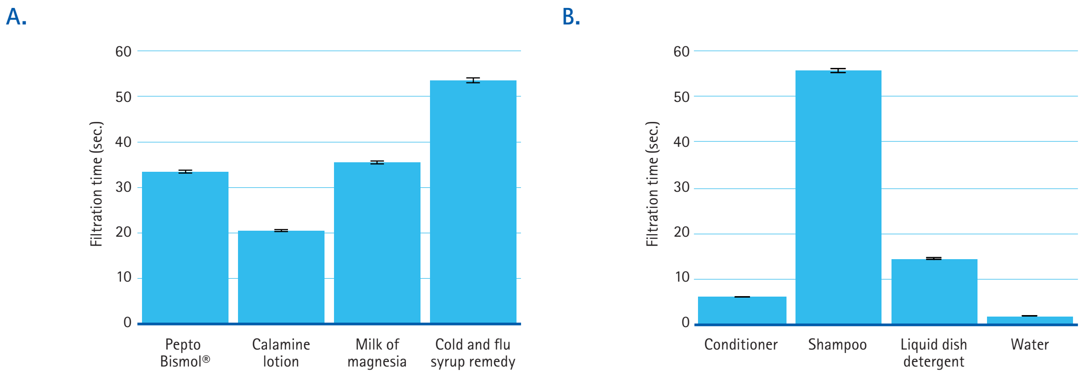
Table 2. Hydrophilic PTFE Millex Samplicity® filters (0.20 µm) efficiently processed hard-to-filter food and beverage samples in seconds. Filtration times were the average of 4 replicates. Water is shown for reference.

Sample	Concentration Filtered, %	Viscosity, Centipoises	Filtration time, seconds	% RSD
Tomato juice	75	90.1	34.5	7
Honey	75	47.1	29.0	3
Yellow mustard	5	4.1	37.0	7
Protein shake	2	2.1	48.3	1
Coconut water	100	2.1	4.5	1
Italian dressing	25	12.1	30.8	6
Ranch dressing	1	2.9	44.3	10
Chocolate syrup	50	147.6	53.8	3
Sports drink	100	2.1	3.5	1
Ketchup	2	3.2	22.8	2
Red wine vinegar	100	2.1	2.5	1
Iced tea with lemon	100	2.1	2.5	1
Tomato soup concentrate	1	201.3	4.0	1
Water	100	2.1	2	0

Table 3. Hydrophilic PVDF Millex Samplicity® filters (0.45 µm) efficiently processed hard-to-filter food and beverage samples in seconds. Filtration times were the average of 4 replicates. Water is shown for reference.

Filtration of pharmaceutical and industrial / chemical samples

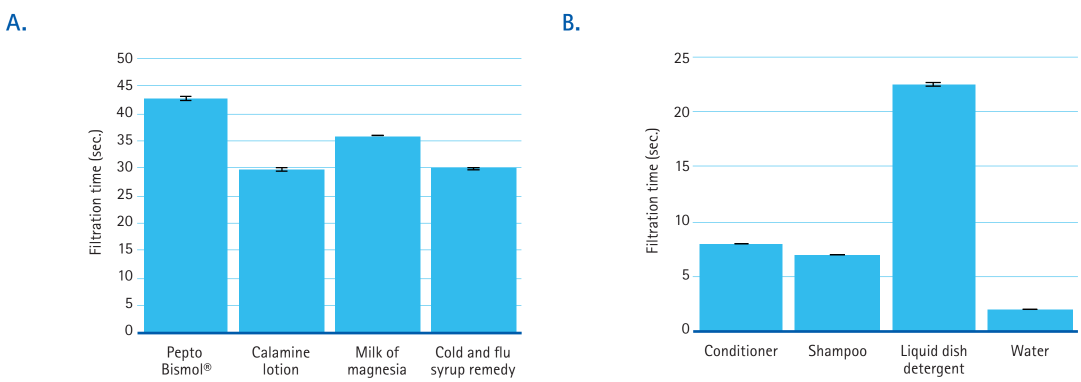
All three Millex Samplicity® filters were able to successfully filter various pharmaceutical and industrial samples, some of which exhibited very high viscosity (Figures 2–4). Depending on membrane and sample characteristics, each filter performed at differing speeds with differences in the maximum concentration of each sample it could efficiently filter. In general, 0.45 µm filters filtered samples faster than 0.2 µm filters (at the same sample concentration) or were able to filter samples at much higher concentration.



Concentration	2%	2%	100%	10%
Viscosity (cP)	2.3	298.3	10088.0	3.6

Concentration	10%	75%	50%	100%
Viscosity (cP)	2946.3	30.8	17.3	2.1

Figure 2. Hydrophilic PTFE Millex Samplicity® filters (0.45 µm) efficiently processed hard-to-filter pharmaceutical samples (A) and industrial/chemical samples (B) in seconds. Filtration times were the average of 4 replicates and error bars represent standard deviation. Water is shown for reference.



Concentration	2%	2%	100%	5%
Viscosity (cP)	2.3	298.3	10088.0	2.2

Concentration	10%	50%	50%	100%
Viscosity (cP)	2946.3	4.5	17.3	2.1

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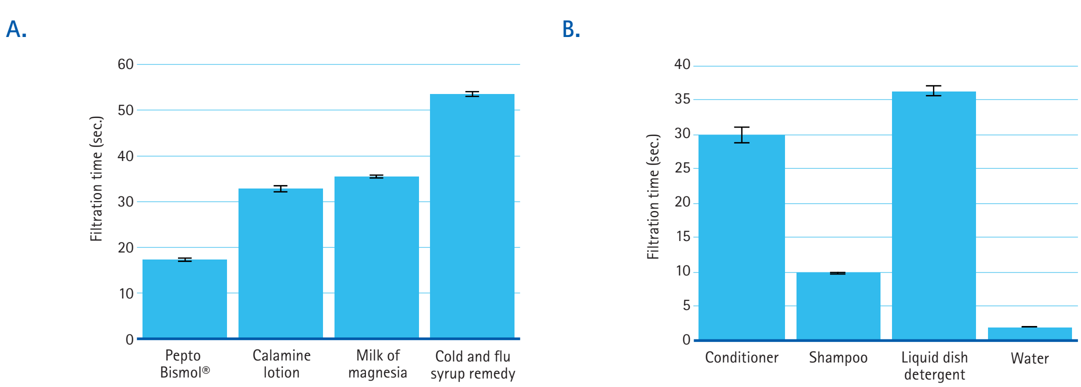

Concentration	2%	5%	100%	10%
Viscosity (cP)	2.3	510	10088.0	3.6

Concentration	1%	75%	75%	100%
Viscosity (cP)	4.5	30.8	140.6	2.1

Figure 4. Hydrophilic PVDF Millex Samplicity® filters (0.45 µm) efficiently processed hard-to-filter pharmaceutical samples (A) and industrial/chemical samples (B) in seconds. Filtration times were the average of 4 replicates and error bars represent standard deviation. Water is shown for reference.

Conclusion

Filtration of particle-laden and viscous samples prior to chromatography is always very challenging, decreases laboratory efficiency, and may require undesirable degrees of sample dilution.

Syringe filters with prefilters are currently used for filtration of such samples. Prefilters are useful when filtering large volumes of sample, because the prefilter prevents the membrane from clogging up. However, when filtering samples for HPLC / UHPLC, samples are typically smaller than 2 mL. For such small volumes, prefiltration media can lead to analyte loss, sample loss and introduction of extractable impurities. If prefilters are not used, membrane-only syringe filtration of these samples is very difficult and places physical strain on the analyst.

We have shown that the Samplicity® filtration system, which allows for vacuum-driven filtration of sample directly into HPLC vials and batch processing of up to 8 samples, is an ergonomic alternative to syringe filtration of viscous and particle laden samples. All three Millex Samplicity® filters allow for filtration of these difficult samples without significant manual force.

More importantly, the Samplicity® system enabled efficient filtration of samples at higher concentrations. Consequently, this sample preparation method can minimize the need for sample dilution, potentially yielding higher signal-to-noise ratios in sensitive downstream analyses, such as HPLC or UHPLC.

Ordering Information

Description	Qty/pk	Catalogue No.
Samplicity® Filtration System	1	SAMPSYSGR (green) or SAMPSYSBL (blue)
Millex Samplicity® Filters, hydrophilic PTFE, 0.45 µm	96	SAMPLCR01
Millex Samplicity® Filters, hydrophilic PTFE, 0.45 µm	384	SAMPLCR04
Millex Samplicity® Filters, hydrophilic PTFE, 0.20 µm	96	SAMPLG001
Millex Samplicity® Filters, hydrophilic PTFE, 0.20 µm	384	SAMPLG004
Millex Samplicity® Filters, hydrophilic PVDF, 0.45 µm	96	SAMPHV001
Millex Samplicity® Filters, hydrophilic PVDF, 0.45 µm	384	SAMPHV004

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