Filtration of high particulate and high viscosity solutions using vacuum-driven Milliflip™ filters

Introduction

Proper filtration of samples, solvents, and buffers leads to higher quality data and more consistent results, while also optimizing instrument performance. Filtration has proved to be an efficient means by which samples can be clarified for downstream analyses. Samples of foods, beverages, and other consumer products are frequently assessed for safety and quality using chromatographic or other analytical techniques. Many of these samples are challenging to filter either because of their high viscosity, which can greatly slow filtration, or because they contain high levels of particulates, which can clog filters.

Filtration of high particulate or high viscosity samples manually using hand pressure-driven devices requires excessive manual force and can lead to poor recovery, increasing fatigue and reducing laboratory efficiency. In contrast, vacuum-driven Milliflip™ filters offer an optimized, ergonomic solution for processing mid-range volumes of hard-to-filter samples. These non-sterile filters feature low hold-up volumes for high recovery, making them ideal for preparing 2 - 50 mL volumes of sample for analysis.

To minimize filtration times for hard-to-filter samples, dilution is often performed. We optimized the concentration of 29 consumer products and other samples for processing with 0.45 µm PVDF Milliflip™ filters. The viscosity of the optimized sample concentrations and filtration time using 10 mL and 50 mL sample volumes were determined for all sample types. The hold-up volume of the Milliflip™ filter was also measured. These results can be used as a guideline for diluting food and beverage, pharmaceutical, nutraceutical, detergent, cosmetic, environmental, and other sample types to streamline analytical workflows using Milliflip™ filters.

Materials and Methods

Optimization of sample concentration

The following samples were analyzed:

- **Food**: Red wine vinegar, Chocolate syrup, Honey, Yellow mustard, Ketchup, Baby food (peas), Italian dressing, Ranch dressing, Tomato soup concentrate
- **Beverage**: Sports drink, Coconut water, Iced tea with lemon, Tomato juice, UF milk (non-fat), Protein shake
- **Pharmaceutical**: Cold and flu syrup remedy, Milk of magnesia, Calamine lotion, Pepto Bismol® liquid
- **Nutraceutical**: Garcinia Cambogia, Vitamin D3, Prenatal vitamin, Green tea extract
- **Industrial**: Laundry detergent
- **Cosmetic**: Shampoo, Conditioner, Body lotion
- **Environmental**: Soil samples (certified reference materials)

To determine the optimum concentration for filtration, each sample was diluted as follows: 100 %, 75 %, 50 %, 25 %, 10 %, 5 %, 1 %, 0.5 %, 0.25 %, 0.1 %, 0.05 %, 0.025 %, and 0.01 % all w/w in distilled water. The viscosity of the final solutions was measured on a Brookfield Model LVDV-II Viscometer fitted with spindle S61 at 60 rpm for low viscosity measurements. Viscosities were calculated as the average of triplicate measurements.

Sample filtration

Filtration studies were conducted by adding 10 mL or 50 mL of each appropriately diluted sample to a double-threaded 50 mL polypropylene tube (Falcon® tube, **Cat. No. CLS352070**) or funnel accessory (**Cat. No. SC50FL025**). For all experiments, samples were filtered through non-sterile Milliflip™ filters with 0.45 µm Durapore® PVDF (polyvinylidene fluoride) membranes (**Cat. No. MFHV00025**). Filtration was performed at room temperature (21-23 °C) using a vacuum pump set to 25" Hg with regulated pressure gauge.

Comparison of filtration times

Low and high-volume processing flow times and recovered volumes were determined for each sample type. An average filtration time at each sample concentration and volume was reported as the average of four replicates.

Results and Discussion

Solutions of samples of varying viscosity and particulate load were tested. We chose samples representative of products of various industries and application areas.

Sample concentration optimization

Most of the samples tested are challenging to filter either because of their high viscosity or high particulate load. To filter within a reasonable time frame, these samples were diluted to an optimum concentration. **Table 1** shows the optimized solution concentrations that were used in the subsequent filtration studies.

Sample		Optimized % Solution	Measured Viscosity, Centipoises (cP)
Diluent	Water (deionized)	100.00 %	1.0
Food	Red wine vinegar	100.00 %	15.0
	Chocolate syrup	1.00 %	3.0
	Honey	1.00 %	2.6
	Yellow mustard	0.50 %	2.2
	Ketchup	0.50 %	3.6
	Baby food-peas	0.25 %	3.3
	Italian dressing	0.25 %	2.6
	Ranch dressing	0.05 %	2.9
	Tomato soup concentrate	0.05 %	7.0
Beverage	Sports drink	100.00 %	9.8
	Coconut water	100.00 %	3.0
	Iced tea with lemon	100.00 %	14.7
	Tomato juice	1.00 %	3.4
	UF milk (non-fat)	1.00 %	3.6
	Protein shake	0.50 %	1.7
Pharmaceutical	Cold and flu syrup remedy	50.00 %	25.3
	Milk of magnesia	10.00 %	4.8
	Calamine lotion	1.00 %	2.3
	Pepto Bismol® liquid	0.10 %	2.8
Detergent/ Cosmetic	Laundry detergent	75.00 %	32.9
	Shampoo	50.00 %	5.1
	Conditioner	0.05 %	10.0
	Body lotion	0.01 %	3.8
Environmental	Soil A	2.50 %	2.12
	Soil B	0.50 %	2.0
Nutraceutical	Garcinia Cambogia	75.00 %	16.2
	Vitamin D3	5.00 %	2.4
	Prenatal vitamin	1.00 %	2.0
	Green tea extract	0.25 %	1.6

Table 1. Optimization of sample concentration and viscosity measurement of optimized samples. All viscosity measurements were reported as average of three measurements.

Filtration time for hard-to-filter solutions

Once the optimum sample concentrations were established, the filtration times for low-volume (10 mL) and high-volume (50 mL) sample solutions were measured (**Figures 1-2**). In general, the 0.45 µm PVDF Milliflip™ filters could process all samples at different concentrations, with the 50 mL samples requiring longer time to filter than the 10 mL sample, as expected. Also, the higher the dilution for a given sample type, the faster the processing. This finding was expected as higher sample concentrations are frequently associated with lower throughput and/or higher rates of clogging. At optimized concentrations, the 0.45 µm PVDF Milliflip™ filter processed even viscous samples such as ketchup, mustard, and chocolate syrup in seconds to minutes without manual force.

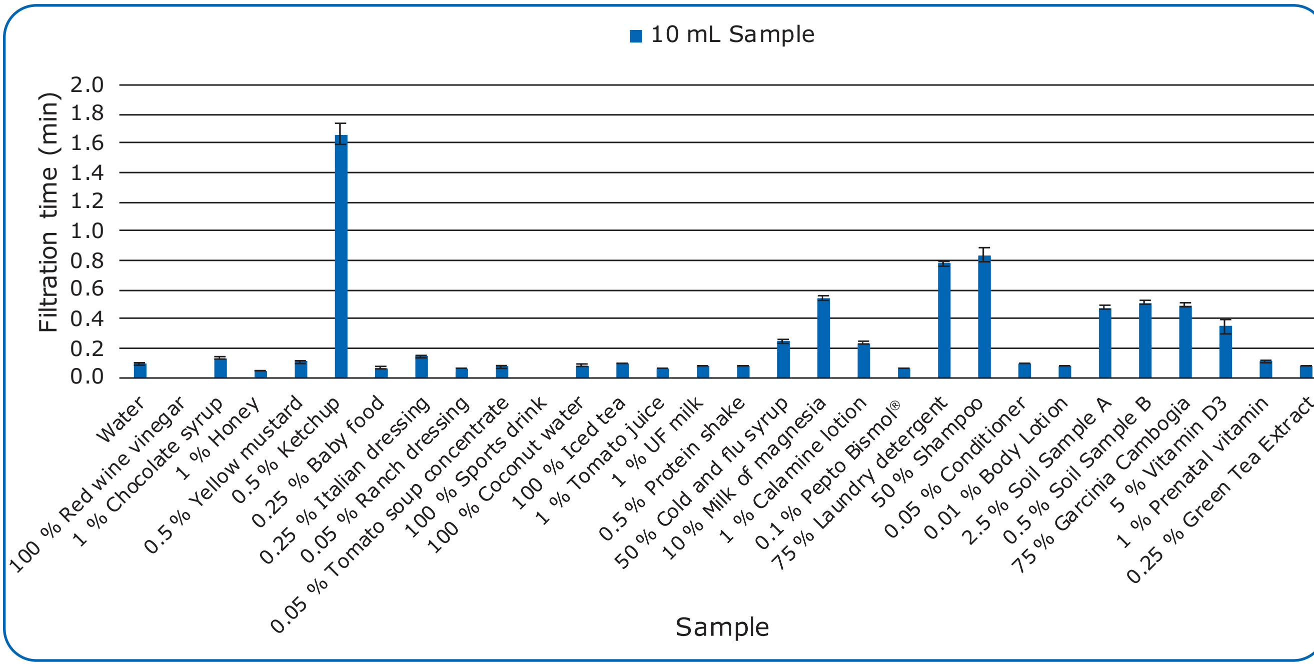

Figure 1. Processing times for 10 mL volumes of hard-to-filter solutions at optimized concentrations using Milliflip™ filters containing 0.45 µm PVDF membranes. Distilled water was used as a control. (n=4)

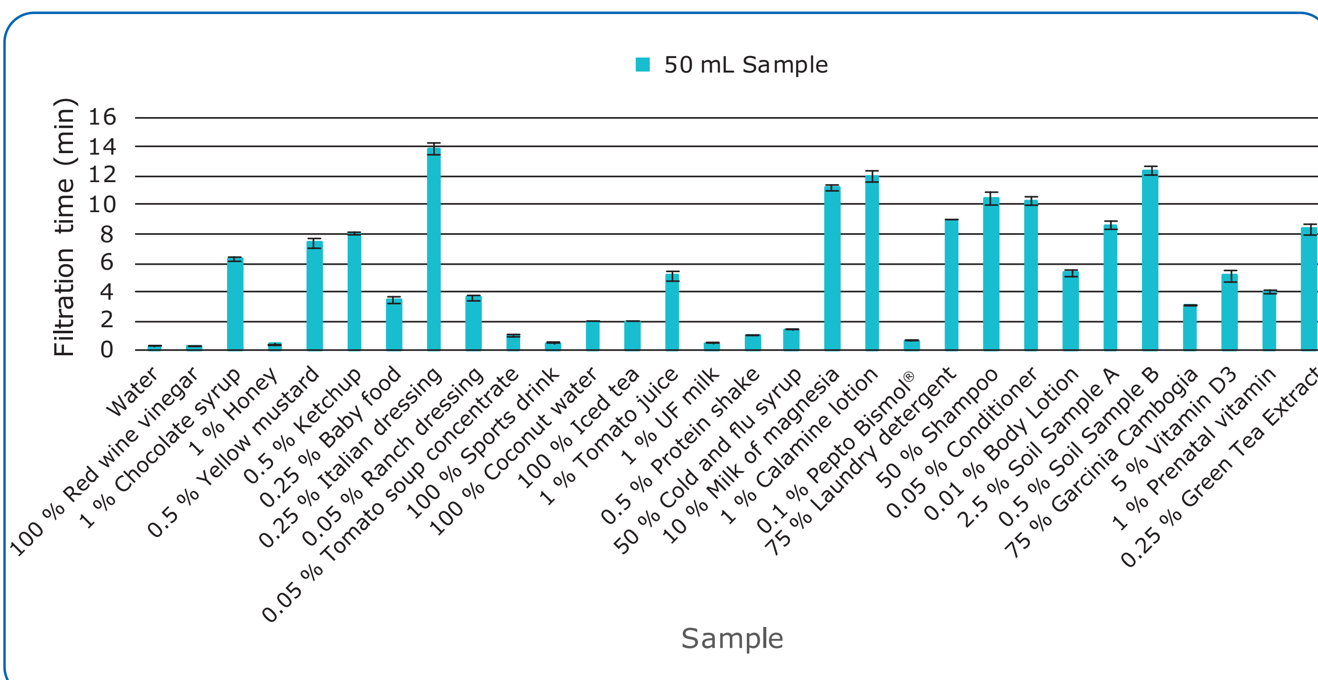

Figure 2. Processing times for 50 mL volumes of hard-to-filter solutions at optimized concentrations using Milliflip™ filters containing 0.45 µm PVDF membranes. Distilled water was used as a control. (n=4)

Hold-up volume of the Milliflip™ filter

During filtration, all systems retain some liquid that cannot be recovered, known as "hold-up" volume. This is the volume of liquid (in this case, "sample") remaining in the filtration device after use. The larger the hold-up volume, the more sample that is lost.

Design elements of the filter housing and filtration device as well as filter diameter or surface area impact hold-up volume. The hold-up volume during application of the 0.45 µm PVDF Milliflip™ filter was measured to be ~0.40 mL, using distilled water (**Figure 3**).

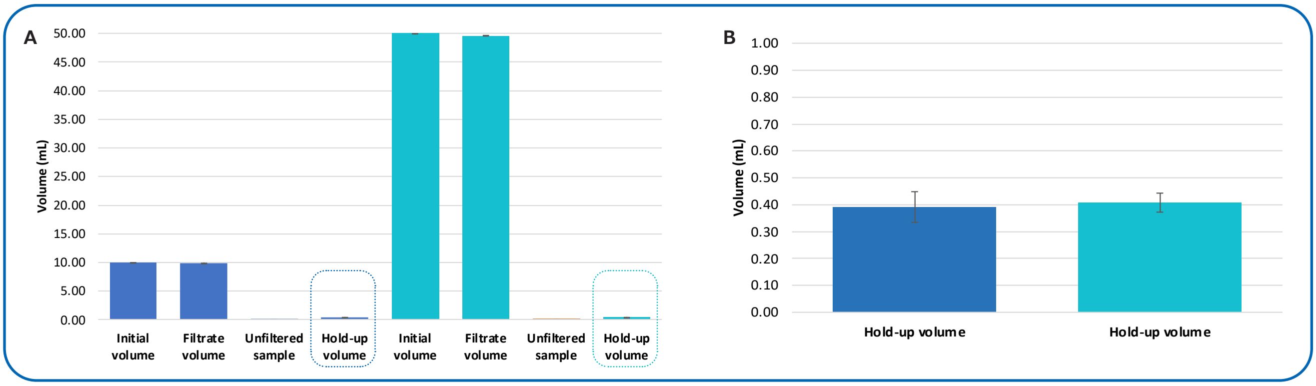

Figure 3. A) Determination of hold-up volume during application for 0.45 µm PVDF Milliflip™ filters with distilled water. B) Y-axis is rescaled for final hold-up volume.

Conclusions

Filtration of particle-laden and viscous samples can be challenging, requiring increased manual force and longer filtration times if using hand pressure-driven devices. We have shown that vacuum-driven Milliflip™ filters can be used for efficient, ergonomic filtration of viscous and particle-laden samples with the appropriate dilution. Milliflip™ filters offer an optimized solution for processing 2 - 50 mL volumes of food and beverage, pharmaceutical, nutraceutical, detergent, cosmetic, environmental, and other sample types in analytical workflows.

Related Products

Description	Cat. No
Milliflip™ filter, Non-sterile Vacuum Filtration Unit, 0.45 µm PVDF	**MFHV00025**
Vacuum Pump (115 V, 60 Hz)	**WP6111560**
Vacuum Pump (220 V, 50 Hz)	**WP6122050**
Vacuum Pump (100 V, 50/60 Hz)	**WP6110060**
Millipore® Vacuum Filtering Side-Arm Flask (1 L)	**XX1014705**
Threaded Side-Arm with Quick Vacuum Disconnect (1 L)	**XX1514706**
Millex®-FG50 filter for vacuum line protection, 0.2 µm hydrophobic polytetrafluoroethylene (PTFE), 10/pk	**SLFG05010**
Steriflip® Funnel Attachment, Non-sterile	**SC50FL025**
Stoppers, No. 8, 9.5 mm (3/8 in.) hole, Silicone with Parylene Coating	**XX2014718**
Stoppers, No. 8, Perforated, Silicone with Parylene Coating	**XX1014708**
BD Falcon Double-Threaded 50 mL Tubes	

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