SPWCC Wet Process Conference, February 13-15, 2006, Santa Clara, CA

Effect of Pump Pulsation and Particle Loading on Membrane Filter Retention

Mark R. Litchy[1], Donald C. Grant[1] and Reto Schoeb[2]

[1]CT Associates, Inc.
7121 Shady Oak Road, Eden Prairie, MN 55344
www.ctassociatesinc.com
(952) 470-0166

[2]Levitronix GmbH
Technoparkstrasse 1
CH-8005 Zurich, Switzerland
www.levitronix.com

Abstract

Hydraulic shocks caused by flow stoppages through microporous membrane filters have been shown to dramatically increase particle release from the filters. The magnitude of the release can be mitigated by techniques like Stabilized Distribution™[1]. In Stabilized Distribution™, a minimum flow rate is always maintained through system filters to minimize particle release. Changes in the flow rate through a filter have also been shown to affect filtrate particle concentrations [2].

This experiment was undertaken to determine if pulsations induced by different pumps also affect filter performance. Three types of pumps (diaphragm, bellows, and centrifugal) with varying degrees of pulsation were tested at similar average flow rates and backpressures. The magnitudes of the flow pulsations from each pump type were measured at all test conditions. Particle retention by 0.1 μm membrane filters was characterized as a function of pump pulsation intensity and particle loadings. At low loading, particle retention decreased with increasing pulsation intensity. Particle retention decreased with increased particle loading. The decrease was most pronounced for the pump with the highest pulsation intensity.

Introduction

Various types of pumps have been used in bulk chemical delivery systems, recirculating etch baths (REBs), and other high purity process applications. Many of these pumps (i.e. bellows, diaphragm, etc.) create flow pulsations that may impact the performance of the filters used in the process loop. This study was undertaken (1) to quantify the magnitude of pump-induced flow pulsations in the pump systems and (2) to correlate those pulsations with filter retention. It was assumed that the flow pulsations were directly related to the pressure variations. Hence, a fast response pressure transducer was used to quantify the magnitude of the flow pulsations. Although a flowmeter could have been used for these measurements, flowmeters generally have much slower response times than pressure transducers and likely would be too slow to adequately monitor the flow variations.

Two experiments were performed. In the first experiment, filter performance was determined by measuring the retention of monodisperse polystyrene latex (PSL) spheres with two types of 0.1 μm membrane filters at relatively low particle loadings. Filter retention was characterized at three flow rates for each type of pump. In the second experiment, particle retention measurements was measured as filters of the same type were challenged with a simulated one-year particle loading using four different pumps.

A bellows pump, a diaphragm pump, and two centrifugal pumps were tested. In some applications, pulsation dampeners are used to minimize the effect of the pulsations on the system. In this test, pulsation dampeners were not utilized to determine the impact of flow pulsations on filter performance under worse case conditions.

Experimental Procedure

Pulsation intensity

A fast response NT® International pressure transducer was installed downstream of the pump being evaluated. This transducer, which has a response time of about 1 millisecond or better, was used to quantify the magnitude of the pressure pulsations. Tests were performed under nearly identical test conditions (flow rate and back pressure) for each pump system. Pressure measurements were collected at 1000 Hz. Data were collected and analyzed over one-minute time intervals at each test condition.

Retention measurements I: filter retention at low loading

A schematic of the test system is presented in Figure 1. A Levitronix BPS-3 pump was used to circulate water through 10” Mykrolis 0.1 μm Microgard filters arranged in parallel. These filters provide a continuous feed of low particle (<0.1/ml ≥ 0.10 μm) deionized water to the pumps being evaluated.

Filter retention measurements were performed by injecting monodisperse PSL spheres into the flow stream upstream of the pump being evaluated. The PSL challenge solutions consisted of particle sizes ranging from 0.1 to 0.5 μm in diameter. Particle retention measurements were conducted at flow rates of 5, 7.5, and 10 gpm with a backpressure of 20 ± 2 psig. Particle concentrations were monitored upstream and downstream of the test filter during each particle injection. In addition, upstream and downstream background measurements were made prior to and after each particle injection test to ensure that particle concentrations were adequately low to perform the particle challenges. In this test, a single filter was used to characterize the retention with each of the pumps at each of the flow rates tested. As a result, the filter loading during these tests was not well controlled, although the tests were performed at relatively low particle loadings. Two different types of 10” filters were tested during this evaluation (see Table I): a Mykrolis 0.1 μm Etchgard HPX and a Pall 0.1 μm Ulti-Etch filter. Filter face velocities ranged from 1-4 cm/min. The particle concentrations were monitored with a Particle Measuring Systems HSLIS S-100. This spectrometer is capable of measuring particles from ≥ 0.1 - ≥ 1.0 μm over 16 size channels. The retention efficiency of each filter was calculated for each pump at each particle challenge size.

Retention measurements II: filter retention with loading

Membrane filter retention typically decreases quickly as a filter is loaded with particles, followed by a slow decrease and eventually leveling off at higher filter loadings [3]. As a result, measurements were made to investigate the effect of particle loading on membrane filter retention subject to varying degrees of pulsation.

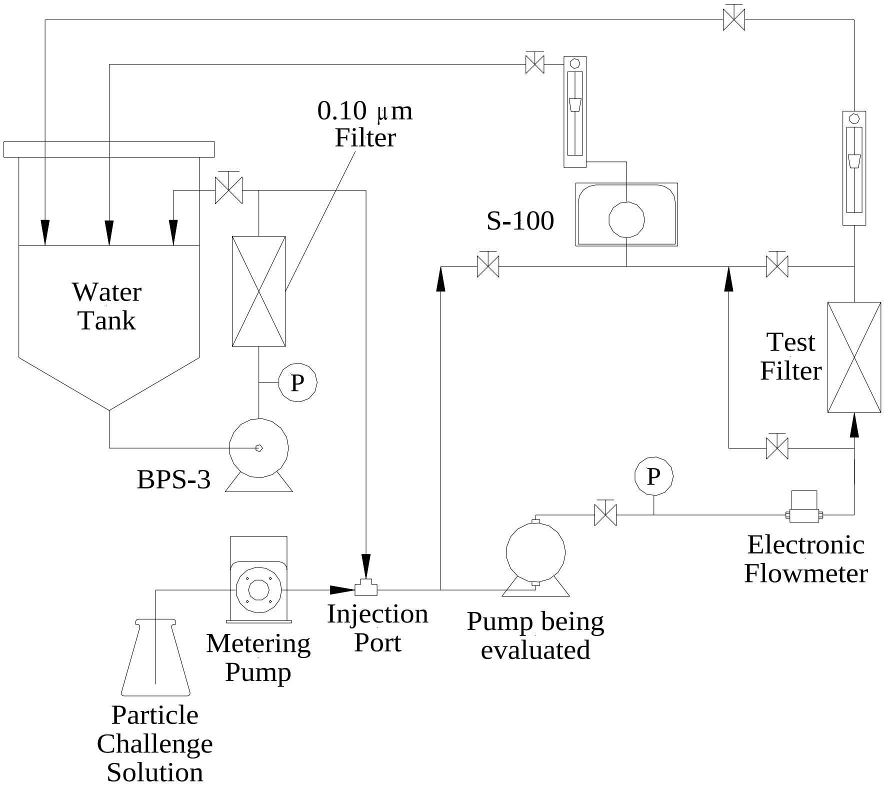

Figure 1. Test system schematic

Table I. Specifications of the two types of filters tested

Manufacturer	Filter	Pore Size	Membrane Material
Mykrolis	Etchgard HPX	0.1 μm	ultra high molecular weight polyethylene
Pall	Ulti-Etch	0.1 μm in recirculation mode	polyvinylidene fluoride

In this test, each pump was tested with a new filter of the same type, unlike the previous test in which all retention measurements were performed with a single filter. A 0.1 μm Pall Ulti-Etch filter was chosen for this test. Since some variability in the initial particle retention from one filter to another was observed, even though the filters were from the same lot, we decided to test as many filters as necessary to obtain four filters (one for each type of pump) with similar initial retention. These initial retention measurements were performed using the same pump. (We chose to use a centrifugal pump for this initial retention characterization since it induced minimal pulsation.) The initial retention measurements were performed by challenging each filter with the following PSL sizes: 102, 126, 152, and 199 nm. After the initial retention measurements were completed, the filter was removed and immediately placed in a class 100 laminar flow hood to dry while the other filters were characterized.

Once four filters with similar initial retention were identified, the pump tests were performed by first installing one of the filters in the test system then briefly challenging the filter with 102 nm PSL particles to ensure that the filter was performing properly (e.g. o-ring was seated properly, filter was dried appropriately, etc.)). The filter was then challenged with a polydisperse suspension of polystyrene latex (PSL) spheres by injecting a concentrated suspension of particles using the peristaltic pump shown in Figure 1. The filter was loaded with a simulated one-year challenge of a polydisperse suspension of PSL particles at normal mixed bed outlet contamination levels (~100 particles/mL ≥ 0.05 μm with a slope of –3 on a log-log plot). The solution was comprised of 11 PSL sizes ranging from 0.05 to 0.5 μm. The loading procedure closely followed that specified in the SEMATECH Provisional Test Method for Determining Particle Contribution and Retention by UPW Distribution System Components (#92010949B-STD). Figure 2 shows the cumulative particle size distribution at the filter inlet during the polydisperse particle challenge. The filter was loaded with the simulated one-year challenge over 16 hours. During loading, particle concentrations were measured continuously downstream of the filter being tested. The challenge concentration was verified near the beginning of the test and at the end of the test by measuring the particle concentration upstream of the filter.

After the simulated one-year challenge was complete, the filter was tested for final particle retention by challenging it with monodisperse PSL sizes ranging from 102 to 299 nm using the same procedure described above.

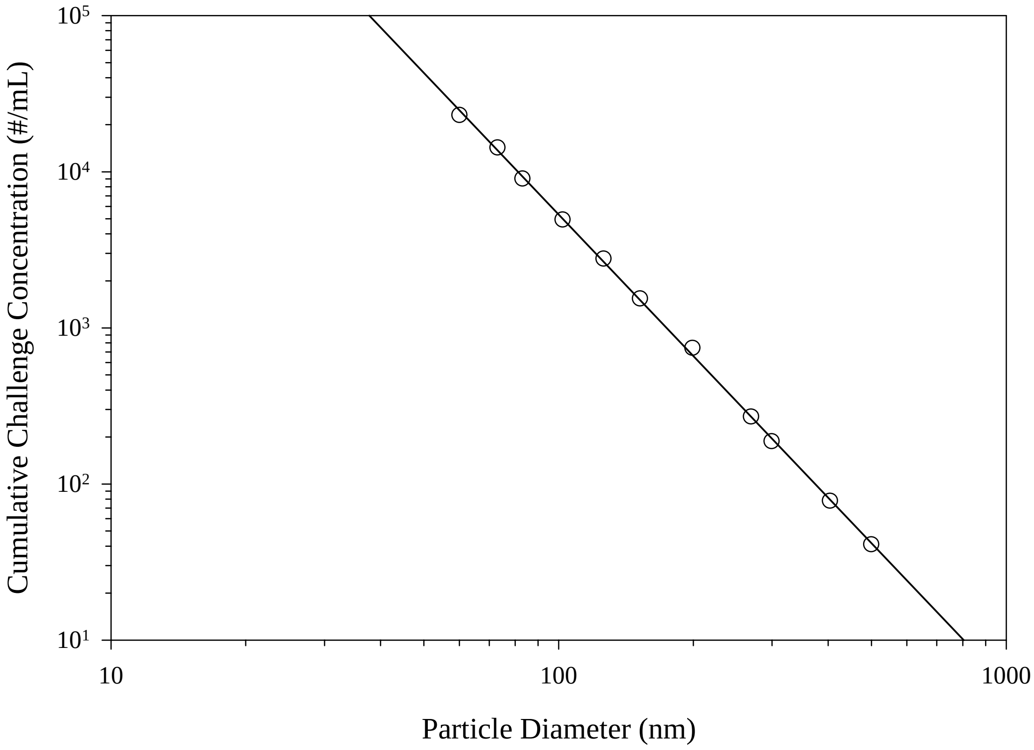

Figure 2. Cumulative particle size distribution of the feed during particle loading

Retention calculations

In this paper, filter retention data are expressed as the filter log reduction value (LRV). For the monodisperse particle challenges, the differential particle concentration was used to calculate a LRV defined as follows:

$$LRV = \log_{10} ((N_I - N_{BI}) / (N_O - N_{BO}))$$

where:

N_I = concentration at the filter inlet during particle injection

N_{BI} = background concentration at the filter inlet after particle injection

N_O = concentration at the filter outlet during particle injection

N_{BO} = background concentration at the filter outlet prior to particle injection

Two points should be made concerning this definition. First, for monodisperse particle challenges, one could use either the differential or cumulative particle concentrations in the calculations and achieve essentially the same results. Second, subtracting the background concentrations at the filter outlet prior to the particle injection and at the filter inlet after the particle injection allows for a more accurate determination of the particle retention at a given particle size if the background feed concentration is not negligible relative to the challenge feed concentration or the filter tends to shed particles without being challenged. In this experiment, the background particle concentrations at the filter inlet were always very low, thus N_{BI} was negligible relative to the N_I and could be excluded. Prior to any significant particle loading, the background particle concentrations at the filter outlet, N_{BO}, was also low and essentially negligible; however, after the one-year polydisperse loading, the concentration at the filter outlet was significant even in the absence of any particle challenge. Thus, this definition of LRV excludes prior particle shedding from the retention calculation.

For the polydisperse particle challenges, the following definition for filter retention was used:

$$\text{LRV (Cumulative)} = \log_{10} (C_I / C_O)$$

where:

C_I = cumulative concentration at the filter inlet during particle injection
C_O = cumulative concentration at the filter outlet during particle injection

Cumulative concentration was used in the retention calculations for the polydisperse challenges because it is independent of the width of the size channel used. For a polydisperse distribution, using cumulative rather than differential concentrations in the calculation effectively causes the retention values to be higher since larger particles that are retained more efficiently are included in the calculation at the smaller size channels.

Furthermore, particle release from the filter tended to increase throughout the polydisperse loading. However, no attempts were made to periodically stop the loading process to monitor the filter release in the absence of the particle challenge.

There is an additional reason why the cumulative LRV results measured with the polydisperse challenges tend to have higher retention than the LRV results measured with the monodisperse challenges. The retention results of the monodisperse challenges were plotted at the actual size of the monodisperse PSL particles being tested rather than the size channels that the particle spectrometer assigned to them. For instance, a properly calibrated spectrometer with a size channel at 0.20 µm will assign 50% of 0.20 µm particles into the size bins above and below 0.20 µm. This effectively shifts the retention results higher than if the actual PSL particle size were used.

Regardless of the definition of LRV used, the relationship between LRV and filter retention is shown in Table II.

Table II: The relationship between LRV and filter retention

LRV	Retention (%)
0.05	10
0.15	30
0.30	50
0.52	70
0.70	80
1.0	90
2.0	99
3.0	99.9

Results and Discussion

Pump pulsation intensity

Figures 3 and 4 show the magnitude of the pressure pulsations for each type of pump at 5 and 10 gpm, respectively. (Measurements were also performed at 7.5 gpm, but are not included here.) As expected, the pulsations from the bellows and diaphragm pumps were substantially higher than the centrifugal pump. Furthermore, the pulsations increased with increasing flow rate for both the bellows and diaphragm pumps.

An analysis of the pulsation data is presented in Table III. The pulsation intensity, defined in this paper as the relative standard deviation (RSD) of the pressure measurements, was calculated over one-minute test intervals for each pump at each test condition. RSD is defined as the standard deviation divided by the mean. Figure 5 shows the pulsation intensity as a function of flow rate for each pump type. The pulsation intensity measurements indicate that the pressure pulsations from the bellows and diaphragm pumps were about 4-7 and 10-20 times higher than the centrifugal pump, respectively. There is some variability in the pressure signal that may be due to electrical noise. Since the pulsation intensities for the centrifugal pump were the lowest of the three types of pumps, the results may have been significantly lower than presented if this noise were eliminated.

The magnitude of the pulsations increased substantially with increasing flow rate for the bellows and diaphragm pumps. The magnitude of the pulsations increased roughly 30% and 100% as the flow rate was increased from 5 to 10 gpm for the bellows and diaphragm pumps, respectively. The pulsations were essentially unchanged for the centrifugal pump.

Table III. Summary of pulsation intensity measurements

Flow Rate	Pulsation Intensity (RSD of Pressure Measurements, %)		
	Centrifugal	Bellows	Diaphragm
5 gpm	2.5	11.8	21.8
7.5 gpm	1.9	12.3	31.8
10 gpm	2.4	15.7	43.8

Pump comparison at low filter loading

Figures 6 and 7 show the retention of the Mykrolis 0.1μm Etchgard HPX and Pall 0.1 μm Ulti-Etch filters as a function of particle size for each pump at flow rates ranging from 5 to 10 gpm. The maximum detectable LRV with this test method was 4, thus LRV values greater than this were plotted at a value of 4.

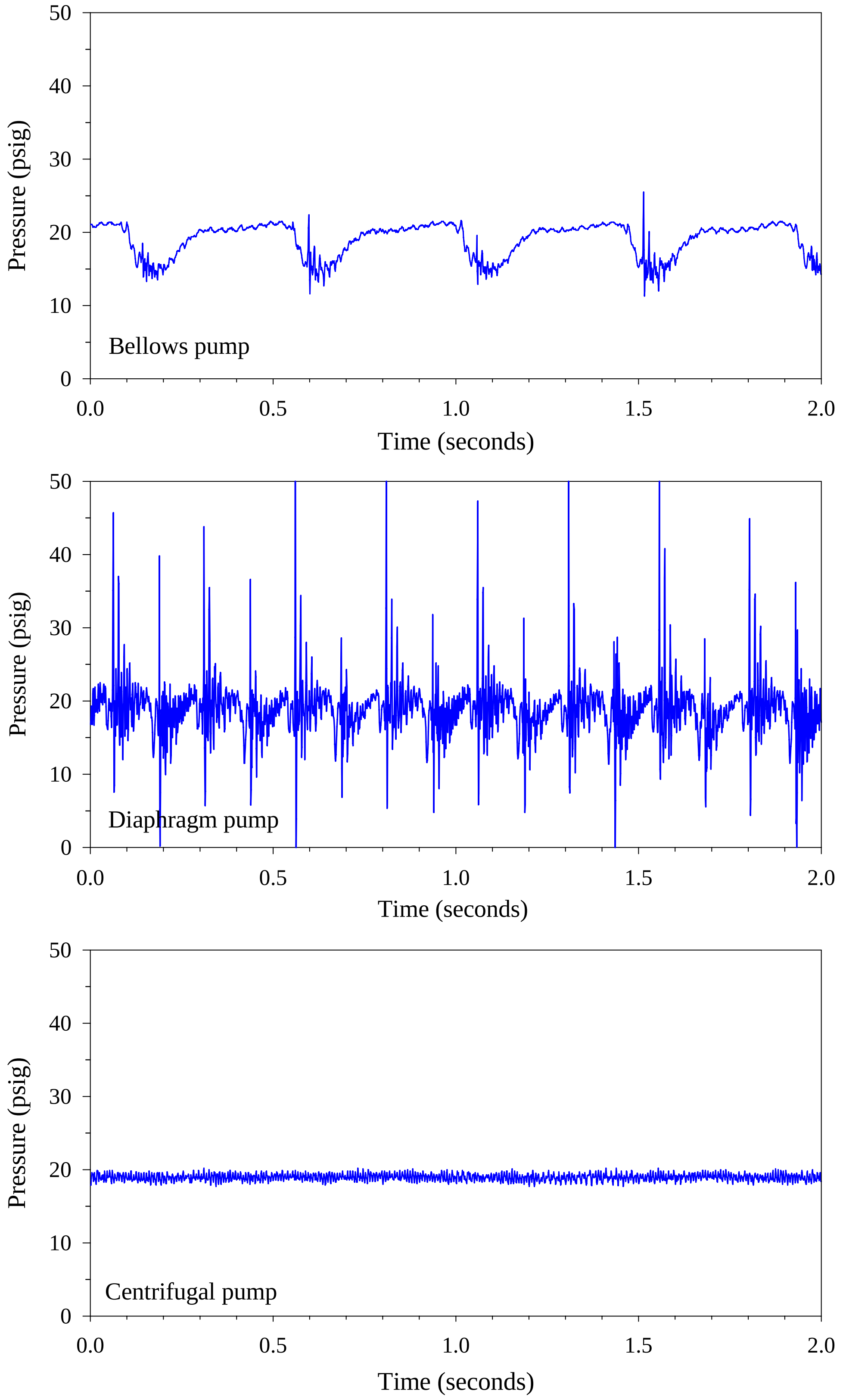

Figure 3. Magnitude of pressure pulsations for each pump at 5 gpm

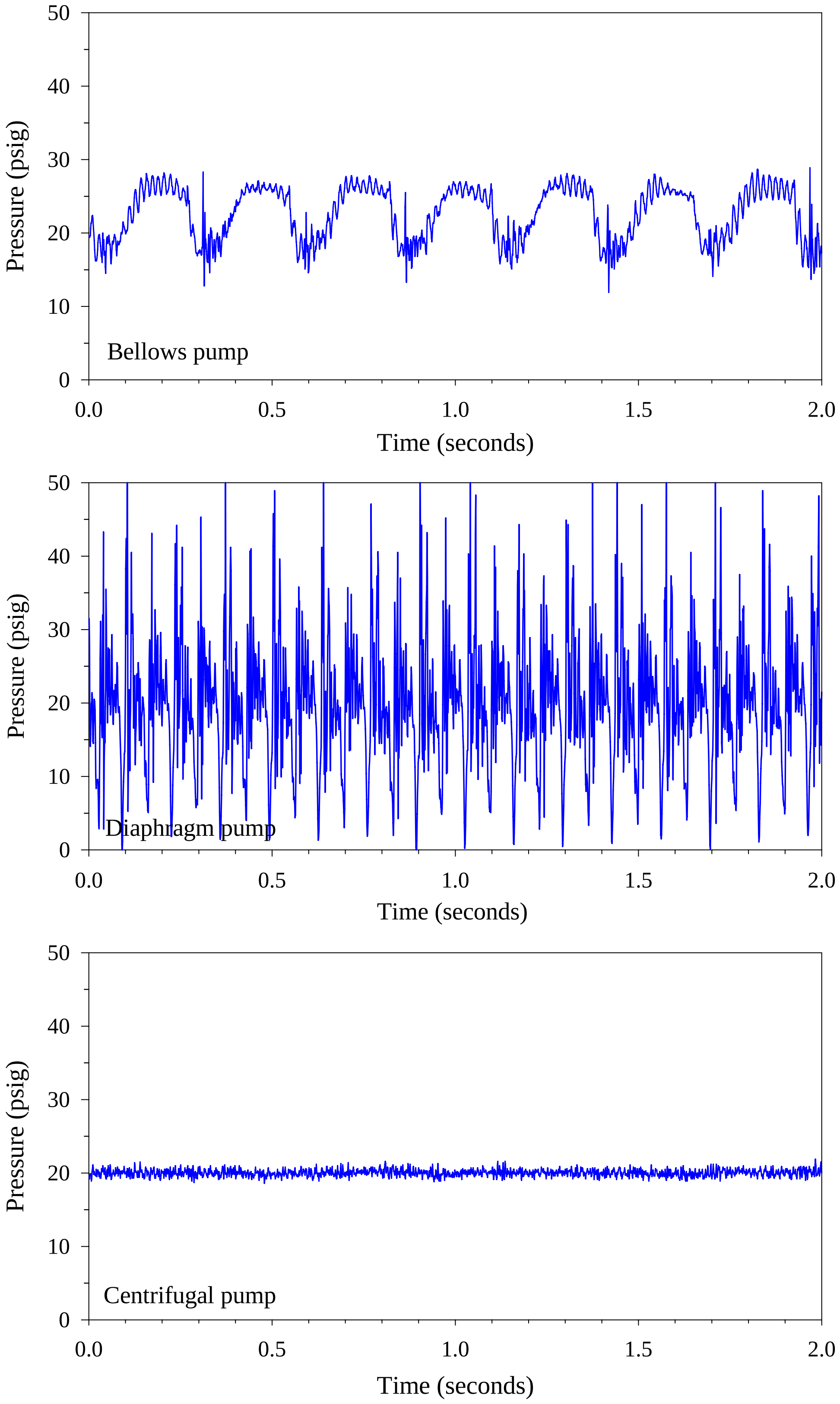

Figure 4. Magnitude of pressure pulsations for each pump at 10 gpm

LRV increased with increasing particle size in each test and was linear when plotted as a function of particle size on a log-log scale. The linear relationship (on a log-log scale) has been observed elsewhere [4].

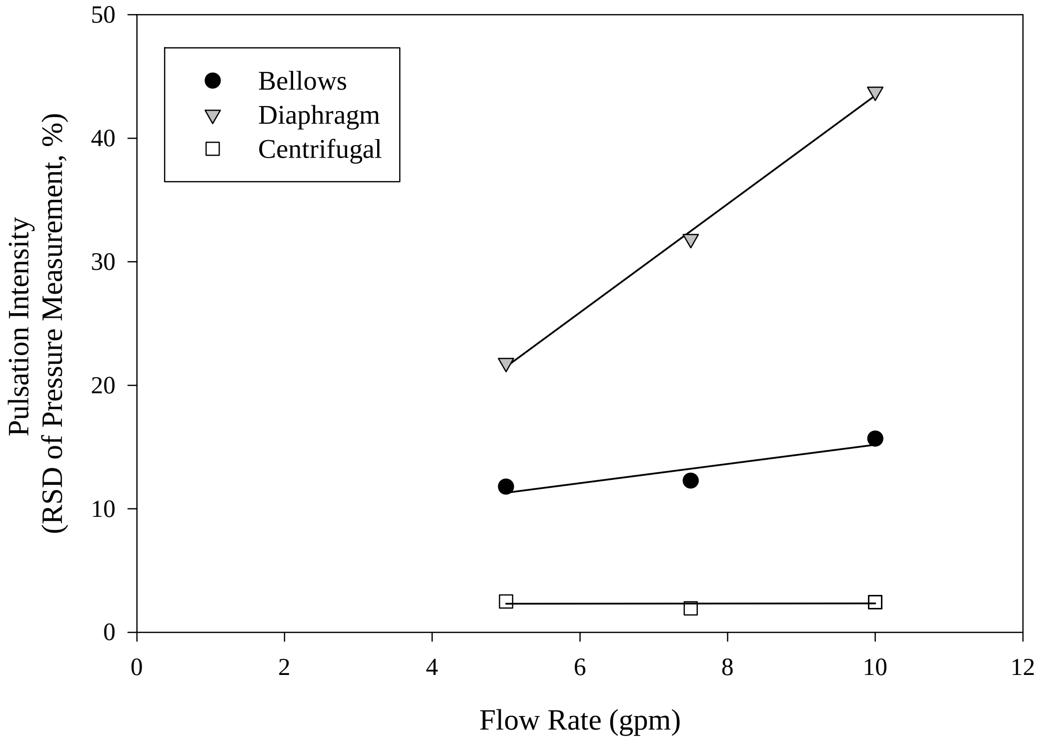

Figure 5. Pulsation intensity as a function of flow rate

The centrifugal pump, which provides the most stable flow of the pumps tested, exhibited the highest particle retention at each flow rate tested. For low flow rates, particle retention with the bellows pump was indistinguishable from the centrifugal pump, even though it delivers flow that has significant pulsation. At 10 gpm, particle retention measured using the bellows pump was slightly lower than that measured using the centrifugal pump. However, particle retention obtained with the diaphragm pump, which exhibited the largest pulsation of the pumps tested, was significantly lower than with the other pumps, particularly at higher flow rates.

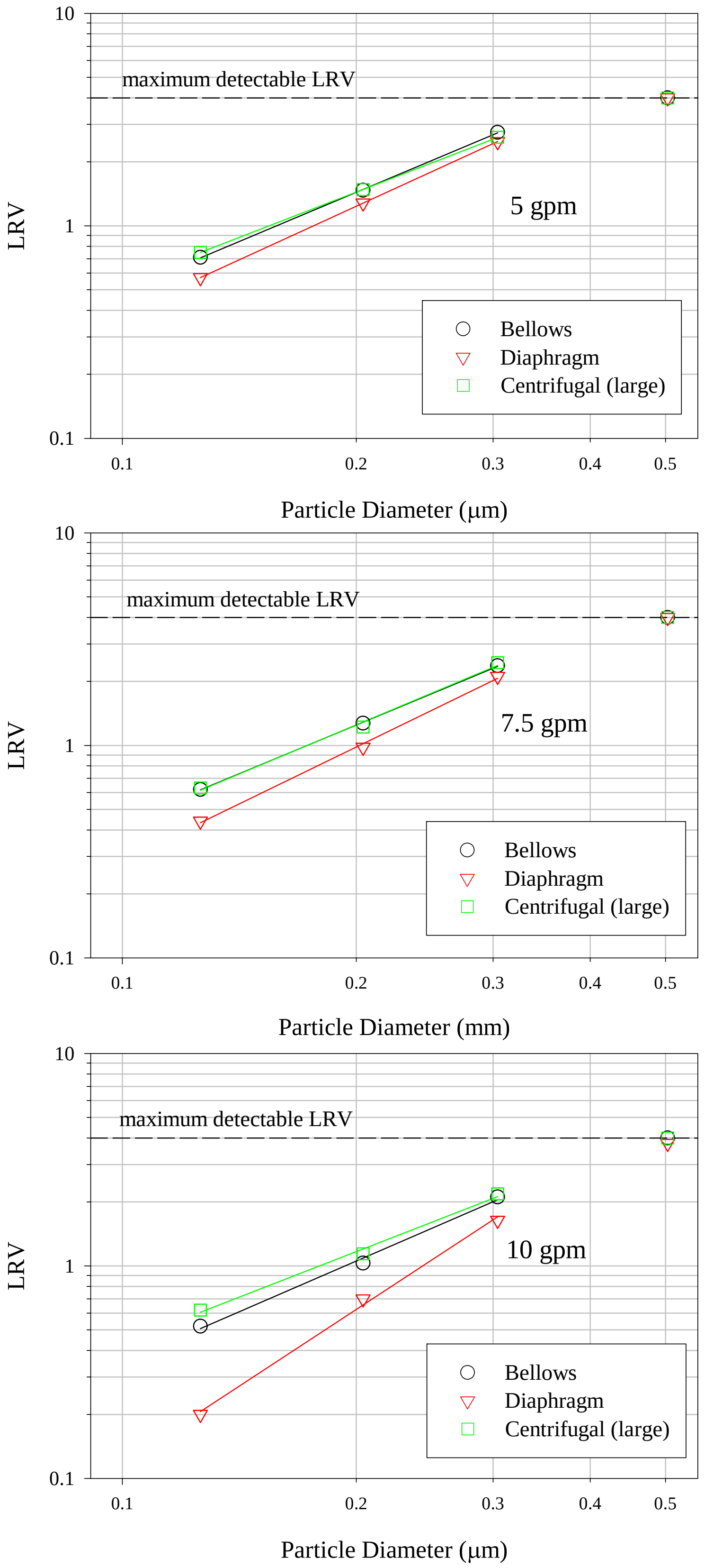

Figure 6. Retention efficiency of 0.1 µm Etchgard HPX at 5, 7.5, and 10 gpm

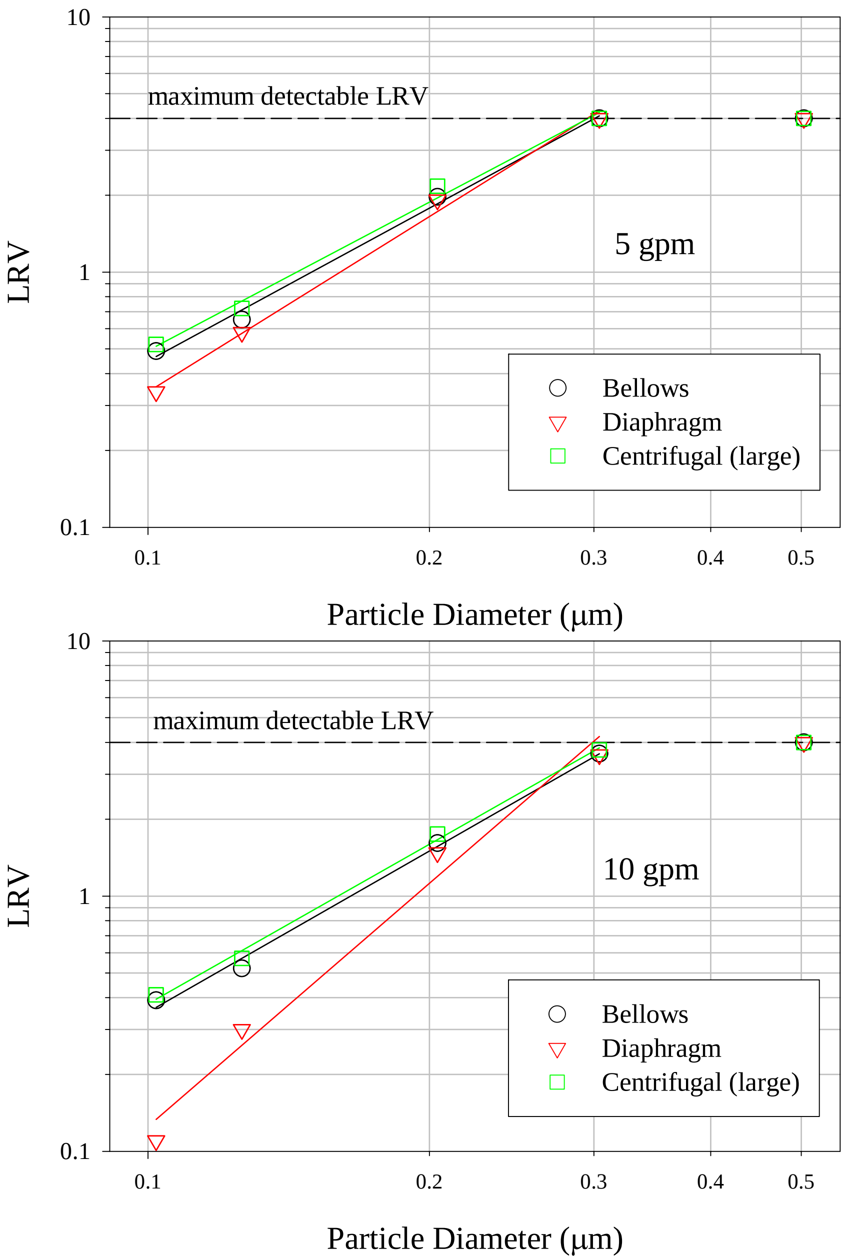

Figure 7. Retention efficiency of 0.1 µm Ulti-Etch at 5 and 10 gpm

In general, the higher the flow rate used, the greater the difference in filter retention between the pumps that deliver flow with and without pulsation. Although it is not easily distinguishable in these figures, the retention values measured using each pump decreased slightly as the flow rate increased.

Although both the Etchgard and Ulti-Etch filters are rated at 0.1 µm, both filters exhibited low retention (LRV ~0.5 or 70% retention) for particles of this size. This is because these filters are typically used in recirculating etch bath (REB) applications. Filters used in REB applications are sometimes rated differently than filters used in other filtration applications. In a REB application, the cleanup time of the bath is not only a function of the retention characteristics of the filter, but also the bath turnover rate. Thus, the most retentive filter may not cleanup a REB the fastest [5]. More open, less retentive filters with lower pressure drop are often more effective at reducing bath particle concentrations.

Pump comparison during filter loading

Figure 8 shows the initial retention as a function of particle size for the four 0.1 µm Ulti-Etch filters used in this evaluation. This figure shows that the initial retention of the four was very similar.

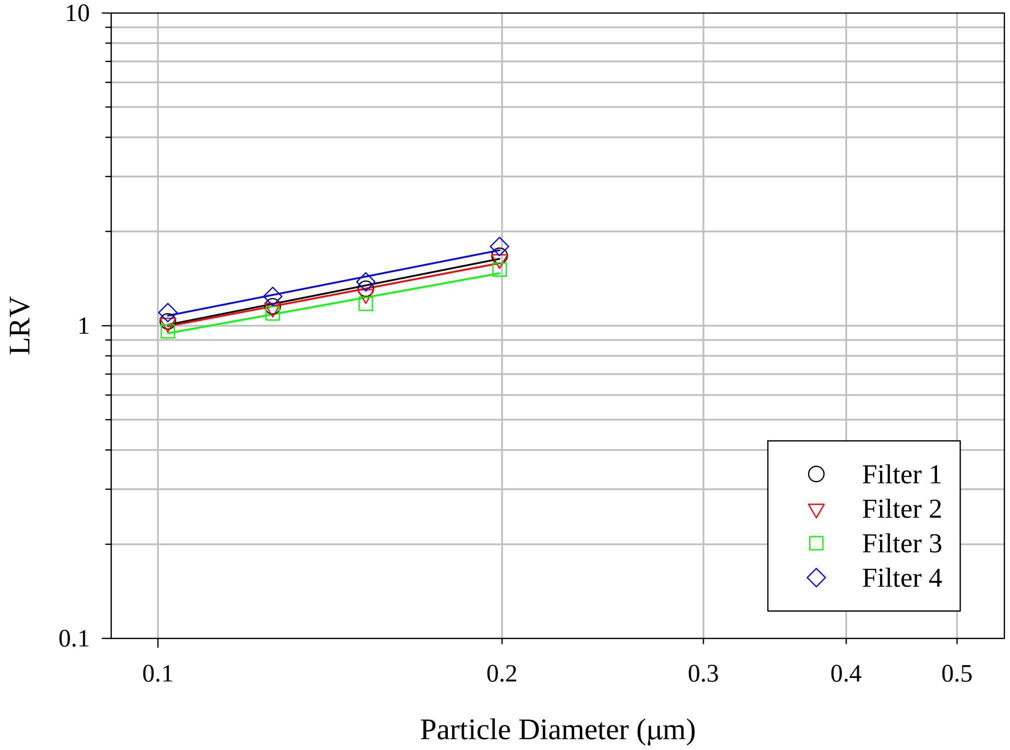

Figure 8. Initial retention of the four 0.1 µm Ulti-Etch filters used in this evaluation

Figure 9 presents retention as a function of particle size at polydisperse loadings equivalent to 0.1, 0.3 and 1.0 years of service while figure 10 presents the final particle retention (using monodisperse particle sizes) as a function of particle size for each pump. As expected, LRV increased with increasing particle size in each test and was linear when plotted as a function of particle size on a log-log scale. Like the previous experiment, filter particle retention the centrifugal pumps was highest at all of the filter loadings in this evaluation. Filter particle retention with the bellows pump was next highest. However, filter particle retention obtained with the diaphragm pump, which exhibited the largest pulsation of the pumps tested, was significantly lower than the other pumps, particularly at high filter loadings. Thus, the magnitude of the reduction in retention due to filter loading was more pronounced for the pumps with pumps having higher pulsation intensity.

Correlation between pulsation intensity and retention efficiency

Figure 11 presents the retention efficiency of both membrane filters from the initial experiment plotted as a function of pulsation intensity. Data from all three pumps are included. The retention efficiency is presented as the LRV of 125 nm PSL particles for each filter. Linear regressions are plotted for each type of filter regardless of the pump used. As anticipated, these data indicate that the LRV decreased as the pulsation intensity increased for both filter types. The LRV decreased from 0.7 (80% retention) to 0.3 (50% retention) when the pulsation intensity increased from 3% to 45%.

Also included in Figure 11 are the retention efficiencies of the Ulti-Etch filters after the one-year polydisperse challenge with each of the pumps. The slope of the regression line was similar to the slopes for the filters tested with low particle loadings, but the LRV values were lower due to the increased loading on the filters. The retention efficiency decreased from 70% to 25% when the pulsation intensity increased from 3% to 45%.

Figure 12 presents the same data as Figure 11, except the regressions are plotted for each pump/filter combination separately. (Regressions for the centrifugal pump data are not included.) These data indicate a good correlation between LRV and pulsation intensity for each pump-filter combination.

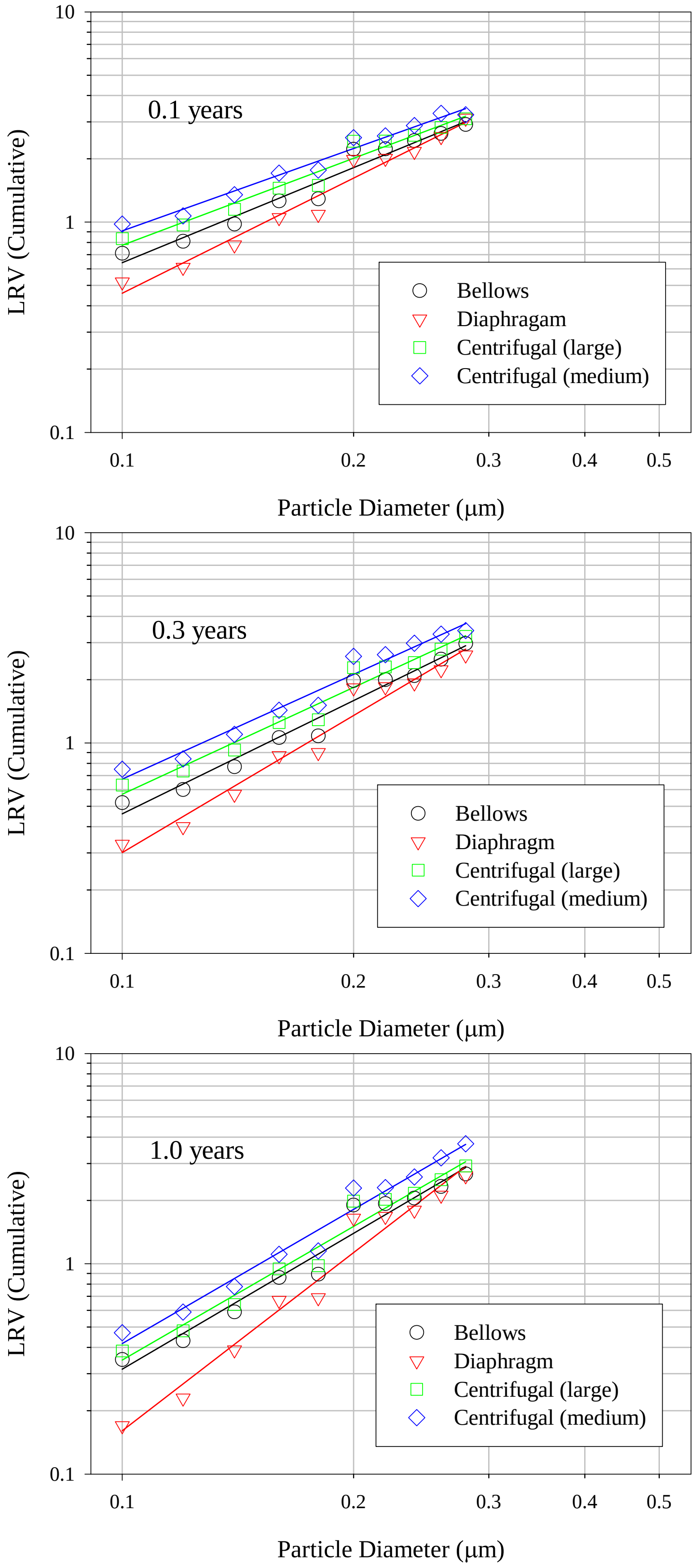

Figure 9. LRV as a function of particle size at loadings of 0.1, 0.3, and 1.0 years

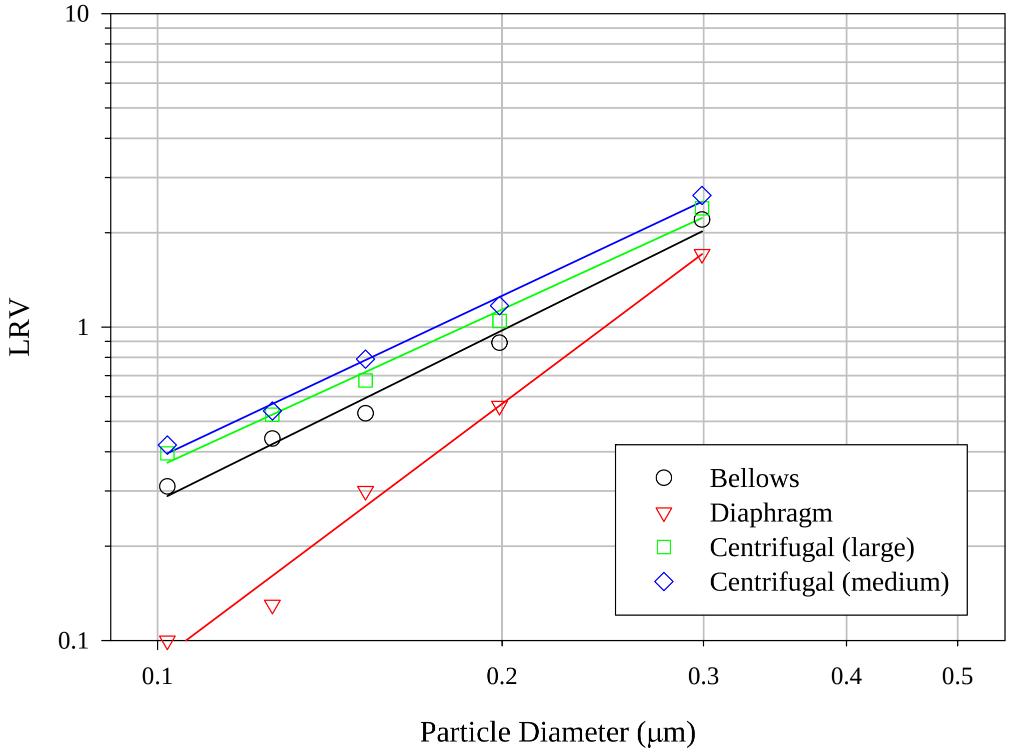

Figure 10. Final retention as a function of particle size for each pump

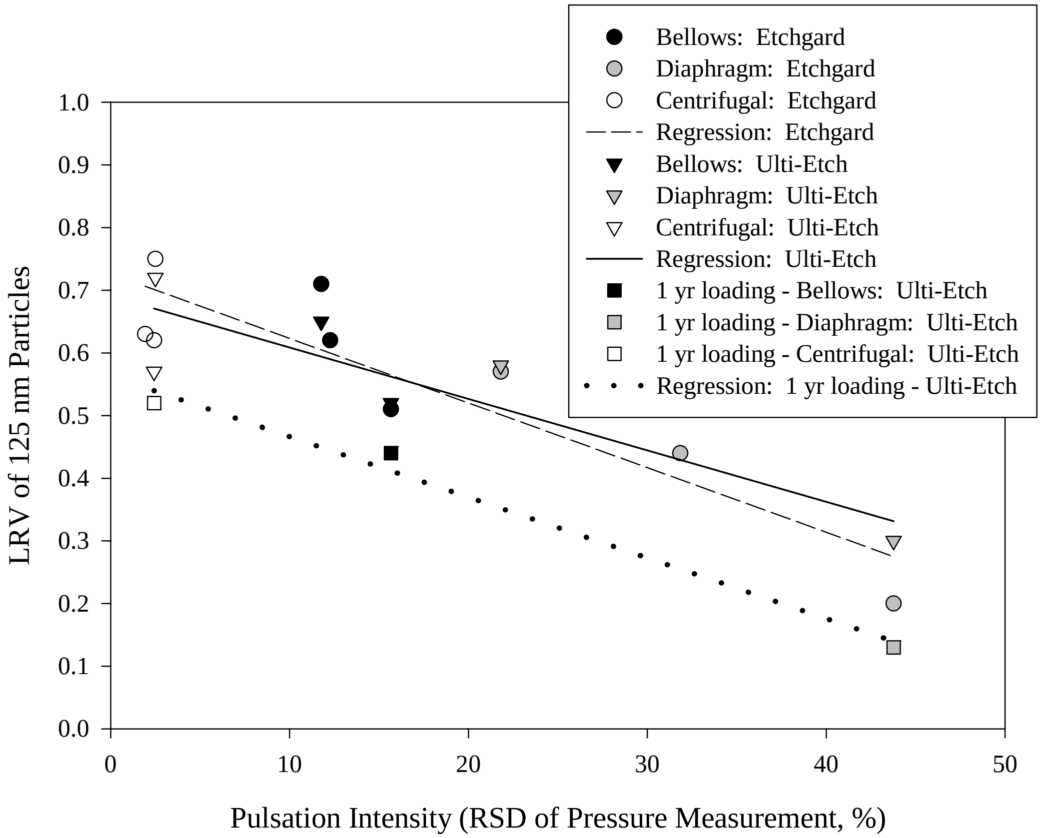

Figure 11. Overall retention efficiency as a function of pulsation intensity

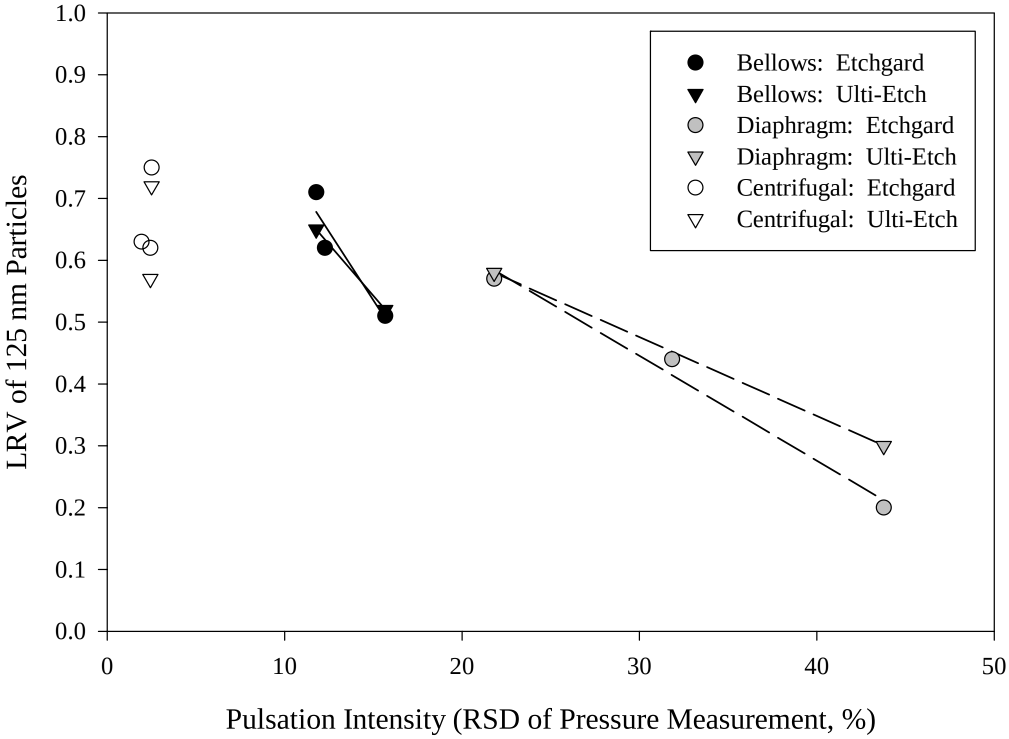

Figure 12. Retention efficiency of each filter type as a function of pulsation intensity

Summary

The effect of flow pulsations in pumping systems on filter particle retention was measured as a function of filter particle loading. Flow pulsation intensity was measured using a fast acting pressure transducer and assuming that flow pulsations are directly related to pressure pulsations. Three types of pumps were tested. Centrifugal pumps provided the lowest pulsation intensity. Pulsation intensities from the bellows and diaphragm pumps were approximately 6 and 15 fold higher, respectively. Pulsation intensity increased as flow rate delivered by the bellows and diaphragm pumps increased.

Filter retention decreased with increasing pulsation intensity and particle loading. At low particle loadings, the retention of 125 nm particles decreased from 80% to 50% as pulsation intensity increased, while at higher loadings, the retention of 125 nm particles decreased from 70% to 25%. The reduction in retention due to filter loading was highest for the pumps with the highest pulsation intensity.

Acknowledgments

Portions of this paper were published in the proceedings of the SCP 9th International Symposium on Wafer Cleaning and Surface Preparation, “Effect of pump pulsation on membrane filter retention”, (2005).

Bios

Mark Litchy is a research engineer for CT Associates. He has several years of experience in particle measurement and control in high purity liquid chemicals and gases and CMP slurry characterization. He is the author of more than 20 publications and presentations. He has a master’s degree in mechanical engineering from the University of Minnesota and a bachelor’s degree in physics from St. John’s University.

Reto Schoeb is the CEO and founding member of Levitronix GmbH, a pioneer in bearingless pump technology.

References

[1] Deal DB and DC Grant (1994). "Chemical Delivery Systems: Past, Present and Future, "Contamination Control and Defect Reduction in Semiconductor Manufacturing III, DN Schmidt, Ed., Proceedings Volume 94-9, The Electrochemical Society, Pennington, NJ, pp. 167-179.

[2] Grant DC and WR Schmidt, “Particle Performance of a Central Chemical Delivery System,” presented at the 7th Annual Millipore Microelectronics Technical Symposium, May 22, 1989.

[3] Grant, DC, and JG Zahka (1990). “Sieving Capture of Particles by Microporous Membrane Filters from Clean Liquids,” 10th International Symposium on Contamination Control (ICCCS 90), Zurich, Switzerland, pp. 160-164.

[4] Zahka JG and DC Grant (1991). "Predicting the Performance of Membrane Filters in Process Liquids Based on their Pore Size Rating," *Microcontamination*, 9(12):23-29.

[5] Zahka JG, DC Grant and C Myhaver (1990). "Modeling of Particle Removal from a Recirculating Etch Bath," Particles in Gases and Liquids: Detection, Characterization and Control, Plenum Press, New York.