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Phase II:
Comparison of Filter Lifetime during Circulation of Cabot Semi-Sperse® 12 Slurry with 3 Types of Pumps

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Introduction

In the first phase of this project, the effect of circulating Semi-Sperse® 12 slurry with three different types of pumps (bellows, diaphragm, and centrifugal) on the size distribution of the particles in the slurry was characterized. The large particle concentrations (≥ 0.56 μm - ≥ 10 μm) increased by at least a factor of 20 within 1,000 system turnovers for both the ASTI and Yamada pump systems. Meanwhile, for the Levitronix pump system, the cumulative particle concentrations ≥ 0.56 μm actually decreased by more than 40%, while the particles ≥ 1.0 μm remained unchanged within 1,000 system turnovers. The complete results of phase I are described in detail in a previous test report, "Comparison of Three Pump Systems on the Particle Size Distribution of Cabot Semi-Sperse® 12 slurry", CTA document #: LTX 775 0962, July, 2004.

In the second phase of this project, filter lifetimes during circulation of Cabot Semi-Sperse® 12 slurry with the same three pump systems were compared. This phase of testing was undertaken to determine whether the large particle generation by the ASTI and Yamada pumps has a significant effect on the lifetime of CMP filters.

Experimental

A few changes were made to the test system that was used in phase I to evaluate the effect of circulating Semi-Sperse® 12 slurry with each pump system on the slurry particle size distribution (PSD). A 10" PFA filter housing was installed immediately downstream of the pump or pump/pulse dampener combination. A new 10" Mykrolis Planargard™ CMP3 filter (cat. no. CMP301E06, lot no. CB4DNP3M2) was used during each test. The pressure drop (ΔP) across the filter was monitored with a differential pressure transducer. The pressure ports were plumbed directly into the filter housing on the upstream and downstream sides of the filter. The slurry was circulated until a ΔP increase of at least 10 psi was achieved or more than 10,000 system turnovers (or passes through the pump) were performed. A filter was considered to reach the end of its life when the ΔP increased by 10 psid.[1] Slurry sample ports on the upstream and downstream sides of the filter were also installed. A schematic of the test system is shown in Figure 1.

The same three types of pumps evaluated in phase I were tested in phase II, an ASTI bellows pump, a Yamada diaphragm pump, and a Levitronix magnetically levitated centrifugal pump. Table I shows the details of each pump. Manufacturer recommended pulse dampeners were installed downstream of both the ASTI and Yamada pumps to minimize pulsation.

Each pump was used to circulate approximately 28.5 liters of slurry at a flow rate of approximately 29.5 lpm (7.8 gpm). Settling of the slurry in the conical bottom tank was minimized by drawing from the bottom of the tank and by turning the volume of slurry in the tank over in less than one minute. The return line to the slurry tank was submerged below the liquid level of the slurry to avoid entraining air into the slurry. The return line was also positioned to minimize the formation of a large vortex in the tank that may entrain air into the slurry. No valves were used to generate back pressure at the outlet of the pump. Instead, ΔP across the filter and a short length of ½" PFA tubing downstream of the filter were sufficient to provide pump outlet pressures ranging from 22-37 psig (depending on the ΔP across the filter), which were similar to those experienced during phase I (30 psig). The air pressure supplied to the Yamada and ASTI pumps was adjusted to maintain a slurry flow rate of 29.5 lpm, while the Levitronix pump was operated over a range 5300-5750 rpm to maintain a constant flow rate. The test system was constructed of PFA, except for the conical bottom tank that was constructed of polyethylene. The slurry used in each test was taken from the same drum of slurry that was used in phase I.

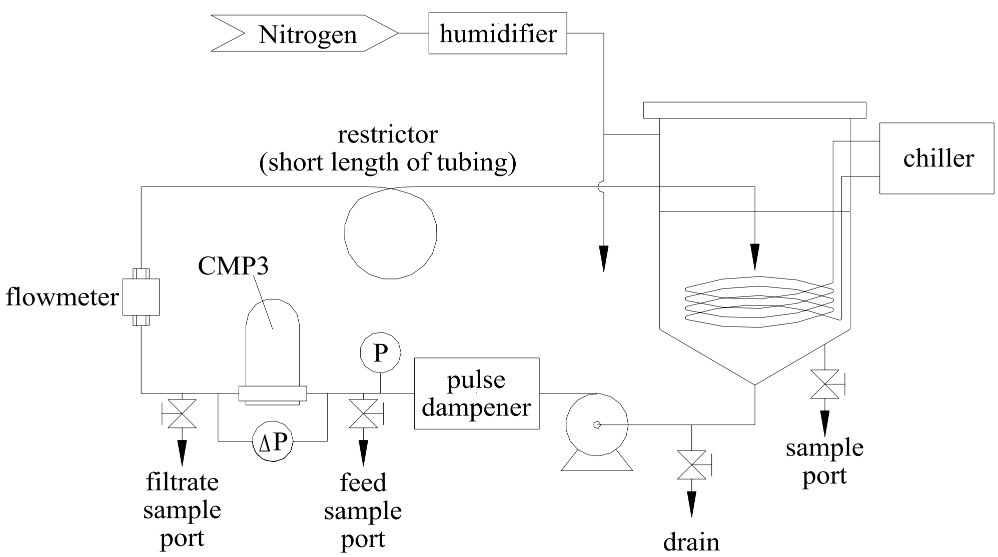
The tank holding the slurry was blanketed with nitrogen to prevent absorption of carbon dioxide from the air that can change the pH and chemical composition of the slurry. The nitrogen was humidified to prevent dehydration of the slurry. Shifts in the pH and dehydration can both result in particle agglomeration in the slurry. A chiller and stainless steel coil were used to maintain the slurry at 20 ± 2°C during the test. The relative humidity in the tank was > 90% throughout the test.

Samples were drawn from the system at selected times for analysis. The particle size distribution (PSD) was measured using 2 techniques. The size of the working particles was measured using a NICOMP 380ZLS (Particle Sizing Systems, Santa Barbara, CA) that determines particle size by dynamic light scattering. The size distribution of the large particle tail was measured using an AccuSizer 780 optical particle counter (Particle Sizing Systems, Santa Barbara, CA).

Table I. Specifications of the three pump systems

Pump and Pulse Dampener Manufacturer	Type of Pump	Model # of Pump	Pulse Dampener Used?	Model # of Pulse Dampener
ASTI	Bellows	PFD3 322S	Yes	AMC3 222A
Yamada	Diaphragm	DP20F-FT	Yes	AD-25TT
Levitronix	Centrifugal	BPS-4	No	NA

Figure 1: Test system schematic



Measurements made using the NICOMP 380ZLS were performed at 23°C on samples of slurry that were diluted approximately 40:1 into deionized water. Each sample was measured for 10 minutes. Triplicate measurements of each sample were made. The size measurement data were analyzed using the instrument's gaussian distribution assumption.

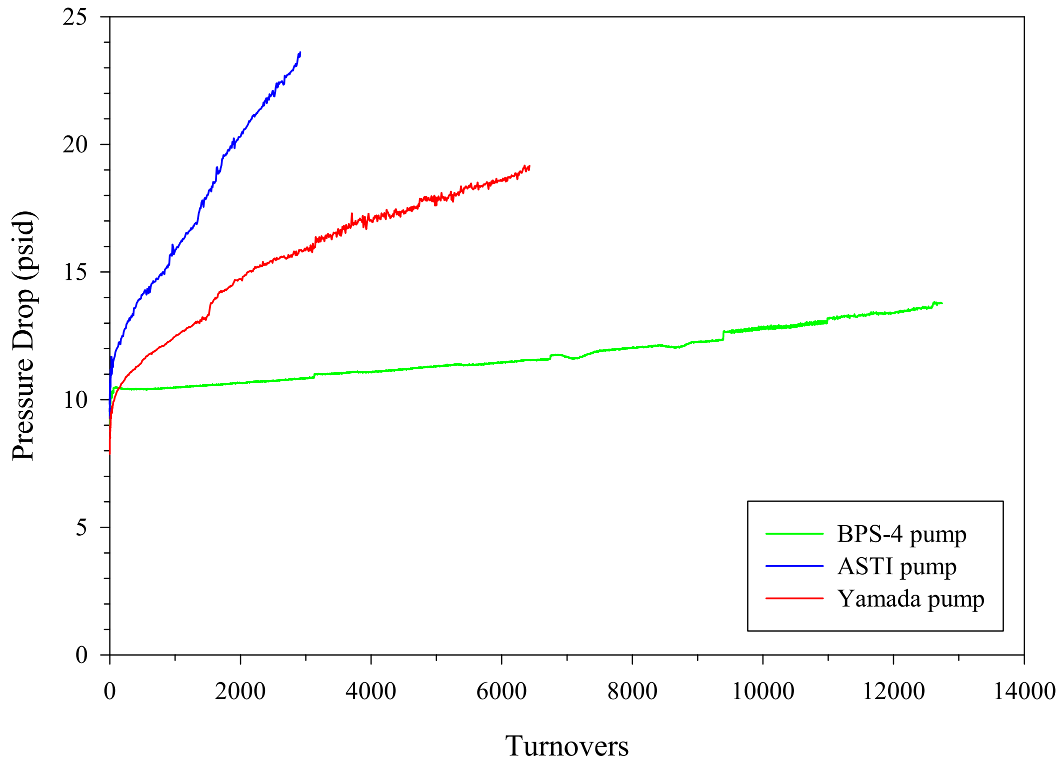
The size distribution of the large particle tail of the slurry was measured with an AccuSizer. This instrument uses a combination of light scattering and light extinction to measure the size distribution of particles ≥ 0.56 µm. The size measurements were performed by diluting the slurry sample by a factor of about 500:1 into deionized water. Between samples, the entire system was thoroughly flushed with deionized water. Data from selected particle size channels were analyzed.

Results

Pressure drop data:
The ΔP measured across each CMP3 filter as a function of tank turnovers (or passes through the pump) are presented in Figure 2 for the ASTI, Yamada, and Levitronix pumps. The data are plotted as a function of turnovers to be consistent with the data presentation in phase I test report referred to above. The initial ΔP measured across each filter was on the order of 10 psid at a flow rate of approximately 29.5 lpm. From this point, the ΔP across each filter increased roughly linearly during each test. The rate of ΔP increase was largest for the ASTI pump and smallest for the Levitronix pump.

Since the ΔP across a filter is a function of the flow rate through the filter, we attempted to maintain a constant flow rate of 29.5 lpm in each pump test. However, small flow rate adjustments were necessary as the filters clogged. These resulted in small variations in the ΔP. Additional details are presented in Appendix A.

Figure 2. ΔP across each filter for all 3 pump tests



In all of the tests, there was a relatively rapid increase in ΔP that occurred during the first 10 turnovers followed by a more gradual rate of increase thereafter. The ΔPs measured across each CMP3 filter during the beginning of each test are shown in Figure 3. The ΔP across each filter was recorded once a flow rate of 29.5 lpm was achieved. Upon closer examination, the 'initial' ΔPs were about 9.2, 8.0, and 9.0 psid for the ASTI, Yamada, and Levitronix pumps, respectively. The variation in the 'initial' ΔP is most likely attributed to variation in the filter media.

Figure 4 presents the increase in ΔP across the filter during the beginning of each test. This graph shows the rapid increase in the ΔP increase across each filter (~1.0-1.5 psid) during the initial 5-10 turnovers of each test. The increase in ΔP across the filter in each test was calculated by subtracting the 'initial' ΔP values from the subsequent ΔP measurements.

Figure 3. ΔP across each filter during the beginning of each test

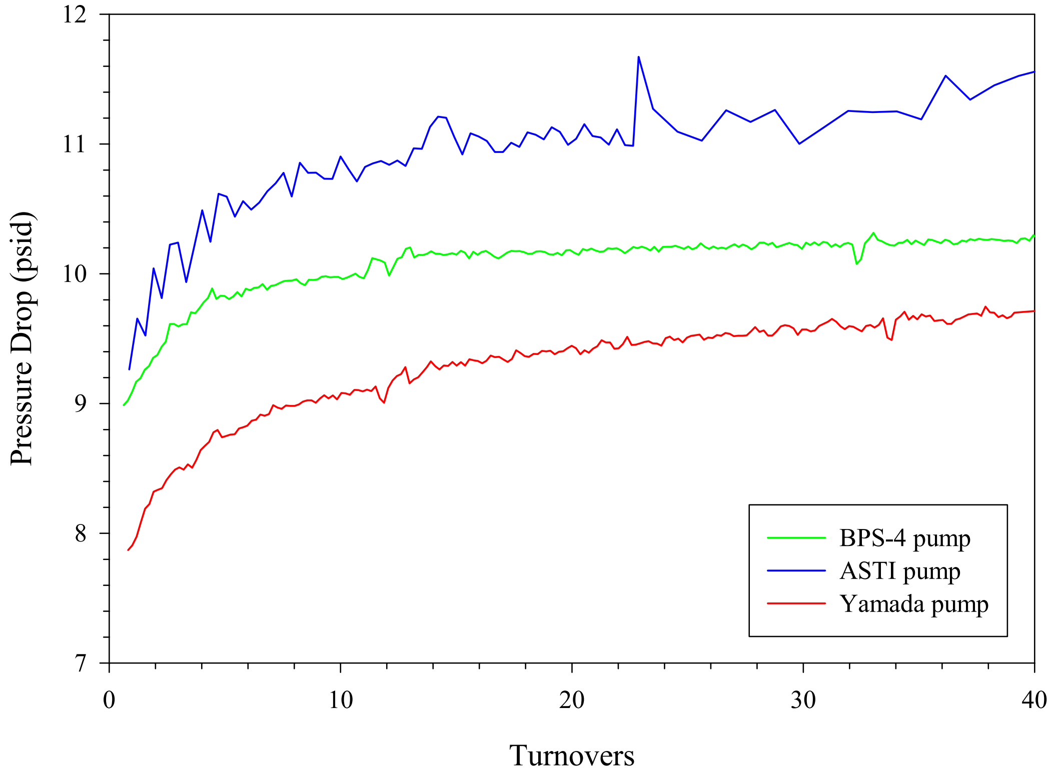


Figure 4. Increase in ΔP across each filter during the beginning of each test

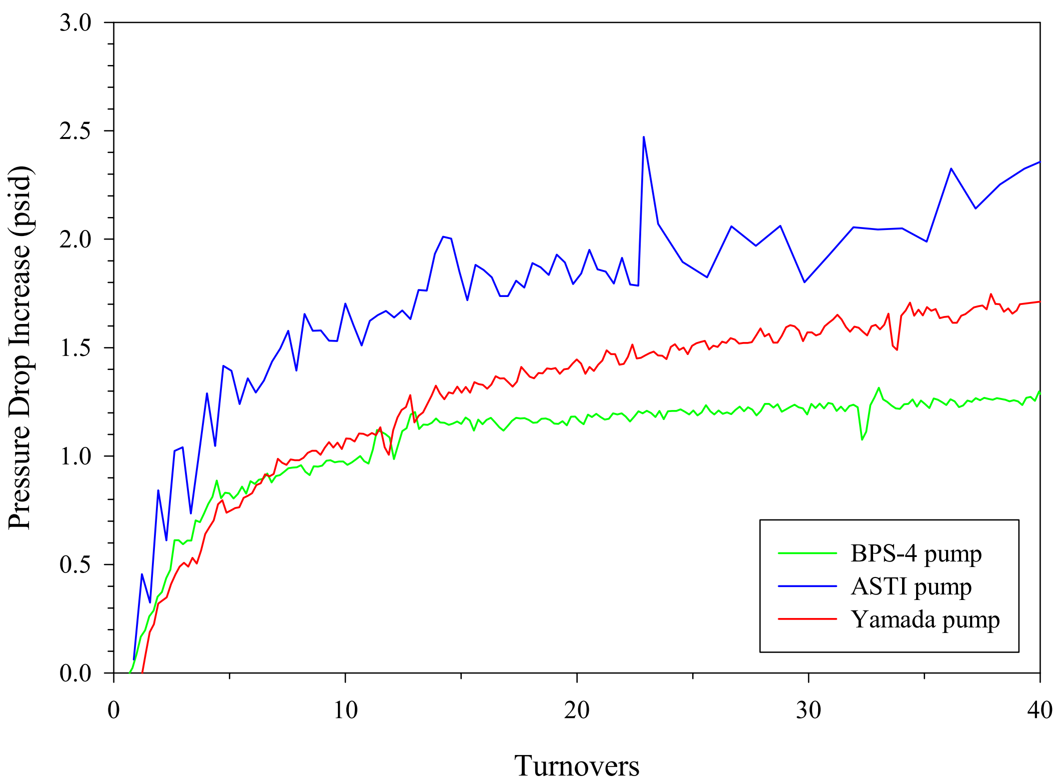


Figure 5 presents the increase in ΔP across the filter in each pump test during the entire test. It shows the rapid increase in the ΔP initially followed by a slow and fairly linear increase in ΔP during the remainder of the test. The increase in ΔP for the Levitronix pump was substantially less than the increase in ΔPs across the filters for the other pump systems. The slopes of linear regressions of each curve (not shown) were used to approximate the ΔP increase per turnover or ΔP increase per 1000 L of slurry passed through each pump as shown in Table II. Based on these slopes the rates of ΔP increase (relative to the Levitronix pump) were a factor 15.1 and 5.5 fold higher for the ASTI and Yamada pumps, respectively.

Figure 5. Increase in ΔP across each filter for all 3 pump tests

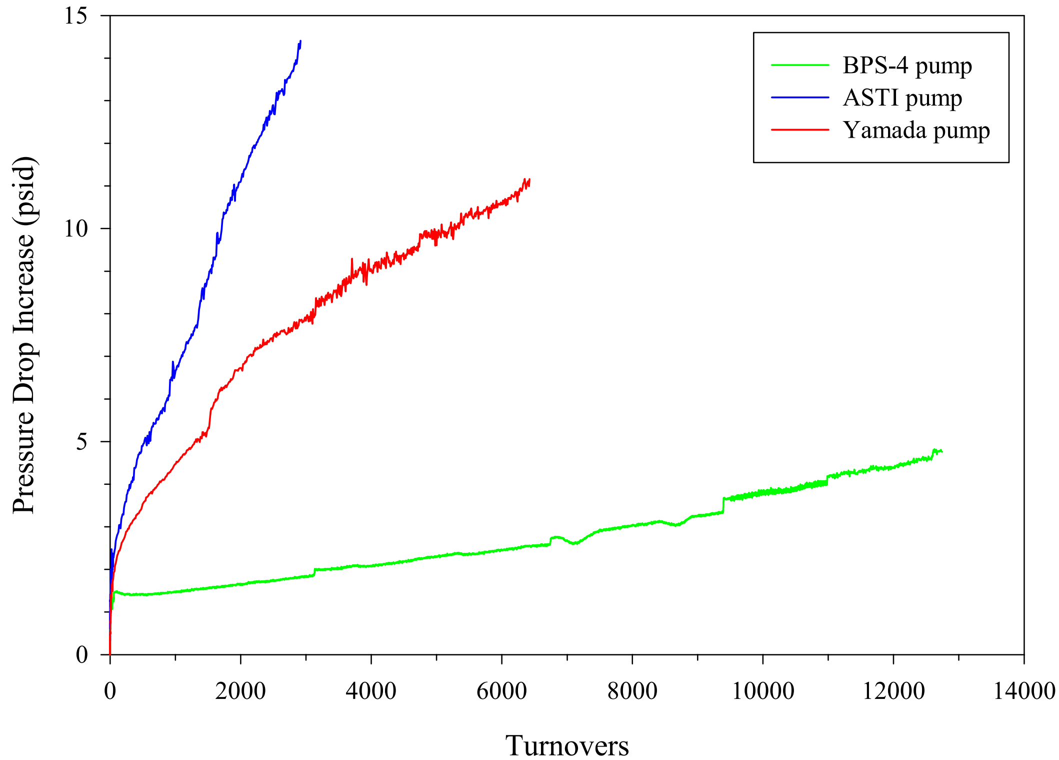


Table II. Rate of increase in ΔP for each pump

Pump Manufacturer	Type of Pump	ΔP Increase per Turnover (psi/turnover)	ΔP Increase per 1000 L (psi/1000L)	Rate of ΔP Increase (relative to Levitronix pump)
ASTI	Bellows	0.0044	0.15	15.1
Yamada	Diaphragm	0.0016	0.055	5.5
Levitronix	Centrifugal	0.00029	0.001	-

The ASTI and Yamada pump tests were continued until the ΔP across the filter increased by at least 10 psid. However, the Levitronix pump test was terminated before the ΔP could reach 10 psid since (1) the working particle size distribution was nearly out of specification (which will be discussed in further detail later), and (2) 10,000 turnovers had been exceeded.

Discussion

Initial ΔP increase:
The ΔP increase measured during the initial 5-10 turnovers was not due to the modification of the slurry due to the pumps, but rather was most likely due to removal of large particles from the as-received slurry. As the large particles were removed from the slurry during the first few passes through the pump, the rate of change in ΔP decreased. Measurements of the large particles in the slurry support this observation. Alternatively, the ΔP increase during the beginning of the test may be due to start up effects with the filter.

Subsequent linear ΔP increase:
The initial increase in ΔP likely depends on the volume of as-received slurry passed through the filter. However, the slow and fairly linear increase in ΔP during the remainder of each test is not only a function of the number of passes through the pump, but also a function of the volume of slurry passed through the pump and the filter. Although the data has been presented as a function of turnovers (or passes though the pump), it could also be presented as the volume passed through the pump.

In phase I of this project, the particle concentration increase was observed to be essentially linear with turnovers for both the Yamada and ASTI pumps. These results suggested that the Yamada and ASTI pumps generated a constant number of large particles per pump stroke, approximately 1.1 and 2.4 million particles ≥ 0.56 μm per pump stroke, respectively. Since the pumps deliver approximately 150 and 200 ml per stroke, this was equivalent to a concentration increase of about 7,030 and 11,800 per ml ≥ 0.56 μm per pump pass, respectively. Meanwhile in the Levitronix pump system, the particle concentration ≥ 0.56 μm remained relatively unchanged. Table III shows the approximate increase in concentration for various particle sizes for these pumps. (No value was included in Table III for particles ≥ 0.56 μm for the Levitronix pump since the concentration decreased during the initial 1000 turnovers, and then increased thereafter.)

Table III. Increase in concentration per pass through the pump

Particle Size	Concentration Increase (#/ml/turnover)		
	ASTI pump	Yamada pump	Levitronix pump
≥ 0.56 μm	11,800	7,030	-
≥ 1.0 μm	2,050	1,340	< 2
≥ 2.0 μm	367	270	< 1
≥ 5.0 μm	57	42	< 0.2
≥ 10 μm	5.4	4.6	< 0.1

Although this appears to be a rather large amount of particle agglomeration, the particle generation must be put into perspective relative to the particle concentration in the filtrate (approximately 150,000 particles/ml ≥ 0.56 μm). It is essentially impossible to distinguish a 5-8% difference between the feed and filtrate, and thus the large particle concentrations measured in the feed and filtrate are essentially indistinguishable after about 3 turnovers. However, the increase has a significant effect on filter lifetime.

Since the concentration increase per pass through each pump generated by the ASTI pump in phase I was 1.2-1.8 times that of the Yamada pump depending on the particle size, we expected the increases in ΔP to be slightly higher for the ASTI pumps. The ASTI pump required about 1725 turnovers to achieve a ΔP increase of 10 psig while the Yamada pump required 5000 turnovers to achieve the same ΔP increase, which is nearly a factor of 3 difference in filter lifetime. Several factors may contribute to this difference including differences in the concentrations and sizes of particles generated by each pump, differences in the shape of the particles or gels generated, slightly different pump operating conditions between phase I and phase II, variation in filters, etc.

Summary

A Yamada diaphragm pump, an ASTI bellows pump, and a Levitronix centrifugal pump were tested to determine how their use affected the lifetime of CMP3 filters in Semi-Sperse® 12 slurry. The tests were continued until the ΔP across the filter increased by at least 10 psig or more than 10,000 turnovers in the system were achieved.

The ASTI pump required about 1725 turnovers to achieve a ΔP increase of 10 psig while the Yamada pump required 5000 turnovers to achieve the same ΔP increase, which is nearly a factor of 3 difference in filter lifetime. Meanwhile, the Levitronix pump required 12,735 turnovers to achieve a ΔP increase of 4.75 psig (the maximum ΔP measured with the Levitronix pump system). The ΔP increases measured with the ASTI and Yamada pumps were approximately 15 and 5 times higher than those experienced with the Levitronix pump.

Previous tests (phase I) showed that the Levitronix pump generated significantly fewer particles ≥ 1.0 µm than the other pumps. The large particle concentrations (≥ 0.56 µm - ≥ 10 µm) increased by at least a factor of 20 within 1,000 system turnovers for both the ASTI and Yamada pump systems. Meanwhile, for the Levitronix pump system, the cumulative particle concentrations ≥ 0.56 µm actually decreased by more than 40%, while the particles ≥ 1.0 µm remained unchanged within 1,000 system turnovers. Hence, these pressure drop results are consistent with changes in the large particle concentration results measured in phase I.

References

1. Z. Lin, G.Vasilopoulos, K. Devriendt, M. Meuris, "Cost effective POU slurry filtration using Solaris™ filter for CMP defect reduction", SEMICON® West 99 Technical Program: CMP Technology for ULSI Interconnection, July 13, 1999.

Appendix A

Effect of variations in flow rate on ΔP:

Figures A1-A3 show the effect of ΔP across the filter and slurry flow rate through the filter during each test. Since the ΔP across a filter is a function of the flow rate through the filter, we attempted to maintain a constant flow rate of 29.5 lpm in each pump test. As the ΔP across a filter increased, the flow rate decreased. Thus, small adjustments in the flow rate were necessary as the filter clogged. The air pressure supplied to the Yamada and ASTI pumps or the speed of the Levitronix pump were periodically adjusted to maintain a constant flow rate of slurry. These resulted in small variations in the ΔP. The mean and standard deviation of the flow rates measured during each pump test were 29.3 ± 0.3, 29.5 ± 0.2, and 29.6 ± 0.2 lpm for the ASTI, Yamada, and Levitronix pumps, respectively.

Figure A1. Effect of ASTI pump on ΔP across a CMP3 filter

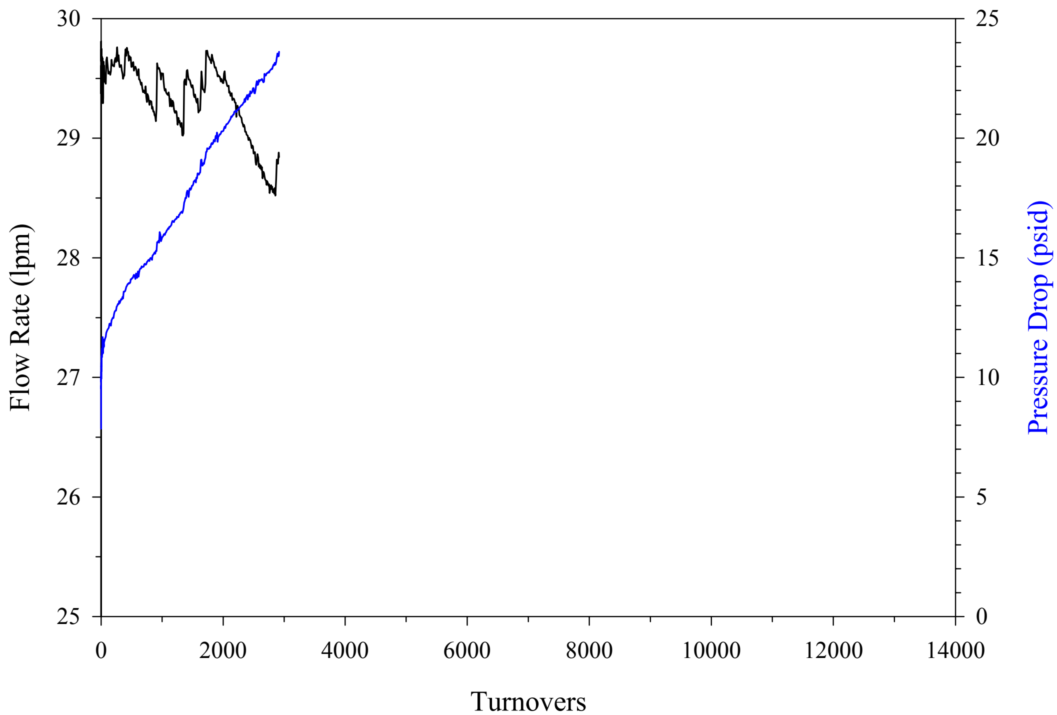


Figure A2. Effect of Yamada pump on ΔP across a CMP3 filter

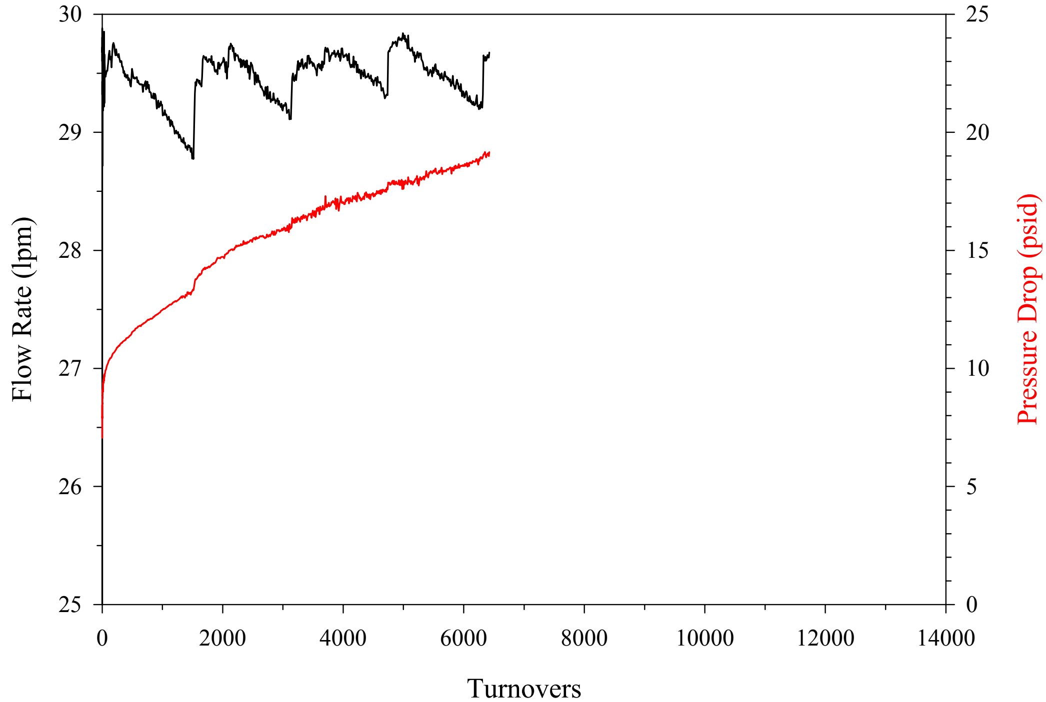
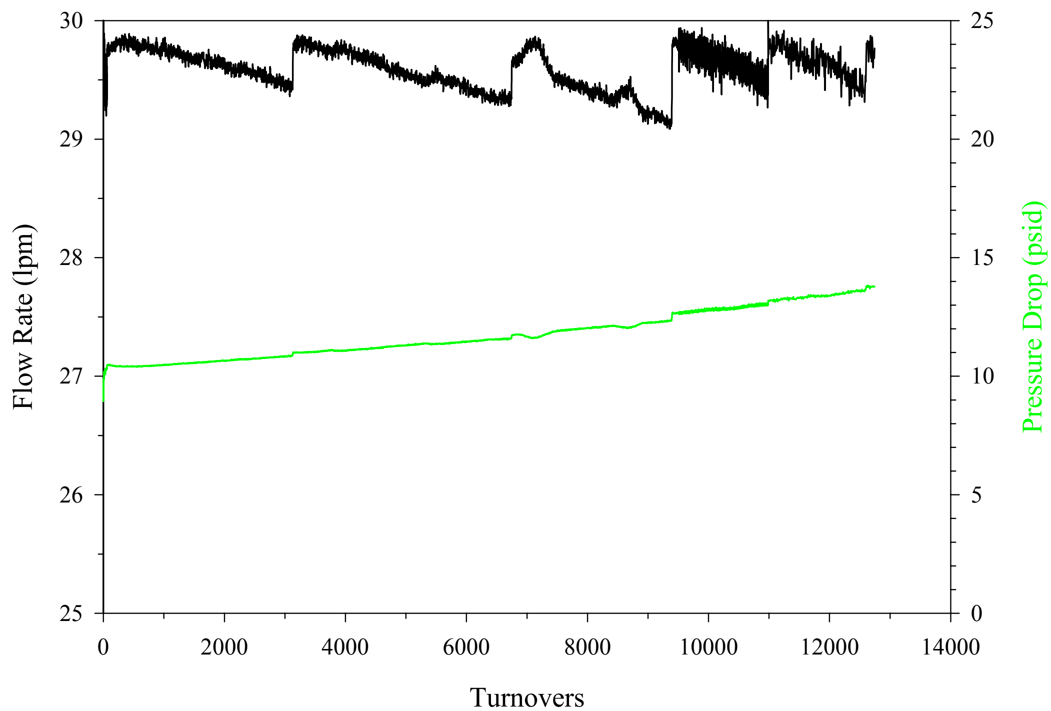


Figure A3. Effect of Levitronix pump on ΔP across a CMP3 filter



The ΔP measured across each CMP3 filter during the beginning of each test are shown in Figures A4-A6 for the ASTI, Yamada, and Levitronix pumps, respectively. Approximately one turnover was needed to adjust the pumps to the desired flow rate of 29.5 lpm. Once this flow rate was achieved, only minor adjustments in the pump air pressure or speed were required.

Figure A4. ΔP across filter during beginning of ASTI pump test

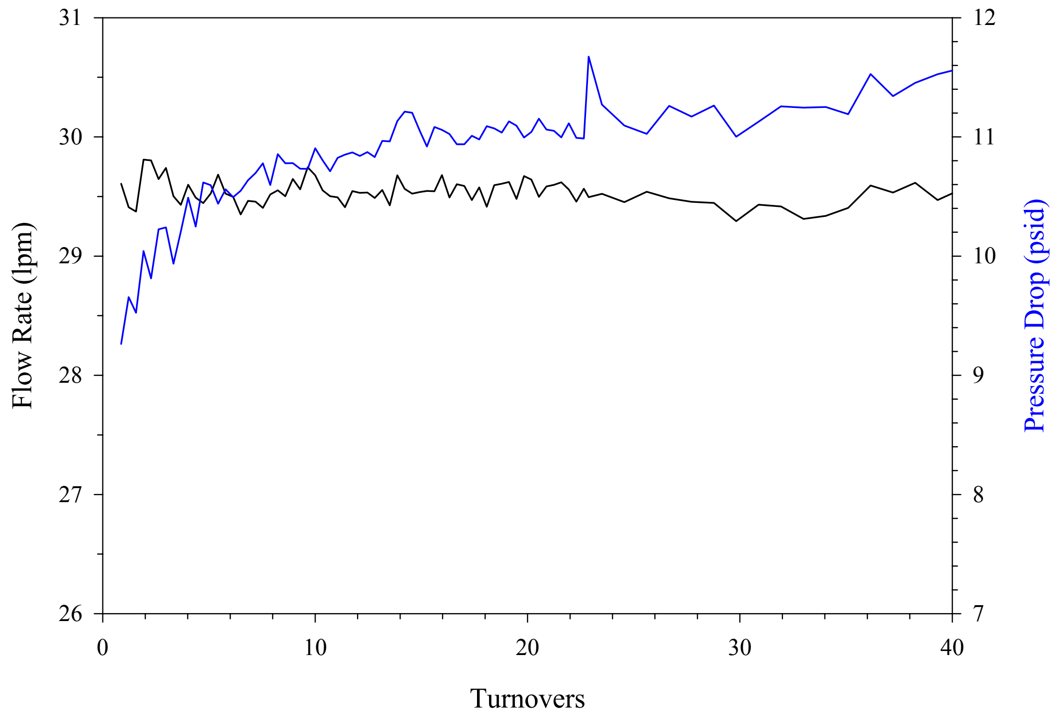


Figure A5. ΔP across filter during beginning of Yamada pump test

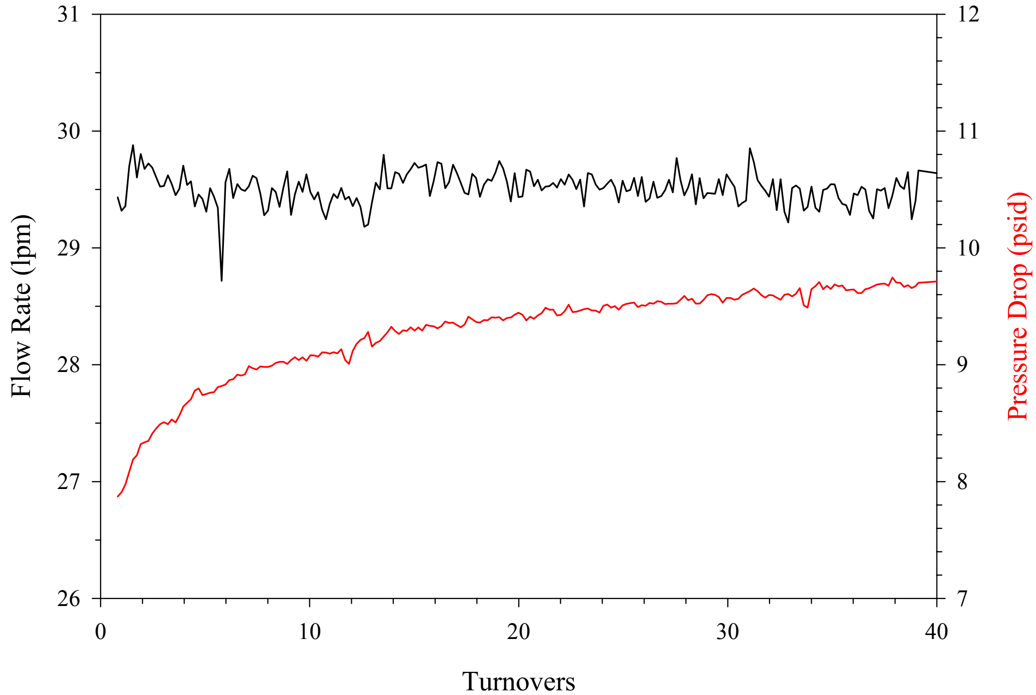


Figure A6. ΔP across filter during beginning of Levitronix pump test