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Comparison of Four Pump Systems on the Particle Size Distribution of Cabot EP-C6618 Slurry

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Introduction

Delivery systems are often used to supply the slurry used to planarize wafers during semiconductor chip manufacturing. These systems pressurize the slurry to deliver it to the tools and circulate it to help keep the particles in suspension. Pressurization and circulation are accomplished by various means including a variety of types of pumps and pressure-vacuum technology. Typically, the slurry passes through the equipment providing the motive force approximately 100 times before it is used to polish wafers, i.e. the slurry is “turned-over” approximately 100 times [1]. Some CMP slurries may be damaged due to the mechanical handling of the slurry. For example, particle agglomerates may form which can limit the life of filters or even reduce yield by causing wafer defects.

Previous experiments have shown significant increases in large particle concentrations when certain types of pumps (diaphragm and bellows) were used to circulate slurry, while magnetically levitated centrifugal pumps (Levitronix BPS-3 and BPS-4) did not [2-5]. However, these tests were performed using only one type of slurry, Cabot Semi-Sperse® 12, which was known to be sensitive to particle agglomeration. In practice, there are many CMP planarization applications incorporating a variety of slurry types.

This test was performed to determine the effect of circulating Cabot EP-C6618 (Cabot Microelectronics Corporation, Aurora, IL), a silica oxide aqueous dispersion, with four different pumps (bellows, diaphragm, and medium and large magnetically levitated centrifugal) on the health of the slurry. During this test, a number of slurry health parameters were monitored including the size distribution of the particles in the slurry.

Significant differences in the large particle tail of the slurry particle size distributions (PSD) were observed after circulation with the different types of pumps, while minimal changes were observed in other slurry health parameters. Large changes in the large particle tail were observed during circulation with diaphragm and bellows pumps, while relatively minimal changes were observed with the centrifugal pumps.

Experimental

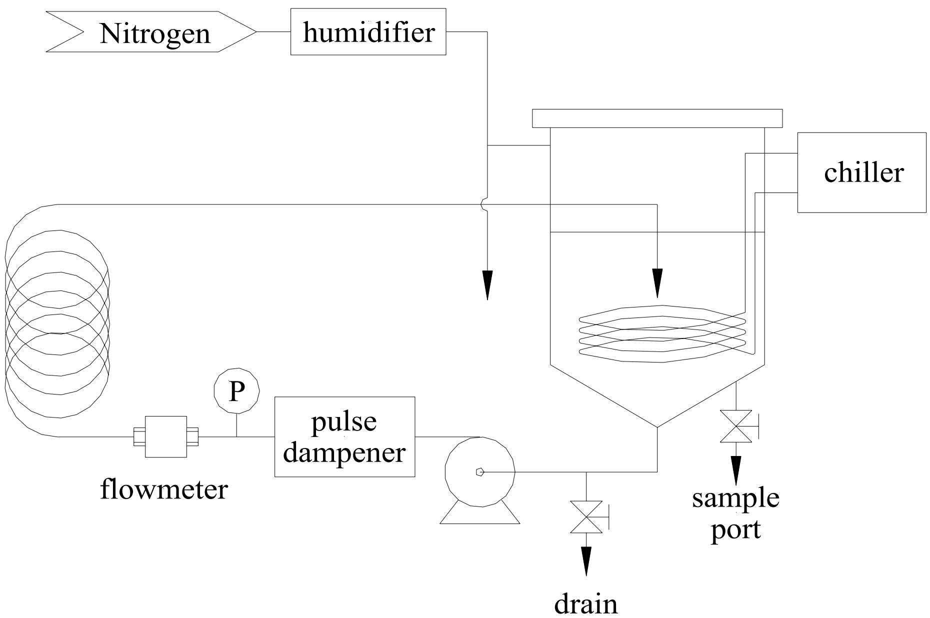
The pumps evaluated in this test were: an ASTI bellows pump, a Yamada diaphragm pump, and two Levitronix magnetically levitated centrifugal pumps. Table I shows the details of each pump. Manufacturer recommended pulse dampeners were installed downstream of both the ASTI and Yamada pumps to minimize pulsation. A schematic of the test system is shown in Figure 1.

Table I. Specifications of the four 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-3	No	NA
Levitronix	Centrifugal	BPS-4	No	NA

Each pump was used to circulate 11 liters of slurry at a flow rate of approximately 26 lpm (6.9 gpm) and outlet pressure of 29 psig (2.0 bar). Settling of the slurry in the tank was minimized by drawing from the bottom of a conical bottom tank and by turning the volume of slurry in the tank over in less than 30 seconds. The return line to the slurry tank was submerged below the liquid level of the slurry to avoid entraining gas into the slurry. The return line was also positioned to minimize the formation of a large vortex in the tank that may entrain gas into the slurry. No valves were used to generate back pressure at the outlet of the pump. Instead, a long length of ½” PFA tubing was used to gradually reduce the pressure from 29 psig at the pump outlet to ambient pressure at the end of the return line to the tank. The air pressure supplied to the Yamada and ASTI pumps was adjusted to achieve a slurry flow rate of 26 lpm with a 29 psig outlet pressure, while the BPS-3 and BPS-4 pumps were operated at 7000 and 5650 rpm, respectively. The slurry was circulated until more than 3,000 tank turnovers were achieved. 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 lot of slurry.

Figure 1: Test system schematic



The tank holding the slurry was blanketed with nitrogen to prevent absorption of carbon dioxide from the air that can change the pH 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 24 ± 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 Particle Sizing Systems NICOMP 380ZLS (Santa Barbara, CA) that determines particle size by dynamic light scattering. The size distribution of the large particle tail was measured using a Particle Sizing Systems AccuSizer 780 optical particle counter.

The AccuSizer 780 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 1600:1 into deionized water. Between samples, the entire system was thoroughly flushed with deionized water. Data from selected particle size channels were analyzed.

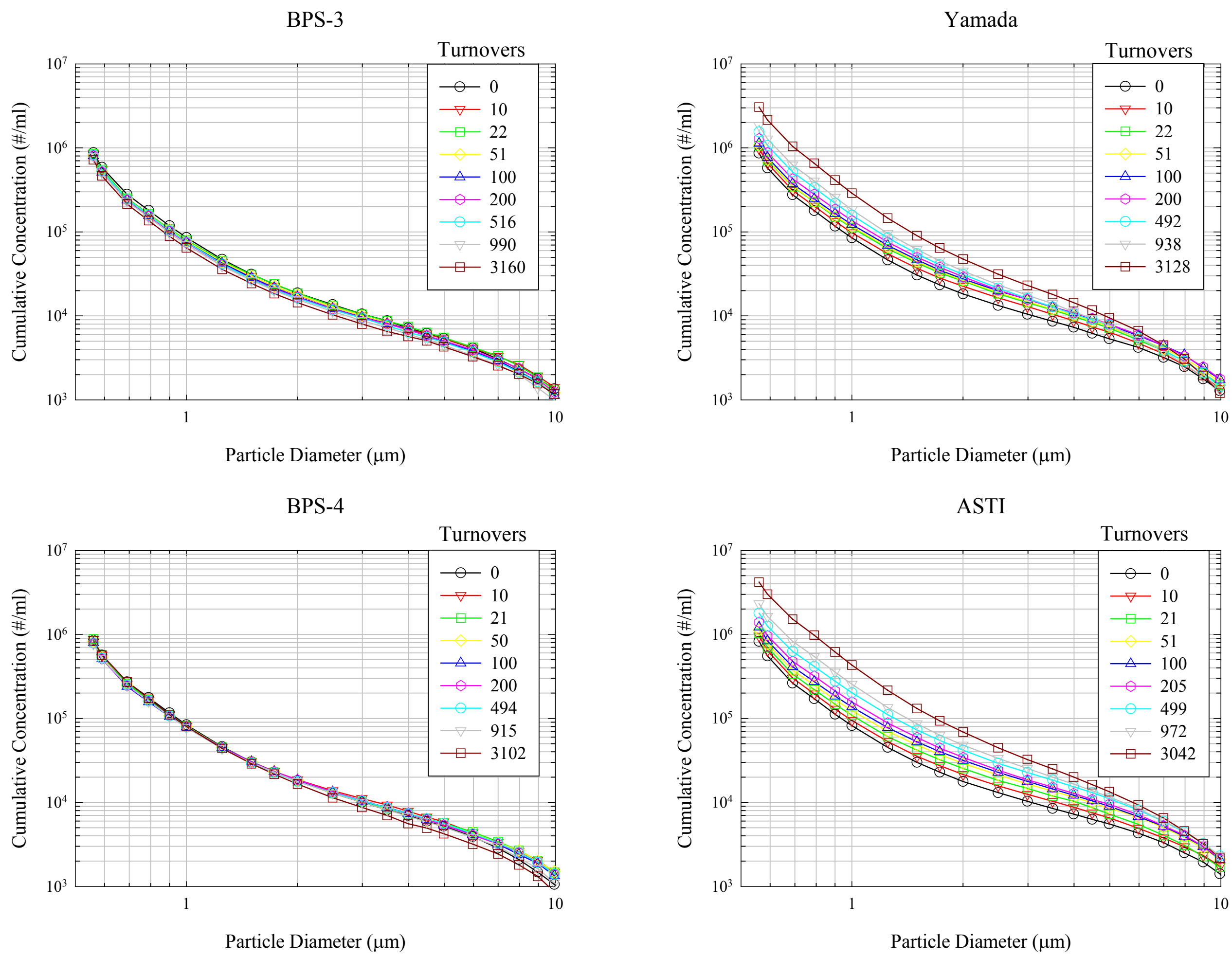
The zeta potential and working PSD measurements were made using the NICOMP 380ZLS. The samples were diluted approximately 40:1 into deionized water and analyzed at 23°C. Each PSD measurement was made over 10 minutes while each zeta potential measurement was made over 2 minutes. The PSD and zeta potential measurements were performed in triplicate quintuplicate, respectively. The size measurement data were analyzed using the instrument's gaussian distribution assumption.

Other slurry health parameters measured included zeta potential, total percent solids, specific gravity, and pH. All measurements were performed in triplicate.

Results and Discussion

The four graphs in Figure 2 show the cumulative particle size distributions (PSD) of the slurry large particle tail measured during each pump test. Each graph presents the results from a different pump. The initial PSD, measured prior to the start of each test, is presented in each graph as well as PSDs after selected numbers of turnovers. Little change in the PSD was observed during the BPS-3 and BPS-4 tests, while concentrations of particles ≥ 0.56 µm increased during the Yamada and ASTI pump tests.

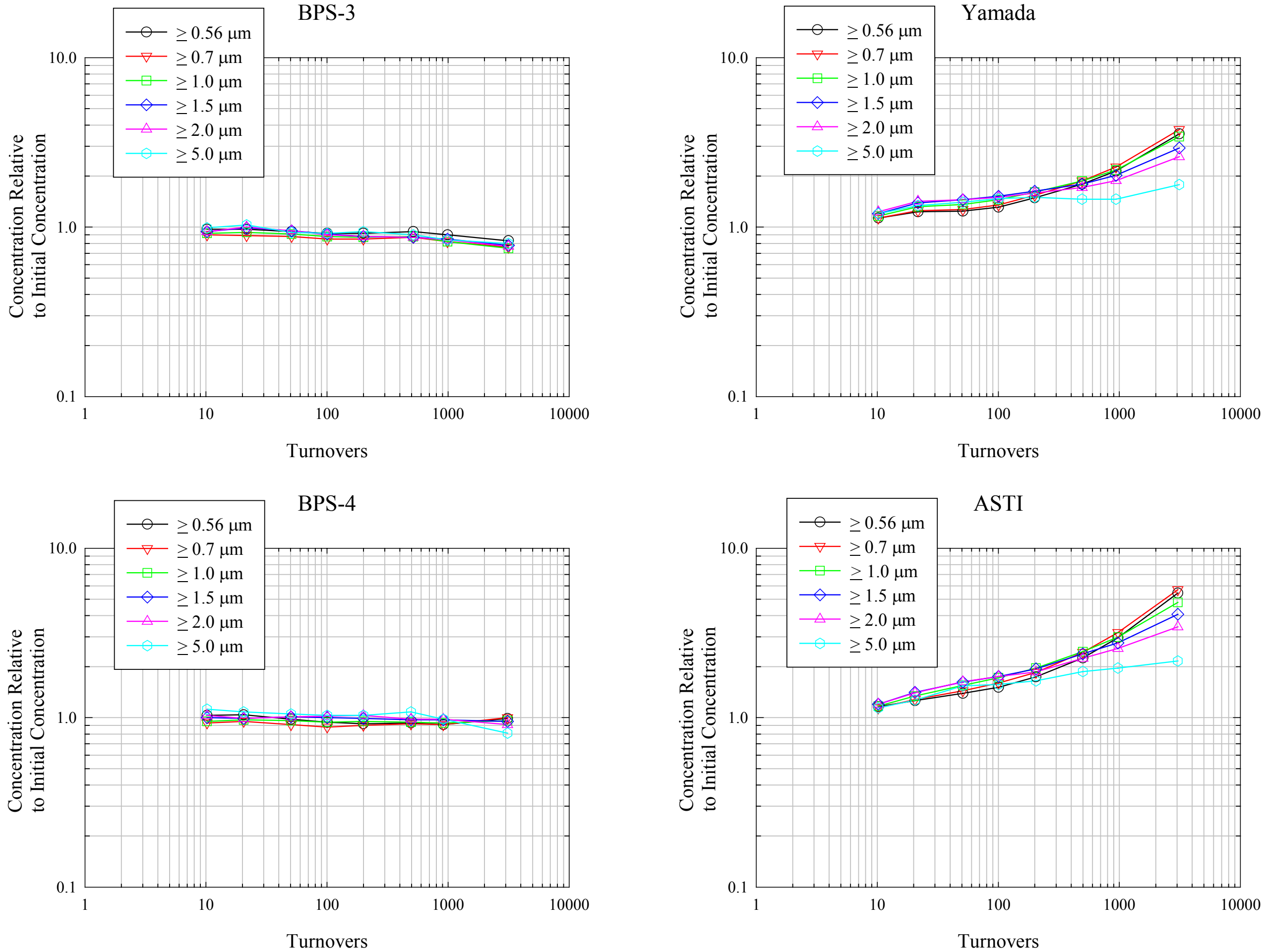
Figure 2: Cumulative PSDs of the large particle tail measured during each pump test



Figures 3 and 4 present the ratio of the particle concentrations at each test point to the initial concentration. Each graph presents the results from each pump for a selection of size channels as a function of tank turnovers. Figure 3 presents the data on logarithmic axes while Figure 4 presents the data on linear axes.

Concentrations of large particles (≥ 0.56 µm) increased with increasing passes through both the Yamada and ASTI pumps. The rate of increase was non-linear with increasing turnovers. For smaller particles, those on the order of 0.56 µm, the concentration increase was nearly linear, but as particle size increased, the rate of change in concentration decreased with increasing turnovers.

Figure 3: Particle concentrations relative to the initial concentration for selected size channels (logarithmic axes)



Relatively small changes were observed in the PSD during the BPS-3 and BPS-4 pump tests relative to the other two pumps. A slight decrease in the particle concentrations (up to 25%) was observed during the BPS-3 and BPS-4 tests. Meanwhile, particle concentrations increased by factors of up to 3.8 and 5.8 depending on particle size during the Yamada and ASTI pump tests, respectively.

Table II shows a summary of the relative particle concentration changes during each test after approximately 100 and 1,000 turnovers for selected particle sizes. The particle concentration increase was largest for smaller particles (~ 0.56-1.0 µm). As the particle sizes increased, the concentration increase became less pronounced. After 1,000 turnovers, concentrations of particles ≥ 1 µm increased by a factor of about 2 and 3 for the Yamada and ASTI pumps, respectively. For particles ≥ 5 µm, the concentration increase was only a factor of 1.5 and 2 and for particles ≥ 10 µm, the concentration increase was 1.0 and 1.6 for the Yamada and ASTI pumps, respectively. Meanwhile, the particle concentrations tended to decrease slightly for both Levitronix pumps.

Figure 4: Particle concentrations relative to the initial concentration for selected size channels (linear axes)

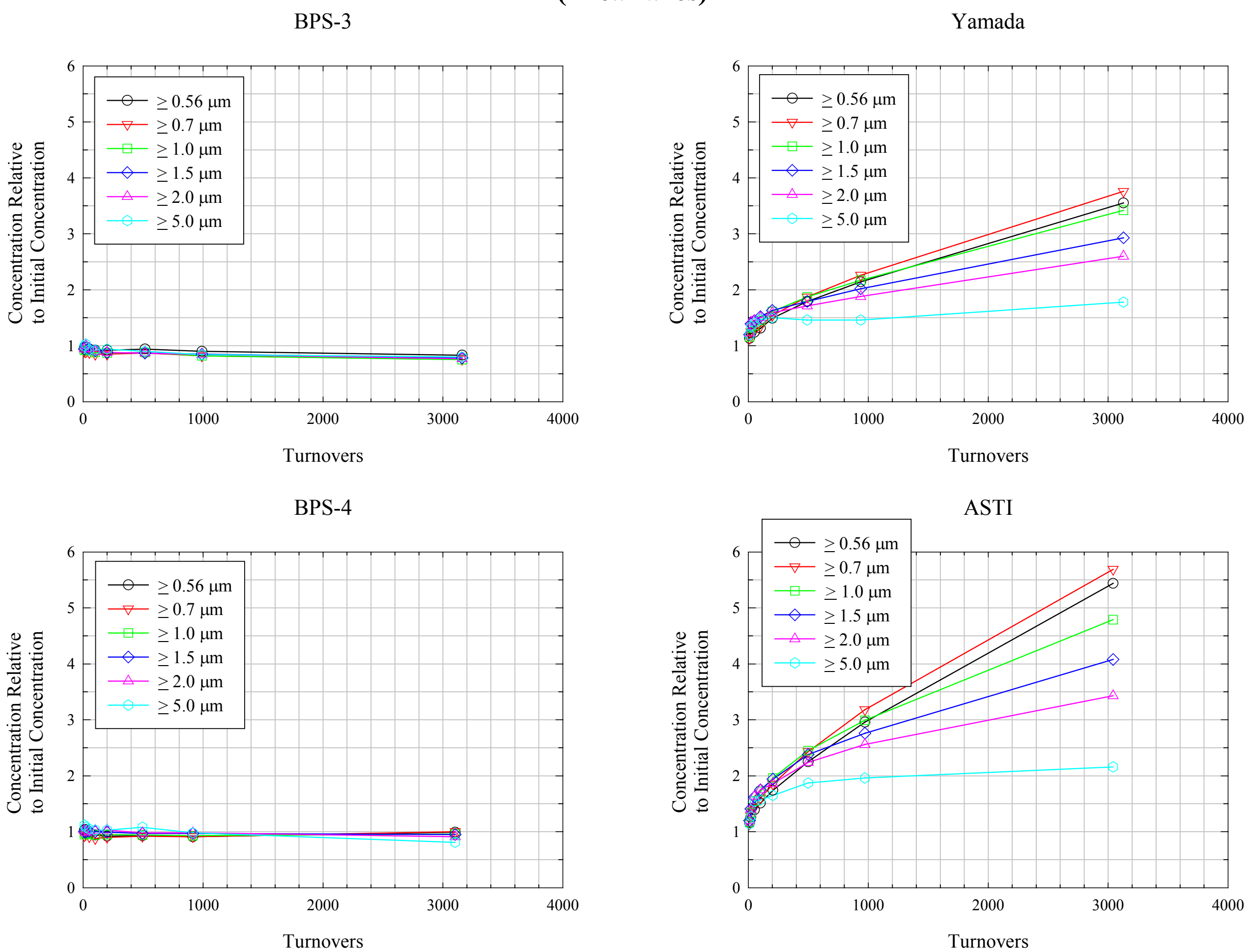


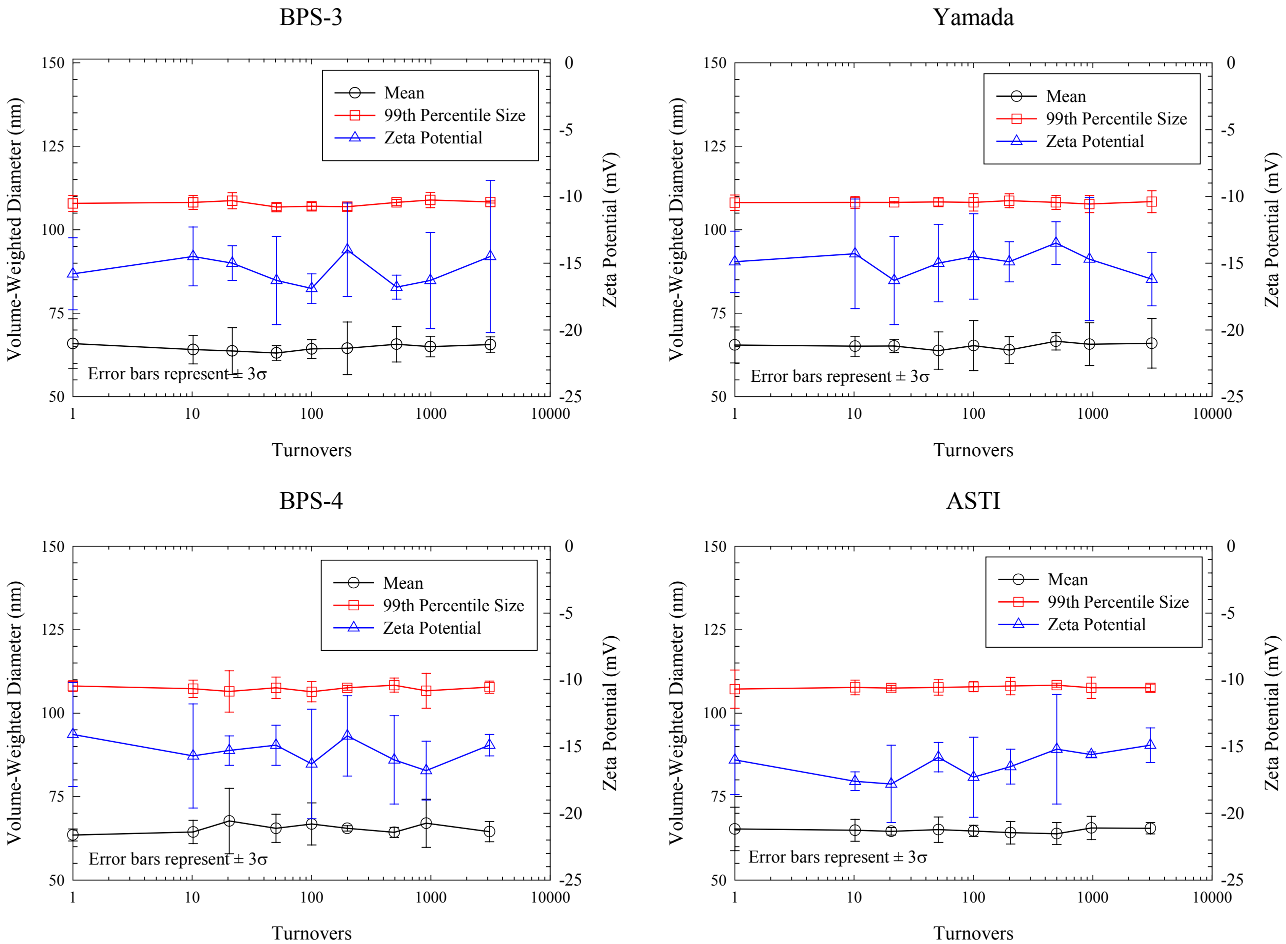
Table II. Summary of the relative particle concentrations changes for selected times and particle sizes

Particle Concentrations Relative to the Initial Particle Concentrations								
Particle Size	100 Turnovers				1,000 Turnovers			
	BPS-3	BPS-4	Yamada	ASTI	BPS-3	BPS-4	Yamada	ASTI
≥ 0.56 µm	0.9	0.9	1.3	1.5	0.9	0.9	2.1	2.8
≥ 1.0 µm	0.9	0.9	1.4	1.7	0.8	0.9	2.2	3.1
≥ 1.5 µm	0.9	1.0	1.5	1.8	0.8	1.0	2.0	2.9
≥ 2.0 µm	0.9	1.0	1.5	1.8	0.8	1.0	1.9	2.7
≥ 5.0 µm	0.9	1.0	1.5	1.6	0.8	1.0	1.5	2.1
≥ 10 µm	0.8	1.3	1.4	1.5	0.7	1.0	1.0	1.6

Figure 5 shows the volume-weighted mean and 99th percentile particle diameters (99% of the particles have diameters less than this size) of the working PSD as a function of tank turnovers. Error bars are included and represent ± 3 standard deviations. The volume-weighted mean and 99th percentile particle diameters at the start of each test were 65 and 108 nm, respectively. No significant changes in the volume-weighted mean or 99th percentile particle diameters were observed for any of the pumps during the tests. Also included in Figure 5 were the zeta potential measurements taken during each test. No significant change in the zeta potential was observed for any of the pumps during the tests.

Figure 6 shows the total percent solids, specific gravity, and pH as a function of tank turnovers during each test. Error bars are included in each graph and represent ± 3 standard deviations. No changes in these slurry health parameters were observed for any of the pumps during the tests.

Figure 5: Working particle size and zeta potential measurements as a function of turnovers



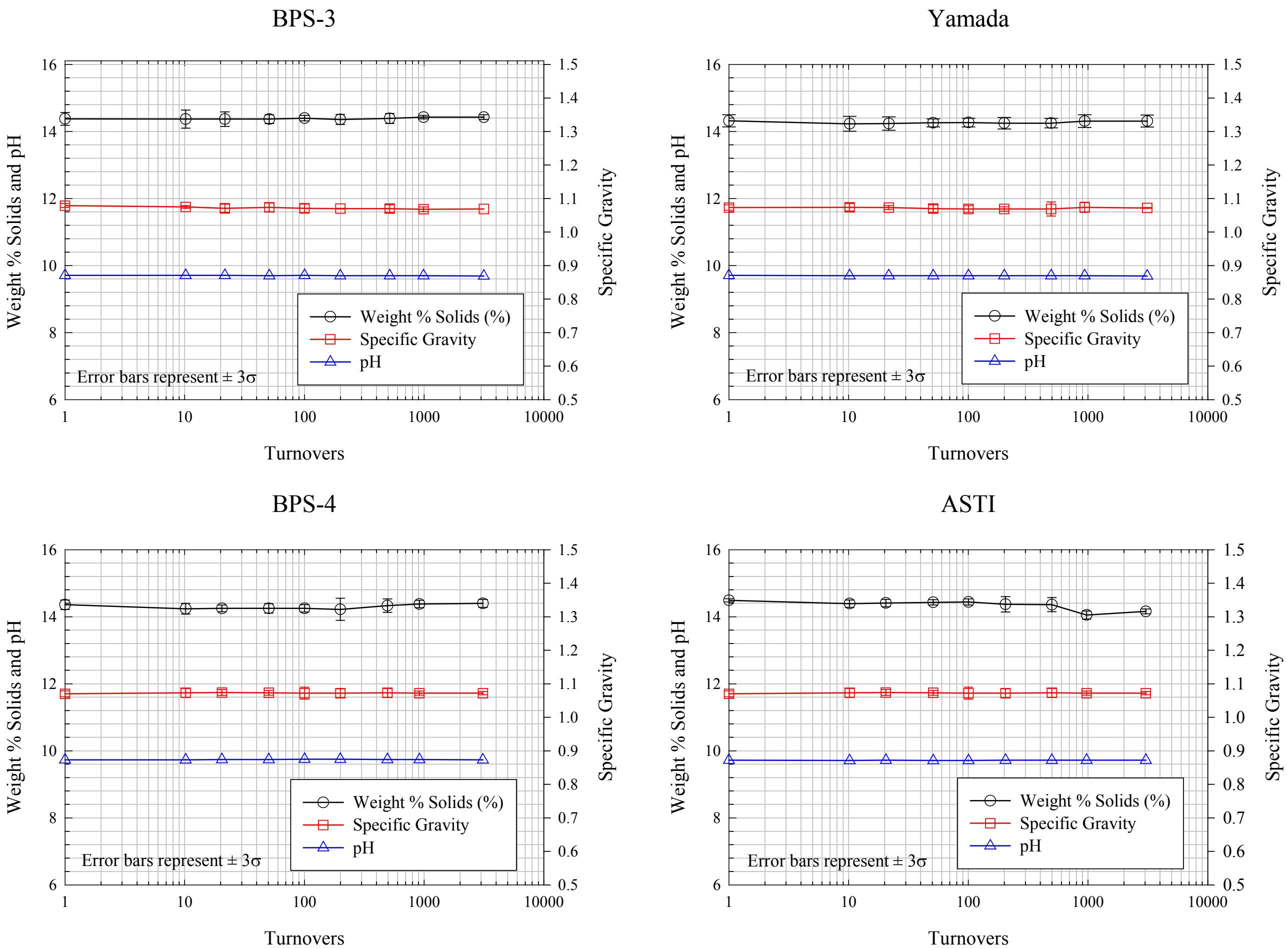
Summary

A Yamada diaphragm pump, an ASTI bellows pump, and two Levitronix centrifugal pumps were tested to determine how their use affected the size distribution of particles in Cabot EP-C6618 slurry. The slurry was circulated until it had passed through each pump a minimum of 3,000 times.

Little change in the large particle tail or working PSD were observed with the BPS-3 and BPS-4 pumps. Concentrations of large particles (≥ 0.56 µm) tended to decrease slightly with increasing turnovers. Increases in large particle concentrations were observed for both the Yamada and ASTI pumps as the number of passes through the pumps was increased. Particle concentrations tended to increase non-linearly with increasing turnovers. Within 1,000 turnovers, concentrations of particles ≥ 1 µm increased by a factor of about 2 and 3 for the Yamada and ASTI pumps, respectively.

No changes were observed in the other slurry health parameters (working PSD, zeta potential, weight percent solids, pH, and specific gravity) for any of the pumps during the tests.

Figure 6: Total % solids, specific gravity and pH measurements as a function of turnovers



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