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Comparison of Four Pump Systems on the Particle Size Distribution of Cabot iCue[®] 600Y75 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 iCue[®] 600Y75 (Cabot Microelectronics Corporation, Aurora, IL), an aluminum 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.

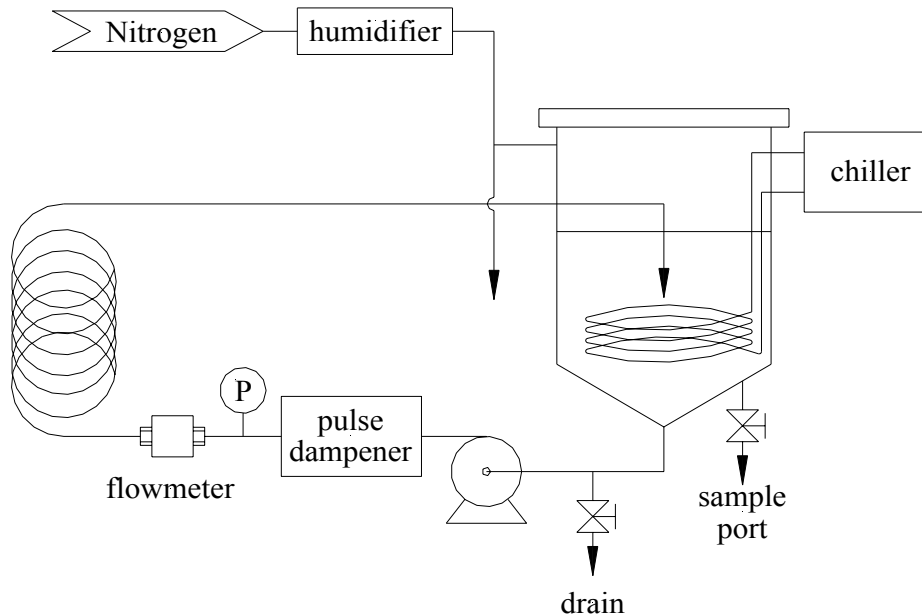
Table I. Specifications of the four pump systems

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 $24 \pm 2^\circ\text{C}$ 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.

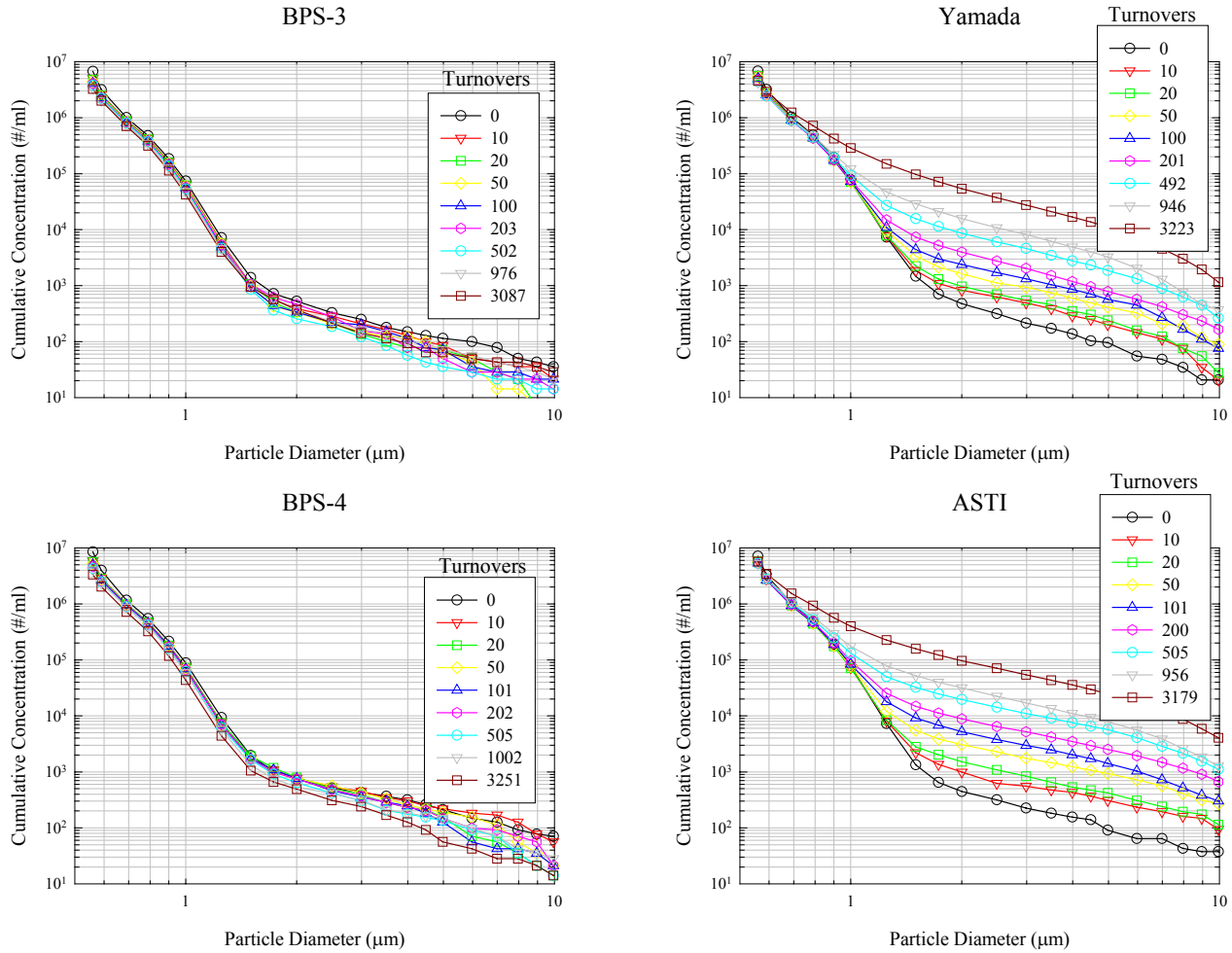
The AccuSizer 780 uses a combination of light scattering and light extinction to measure the size distribution of particles $\geq 0.56 \mu\text{m}$. The size measurements were performed by diluting the slurry sample by a factor of 1600:1 into deionized water. Between samples, the entire system was thoroughly 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

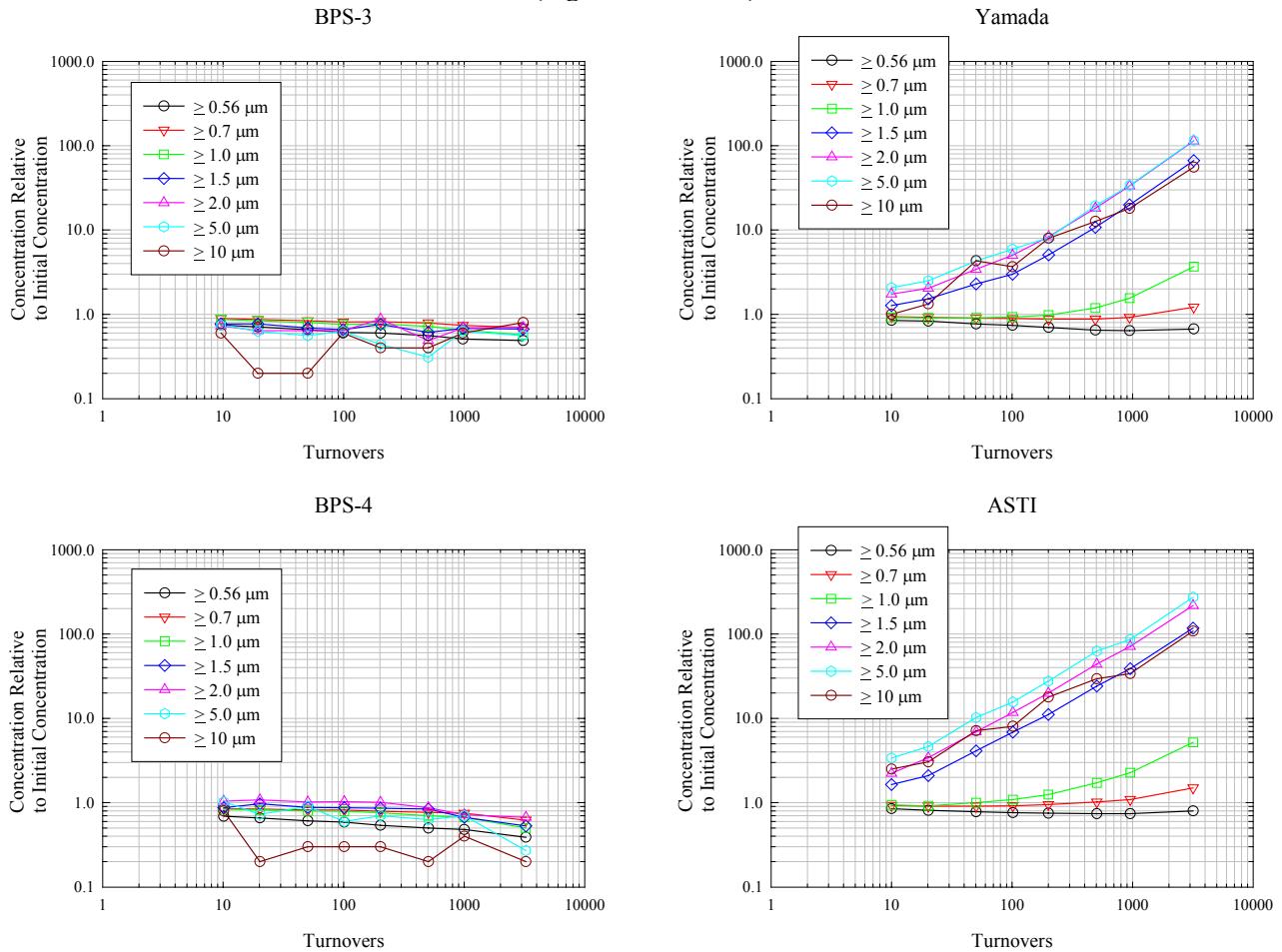
The four graphs in Figure 2 show the cumulative particle size distributions (PSD) of the slurry large particle tail measured during each pump test. Each graph presents the results from a different pump. The initial PSD, measured prior to the start of each test, is presented in each graph as well as PSDs after selected numbers of turnovers. Little change in the PSD was observed during the BPS-3 and BPS-4 tests, while large increases in the large particle concentrations, particularly for particles > 1 μm, were observed 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. Linear regressions are plotted on each curve in Figure 4.

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, on the order of 30-50%, was observed for particles sizes ranging from 0.56 μm to more than 10 μm. Much of the decrease occurred within the initial 10 turnovers. Up to a 30% decrease in the particle concentrations was also observed with the ASTI and Yamada pumps, but only for particle sizes on the order of 0.56-0.60 μm.

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. For particle sizes ≥ 1 μm, significant differences between the pumps were apparent. During the ASTI and Yamada pump tests, particle concentrations ≥ 1 μm tended to increase linearly with increasing turnovers (see Figure 4). For particles ≥ 1.5 μm, concentration increases were very large for the ASTI and Yamada pumps. After 1,000 turnovers, the particle concentrations ≥ 2 μm increased by a factor of 70 and 33 for the ASTI and Yamada pumps, respectively. This equates to about 7% and 3% increases in particle concentrations each time the slurry passes through the ASTI and Yamada pumps, respectively. Meanwhile, particle concentrations decreased slightly for these larger particle sizes during both Levitronix pump tests.

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

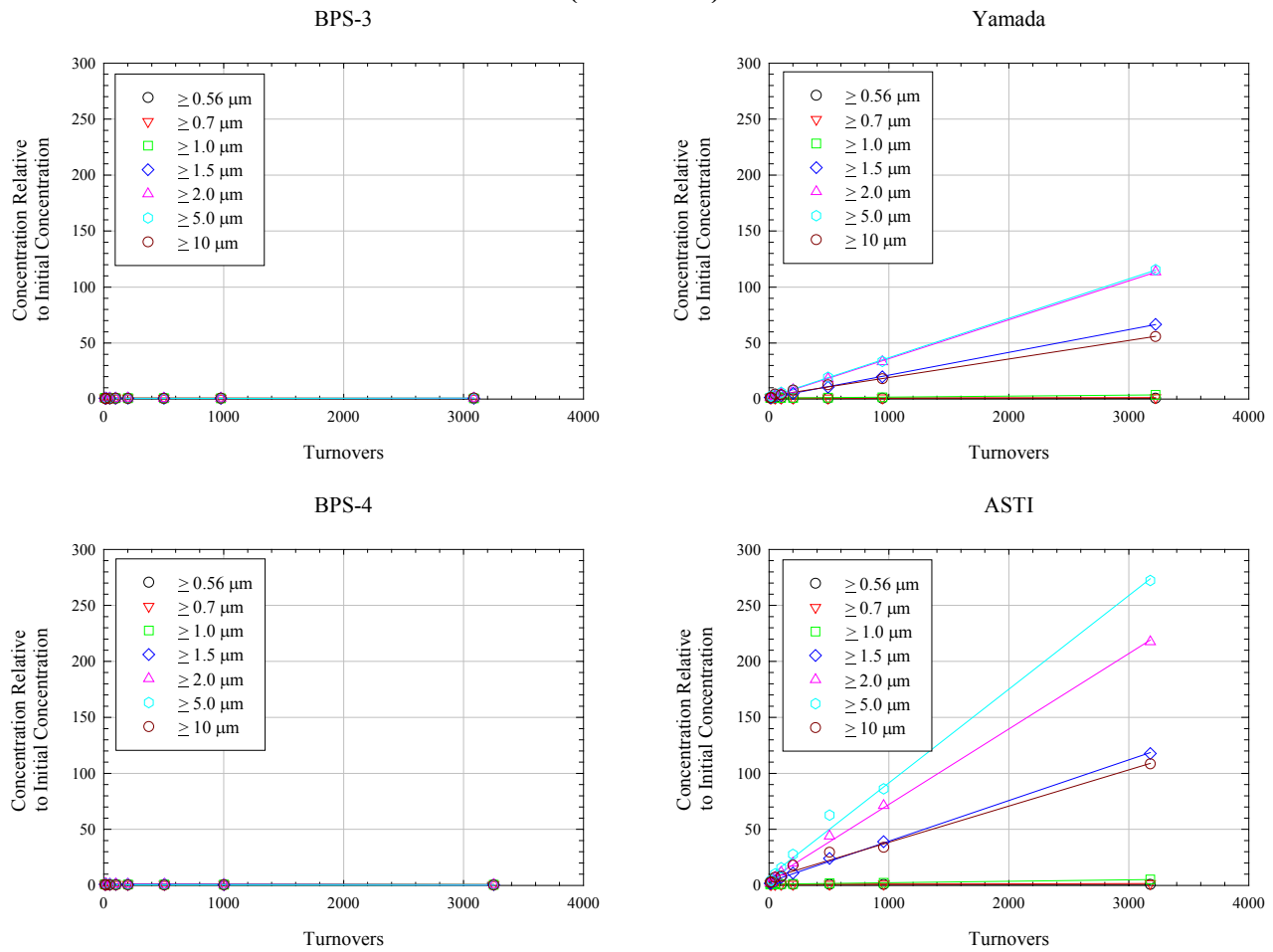


Table II. Summary of the relative particle concentrations changes for selected times and particle sizes

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
$\geq 0.56 \mu\text{m}$	0.6	0.6	0.7	0.8	0.5	0.5	0.6	0.7
$\geq 1.0 \mu\text{m}$	0.8	0.8	0.9	1.1	0.6	0.7	1.6	2.3
$\geq 1.5 \mu\text{m}$	0.7	0.9	3.0	6.9	0.7	0.7	20	39
$\geq 2.0 \mu\text{m}$	0.6	1.0	5.0	11.8	0.7	0.7	33	71
$\geq 5.0 \mu\text{m}$	0.6	0.6	6.0	15.6	0.6	0.7	34	86
$\geq 10 \mu\text{m}$	0.6	0.3	3.7	8.0	0.6	0.4	18	34

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 247 nm and 556 nm, respectively. No significant changes in the volume-weighted mean were observed for any of the pumps during the tests; however, after more than 3,000 turnovers, the 99th percentile particle diameter decreased by about 5-10% during each test. This is consistent with the decreases in the particle concentration $\geq 0.56 \mu\text{m}$ that were observed with the AccuSizer during these tests. Also

included in Figure 5 are zeta potential measurements taken during each test. No significant change in the zeta potential was observed during any of the tests.

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

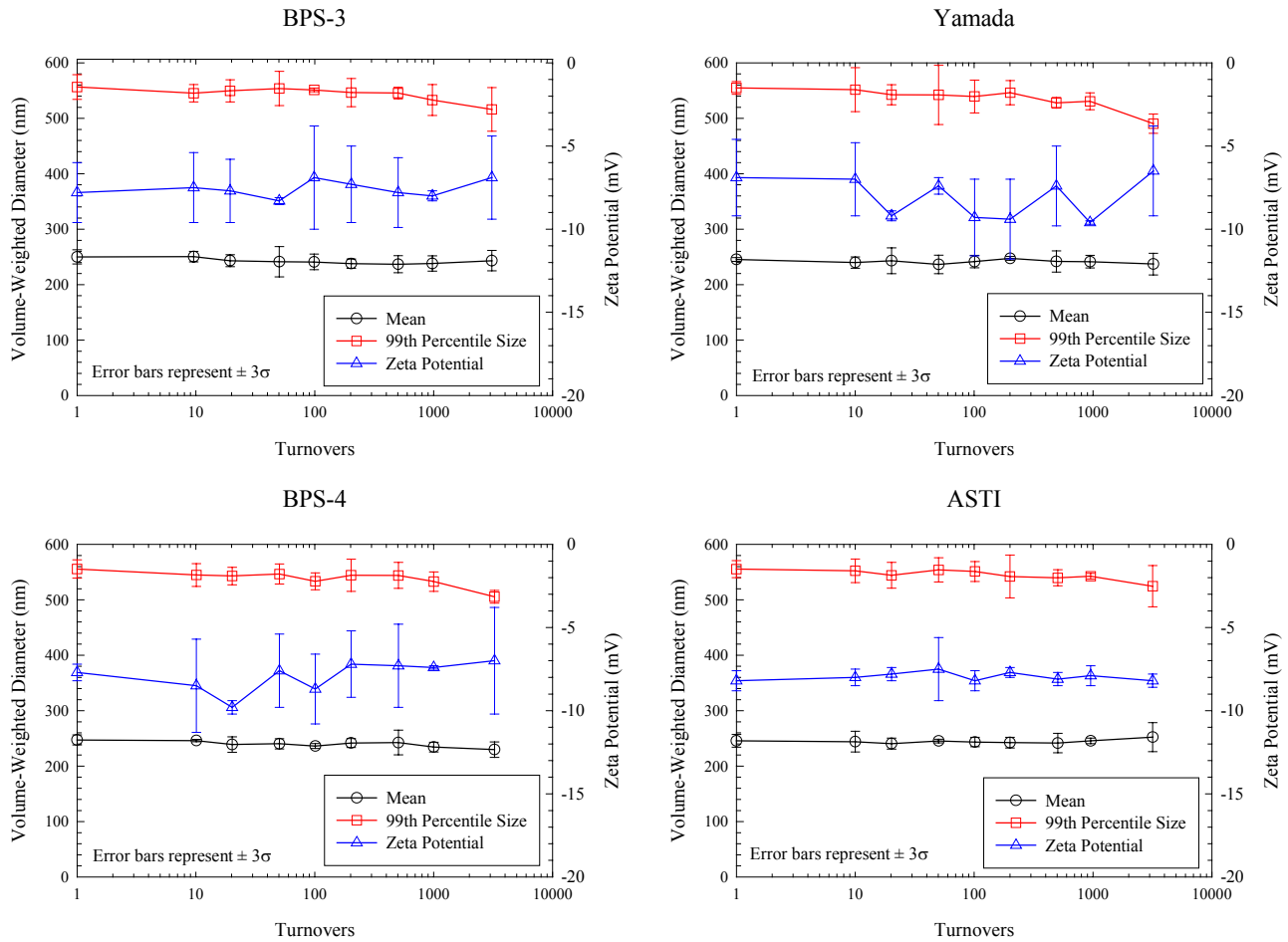


Figure 6 shows the total percent solids, specific gravity, and pH as a function of tank turnovers during each test. Error bars are included in each graph and represent ± 3 standard deviations. No changes in these slurry health parameters were observed for any of the pumps during the tests.

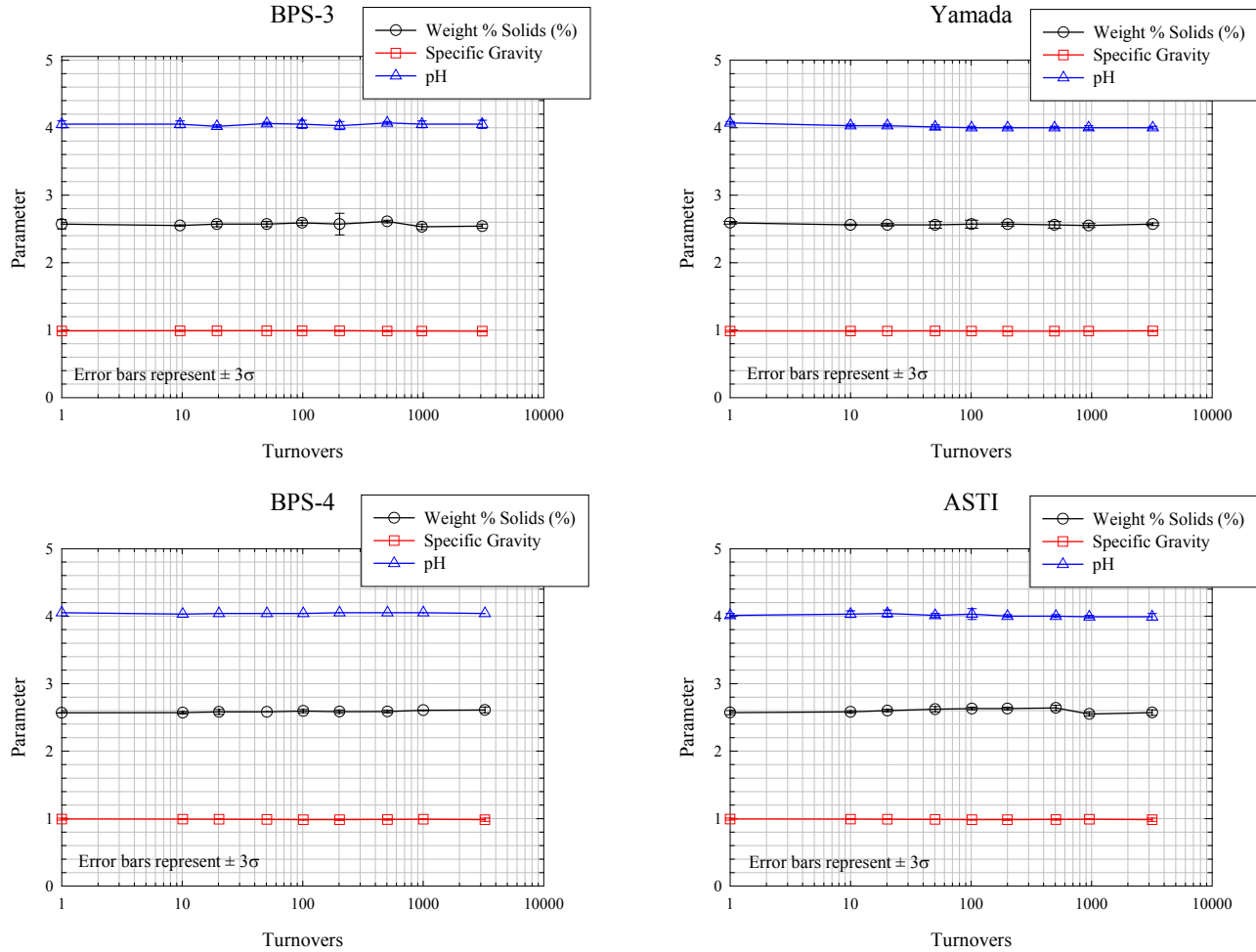
Summary

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 iCue® 600Y75 slurry. The slurry was circulated until it had passed through each pump a minimum of 3,000 times.

Little change in the PSD was observed during the BPS-3 and BPS-4 tests regardless of particle size, while significant increases in the large particle concentrations, particularly for particles > 1 μm, were observed during the Yamada and ASTI pumps tests. During the ASTI and Yamada pump tests, concentrations of particles ≥ 1 μm tended to increase linearly with increasing turnovers. For particles ≥ 1.5 μm, the concentration increases were very large for the both the ASTI and Yamada pumps. After 1,000 turnovers, concentrations of particles ≥ 2 μm increased by a factor of 70 and 33 for the ASTI and Yamada pumps, respectively. Meanwhile, concentrations of particles of these sizes decreased up to 50% during tests with the BPS-3 and BPS-4 pumps.

Relatively little change was observed in the working particle size distribution during the tests. No significant change in the volume-weighted mean was observed during any of the pump tests; however, the 99th percentile particle diameter decreased 5-10% after 3,000 turnovers during each test. No significant changes were observed in the other slurry health parameters measured during each test.

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



References

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3. M. Litchy and R. Schoeb, "Effect of shear stress and pump method on CMP slurry," *Semiconductor International* 27 (12), 87-90 (2004).
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