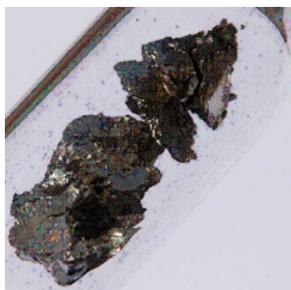




Sample Dispersion Case Study

The praseodymium (Pr_6O_{11}) powder supplied to the applications lab was a dark brown powder. Praseodymium is a soft, silvery, malleable and ductile metal in the lanthanide group. The apparent density is 6.8 kg/m^3 and is paramagnetic at any temperature above 1 K. This material is insoluble in water, but soluble in strong acid.



Praseodymium has several commercial applications including being used as a pigment, and as a varistor – a complex polycrystalline

ceramic system containing several dopants. When used as a varistor good average grain size leads to electro-thermal stability and energy absorption capability.

Based in the information known about this sample experiments were designed to investigate how reported particle size changed as a function of

pH, pump speed, ultrasound. All measurements were made on the HORIBA LA-950 laser diffraction analyzer.

pH

Sample dispersion is often a function of surface chemistry. Common approaches used to modify the surface chemistry of powder suspensions include the use of surfactants, admixtures, and altering the pH. This sample easily wetted in water so no surfactant was necessary. The pH of a base suspension was adjusted to 11, 4 and 1.2. Figure 1 shows the particle size distribution (PSD) at these pH values. The pH value of 1.2 appears most suitable for this sample.

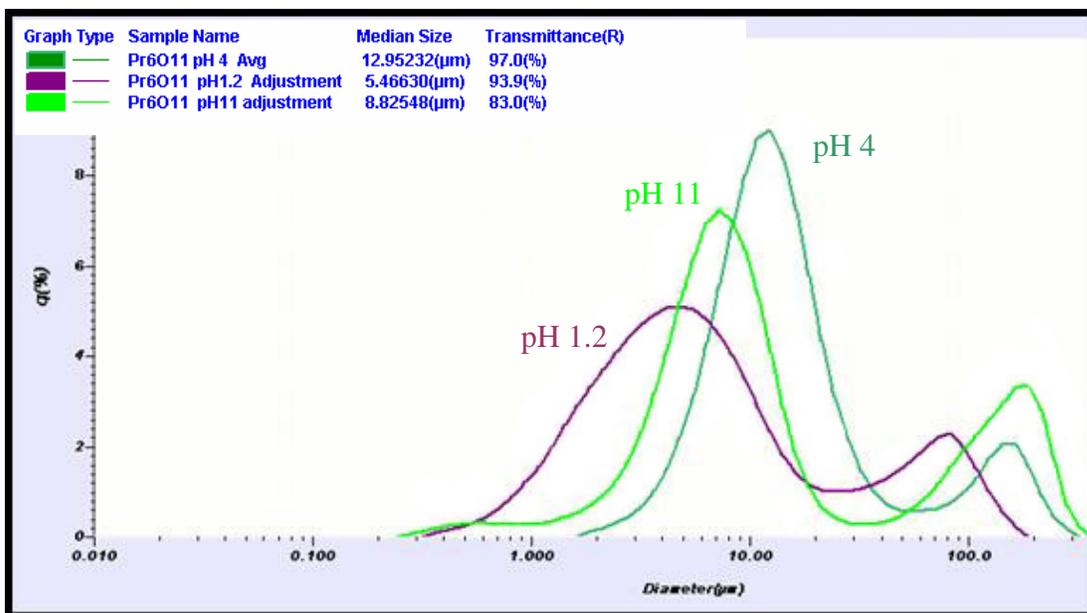


Figure 1: PSD vs. pH



Pump Speed

Particles that are either very large or very dense in contrast to the liquid dispersant are the most dependent upon pumping speed. When developing a method for dense particles it is recommended to examine the distribution graphs at slower speeds to determine if slow speeds report finer particle sizes. This is a strong indicator that those speeds are unable to suspend and re-circulate the larger particles. Care should also be taken when pumping at very high speeds that may create bubbles and exhibit a new peak around 100 microns. Looking at the D90 and D10 values in addition to the particle size distribution can provide insight into the state of dispersion. If the D90 value decreases with decreasing pump speed, then the largest particles are very likely settling out of suspension. When this is occurring the distributions should appear almost exactly the same

except that the coarsest particles are missing. Likewise, if the D10 size value decreases with increasing pump speed, then particles might be breaking (unlikely for this sample). If the agitation is breaking particles the distributions will appear very similar except the finest particles will increase, possibly as a new peak. Once the speeds that are too slow or too fast are identified it is a trivial process of choosing any speed between those values (but not too close).

Figure 2 shows the particle size distribution for the sample as pump speed is varied from 5 to 13 on the LA-950 system. The dramatic shift in the coarse tail of the distribution indicates that pump speed 5 did not adequately suspend the larger particles. Note the change in concentration (%T) as a function of pump speed – another indicator of changing dispersion conditions.

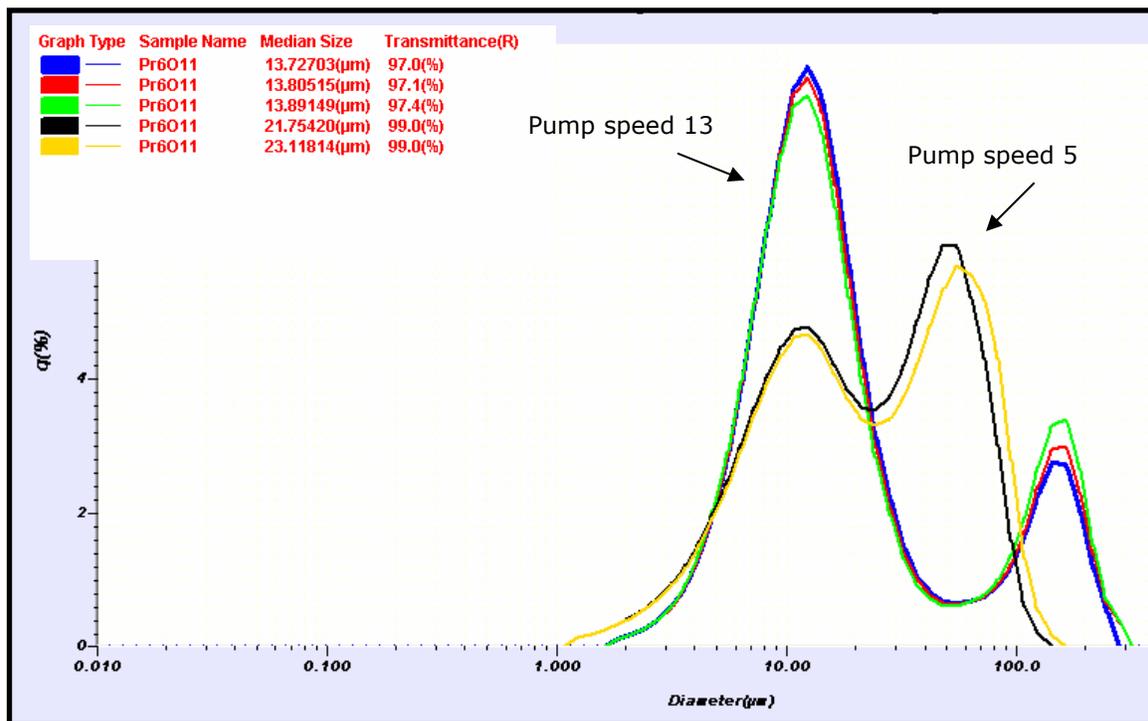


Figure 2: PSD vs. pump speed



Ultrasound

Ultrasound is frequently used as an energy source for the dispersion of agglomerated particles to the single (or primary) particle state. The LA-950 has a built in ultrasonic probe to aid dispersion. The amount of energy added to the system can have a significant effect on the reported PSD. In this case the instrument setting (amount of ultrasound) is altering the state of the particles being measured. The goal in method development is typically to disperse the sample to the single particle state without fracturing

individual particles. The choice of optimum energy level can often be made by examining the two tails of the distribution. Larger particles disappearing with the addition of ultrasonic energy typically indicates the desired dispersion of agglomerates. Smaller particles appearing with the addition of energy typically indicates the unwanted breakage of individual particles.

Figure 3 shows the sample PSD with, during, and post exposure to 30 seconds of ultrasound.

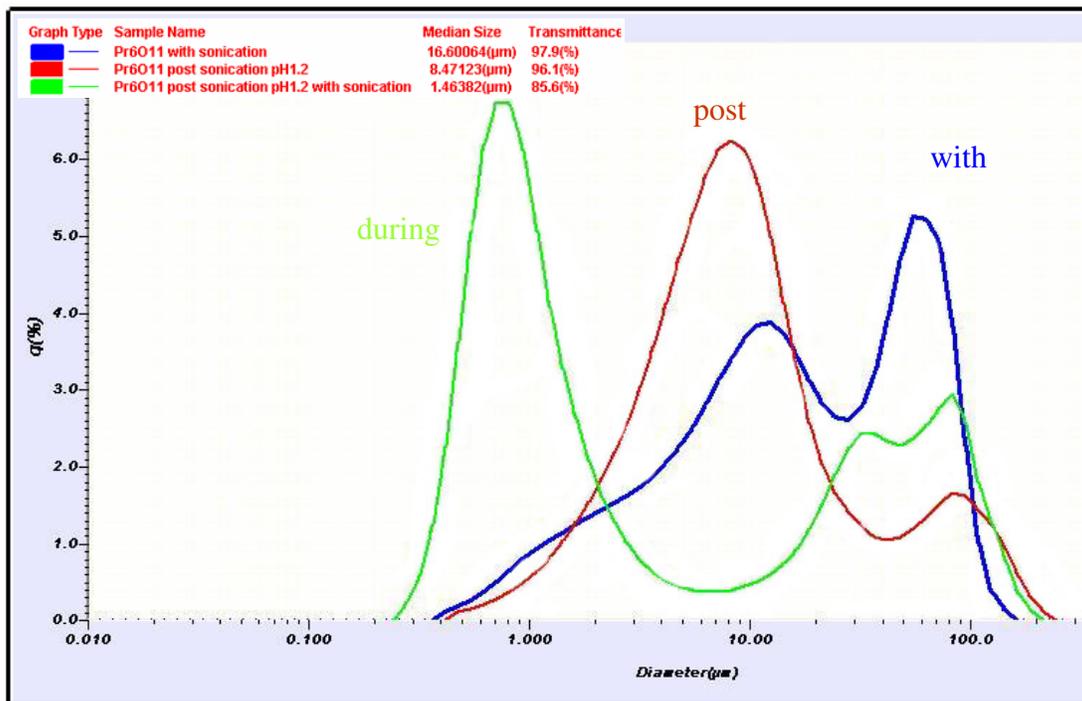


Figure 3: With, during, and post sonication at pH 1.2

The blue result marked "with" was measured by applying 30 sec of ultrasound and then measuring the sample. The green result marked "during" was measured by applying 25 sec of ultrasound and then measuring the sample while the ultrasound was still turned on – keeping the total exposure to ultrasound close to 30 sec.

The result marked "post" was measured 10 seconds after the result marked "during". The ultrasound was turned off at the end of the measurement, 10 sec elapsed, and then this measurement was made. The particles re-agglomerated in 10 sec period between turning off the ultrasound and the measurement.



The same experiment was performed with sample prepared at pH 4 as seen in Figure 4. In this case the dispersion

the ultrasonic probe left on. The LA-950 then generated accurate, reproducible results for this sample.

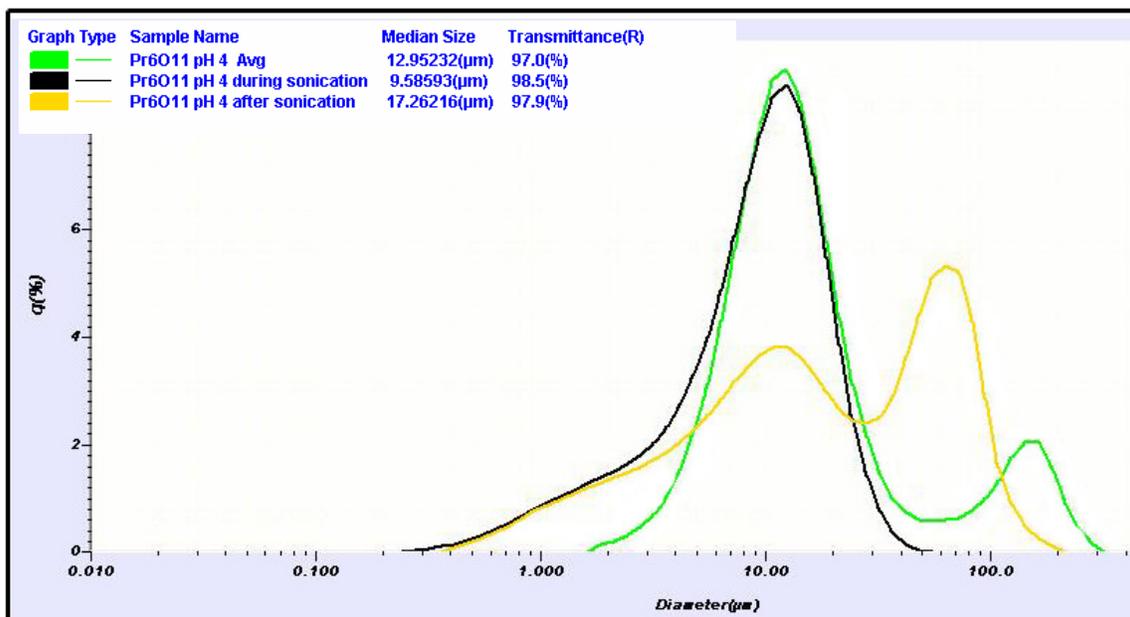


Figure 4: Pre, during, and post sonication at pH 4

and reformation of aggregates is even more obvious. Sonication provided the energy to disperse aggregates, but they re-formed after the ultrasound was turned off. In this situation a dispersing aid such as sodium hexametaphosphate should be added to suppress reformation of aggregates. Another possible approach to this sample would be to measure while the ultrasound is turned on.

Conclusions

The praseodymium powder was a challenging sample to disperse, but the experiments described here determined the optimum conditions for particle size analysis using laser diffraction. The pH was adjusted to 1.2, the pump speed was set at 13, the sample was exposed to 25 seconds of ultrasound and was then analyzed with

A structured approach to method development may take some additional time, but is worth the investment in order to understand how sample preparation and system settings influence results.

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