White Paper: Scaling-up Pixelligent Nanocrystal Dispersions

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Introduction

This white paper will discuss Pixelligent Technologies' success in scaling-up its proprietary nano-dispersions from lab to full-scale production. It is well known that nanoscale zirconium oxide particle dispersions and formulations have a wide array of applications in solid state lighting (SSL), optical components and films (OCF), and numerous other industries. The scaling-up of nanomaterials that maintain all of the unique and valuable benefits, while also delivering the economies of scale required by large end-users, has been the single biggest challenge to widespread market adoption of nanotechnology. The careful control of processes during all aspects of production is necessary to retain key product characteristics (KPCs)[1]. In combination with dramatic improvements in manufacturing efficiencies, yield and robust supply chain management is required to deliver cost-competitive market leading products. Developing these unique manufacturing processes all within a carefully constructed environmental, health, and safety (EHS) framework has resulted in Pixelligent delivering numerous product families of next generation and scaled nano-zicronia dispersions with un-paralleled quality and performance for our customers.

Scale-up of Zirconium Oxide Nano-Dispersions

Over the past 3 years Pixelligent has successfully scaled its unique liquid synthesis process for Zirconium Oxide nano-dispersions from laboratory gram-scale quantities to metric-ton production scale volumes. This cycle is repeated frequently as new products are launched to satisfy market needs. When scaling Pixelligent's production process, several techniques based on the DMAIC model are employed to insure quality is maintained while optimizing for cost.[2] First, product KPCs are established based on the combination of customer or market requirements and development scale statistical performance. Ranges are based on the upper and lower control limits in the lab data and are incorporated into a quality control plan. Samples are taken at each step of the scaled process to confirm the scalability of each process step. Additionally, in-process samples are collected to give further insight to engineers regarding the performance of each unit operation.[3] If significant discrepancies are found in the scaling of any step, process parameters are adjusted to match the target KPCs.

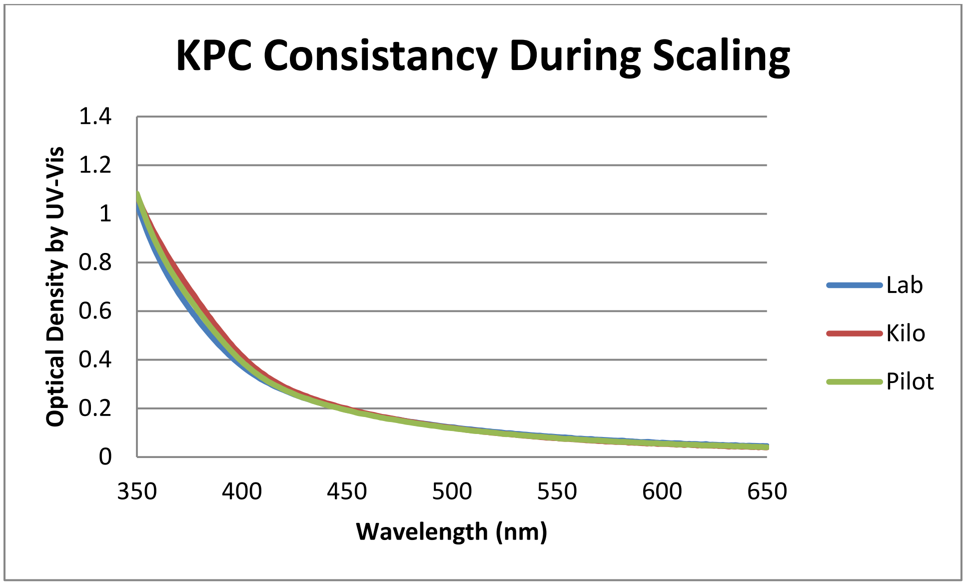

Figure 1: Typical variations in UV observed at differing scales, controlled well below the specification level of 1.4 OD at 350nm.

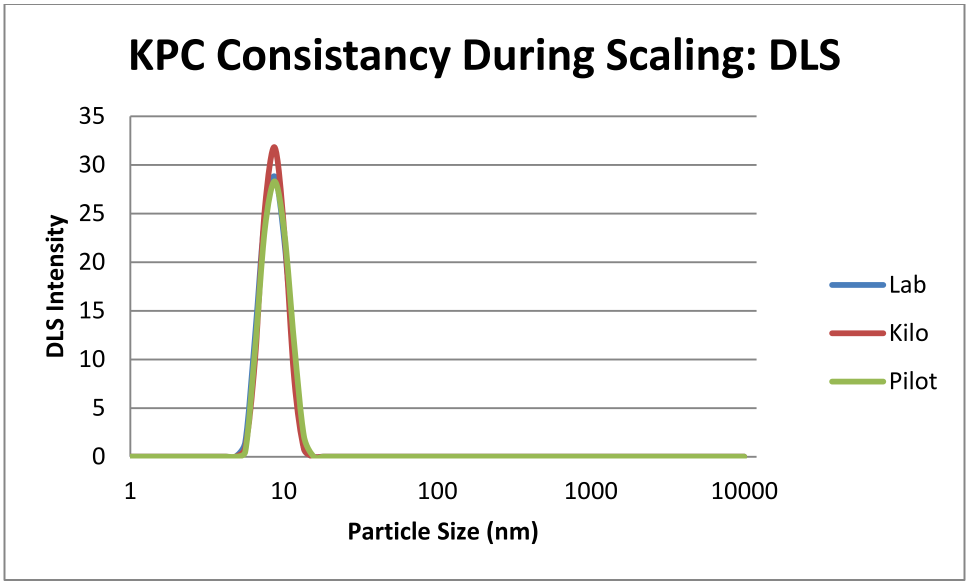

Figure 2: Typical variations in DLS at differing scales, specification. The specification is between 8-11nm

Quality Control

After scale-up is completed KPCs are then monitored using statistical process control.[4] Control charts incorporate KPC limits, as well as upper and lower control limits to ensure process performance. Quality approval is predicated both on KPCs and control limits. Statistical methods such as T-tests, regression analysis, and step-change analysis are used to catch potential process upsets that could affect manufacturing cost and on time delivery.

Thermal gravimetric analysis (TGA) provides solids loading and Zirconium Oxide content, which correlates to refractive index in application. UV-Vis gives a measurement of absorbance and clarity, two features our customers rely on for performance. Dynamic Light Scattering (DLS) provides a measure of particle size and distribution, proving our material is nano-scale.

KPCs are tested immediately after production, and material is not entered into inventory until after quality metrics have passed. Materials are tested and re-certified on a monthly basis. Standard Pixelligent products have shown stability for up to 6 months. Certificates of analysis (CoAs) are provided to the customer with each shipment showing the results of the most recently measured metrics.

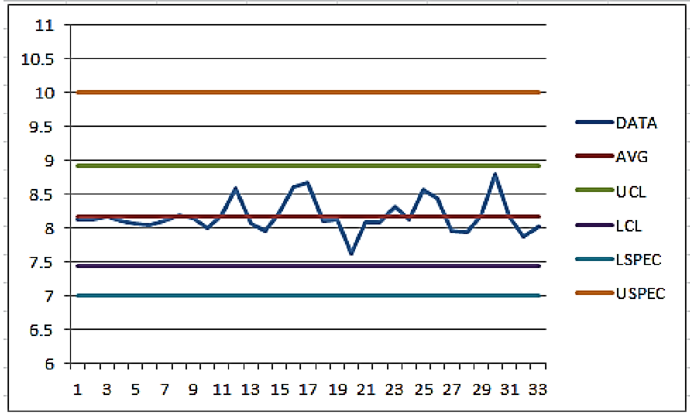

Figure 3: Typical SPC chart of mean particle size, spec 7-10nm in diameter

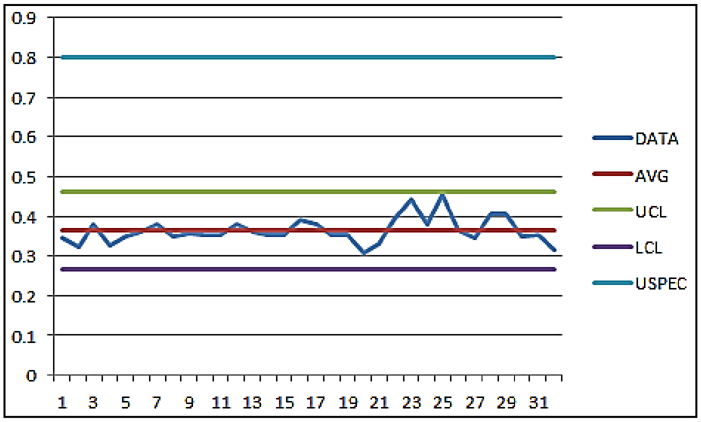

Figure 4: Typical SPC chart of Optical density, spec <1.4

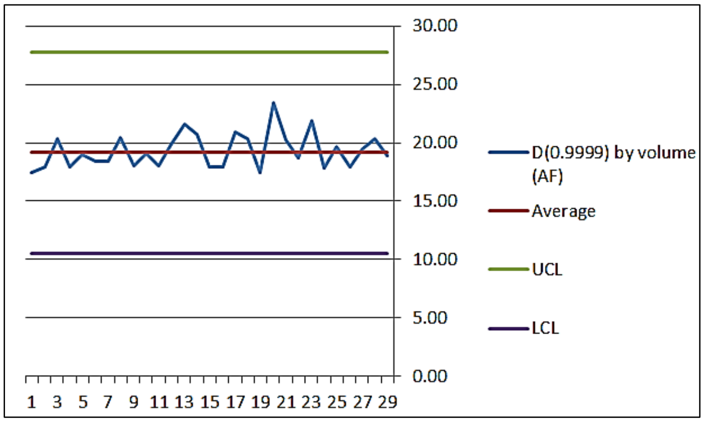

Figure 5: Typical SPC chart of D9999, the largest diameter in nm of 99.99% of particles by volume.

Environmental Health and Safety

Pixelligent's EHS strategy takes a proactive, layered approach to hazard identification and control.[5] Pixelligent avoids the potential health risk of nano-scale particles through an intrinsically safe liquid synthesis process design, which eliminates the need for handling of powders. Since 2012, Pixelligent has partnered with the National Institute for Occupational Safety and Health (NIOSH) to study the occupational exposure of nanomaterial processing. Through this partnership Pixelligent has confirmed exposure levels are a factor of ten or more below OSHA limits. Process safety is addressed by the voluntary adoption of the 14 elements of OSHA PSM such as PHA and PSSR.[6] Environmental compliance is incorporated into the product scaling process, to insure TSCA compliance. Nanomaterial waste is processed above the federal requirement levels as a conservative approach to environmental stewardship. Audits are regularly completed both internally and by third parties to improve our programs. In the summer of 2015 Pixelligent invited Maryland Occupational Safety and Health (MOSH) through the small business consultation program, which led to no major actions required, but did produce meaningful improvements to the Health and Safety program.

Conclusion

By employing, and in some cases establishing, best practices in EHS, process control, and cost management, Pixelligent has successfully scaled its Zirconia nano-dispersion technology to commercial scale with systems in place to sustain this as new materials and technologies are introduced. The end results are robust scaled nano-zicronia dispersions and formulations with un-paralleled quality and performance for customers.

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[1] Zhao, Q.; Boxman, A.; Chowdhry, U. (2003) "Nanotechnology in the Chemical Industry- Opportunities and Challenges" in *Journal of Nanoparticle Research* Vol. 5, Iss 5, pp. 567-572

[2] Kumar, S.; Sosnoski, M. (2009) "Using DMAIC Six Sigma to systematically improve shop floor production quality and costs" in *International Journal of Productivity and Performance Management*, Vol. 58 Iss 3, pp.254 - 273

[3] Moen, Ronald D., Thomas W. Nolan, and Lloyd P. Provost. *Quality Improvement through Planned Experimentation.* New York: McGraw-Hill, 1999. Print.

[4] Moen, Ronald D., Thomas W. Nolan, and Lloyd P. Provost. *Quality Improvement through Planned Experimentation.* New York: McGraw-Hill, 1999. Print.

[5] Edwards, Victor H., P.E. "Designing Safer Process Plants." *Chemical Engineering* Apr. 2011: 44-48. Print.

[6] United States. OSHA. *Process Safety Management*. Washington, D.C.: U.S. Dept. of Labor, Occupational Safety and Health Administration, 2000. Print.