THE INVESTIGATION OF TITANIUM DIOXIDE NANOPARTICLE FILMS CREATED THROUGH ELECTROPHORETIC DEPOSITION

By

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CHAPTER 1

INTRODUCTION

The creation of thin films of TiO_2 nanoparticles is of particular interest to scientists and industry due to its properties and the variety of applications. As a photocatalyst, TiO_2 is the preferred material due to its photocatalytic activity, chemical stability, nontoxicity, and low cost compared to other materials.[1] Additionally, TiO_2's important properties include a large band gap, high electric resistivity, a high dielectric constant, and high oxidative power.[2, 3] These properties lead to applications as capacitors in microelectronic devices, gas sensors, dye-based solar cells, optical filters, antireflection coatings, and sterilization materials.[1, 3, 4]

To create these thin films with TiO_2 nanoparticles, two major processes must be employed: 1) the creation of nanoparticles that have the desired shape and properties needed for the specific use for which they are produced; and 2) deposition to create the films of these particles. For both of these major processes, there are numerous methods to achieve the desired result. The scope of this paper is limited to the particle synthesis produced through the sol-gel process, the size separation of particles using centrifugation techniques, and the creation of films using electrophoretic deposition.

This paper is organized in three separate chapters. Chapter II will cover the basic properties and a brief synopsis of the process to synthesize the

nanoparticles used in the following research. Chapter III focuses on the centrifugation techniques employed to separate particles by size, making particles greater than 30nm precipitate out of the suspension, and particles 30nm and below remain in the suspension. Chapter IV discusses the films created through electrophoretic deposition for particles commercially produced by Nanostructured & Amorphous Materials, Inc (Nanoamor) and through the sol-gel method by our collaborators in Spain.*[1] The results show that the suspension produced by Professor Rodrigo Moreno and his group have better size characteristics and are a better suspension to perform electrophoretic deposition than the suspension produced with water or ethanol from the nanoparticles purchased from Nanoamor.

*Professor Rodrigo Moreno, Instituto de Cermamica y Vidrio, Consejo Superior de Investigaciones Científicas, Madrid, Spain.

CHAPTER II

CHARACTERISTIC PROPERTIES AND THE SYNTHESIS OF TITANIUM DIOXIDE NANOPARTICLES

The unique crystal structure, photocatalytic activity, and photoluminescence properties of titanium dioxide nanoparticles lead to the variety of applications previously mentioned. Titanium dioxide is found to have three prominent crystal phases: anatase, rutile, and brookite.[5] In nature, the most commonly found crystal phase is rutile due to its stability. However, due to temperatures most commonly used in the heating process during nanoparticle synthesis, the anatase phase becomes the most stable phase.[5, 6] Because the anatase and rutile phases are the most commonly phases used in the preparation of suspensions for electrophoretic deposition, only the properties and synthesis of these two phases will be analyzed in this paper.

Particle Synthesis

There are several methods used to synthesize TiO_2, including the hydrothermal method, chemical vapor deposition, electrodeposition, and the sol-gel method.[5, 7, 8] Because the sol-gel method is the most employed nanoparticle preparation method, the research in this section will focus on that method of synthesis. The sol-gel method is the most common nanoparticle preparation method because it is an easily controlled and variable process that is effective,

efficient, and relatively inexpensive.[9] The sol-gel method involves four separate steps: formulation of a sol containing the TiO_2 precursor, deposition of the sol on the desired mold, evaporation of the solvent to create the gel, and application of the heat treatment to achieve the desired phase and size of TiO^2 particles.[10] A flow chart of this process is depicted in Figure 1. The addition of nitric acid is used to adjust the pH of the suspension, increasing the surface charge of the particle and thereby maximizing the zeta potential of the suspension.[7]

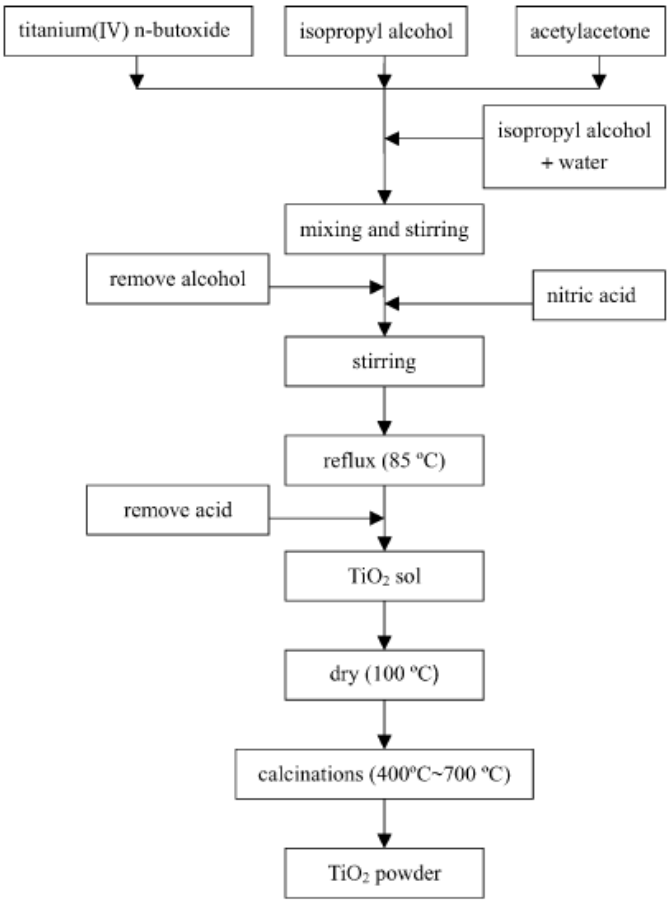


Figure 1 Flow chart depicting the sol-gel particle synthesis process.[7]

During the heating processes, temperatures are chosen that affect both the particle size and the crystal phase of the nanoparticles.[4] Temperatures kept between 300°C and 500°C produce only TiO_2 particles that are in the anatase phase and are 20nm in diameter. Between 500°C and 900°C, the nanoparticles begin to transition from the anatase phase to the rutile phase. This phase of transition is complete at temperatures above 900°C, where all the nanoparticles are in the rutile phase and are 110nm or larger in diameter.[4] Because particle

sizes that are less than 30nm in diameter are preferred in the research in this paper, the phase of interest for this research is on particles in the anatase phase.

Properties of TiO_2 Nanoparticles in the Anatase Phase

One of the major differences between the nanoparticle and bulk material properties is the difference in the surface area to volume ratio, which is inversely related to the particle size: for a sphere this ratio is $\frac{3}{r}$. This increased surface area to volume ratio changes the surface charge of the particle the affects the physical and chemical behaviors of the particle.[4] The most prevalent of these changes is the difference in bond lengths of TiO_2. This change in bond length changes the lattice structure of the particle, causing different mass densities and different electronic band structures.[5] As the particle size decreases, the band gap energy increases; in the anatase phase at room temperature, this band gap change is 0.1-0.2eV for particles 2nm in diameter.[5, 11, 12]

This change in the band gap has an impact on the optical properties of the particles based on their sizes. A property of interest in the scientific community is the absorption of the material, which is important in the production of solar cell technology to minimize the amount of light reflected from the material and maximize the amount of light absorbed by the material. Because TiO_2 is an indirect band gap material, the absorption is proportional to the photon energy and the band gap energy based on the relationship[13]

$$\alpha \propto \frac{\left(\hbar\omega - E_{gap}\right)^2}{\hbar\omega}$$

Because the change of the band gap is directly related to the size of the nanoparticle, the ability to create a film of a specific absorption quality is possible and provides promise for future research and development.

CHAPTER III

THE USE OF CENTRIFUGATION FOR SIZE DEPENDENT PARTICLE SEPARATION

Because the strength of the films created through electrophoretic deposition is largely dependent on the particle size in the suspension, the ability to separate the particles in a suspension by size is of particular interest to the scientific community.[14] Numerous methods have been employed to separate suspended nanoparticles by size, including magnetic separation, selective precipitation, filtration, centrifugation, electrophoresis, and chromatographic methods.[15] In the experiments conducted in this paper, the desired particle sizes were those less than 30nm. Due to its non-destructive nature and the availability of equipment in most labs, centrifugation became the focus of particle size separation methods for this paper.

Calculations for Particle Separation by Centrifugation

Treating the particles as spheres, one can analyze the forces that are acting on the particle through the centrifugation process. Looking at the kinematics of a particle in a suspension prior to centrifugation, three different forces can be identified: gravity, buoyancy, and drag (Figure 2).[16] The combination of the force due to gravity and the buoyancy force can be found through the equation

$$F = \frac{4}{3}\pi\left(\frac{x}{2}\right)^3(\rho_p - \rho_f)g$$

where ρ_p is the known density of the TiO_2 particle and ρ_f is the density of the

liquid the particle is suspended in.[16] To simplify the calculation for the force of drag, the assumption is made that there is little fluid turbulence, and the particle is small and moving with a slow velocity. These assumptions cause the Reynolds number, the number that expresses the ratio of inertial forces to the viscous forces associated with the motion of the particle through the liquid, to be approximately one and insignificant in the calculation of the drag forces. With these assumptions, the force of drag can be defined as $F_d = 3\pi v_p x \mu$, where v_p is the velocity of the particle and μ is the viscosity of the suspension.[16, 17] Replacing the gravitational acceleration with the acceleration due to centrifugation and solving for the particle diameter, we arrive at the equation

$$x = \sqrt{\frac{18\mu v_r}{(\rho_p - \rho_f) r \omega^2}}$$

This equation defines the size of the particle that is in equilibrium, known as the cut size, at a given radius from the center of the rotating axis, known as the locus of zero vertical velocity.[16, 18] However, these calculations are based on processes using a hydrocyclone, where the suspension is able to be inserted into the spinning centrifuge with a set angular velocity at a desired radius from the axis of rotation, thus allowing the larger particles to sediment and be removed from the liquid, and the smaller particles to be left in the suspension and removed with the overflow liquid during the

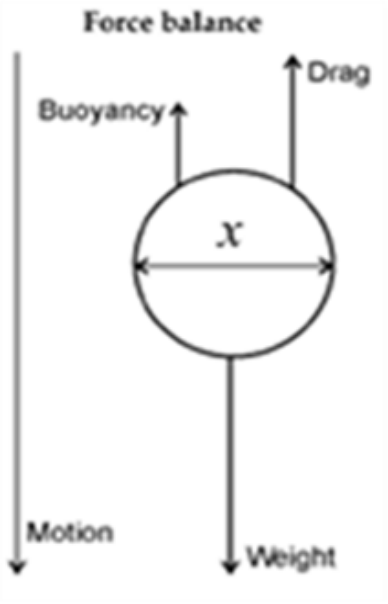


Figure 2 Forces acting on a particle in a suspension during centrifugation: buoyancy force, drag force, gravitational force.[16]

centrifugation.[16] Using the Drucker centrifuge, the suspension must be placed in centrifuge tubes during the experiments. This process creates a gradient of force due to the distance from the axis of rotation, where the forces at the bottom of the tube greater than at the top. Because the suspensions were uniformly distributed prior to the centrifugation, particles of the same size would be subjected to different forces based on their initial position in the centrifuge tube.

Results from Previous Centrifugation Studies

The goal of the research from this chapter was to develop a process to consistently obtain particles of a given size through simple centrifugation. While there are studies on the size separation of particles by centrifugation for gold nanoparticles and nanorods and the use of density gradients in the suspensions, one study was of particular interest.[19, 20] Doctor Ralf Kaegi, the head of the particle laboratory at the Swiss Federal Institute of Aquatic Science, conducted an experiment to investigate how TiO_2 nanoparticles used in

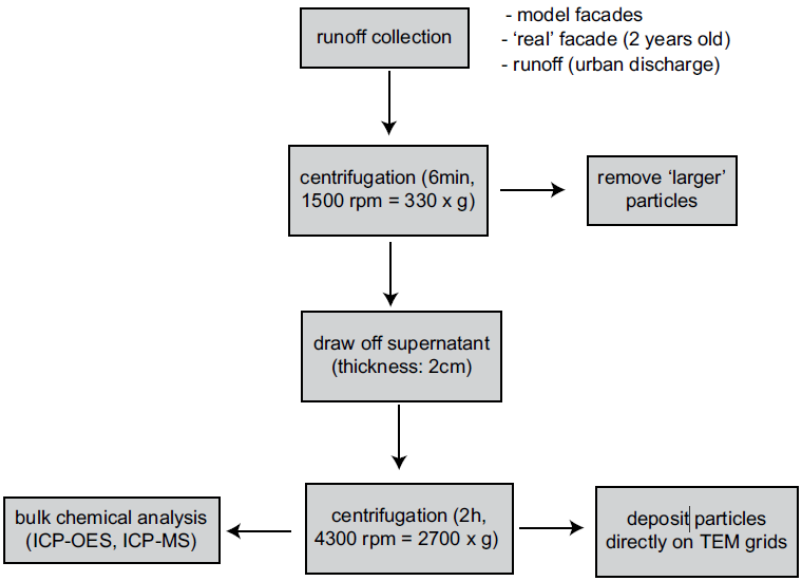


Figure 3 Doctor Ralf Kaegi's centrifugation data for the extraction of TiO2 nanoparticles from rainwater.[20]

paint were introduced to the natural waterways by rains.[20]

Doctor Kaegi and his team collected the water that ran off the painted surface and used a two-step centrifugation process, as indicated by Figure 3. In the first step, Kaegi and his team were able to reduce the particle size down to 300nm using a Megafuge swing bucket rotor and at a speed of 1500rpm, thus applying and average centrifugal acceleration 330 times that of gravity. He then removed the top 40mL of the centrifuged liquid, and ran in the centrifuge again at a speed of 4300rpm, thus applying and average centrifugal acceleration 2700 times that of gravity and reducing the particle size in the suspension to 20nm. This approach was the foundation for the research that follows in this chapter.

Experiments with Centrifugation

Extensive experiments were conducted in the laboratory to determine the whether we could separate the commercially prepared nanoparticles by size through centrifugation. In the following section, the suspension preparation method, the parameters that were examined, and the results of the experiments are discussed. The centrifugation experiments were conducted with both the Drucker Model 755VES centrifuge, capable of angular speeds up to 3500rpm and a maximum centrifugal acceleration of 2200g, and the Beckman Coulter Allegra 64R centrifuge, capable of speeds up to 21,000rpm and a maximum centrifugal acceleration of 41,420g using the 50mL centrifuge tube and the associated rotor. The Malvern Zetasizer Nano series is an instrument that uses the Dynamic Light Scattering technique to determine the size of particles

suspended in a liquid, with published accuracy as low as 0.6nm. In order to find the diameter of the particles in the suspension, there must be a concentration that is high enough to measure the motion of the particles within the suspension. The experiments using the Beckman Coulter centrifuge are not included in this paper, as the suspensions were too dilute for the Malvern Zetasizer to produce accurate size reports for the particles in the suspension. The desired outcome of these experiments is to remove the nanoparticles that are greater than 30nm or agglomerations that form to achieve suspensions that are comparable to particle sizes that are seen in the sol-gel nanoparticles.

Suspension Preparation

The suspension preparation method was kept as uniform as possible throughout the course of the experiments. The suspensions were prepared with 40mL of ethanol or water added to 45 mg of TiO_2 nanopowder from a commercial supplier, higher than previously used by the research group to ensure that the sample was not too dilute by the end of the centrifugation process. Certain iterations of the experiment required more of the suspension to be extracted at the desired height of the centrifuge tube, but the concentration was kept the same for these iterations. Once the nanopowders and solvent were combined in the centrifuge tube, the mixture was manually agitated for 30 seconds and placed in the ultrasonicator for 15 minutes.

Parameters Examined During Centrifugation

Using the equation derived above for the particle diameter during centrifugation, $x = \sqrt{\frac{18\mu v_r}{(\rho_p - \rho_f) r\omega^2}}$, the parameters to be analyzed can be extracted.[16] Both the distance from the axis of rotation (r) and angular velocity are inversely proportional to the particle size and were analyzed in the experiments below. The density of the fluid was a factor analyzed during initial centrifugation experiments. This was done by trying two different liquids: ethanol and water. The difference in the density, viscosity, and dielectric constant of the two solvents were of interest to us for electrophoretic deposition. In initial experiments, the attempts to create suspensions with water were not successful due to its instability because the suspensions would precipitate at low rpm settings or if left to the effects of gravity overnight. Using the Zetasizer to determine the diameter of the particles remaining in the suspension, the size reports indicated a concentration of TiO_2 that was too low to be measured, as shown in the size quality reports in Appendix A. Therefore, all experiments conducted below use ethanol as the solvent to prepare the suspension. Additionally, because one of the variables in the equation for the desired particle diameter is the radial velocity of the particle during the centrifugation, there is an implicit time variable that must be explored.[16]

Experiments on the Effect of Radial Distance on the Particle Diameter

Because the prepared suspensions must be placed in a plastic centrifuge as a part of a closed system, there is no effective way to change the distance of the

suspension from the axis of rotation. To analyze the effect this change had on the particle size, a syringe was used to extract the suspension from two locations in the centrifuge tube: the 30mL and the 15mL mark on the centrifuge tube. These two values were chosen to be far enough into the solution to avoid any sort of surface effects of particles on top of the liquid and to be far enough from the bottom of the centrifuge tube to avoid extracting some of the particles that were part of the sedimentation.

Using the equation $g = (1.118 \times 10^{-5}) r\omega^2$ to determine the centrifugal acceleration in units of earth's gravity, and measuring the radial difference between the 30mL and the 15mL mark on the centrifuge to be 4.7 cm, the change in location that the sample was extracted was subjected to would differ by a factor of 4.7 g's. To isolate effect that the 4.7cm difference of radial distance has on the particle, samples were obtained from the 30mL and 15mL marks at four distinct angular velocities for 15 minutes each (Figure 4). Initially, the particle size from the sample extracted from the 30mL mark was larger than that of the particle extracted from the 15mL mark, which does not coincide with the expected results using the calculations above. However, as the angular velocities were increased, the difference between the sizes of the particles at each of the two positions was essentially negligible given the associated standard deviation. Appendix B contains the reports generated by the Malvern Zetasizer for these results. By this experiment, the conclusion is made that the particle size is not dependent upon its location within the centrifuge tube so long as these particles are not a part of the sedimentation at the bottom of the tube.

	RPM	Z-Ave (d.nm)	PdI	σ
Sample drawn from the 30mL mark on the centrifuge tube	1000	411.8	0.342	240.8
	2000	217.4	0.145	82.8
	3000	173.1	0.120	60.0
	3500	151.6	0.083	43.7
Sample drawn from the 15mL mark on the centrifuge tube	1000	329.5	0.183	141.0
	2000	209.9	0.152	81.8
	3000	171.9	0.113	57.8
	3500	151.2	0.146	57.8

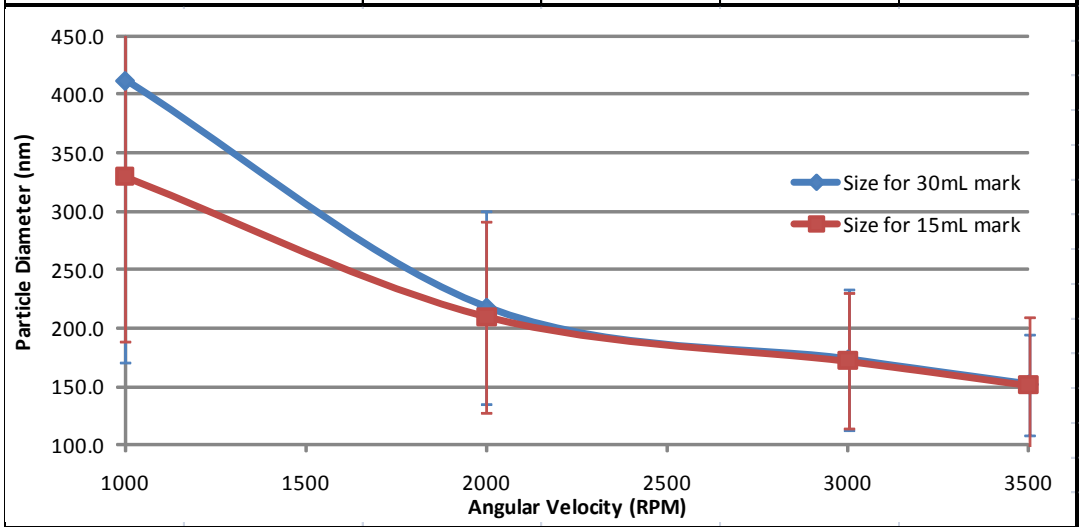


Figure 4 Numerical and graphical data for the particle size, polydispersion index, and the associated standard deviation based on the radial distance from the axis of rotation during centrifugation.

Experiments on the Effect of Angular Velocity on Particle Diameter

To isolate the effects of the angular velocity on the particle size, the same suspension was used for centrifugation, the sample was extracted from 15mL mark on the centrifuge tube, and the time period that each suspension underwent centrifugation was kept constant at 15 minutes. The diameter of the particles still in suspension was measured as the angular velocity of the centrifuge was varied from 1500rpm to 3500rpm in 500rpm intervals. The results show that the change in sampled particle size when centrifuged from 1500rpm to 2000rpm is significant, but that subsequent intervals do not seem to make a significant

change, especially with respect to the standard deviation of the particle size, as indicated by the error bars in the graph in Figure 5. Appendix C contains the reports generated by the Malvern Zetasizer for these results.

	RPM	Z-Ave (d.nm)	PdI	σ
Sample drawn from the 15mL mark on the centrifuge tube, variable RPM, constant time (15min)	1500	319.3	0.286	170.8
	2000	220.0	0.145	83.8
	2500	218.4	0.191	95.4
	3000	178.2	0.131	64.5
	3500	172.1	0.153	67.3

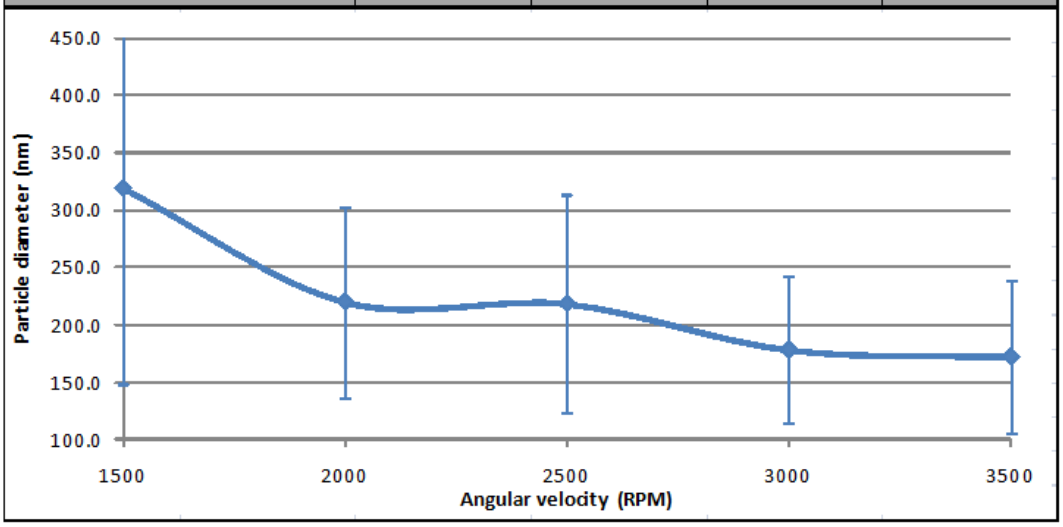


Figure 5 Numerical and graphical data for the particle size, polydispersion index, and the associated standard deviation based on the angular velocity during centrifugation.

Experiments on the Effect of Centrifugation Time on Particle Diameter

The last parameter analyzed was the amount of time that a suspension endured centrifugation. This was done by using the same suspension, extracting the sample from the 15mL mark on the centrifuge tube, and keeping the angular velocity constant at 3500rpm. The time interval was measured in 30 minute intervals, ranging from 30 minutes to 120 minutes. The results indicate that there is little effect on the ability to separate the commercial nanoparticles by size

using a constant angular velocity and increasing the amount of time. These results are depicted in Figure 6 and detailed reports printed in Appendix D.

	Time (min)	Z-Ave (d.nm)	PdI	σ
Sample drawn from the 15mL mark on the centrifuge tube, constant RPM (3500), variable time	30	142.9	0.142	53.8
	60	124.5	0.109	41.1
	90	116.1	0.116	39.5
	120	109.7	0.179	46.4

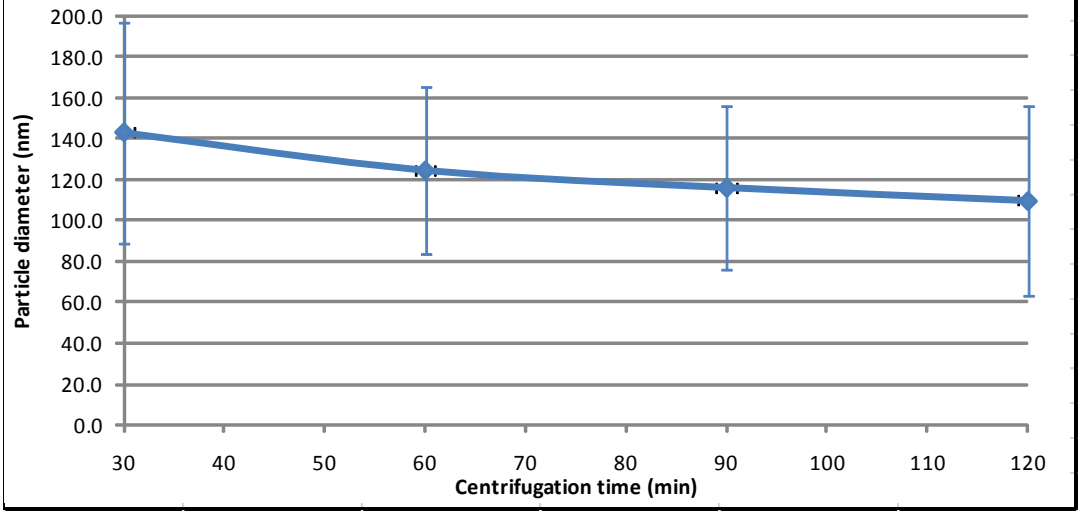


Figure 6 Numerical and graphical data for the particle size, polydispersion index, and the associated standard deviation based on the amount of time the suspension underwent centrifugation.

Effectiveness of Size Separation through Centrifugation

Through the centrifugation techniques employed, nanoparticles less than 30nm in diameter were not able to be separated and stay suspended in the solvent at concentrations that could be measured with the Malvern Zetasizer. It was possible to separate nanoparticles down to 150nm with concentrations high enough to be evaluated by the Malvern Zetasizer. However, achieving particles smaller than 30nm at a sufficient concentration for EPD was not possible with the centrifugation methods used in these experiments.

CHAPTER IV

ELECTROPHORETIC DEPOSITION OF THIN FILMS USING TITANIUM DIOXIDE SUSPENSIONS

Scientists and industrial leaders have been using various deposition techniques to make thin films of nanoparticles, including chemical vapor deposition, sputtering, dip casting, and electrophoretic deposition.[21] In the creation of films made from titanium dioxide nanoparticles, electrophoretic deposition is a favored method due the short time period required for deposition, the ability to deposit a film on a non uniform surface, the small equipment footprint and associated costs, and the ability to control the thickness of the films.[22] This section will provide an overview of the fundamentals of electrophoretic deposition, how each of the variables in the deposition process affects the creation of thin films, what variables were analyzed in the course of this research, and an analysis of the films created with the sol-gel suspension from our collaborators and the suspensions prepared with the commercially produced titanium dioxide nanoparticles.

Fundamentals of Electrophoretic Deposition

The basic concept behind electrophoretic deposition for TiO_2 nanoparticles, as shown in Figure 7, is simple: voltage is applied to a suspension; the charged nanoparticles move towards the electrode of opposite charge (electrophoresis) and form a stable deposition of a desired thickness on its surface (deposition).[23]

While the concept sounds simple, the physics behind the process has proven to be more difficult to explain.

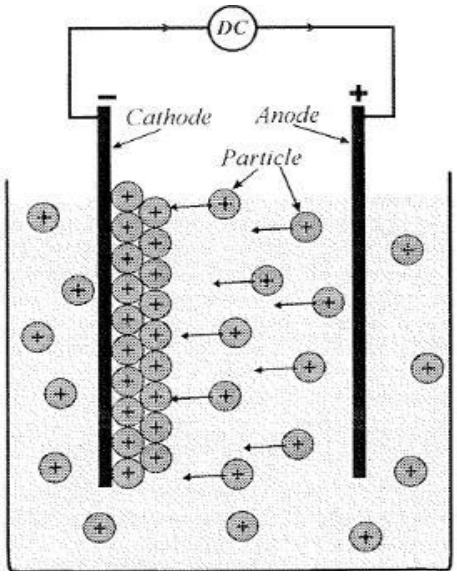


Figure 7 Schematic of electrophoretic deposition process.[23]

Electrophoresis is governed by four different forces. When the voltage is applied to the electrodes, an electric field is created that interacts with the surface charge of the nanoparticle, producing a force that moves the particle towards the electrode of the opposite charge.[24] The other three forces work to counter the force due to the electric field. The drag created by the viscosity of the fluid reduces the ability of the particle to move in the suspension. Additionally, the ions from the solvent used in the suspension surround the particle, and exert a force due to the electric field in the opposite direction, which retards motion. Lastly, the distortion in the double layer, described later in this chapter, caused by the displacement between the center of the positive and negative charges cause the particle's motion toward the electrode to be inhibited.[24]

The combination of all these forces is used to calculate the electrophoretic mobility of the particles, which describes the particles' ability to move in a specific solvent under an electric field, as defined by the equation

$$\mu_e = \frac{v}{E} = \frac{\epsilon\zeta}{\eta}$$

where *v* is the velocity of a particle in the electric field (E), ϵ is the dielectric constant for the solvent used, ζ is the zeta-potential of the particles in the

suspension, and η is the viscosity of the liquid in the suspension.[25] The viscosity of the liquid is one of the characteristics of the suspension that inhibits the free movement of the particles towards the electrode, as indicated by the equation above.[24]

The electrophoretic mobility of a particle depends on these interrelated properties of the nanoparticles and the solvent used to prepare the suspension. The size of the particles in the suspension is directly related to the stability of the suspension and the quality of the films created by the deposition. The larger the particles are, the more they tend to sediment due to the effects of gravity. To make a uniform film, the effects of gravity must be overcome to deposit the particles onto the electrode and avoid the creation of a film with a thickness gradient as the particles settle. The electric field must be increased to avoid the creation of a nonuniform film on the electrode due to force of gravity. This can be done in one of three ways: use a particle with a larger charge to mass ratio, increase the size of the electric double layer of the particle, or increase the voltage applied across the electrodes. Additionally, the smaller the particles are, the higher the ratio is between the surface charge and the mass of the particle, increasing the mobility of the particle.[26] While the charged particle is suspended in a liquid, ions of the opposite charge are attracted to the particle, and ions with the same charge of the particle are attracted to the first layer of ions, essentially creating two concentric spheres of opposite charge about the particle called the double layer, as depicted in Figure 8. The potential difference between the two ionic layers is known as the zeta potential, another suspension characteristic that

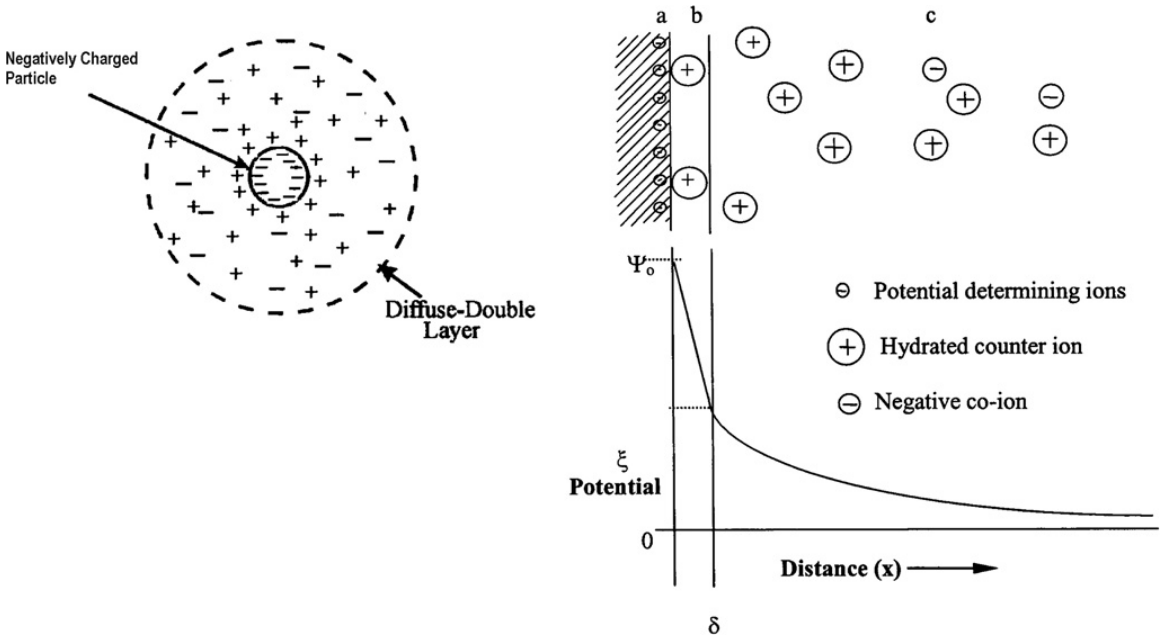


Figure 8 Graphical depiction of the diffuse double layer surrounding a particle and the zeta potential measurement locations, where (a) is the surface layer, (b) is the Stern layer, and (c) is the diffuse layers of the counter-ions.[22]

plays a vital role in electrophoretic deposition.[23] The zeta potential, related to the suspension characteristics through the equation $\mu_e = \frac{v}{E} = \frac{\epsilon\zeta}{\eta}$, is used to determine the stability of the suspension, the direction and velocity of a particle during EPD, and can predict which electrode the particles would be deposited on.[22]

The dielectric constant of the liquid used for the suspension must be in a range that optimizes the particles ability to move towards the electrode. The dielectric constant of a body is the ratio of capacitance of two plates in a vacuum to the capacitance of two plates with the body in between, thus getting a mathematical factor of the ability of the medium to hold a charge.[25] Using a liquid with dielectric constant that is too low will cause little to no deposition due to the insufficient dissociative energy, and those liquids with high dielectric constants reduce the size of the double layer region in the particles.[22] The conductivity of the suspended particle proves to be an important factor in the ability of the

particle to be deposited. In studies by Ferrari and Moreno, they found that particles had to be within a specific range of conductivity values for both polar and nonpolar suspensions.[27] If the conductivity values were too low or two high, the particles would not deposit on the electrode, no matter how strong the intensity of the electric field was or the length of time the electric field was applied.[27]

Additionally, attention must be paid to the solvents' ability to keep the nanoparticles suspended. Stable suspensions allow for particles to maintain a uniform dispersion throughout the liquid, limiting agglomeration, and allowing the particles to move toward, and deposit on, the electrode separately.[28] The stability is widely characterized by the zeta potential, where the higher the absolute value of the zeta potentional is, the more stable the suspension is; though this is not a steadfast rule.[22] The best was to characterize the stability of a suspension is to observe that it has a slow rate of settling, shows little propensity to flocculate, and forms dense and strongly adhering deposits.[22, 23] Once we take all of the suspension characteristics together, we find that the best solvents to use are ones that allow for a stable suspension of the desired particles, have low viscosity, high dielectric constants, and low conductivity.[22]
The deposition process is the coagulation of the particles on the electrode, producing a stable film of dense mass.

Theories to explain the deposition mechanisms at the electrode include particle neutralization at the electrode, coagulation at the electrode due to the formation of ions from the reactions of the particle and the electrode, and the

effects of double-layer distortion during electrophoresis followed by the coagulation of the particles at the electrode due to the applied electric field.[28] While the effects of the interactions between the particle and the electrode do cause some coagulation play role in the deposition process, Sarkar and Nicholson searched for definitive proof and conducted an experiment to determine if one or all of these theories were correct. After placing a dialysis membrane between the electrodes during an EPD, they were able to get a deposition on the membrane and still register a current between the plates. Because deposition was seen on the membrane and a current was still measured between the two plates due to the ions passing through the membrane, Sarkar and Nicholson determined that the particle/electrode interaction and the charge neutralization at the electrode did not play a part in the deposition of materials[23]

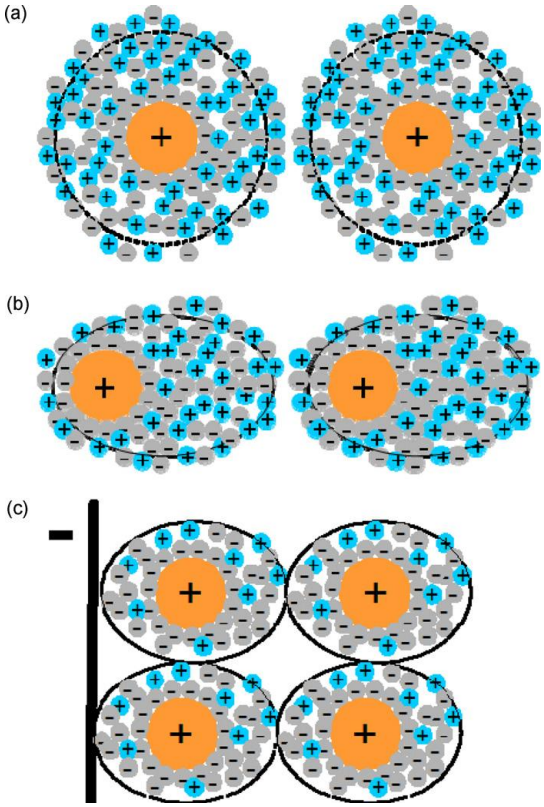


Figure 9 Depiction of the particle dispersed electrostatically(a), the distorted doubly layer due to the motion of the particle during electrophoretic deposition(b), and the change in ion distribution at the electrode(c).[28]

This section will explain the basics of the effects of double-layer distortion and how it affects coagulation at the electrode surface. As briefly noted earlier, charged particles suspended in a liquid will attract ions of opposite charge and create a sphere around the charged

particle. As the particle moves in the liquid, this sphere becomes distorted and the distribution of charge changes, causing the ions to be more heavily distributed on the trailing side. As a result of this distortion, the tail will become so thin and stretched out that the next particle in motion will approach close enough for van der Waals attractive force to be stronger than that of the repulsion force cause by the double-layer interaction and will induce deposition (Figure 9).[22] This is what gives the nanoparticle films created through EPD their strength.

Mathematically, this process is described by the DLVO theory, named after Derjaguin, Landau, Verwey, and Overbeek.[29, 30] This theory combines the effect of van der Waals attractive forces and the counter forces of the ion surrounding the particle to describe the motion of a charged particle through a liquid. Mathematically, this is often depicted as

$$V_{TOTAL} = V_A + V_R$$

where V_A is the van der Waals attractive force and V_R is the electrostatic repulsive force.[22, 31, 32] After applying Derjaguin's approximation, V_A and V_R are reduced to the following two equations:

$$V_A = -\frac{A_H}{6D}\left(\frac{a_1 a_2}{(a_1 + a_2)}\right);\ V_R = 2\pi r \varepsilon \Psi_0^2 ln\left[1 + e^{(-\kappa D)}\right]$$

where A_H is Hamaker's constant, $a_{1,2}$ are the radius of two different sized particles that are interacting, D is the distance between the surfaces of the two interacting particles, ε is the permittivity of the solvent, Ψ_0 is the surface potential, and κ is the reciprocal of the double layer thickness.[31, 32] As shown in Figure 10, there is an energy barrier that particles must overcome to irreversibly adhere to

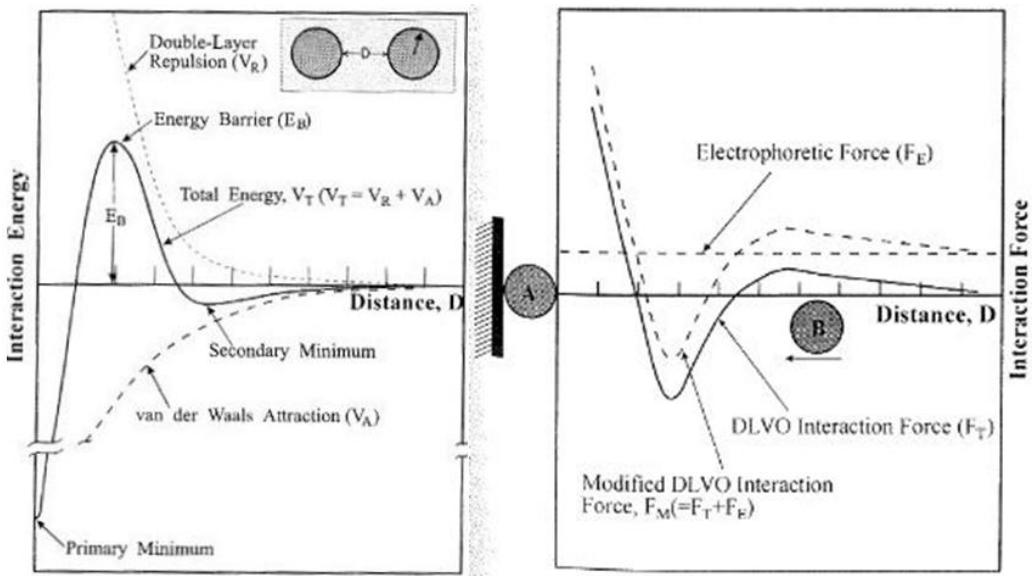


Figure 8 Particle-particle interaction energy and force diagrams based on the DLVO theory.[23]

each other, and the graph indicates the minimum electric field required for coagulation at the electrode.[23] As the deposition is occurring on the electrode, the electric field will become weaker due to the increased resistance of the electrode caused by the deposited material as well as the decreased concentration of the charged particles near the electrode. Therefore, it is common practice to apply an electric field that maintains a constant current between the electrodes, thus keeping a constant electric field and constant rate of deposition.[23]

Variables and Parameters for Electrophoretic Deposition

In 1940, Hugo Christiaan Hamaker studied the process of electrophoretic deposition and, based on the principle of conservation of mass, derived the equation

$$m = \mu ESct$$

This equation defines the mass that is deposited on an electrode based on the

electrophoretic mobility of the particles in the suspension (μ), the electric field across the electrodes (E), the surface area of the electrodes (S), and the concentration of the particles in the suspension (c), and the time that the deposition was occurring (t).[33] While this equation may not be exact in determining the amount of deposition that occurs, because of changing concentrations of particles around the electrode or a change in the electric field due to previously deposited material on the electrode, it does provide a relationship between the key factors of EPD that can be varied in the deposition process.

For the scope of the research discussed in this paper, the electrophoretic mobility of the particles in each suspension was noted but was not varied in order to research its effect on the characteristics of the deposited materials. A change in the electrophoretic mobility of the particles would have required adjusting the pH of the suspension, and adjusting both the pH and the electrophoretic mobility were omitted from the research parameters. The electric field used for the deposition was created by applying a constant voltage to the electrodes as opposed to the constant current setting. While deposition with constant current is the preferred method amongst the leaders in electrophoretic deposition, research performed in our lab was done with a constant voltage applied to avoid the effects of electrolysis that would be prevalent at the electrode and would cause an environment that would not be conducive to quality film formation.[34] The surface area of the electrodes was not varied during the research on the electrophoretic deposition iterations using the commercially prepared

nanopowders or the iterations using the sol-gel suspensions, as the electrode sizes, deposition depth, and volume of the suspension that the electrodes were exposed to remained constant. However, the surface areas of the electrodes were not the same between each of those two experiments. Similarly, the sol-gel suspensions had the same concentration for all performed deposition experiments and the concentration of the suspensions made from the Nanoamor nanopowders were the same for all deposition experiments conducted with the ethanol based suspensions. Time was the other variable used to see how the deposition was affected. The time of deposition was changed in both a continuous and continual manner; that is, the length of the time interval was changed, as was the number of times the interval was repeated. Additionally, an artificial field gradient was created for a set of electrodes by making an indentation on the surface of the steel electrode to see if there was any change in the nanoparticles propensity to deposit where a field gradient existed. The details of the parameters associated with each experiment will be elaborated on in the following sections.

Electrophoretic Deposition Using Commercially Purchased TiO_2 Nanopowder

As indicated in the previous chapter, numerous attempts were made to achieve a suspension made from TiO_2 nanopowder (Nanoamor, 99% purity, 10nm APS) that was stable, that had small particle diameters, and that were concentrated enough to make quality depositions after the centrifugation processes. Because the suspensions prepared with water were unstable and

flocculation was visually evident, the solvent chosen for the preparation of the nanopowder suspension was ethanol. Three suspensions were used to analyze the difference between two different voltages applied to the electrodes, the effects of an electric field gradient produced by the indentation in the electrode, and the effects of multiple depositions on the same electrode. The rest of this section will describe the procedure, expected results, and the actual results.

Suspension preparation

All three suspensions were created by adding 50mL of ethanol to 50mg of TiO_2 nanopowder purchased from a Nanoamor that was placed in a 50mL centrifuge tube. The mixture was manually agitated for 30 seconds and placed in the ultrasonicator for 15 minutes. Three of the suspensions were then placed in the centrifuge to separate out the large agglomerations. Suspension 1 was then placed in the centrifuge for 30 minutes at 2000rpm, suspension 2 was placed in the centrifuge for 90 minutes at 3500rpm, and suspension 3 was placed in the centrifuge for 90 minutes at 3500 followed by a 30 minutes in a larger centrifuge for 30 minutes at 15,000rpm. Due to the extra centrifugation in suspension 3, the concentration of the nanoparticles became too low for the Malvern DLS system to get accurate readings on particle size and zeta potential. The characteristics of these three suspensions are

Table 1 Electrophoretic mobility, Zeta potential, particle size, and the polydispersion index values for the three suspensions used for the electrophoretic deposition experiment.

	Electrophoretic Mobility (μ)	Zeta Potential (ζ)	Particle Size (nm)	Quality of Size Data (PDI)
Suspension 1	-0.3672	-18.40	268.0	0.352
Suspension 2	-0.2023	-10.10	179.1	0.104
Suspension 3	-0.0053	-0.27	379.4	0.465**

shown in Table 1 and the full data reports are shown in Appendix E. By analyzing the data at in this table, the high value of zeta potential and electrophoretic mobility of suspension 1 should indicate better suspension stability and better particle mobility in the suspension. Therefore, electrophoretic deposition experiments using suspension 1 should have a larger amount of deposited materials on the electrodes than the electrodes from the other two suspensions.

Deposition Process

To conduct the electrophoretic deposition experiments in the lab, the electrodes, thin sheets of steel cut into one inch long by one-half inch wide strips, were placed between microscope slides to create a separation distance of one centimeter separation. Voltage was applied to these electrodes through copper tape on the inside of the glass slides. The voltage was checked at the leads and the electrode with a voltmeter to ensure that the proper voltage was applied to the electrodes. Beginning with suspension 1, a 20mL beaker was filled with 12.5mL of the prepared suspension and placed in the EPD chamber. Working through the Labview software, 50 Volts DC was applied to the electrodes and the electrodes were lowered into the suspension for 15 minutes. After the 15 minute time period, the electrodes were extracted and allowed to dry with the voltage applied for 5 minutes, at which point the voltage is turned off.

This process is repeated with a second set of steel electrodes, except this time, 70 Volts DC was applied to the system. For the next set of electrodes, this

process was repeated three times with the same set of electrodes, with a one minute time period between each interval where the voltage was not applied. These three processes were performed with each of the three prepared suspensions, and were repeated again with electrodes with intentional indentations to observe the effects of an electric field gradient.

Results and Discussion

The results for electrophoretic deposition using the suspensions created from the nanopowders failed to produce uniform films on the steel electrodes. During the EPD process, suspension 1 had a large amount of flocculation and associated sedimentation around both electrodes, indicating a lack of stability in that suspension. The electrodes showed a faint white deposition around the edges and a heavy white deposition at the meniscus for each of the depositions. The EPD performed with 70 Volts caused many more particles to adhere to the electrodes, however with no significant changes were observed for the electrodes that were exposed to multiple depositions or experienced an electric field gradient.

The SEM images from the electrodes using suspension one showed large agglomerations on the electrode, but no densely pack materials. Depositions with suspension 2 produced a small amount of flocculation and precipitation, though this was concentrated beneath the negative electrode. Again, the electrodes that underwent electrophoretic deposition in suspension 2 only had visual indications of a film on the outer edges of the negative electrodes and on

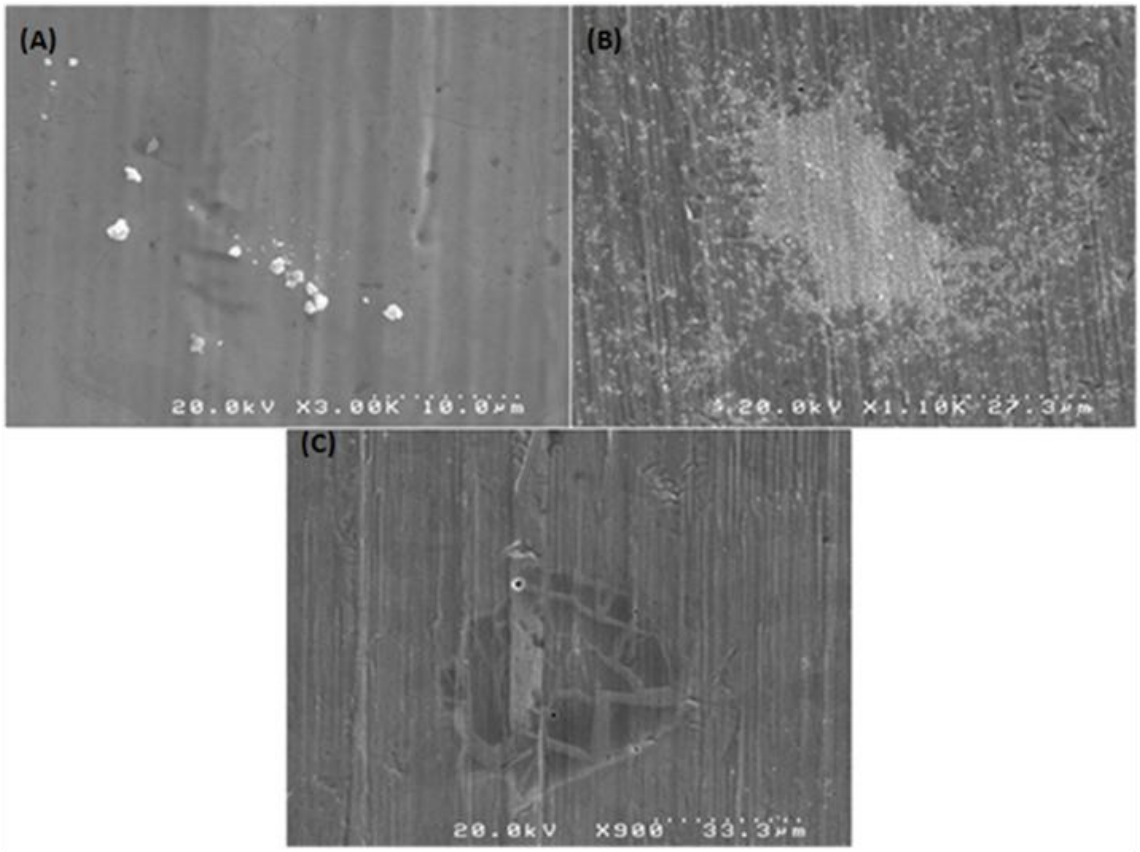


Figure 11 SEM images of the deposition on the electrodes from suspension 1 (A), suspension 2 (B), and suspension 3 (C). Deposition from suspension 1 shows only a few large agglomerations of particles on the electrode. Deposition from suspension 2 shows a patch of densely packed particles approximately 200nm each in diameter while suspension 3 produced no deposition

the meniscus for both the positive and negative electrodes with the only changes in the amount deposited using multiple depositions or different voltages during the EPD. Depositions with suspension 2 showed small patches of densely packed particles, but were only seen using the higher voltages. Again, no change was seen when using multiple depositions or when an electric field gradient was created. Suspension 3 showed no flocculation or sedimentation during the experiment, and showed no signs of deposition on the electrode visually or with under SEM. Most of results meet expectations due to the characteristics of the suspensions used.

The zeta-potentials of the suspensions were relatively small and sedimentation was evident after periods of inactivity or a lack of agitation, indicating a lack of stability of the suspensions. Thus, the flocculation observed in during the electrophoretic deposition was anticipated. Another anticipated result was that there was more deposition on the edges of the electrodes, as there were more particles near the edges of the electrodes that were subject to the effects of the electric field applied. Additionally, the deposition occurred primarily on the positive electrode, as expected given the negative zeta potential. I would have expected there to have been an increased deposition either on the peak of the created indentation on the positive electrode, or a concentrated group of particles opposite the peak on the negative electrode, since the electric field gradient would be larger at the point of indentation, and the electric field would be greater due to the decreased distance between the plates. Due to the instability of the suspension using the nanopowders and the consequential inability to make consistent uniform films with these suspensions, the focus of the researched shifted to the sol-gel suspensions provided by our collaborators.

Electrophoretic Deposition Using Eu doped TiO_2 Sol-gel Suspension

The sol-gel suspensions used for electrophoretic deposition were obtained from Dr. Rodrigo Moreno and his group, from the Institute of Ceramics and Glass(CSIC), in Madrid, Spain. This suspension had a ratio of water to Ti(IV)-isopropoxide that was 1:50, with a small amount of acetate and nitric acid that was used to adjust the pH and introduce europium (2%) into the suspension.

Due to the high water content of the suspension, low voltage was used to prevent electrolysis at the electrode. The first experiments were performed using steel electrodes, where it became evident that films could be created that were of much higher quality compared to the previous nanopowder suspensions. The original parameters for the sol-gel deposition process remained the same as the process for the nanopowders: one inch long by one-half inch wide steel electrodes separated by one centimeter. The first deposition was performed with

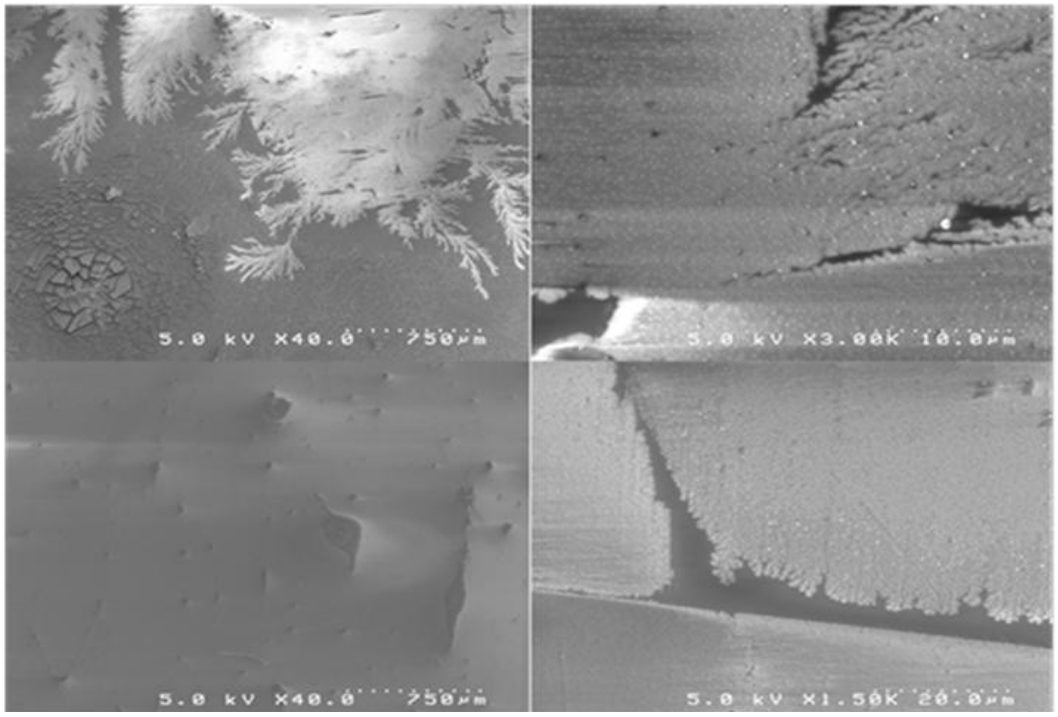


Figure 12 SEM imagery of the results from electrophoretic deposition experiments and dipcasting of two different electrodes using the sol-gel suspensions from our collaborators.

an applied voltage of 2.5 Volts, a 10 minute deposition time, and a 5 minute annealing time, based on preliminary research performed by a former group member. Next, a film was produced by dip casting to observe the similarities and differences between these films and the films produced through electrophoretic deposition. Looking at the films with the SEM, there was very little visual

difference between the two films. In fact, the film created through dip-casting seemed to be more uniform and less brittle than the film created through EPD. After consulting one of my group members, Dr. Isabel Gonzalo de Juan, she suggested that the brittleness of the film possibly could be attributed to electrolysis at the electrode and that I needed observe into the change in pH and the change in conductivity of the suspension compared with the voltage applied during an electrophoretic deposition experiment.

Due to a small supply of the europium doped TiO_2 sol-gel suspension, the electrophoretic deposition was conducted using the same 40mL sample, using steel electrodes, and a deposition time of 10 minutes. The experiments were done repeatedly, decreasing the voltage first in .25V increments, then in .05V increments until the pH of the suspension remained unchanged. After completing the experiments, the optimal voltage to maximize the electric field and not encounter electrolysis was determined to be 1.9V. Additionally, the substrate used for deposition was changed from using steel electrodes to ITO to eliminate the flexibility or deformations of the steel as a source of the brittleness and to better observe the optical properties of the film.

To examine the qualities under the SEM and using spectrophotometric analysis, films were created using both dip casting and electrophoretic deposition. The electrodes used to create the dip cast were placed in the electrode holder with one centimeter separation. They were then lowered into a 25mL beaker for 10 minutes and raised out of the suspension to dry for 25 minutes. The electrodes that were used create films through EPD underwent the

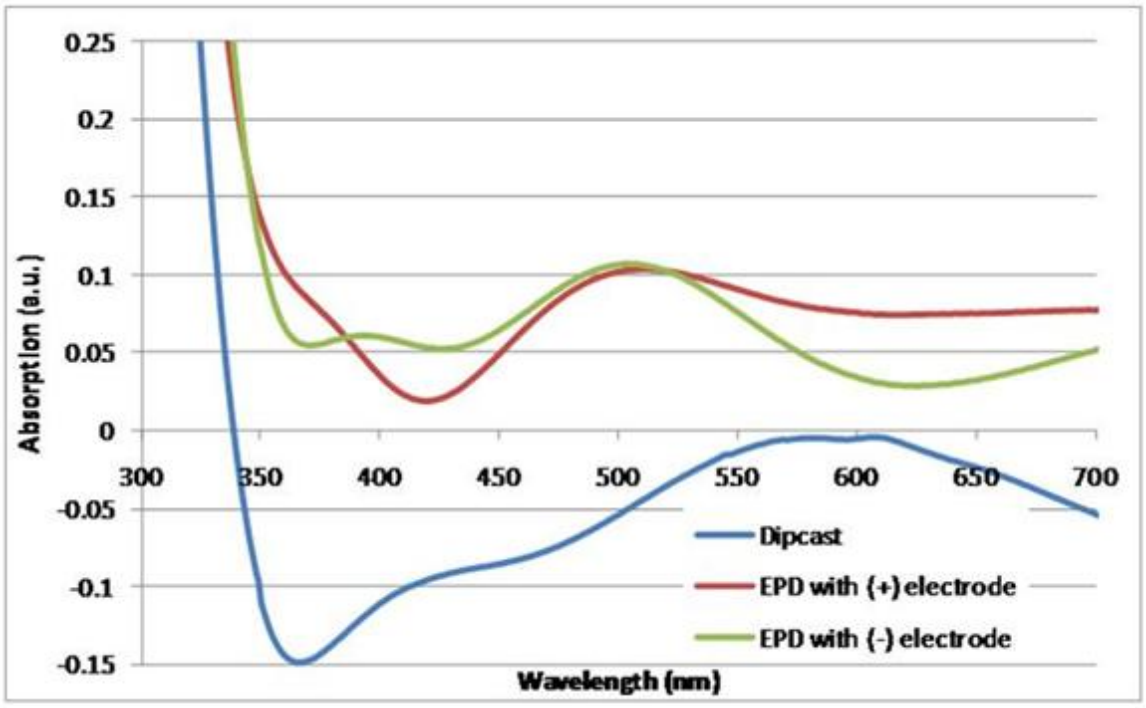


Figure 13 Spectrophotometer results from the electrophoretic deposition experiments and dipcasting of two different electrodes using the sol-gel suspensions from our collaborators, showing different absorption properties between the films.

same process except also had 1.9V applied throughout the process. Visually, both films looked uniform and did not appear to have much cracking or flaking with the exception of the bottom of the electrode, where the liquid accumulated during the drying process. Looking at the films under the SEM, both films appear to have a deposition of the TiO_2 nanoparticles. Both the dip cast and the films produced with EPD have TiO2, but the dip cast film looks life it precipitated in the shape of tree branches where the EPD film appeared to be more uniformly deposited, as seen in Figure 12. Spectrophotometric analysis was performed on both the films using the Varian Cary 5000 UV-VIS-NIR Spectrophotometer to compare the absorption of the two films (Figure 13). The results show that there is a distinct difference in the absorption characteristics of the two films, where the films created through EPD have an absorption peak that is shifted towards the

UV range. This suggests that the electrophoretic deposition either attracts or repels material that is not attracted or repelled through the dip casting process. This is the case for both the positive and negative electrode, so the material that is present in the dip cast film is likely charge neutral.

CHAPTER V

CONCLUSION

The research in this paper discussed the characteristics of titanium dioxide nanoparticles, the size separation techniques employed through centrifugation, and the electrophoretic deposition of thin film of TiO_2 nanoparticles onto a substrate. Reviewing the results from this work, size separation of nanoparticles below 100nm was not achieved with the centrifugation methods employed, suspensions prepared with the TiO2 nanopowders were not stable and proved problematic in electrophoretic deposition experiments, and the films created through electrophoretic deposition had fundamental differences than those created by dip casting when using sol-gel suspensions.

Future research using centrifugation for size separation of nanoparticles in a suspension prepared using a solvent and nanopowders should focus on the creation of a stable suspension. Suspension stability could be achieved through a different solvent being used, or using water with another solvent that would adjust the pH in the suspension high enough so that the zeta potential would be increased, thereby increasing the stability of the suspension.

Continued research in the electrophoretic deposition experiments using the sol-gel suspension provides many opportunities for growth in the scientific and industrial communities. Because the band gap of materials increases as the size of the nanoparticle decreases, it would be possible to tailor a material, whether it

is with one layer of nanoparticles or multiple layers of nanoparticles, to achieve a desired band gap. Such technology would be welcome in optics and in photovoltaics, where the band gap of the material is essential in the efficiency and cost effectiveness of the solar cell.

APPENDIX A

SIZE DATA REPORTS FOR SUSPENSION PREPARATION USING WATER

Size Distribution Report by Intensity

Sample Details

Sample Name	TiO2 in Water, 1500rpm, 15min
SOP Name:	TiO2 Size in water glass.sop
General Notes:	

File Name:	DATA.dts	**Dispersant Name:**	Water
Record Number:	22	**Dispersant RI:**	1.330
Material RI:	2.49	**Viscosity (cP):**	0.8874
Material Absorbtion:	0.40	**Measurement Date and Time:**	Thursday, July 15, 2010 11:2.

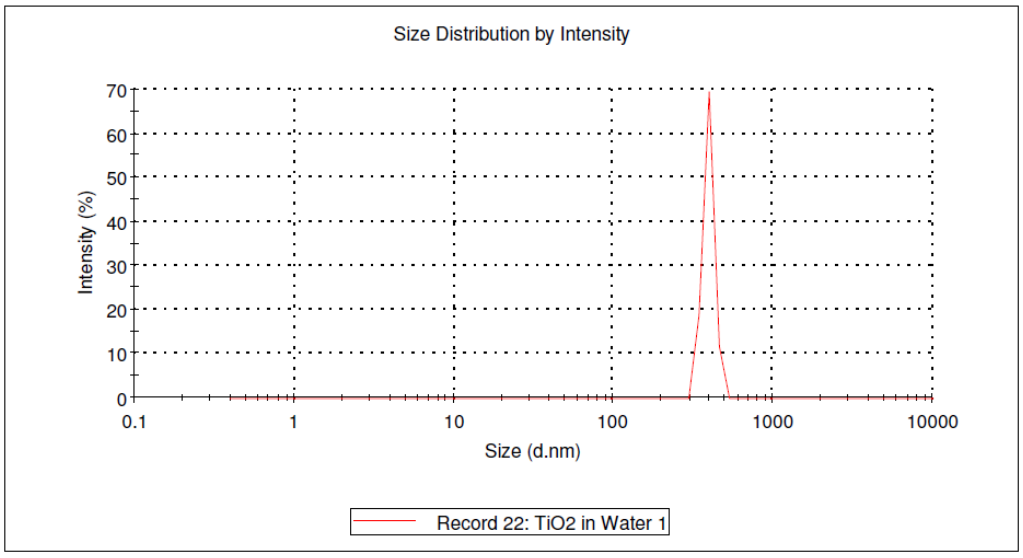
System

Temperature (°C):	25.1	**Duration Used (s):**	70
Count Rate (kcps):	117.3	**Measurement Position (mm):**	4.65
Cell Description:	Glass cuvette with round apert...	**Attenuator:**	7

Results

			Diam. (nm)	% Intensity	Width (nm)
Z-Average (d.nm):	1985	**Peak 1:**	393.3	100.0	31.67
PdI:	0.985	**Peak 2:**	0.000	0.0	0.000
Intercept:	1.10	**Peak 3:**	0.000	0.0	0.000

Result quality : POOR - see result quality report



Size Results Export Report

Sample Name TiO2 in Water, 1500rpm, 15min
Record Number: 22

Results Table

Size d.nm	Intensity %	Size d.nm	Intensity %
0.4000	0.0	68.06	0.0
0.4632	0.0	78.82	0.0
0.5365	0.0	91.28	0.0
0.6213	0.0	105.7	0.0
0.7195	0.0	122.4	0.0
0.8332	0.0	141.8	0.0
0.9649	0.0	164.2	0.0
1.117	0.0	190.1	0.0
1.294	0.0	220.2	0.0
1.499	0.0	255.0	0.0
1.736	0.0	295.3	0.0
2.010	0.0	342.0	18.8
2.328	0.0	396.1	69.4
2.696	0.0	458.7	11.8
3.122	0.0	531.2	0.0
3.615	0.0	615.1	0.0
4.187	0.0	712.4	0.0
4.849	0.0	825.0	0.0
5.615	0.0	955.4	0.0
6.503	0.0	1106	0.0
7.531	0.0	1281	0.0
8.721	0.0	1484	0.0
10.10	0.0	1718	0.0
11.70	0.0	1990	0.0
13.54	0.0	2305	0.0
15.69	0.0	2669	0.0
18.17	0.0	3091	0.0
21.04	0.0	3580	0.0
24.36	0.0	4145	0.0
28.21	0.0	4801	0.0
32.67	0.0	5560	0.0
37.84	0.0	6439	0.0
43.82	0.0	7456	0.0
50.75	0.0	8635	0.0
58.77	0.0	1.000e4	0.0

Right click the table to change the displayed parameters and/or change the number of columns. Use the Edit - Copy Table command to copy the data for pasting into a third party spreadsheet or graphing package.

Trend Table

Rec #	Z-Avg d.nm	Pk 1 Avg (I) d.nm	Pk 2 Avg (I) d.nm	Pk 3 Avg (I) d.nm	DCR kcps
22	1985	393.3	0.000	0.000	13586.4

Size Distribution Report by Intensity

Sample Details

Sample Name: TiO2 in Water, 2500rpm, 15 min
SOP Name: TiO2 Size in water glass.sop
General Notes:

File Name:	DATA.dts	**Dispersant Name:**	Water
Record Number:	18	**Dispersant RI:**	1.330
Material RI:	2.49	**Viscosity (cP):**	0.8861
Material Absorbtion:	0.40	**Measurement Date and Time:**	Thursday, July 15, 2010 9:19:

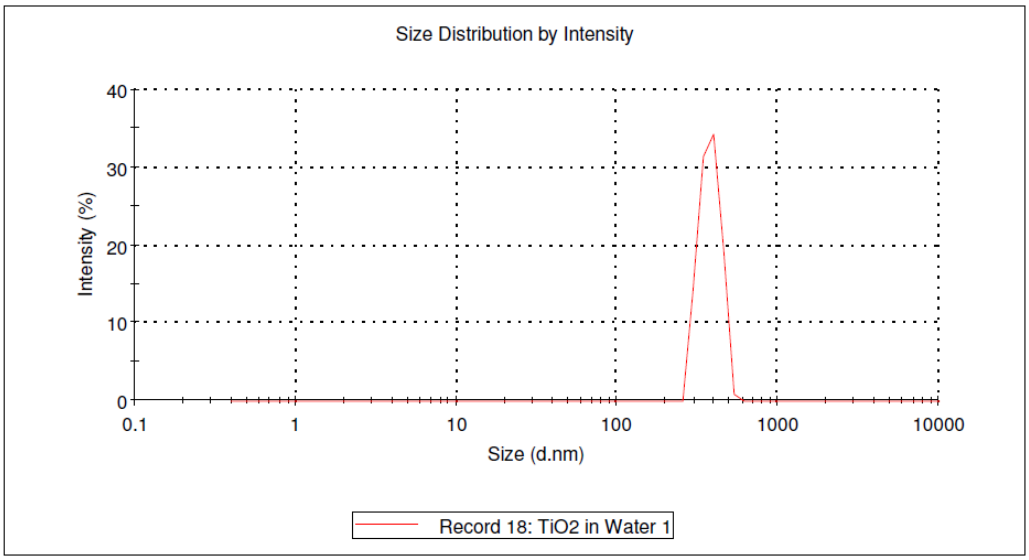
System

Temperature (°C):	25.0	**Duration Used (s):**	80
Count Rate (kcps):	227.3	**Measurement Position (mm):**	4.65
Cell Description:	Glass cuvette with round apert...	**Attenuator:**	8

Results

			Diam. (nm)	% Intensity	Width (nm)
Z-Average (d.nm):	877.9	**Peak 1:**	377.7	100.0	54.33
PdI:	0.903	**Peak 2:**	0.000	0.0	0.000
Intercept:	1.02	**Peak 3:**	0.000	0.0	0.000

Result quality : POOR - see result quality report



Size Results Export Report

Sample Name TiO2 in Water, 2500rpm, 15 min
Record Number: 18

Results Table

Size d.nm	Intensity %	Size d.nm	Intensity %
0.4000	0.0	68.06	0.0
0.4632	0.0	78.82	0.0
0.5365	0.0	91.28	0.0
0.6213	0.0	105.7	0.0
0.7195	0.0	122.4	0.0
0.8332	0.0	141.8	0.0
0.9649	0.0	164.2	0.0
1.117	0.0	190.1	0.0
1.294	0.0	220.2	0.0
1.499	0.0	255.0	0.0
1.736	0.0	295.3	14.4
2.010	0.0	342.0	31.4
2.328	0.0	396.1	34.3
2.696	0.0	458.7	19.1
3.122	0.0	531.2	0.9
3.615	0.0	615.1	0.0
4.187	0.0	712.4	0.0
4.849	0.0	825.0	0.0
5.615	0.0	955.4	0.0
6.503	0.0	1106	0.0
7.531	0.0	1281	0.0
8.721	0.0	1484	0.0
10.10	0.0	1718	0.0
11.70	0.0	1990	0.0
13.54	0.0	2305	0.0
15.69	0.0	2669	0.0
18.17	0.0	3091	0.0
21.04	0.0	3580	0.0
24.36	0.0	4145	0.0
28.21	0.0	4801	0.0
32.67	0.0	5560	0.0
37.84	0.0	6439	0.0
43.82	0.0	7456	0.0
50.75	0.0	8635	0.0
58.77	0.0	1.000e4	0.0

Right click the table to change the displayed parameters and/or change the number of columns. Use the Edit - Copy Table command to copy the data for pasting into a third party spreadsheet or graphing package.

Trend Table

Rec #	Z-Avg d.nm	Pk 1 Avg (I) d.nm	Pk 2 Avg (I) d.nm	Pk 3 Avg (I) d.nm	DCR kcps
18	877.9	377.7	0.000	0.000	6615.6

Size Distribution Report by Intensity

Sample Details

Sample Name: TiO2 in Water, 3500rpm, 15min

SOP Name: TiO2 Size in water glass.sop

General Notes:

File Name:	DATA.dts	**Dispersant Name:**	Water
Record Number:	23	**Dispersant RI:**	1.330
Material RI:	2.49	**Viscosity (cP):**	0.8883
Material Absorbtion:	0.40	**Measurement Date and Time:**	Thursday, July 15, 2010 11:2.

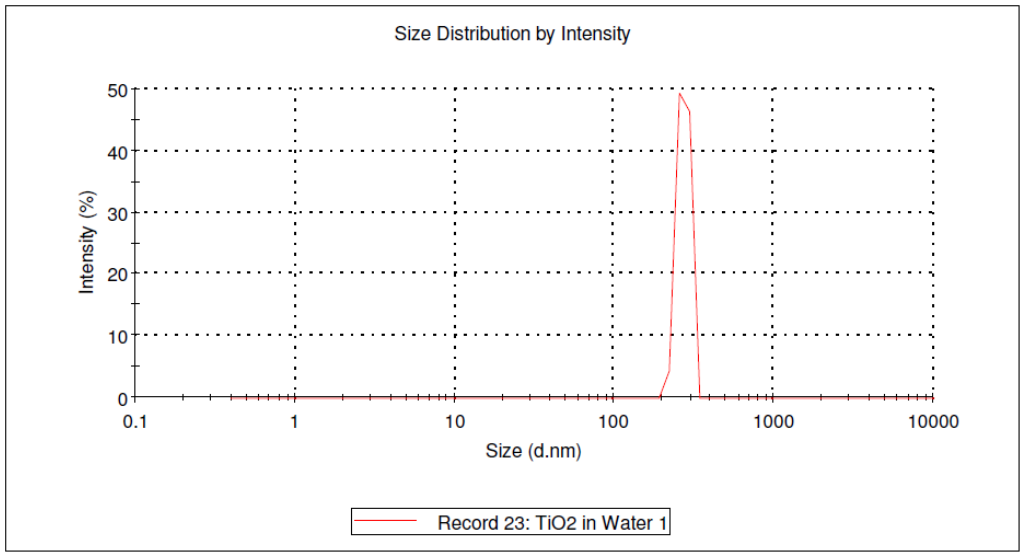
System

Temperature (°C):	25.0	**Duration Used (s):**	80
Count Rate (kcps):	86.6	**Measurement Position (mm):**	4.65
Cell Description:	Glass cuvette with round apert...	**Attenuator:**	9

Results

			Diam. (nm)	% Intensity	Width (nm)
Z-Average (d.nm):	1565	**Peak 1:**	272.1	100.0	22.63
PdI:	0.896	**Peak 2:**	0.000	0.0	0.000
Intercept:	1.25	**Peak 3:**	0.000	0.0	0.000

Result quality : POOR - see result quality report



Size Results Export Report

Sample Name TiO2 in Water, 3500rpm, 15min
Record Number: 23

Results Table

Size d.nm	Intensity %	Size d.nm	Intensity %
0.4000	0.0	68.06	0.0
0.4632	0.0	78.82	0.0
0.5365	0.0	91.28	0.0
0.6213	0.0	105.7	0.0
0.7195	0.0	122.4	0.0
0.8332	0.0	141.8	0.0
0.9649	0.0	164.2	0.0
1.117	0.0	190.1	0.0
1.294	0.0	220.2	4.4
1.499	0.0	255.0	49.3
1.736	0.0	295.3	46.3
2.010	0.0	342.0	0.0
2.328	0.0	396.1	0.0
2.696	0.0	458.7	0.0
3.122	0.0	531.2	0.0
3.615	0.0	615.1	0.0
4.187	0.0	712.4	0.0
4.849	0.0	825.0	0.0
5.615	0.0	955.4	0.0
6.503	0.0	1106	0.0
7.531	0.0	1281	0.0
8.721	0.0	1484	0.0
10.10	0.0	1718	0.0
11.70	0.0	1990	0.0
13.54	0.0	2305	0.0
15.69	0.0	2669	0.0
18.17	0.0	3091	0.0
21.04	0.0	3580	0.0
24.36	0.0	4145	0.0
28.21	0.0	4801	0.0
32.67	0.0	5560	0.0
37.84	0.0	6439	0.0
43.82	0.0	7456	0.0
50.75	0.0	8635	0.0
58.77	0.0	1.000e4	0.0

Right click the table to change the displayed parameters and/or change the number of columns. Use the Edit - Copy Table command to copy the data for pasting into a third party spreadsheet or graphing package.

Trend Table

Rec #	Z-Avg d.nm	Pk 1 Avg (I) d.nm	Pk 2 Avg (I) d.nm	Pk 3 Avg (I) d.nm	DCR kcps
23	1565	272.1	0.000	0.000	838.6

APPENDIX B

SIZE DATA REPORTS FOR CENTRIFUGATION EXPERIMENTS EVALUATING THE EFFECTS OF DISTANCE FROM THE AXIS OF ROTATION ON PARTICLE DIAMETER

Size Distribution Report by Intensity

Sample Details

Sample Name TiO2 in ethanol, 15min, 1000rpm, 30mL mark

SOP Name: TiO2 Size Ethanol Glass.sop

General Notes:

File Name:	Radius Comparison.dts	**Dispersant Name:**	Ethanol
Record Number:	1	**Dispersant RI:**	1.361
Material RI:	2.59	**Viscosity (cP):**	1.1170
Material Absorbtion:	0.10	**Measurement Date and Time:**	Wednesday, July 21, 2010 1..

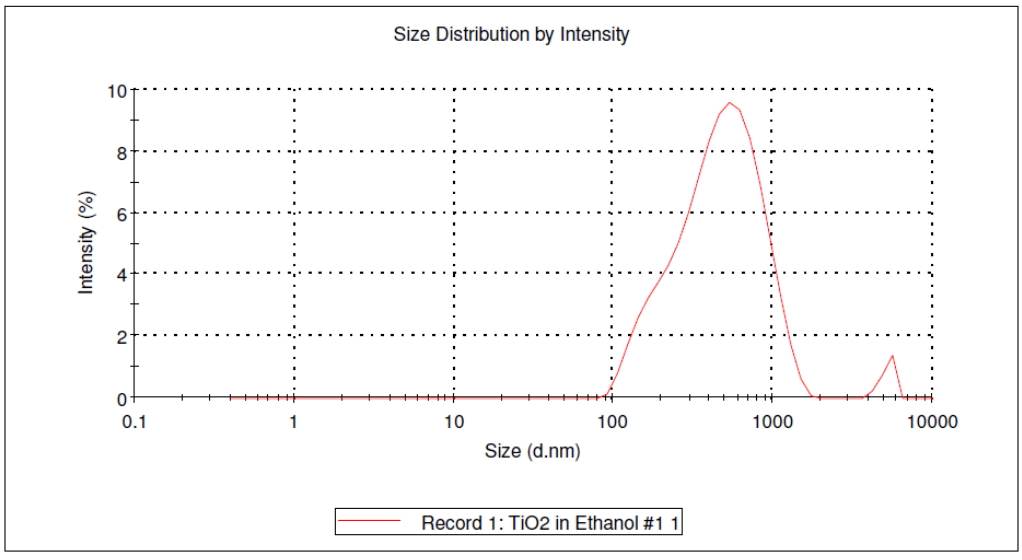
System

Temperature (°C):	20.0	**Duration Used (s):**	60
Count Rate (kcps):	326.2	**Measurement Position (mm):**	1.25
Cell Description:	Glass cuvette with round apert...	**Attenuator:**	4

Results

			Diam. (nm)	% Intensity	Width (nm)
Z-Average (d.nm):	411.8	**Peak 1:**	516.7	97.6	285.7
PdI:	0.342	**Peak 2:**	5176	2.4	488.0
Intercept:	0.921	**Peak 3:**	0.000	0.0	0.000

Result quality : Good



Size Results Export Report

Sample Name TiO2 in ethanol, 15min, 1000rpm, 30mL mark
Record Number: 1

Results Table

Size d.nm	Intensity %	Size d.nm	Intensity %
0.4000	0.0	68.06	0.0
0.4632	0.0	78.82	0.0
0.5365	0.0	91.28	0.1
0.6213	0.0	105.7	0.8
0.7195	0.0	122.4	1.7
0.8332	0.0	141.8	2.6
0.9649	0.0	164.2	3.2
1.117	0.0	190.1	3.8
1.294	0.0	220.2	4.3
1.499	0.0	255.0	5.0
1.736	0.0	295.3	6.0
2.010	0.0	342.0	7.2
2.328	0.0	396.1	8.3
2.696	0.0	458.7	9.2
3.122	0.0	531.2	9.6
3.615	0.0	615.1	9.3
4.187	0.0	712.4	8.4
4.849	0.0	825.0	7.0
5.615	0.0	955.4	5.2
6.503	0.0	1106	3.3
7.531	0.0	1281	1.7
8.721	0.0	1484	0.6
10.10	0.0	1718	0.1
11.70	0.0	1990	0.0
13.54	0.0	2305	0.0
15.69	0.0	2669	0.0
18.17	0.0	3091	0.0
21.04	0.0	3580	0.0
24.36	0.0	4145	0.2
28.21	0.0	4801	0.8
32.67	0.0	5560	1.4
37.84	0.0	6439	0.0
43.82	0.0	7456	0.0
50.75	0.0	8635	0.0
58.77	0.0	1.000e4	0.0

Right click the table to change the displayed parameters and/or change the number of columns. Use the Edit - Copy Table command to copy the data for pasting into a third party spreadsheet or graphing package.

Trend Table

Rec #	Z-Avg d.nm	Pk 1 Avg (I) d.nm	Pk 2 Avg (I) d.nm	Pk 3 Avg (I) d.nm	DCR kcps
1	411.8	516.7	5176	0.000	546877.4

Size Distribution Report by Intensity

Sample Details

Sample Name TiO2 in ethanol, 15min, 2000rpm, 30mL mark

SOP Name: TiO2 Size Ethanol Glass.sop

General Notes:

File Name:	Radius Comparison.dts	**Dispersant Name:**	Ethanol
Record Number:	3	**Dispersant RI:**	1.361
Material RI:	2.59	**Viscosity (cP):**	1.1170
Material Absorbtion:	0.10	**Measurement Date and Time:**	Wednesday, July 21, 2010 1...

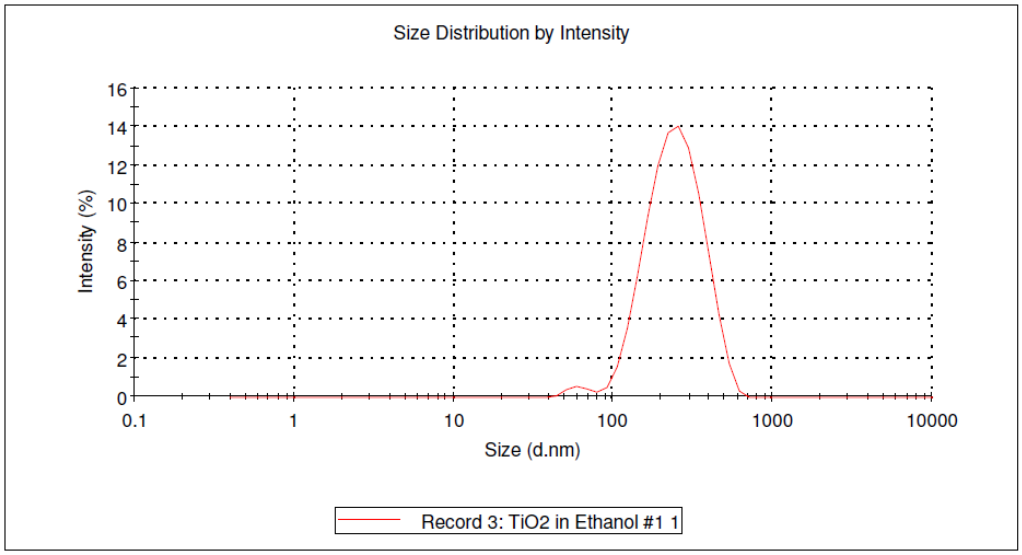
System

Temperature (°C):	20.0	**Duration Used (s):**	60
Count Rate (kcps):	339.4	**Measurement Position (mm):**	4.65
Cell Description:	Glass cuvette with round apert...	**Attenuator:**	6

Results

			Diam. (nm)	% Intensity	Width (nm)
Z-Average (d.nm):	217.4	**Peak 1:**	258.0	98.3	97.97
PdI:	0.145	**Peak 2:**	61.71	1.7	9.898
Intercept:	0.953	**Peak 3:**	0.000	0.0	0.000

Result quality : Good



Size Results Export Report

Sample Name TiO2 in ethanol, 15min, 2000rpm, 30mL mark
Record Number: 3

Results Table

Size d.nm	Intensity %	Size d.nm	Intensity %
0.4000	0.0	68.06	0.4
0.4632	0.0	78.82	0.3
0.5365	0.0	91.28	0.5
0.6213	0.0	105.7	1.6
0.7195	0.0	122.4	3.6
0.8332	0.0	141.8	6.3
0.9649	0.0	164.2	9.3
1.117	0.0	190.1	12.0
1.294	0.0	220.2	13.7
1.499	0.0	255.0	14.0
1.736	0.0	295.3	12.9
2.010	0.0	342.0	10.5
2.328	0.0	396.1	7.4
2.696	0.0	458.7	4.3
3.122	0.0	531.2	1.7
3.615	0.0	615.1	0.3
4.187	0.0	712.4	0.0
4.849	0.0	825.0	0.0
5.615	0.0	955.4	0.0
6.503	0.0	1106	0.0
7.531	0.0	1281	0.0
8.721	0.0	1484	0.0
10.10	0.0	1718	0.0
11.70	0.0	1990	0.0
13.54	0.0	2305	0.0
15.69	0.0	2669	0.0
18.17	0.0	3091	0.0
21.04	0.0	3580	0.0
24.36	0.0	4145	0.0
28.21	0.0	4801	0.0
32.67	0.0	5560	0.0
37.84	0.0	6439	0.0
43.82	0.1	7456	0.0
50.75	0.4	8635	0.0
58.77	0.6	1.000e4	0.0

Right click the table to change the displayed parameters and/or change the number of columns. Use the Edit - Copy Table command to copy the data for pasting into a third party spreadsheet or graphing package.

Trend Table

Rec #	Z-Avg d.nm	Pk 1 Avg (I) d.nm	Pk 2 Avg (I) d.nm	Pk 3 Avg (I) d.nm	DCR kcps
3	217.4	258.0	61.71	0.000	109430.9

Size Distribution Report by Intensity

Sample Details

Sample Name TiO2 in ethanol, 15min, 3000rpm, 30mL mark

SOP Name: TiO2 Size Ethanol Glass.sop

General Notes:

File Name:	Radius Comparison.dts	**Dispersant Name:**	Ethanol
Record Number:	5	**Dispersant RI:**	1.361
Material RI:	2.59	**Viscosity (cP):**	1.1170
Material Absorbtion:	0.10	**Measurement Date and Time:**	Wednesday, July 21, 2010 1..

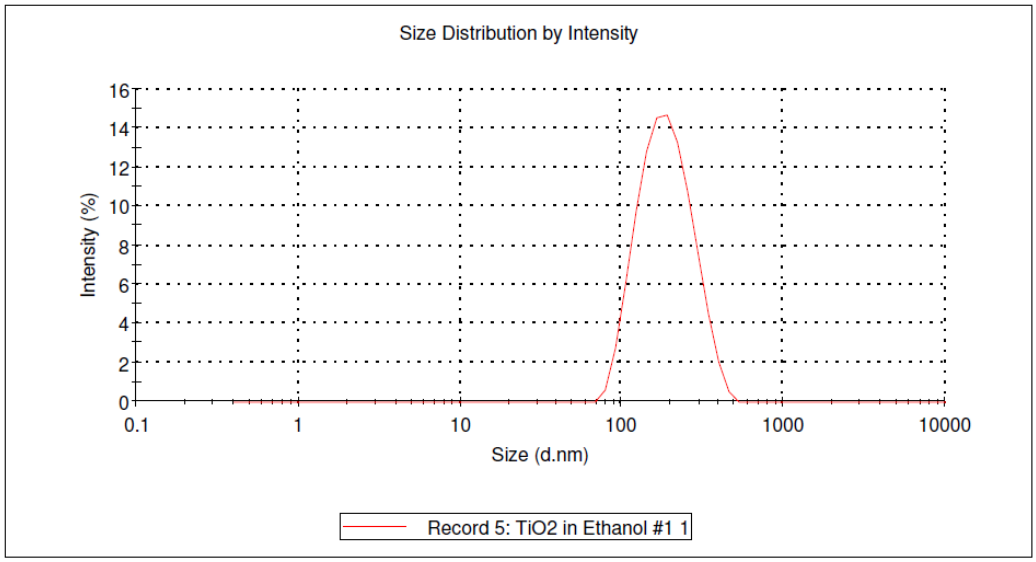
System

Temperature (°C):	20.0	**Duration Used (s):**	70
Count Rate (kcps):	226.5	**Measurement Position (mm):**	4.65
Cell Description:	Glass cuvette with round apert...	**Attenuator:**	6

Results

			Diam. (nm)	% Intensity	Width (nm)
Z-Average (d.nm):	173.1	**Peak 1:**	196.5	100.0	72.89
PdI:	0.120	**Peak 2:**	0.000	0.0	0.000
Intercept:	0.964	**Peak 3:**	0.000	0.0	0.000

Result quality : Good



Size Results Export Report

Sample Name TiO2 in ethanol, 15min, 3000rpm, 30mL mark
Record Number: 5

Results Table

Size d.nm	Intensity %	Size d.nm	Intensity %
0.4000	0.0	68.06	0.0
0.4632	0.0	78.82	0.6
0.5365	0.0	91.28	2.8
0.6213	0.0	105.7	6.1
0.7195	0.0	122.4	9.8
0.8332	0.0	141.8	12.8
0.9649	0.0	164.2	14.5
1.117	0.0	190.1	14.7
1.294	0.0	220.2	13.3
1.499	0.0	255.0	10.7
1.736	0.0	295.3	7.6
2.010	0.0	342.0	4.5
2.328	0.0	396.1	2.1
2.696	0.0	458.7	0.5
3.122	0.0	531.2	0.0
3.615	0.0	615.1	0.0
4.187	0.0	712.4	0.0
4.849	0.0	825.0	0.0
5.615	0.0	955.4	0.0
6.503	0.0	1106	0.0
7.531	0.0	1281	0.0
8.721	0.0	1484	0.0
10.10	0.0	1718	0.0
11.70	0.0	1990	0.0
13.54	0.0	2305	0.0
15.69	0.0	2669	0.0
18.17	0.0	3091	0.0
21.04	0.0	3580	0.0
24.36	0.0	4145	0.0
28.21	0.0	4801	0.0
32.67	0.0	5560	0.0
37.84	0.0	6439	0.0
43.82	0.0	7456	0.0
50.75	0.0	8635	0.0
58.77	0.0	1.000e4	0.0

Right click the table to change the displayed parameters and/or change the number of columns. Use the Edit - Copy Table command to copy the data for pasting into a third party spreadsheet or graphing package.

Trend Table

Rec #	Z-Avg d.nm	Pk 1 Avg (I) d.nm	Pk 2 Avg (I) d.nm	Pk 3 Avg (I) d.nm	DCR kcps
5	173.1	196.5	0.000	0.000	73012.0

Size Distribution Report by Intensity

Sample Details

Sample Name TiO2 in ethanol, 15min, 3500rpm, 30mL mark

SOP Name: TiO2 Size Ethanol Glass.sop

General Notes:

File Name:	Radius Comparison.dts	**Dispersant Name:**	Ethanol
Record Number:	7	**Dispersant RI:**	1.361
Material RI:	2.59	**Viscosity (cP):**	1.1170
Material Absorbtion:	0.10	**Measurement Date and Time:**	Wednesday, July 21, 2010 1..

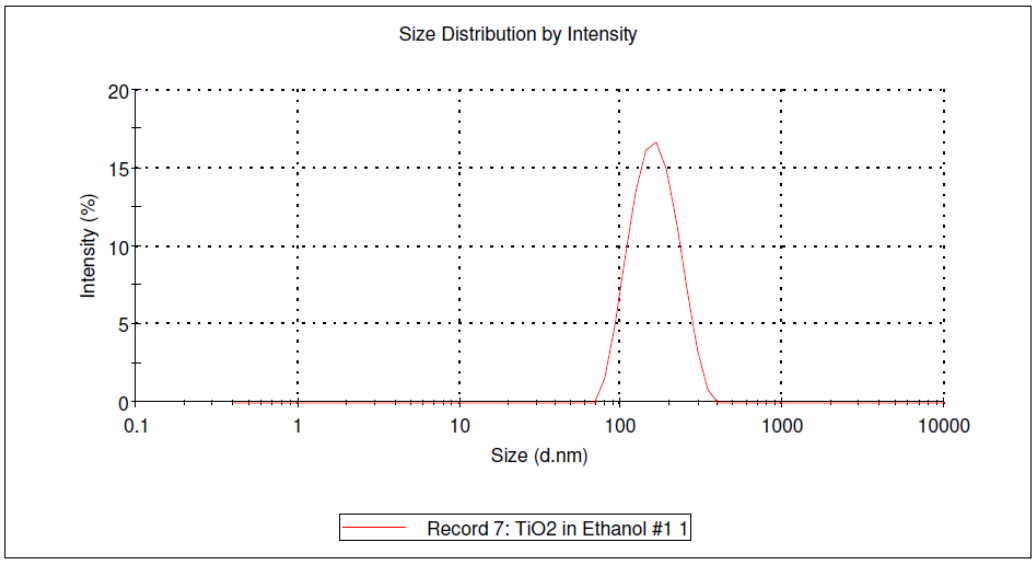
System

Temperature (°C):	20.0	**Duration Used (s):**	60
Count Rate (kcps):	339.7	**Measurement Position (mm):**	4.65
Cell Description:	Glass cuvette with round apert...	**Attenuator:**	7

Results

			Diam. (nm)	% Intensity	Width (nm)
Z-Average (d.nm):	151.6	**Peak 1:**	167.4	100.0	54.04
PdI:	0.083	**Peak 2:**	0.000	0.0	0.000
Intercept:	0.957	**Peak 3:**	0.000	0.0	0.000

Result quality : Good



Size Results Export Report

Sample Name TiO2 in ethanol, 15min, 3500rpm, 30mL mark
Record Number: 7

Results Table

Size d.nm	Intensity %	Size d.nm	Intensity %
0.4000	0.0	68.06	0.0
0.4632	0.0	78.82	1.6
0.5365	0.0	91.28	5.0
0.6213	0.0	105.7	9.3
0.7195	0.0	122.4	13.5
0.8332	0.0	141.8	16.1
0.9649	0.0	164.2	16.6
1.117	0.0	190.1	14.9
1.294	0.0	220.2	11.5
1.499	0.0	255.0	7.2
1.736	0.0	295.3	3.4
2.010	0.0	342.0	0.9
2.328	0.0	396.1	0.0
2.696	0.0	458.7	0.0
3.122	0.0	531.2	0.0
3.615	0.0	615.1	0.0
4.187	0.0	712.4	0.0
4.849	0.0	825.0	0.0
5.615	0.0	955.4	0.0
6.503	0.0	1106	0.0
7.531	0.0	1281	0.0
8.721	0.0	1484	0.0
10.10	0.0	1718	0.0
11.70	0.0	1990	0.0
13.54	0.0	2305	0.0
15.69	0.0	2669	0.0
18.17	0.0	3091	0.0
21.04	0.0	3580	0.0
24.36	0.0	4145	0.0
28.21	0.0	4801	0.0
32.67	0.0	5560	0.0
37.84	0.0	6439	0.0
43.82	0.0	7456	0.0
50.75	0.0	8635	0.0
58.77	0.0	1.000e4	0.0

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Trend Table

Rec #	Z-Avg d.nm	Pk 1 Avg (I) d.nm	Pk 2 Avg (I) d.nm	Pk 3 Avg (I) d.nm	DCR kcps
7	151.6	167.4	0.000	0.000	39349.0

Size Distribution Report by Intensity

Sample Details

Sample Name: TiO2 in ethanol, 15min, 1000rpm, 15mL mark

SOP Name: TiO2 Size Ethanol Glass.sop

General Notes:

File Name:	Radius Comparison.dts	**Dispersant Name:**	Ethanol
Record Number:	2	**Dispersant RI:**	1.361
Material RI:	2.59	**Viscosity (cP):**	1.1170
Material Absorbtion:	0.10	**Measurement Date and Time:**	Wednesday, July 21, 2010 1..

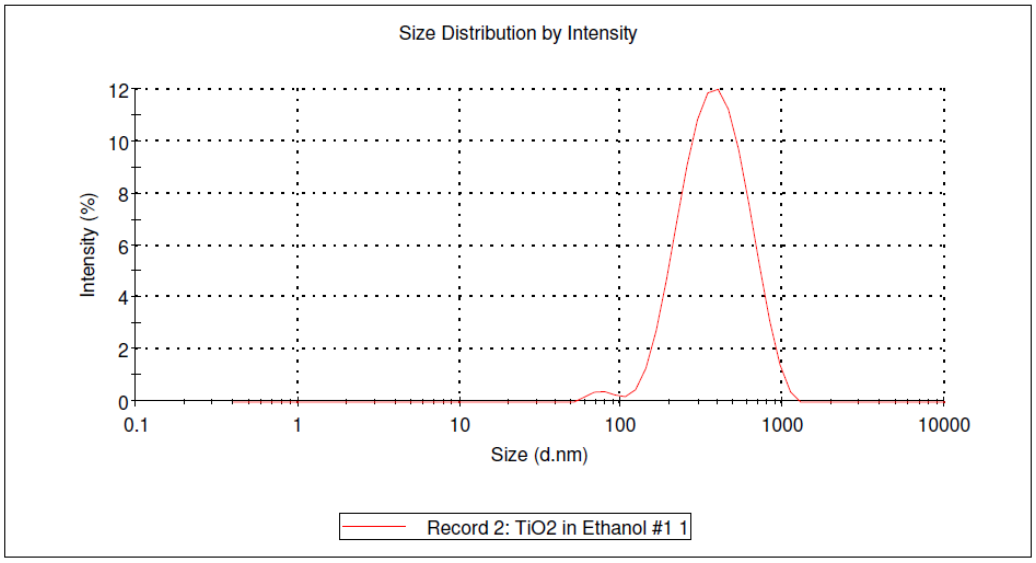
System

Temperature (°C):	20.0	**Duration Used (s):**	70
Count Rate (kcps):	236.6	**Measurement Position (mm):**	4.65
Cell Description:	Glass cuvette with round apert...	**Attenuator:**	7

Results

			Diam. (nm)	% Intensity	Width (nm)
Z-Average (d.nm):	329.5	**Peak 1:**	410.3	98.5	183.9
PdI:	0.183	**Peak 2:**	79.66	1.5	14.67
Intercept:	0.918	**Peak 3:**	0.000	0.0	0.000

Result quality : Good



Size Results Export Report

Sample Name TiO2 in ethanol, 15min, 1000rpm, 15mL mark
Record Number: 2

Results Table

Size d.nm	Intensity %	Size d.nm	Intensity %
0.4000	0.0	68.06	0.4
0.4632	0.0	78.82	0.4
0.5365	0.0	91.28	0.3
0.6213	0.0	105.7	0.2
0.7195	0.0	122.4	0.5
0.8332	0.0	141.8	1.3
0.9649	0.0	164.2	2.7
1.117	0.0	190.1	4.7
1.294	0.0	220.2	6.9
1.499	0.0	255.0	9.1
1.736	0.0	295.3	10.8
2.010	0.0	342.0	11.8
2.328	0.0	396.1	12.0
2.696	0.0	458.7	11.2
3.122	0.0	531.2	9.7
3.615	0.0	615.1	7.6
4.187	0.0	712.4	5.3
4.849	0.0	825.0	3.1
5.615	0.0	955.4	1.4
6.503	0.0	1106	0.4
7.531	0.0	1281	0.0
8.721	0.0	1484	0.0
10.10	0.0	1718	0.0
11.70	0.0	1990	0.0
13.54	0.0	2305	0.0
15.69	0.0	2669	0.0
18.17	0.0	3091	0.0
21.04	0.0	3580	0.0
24.36	0.0	4145	0.0
28.21	0.0	4801	0.0
32.67	0.0	5560	0.0
37.84	0.0	6439	0.0
43.82	0.0	7456	0.0
50.75	0.0	8635	0.0
58.77	0.2	1.000e4	0.0

Right click the table to change the displayed parameters and/or change the number of columns. Use the Edit - Copy Table command to copy the data for pasting into a third party spreadsheet or graphing package.

Trend Table

Rec #	Z-Avg d.nm	Pk 1 Avg (I) d.nm	Pk 2 Avg (I) d.nm	Pk 3 Avg (I) d.nm	DCR kcps
2	329.5	410.3	79.66	0.000	27412.5

Size Distribution Report by Intensity

Sample Details

Sample Name TiO2 in ethanol, 15min, 2000rpm, 15mL mark

SOP Name: TiO2 Size Ethanol Glass.sop

General Notes:

File Name:	Radius Comparison.dts	**Dispersant Name:**	Ethanol
Record Number:	4	**Dispersant RI:**	1.361
Material RI:	2.59	**Viscosity (cP):**	1.1170
Material Absorbtion:	0.10	**Measurement Date and Time:**	Wednesday, July 21, 2010 1..

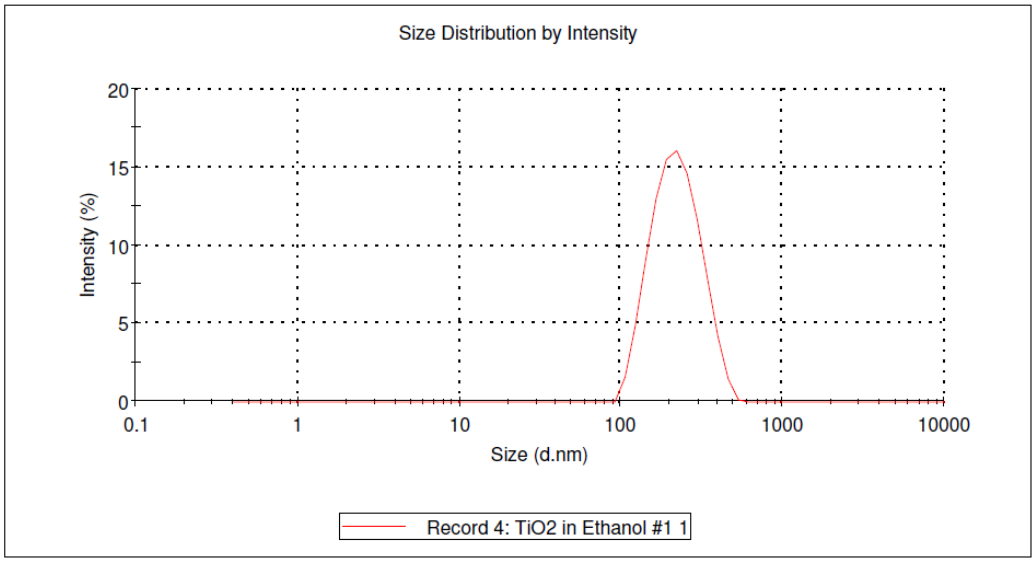
System

Temperature (°C):	20.0	**Duration Used (s):**	60
Count Rate (kcps):	337.7	**Measurement Position (mm):**	4.65
Cell Description:	Glass cuvette with round apert...	**Attenuator:**	6

Results

			Diam. (nm)	% Intensity	Width (nm)
Z-Average (d.nm):	209.9	**Peak 1:**	229.2	100.0	77.36
PdI:	0.152	**Peak 2:**	0.000	0.0	0.000
Intercept:	0.950	**Peak 3:**	0.000	0.0	0.000

Result quality : Good



Size Results Export Report

Sample Name TiO2 in ethanol, 15min, 2000rpm, 15mL mark
Record Number: 4

Results Table

Size d.nm	Intensity %	Size d.nm	Intensity %
0.4000	0.0	68.06	0.0
0.4632	0.0	78.82	0.0
0.5365	0.0	91.28	0.0
0.6213	0.0	105.7	1.6
0.7195	0.0	122.4	4.9
0.8332	0.0	141.8	9.1
0.9649	0.0	164.2	13.0
1.117	0.0	190.1	15.5
1.294	0.0	220.2	16.0
1.499	0.0	255.0	14.6
1.736	0.0	295.3	11.6
2.010	0.0	342.0	7.8
2.328	0.0	396.1	4.2
2.696	0.0	458.7	1.5
3.122	0.0	531.2	0.2
3.615	0.0	615.1	0.0
4.187	0.0	712.4	0.0
4.849	0.0	825.0	0.0
5.615	0.0	955.4	0.0
6.503	0.0	1106	0.0
7.531	0.0	1281	0.0
8.721	0.0	1484	0.0
10.10	0.0	1718	0.0
11.70	0.0	1990	0.0
13.54	0.0	2305	0.0
15.69	0.0	2669	0.0
18.17	0.0	3091	0.0
21.04	0.0	3580	0.0
24.36	0.0	4145	0.0
28.21	0.0	4801	0.0
32.67	0.0	5560	0.0
37.84	0.0	6439	0.0
43.82	0.0	7456	0.0
50.75	0.0	8635	0.0
58.77	0.0	1.000e4	0.0

Right click the table to change the displayed parameters and/or change the number of columns. Use the Edit - Copy Table command to copy the data for pasting into a third party spreadsheet or graphing package.

Trend Table

Rec #	Z-Avg d.nm	Pk 1 Avg (I) d.nm	Pk 2 Avg (I) d.nm	Pk 3 Avg (I) d.nm	DCR kcps
4	209.9	229.2	0.000	0.000	108881.0

Size Distribution Report by Intensity

Sample Details

Sample Name TiO2 in ethanol, 15min, 3000rpm, 15mL mark

SOP Name: TiO2 Size Ethanol Glass.sop

General Notes:

File Name:	Radius Comparison.dts	**Dispersant Name:**	Ethanol
Record Number:	6	**Dispersant RI:**	1.361
Material RI:	2.59	**Viscosity (cP):**	1.1170
Material Absorbtion:	0.10	**Measurement Date and Time:**	Wednesday, July 21, 2010 1..

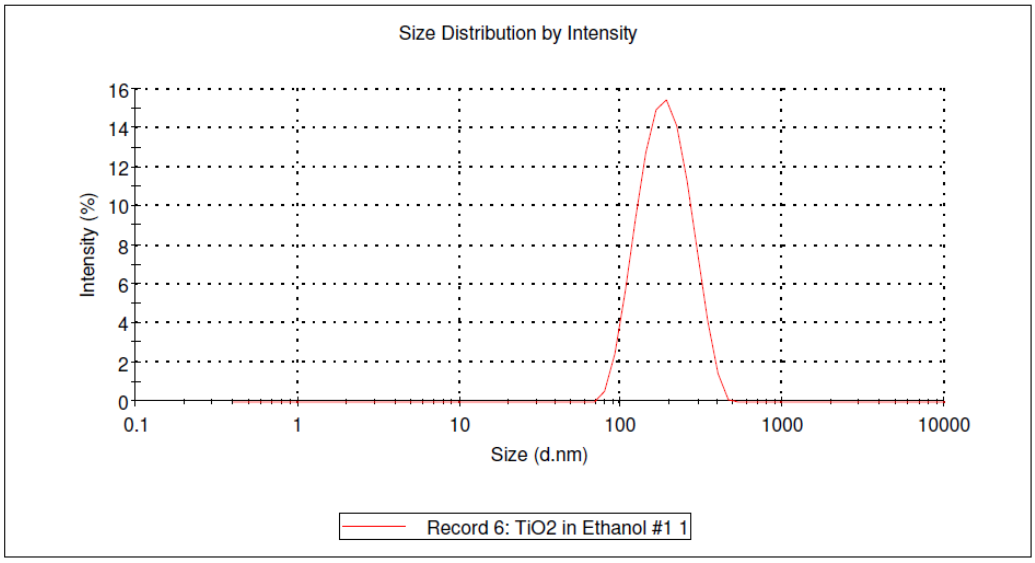
System

Temperature (°C):	20.0	**Duration Used (s):**	70
Count Rate (kcps):	235.0	**Measurement Position (mm):**	4.65
Cell Description:	Glass cuvette with round apert...	**Attenuator:**	6

Results

			Diam. (nm)	% Intensity	Width (nm)
Z-Average (d.nm):	171.9	**Peak 1:**	195.3	100.0	68.18
PdI:	0.113	**Peak 2:**	0.000	0.0	0.000
Intercept:	0.966	**Peak 3:**	0.000	0.0	0.000

Result quality : Good



Size Results Export Report

Sample Name TiO2 in ethanol, 15min, 3000rpm, 15mL mark
Record Number: 6

Results Table

Size d.nm	Intensity %	Size d.nm	Intensity %
0.4000	0.0	68.06	0.0
0.4632	0.0	78.82	0.6
0.5365	0.0	91.28	2.5
0.6213	0.0	105.7	5.6
0.7195	0.0	122.4	9.4
0.8332	0.0	141.8	12.7
0.9649	0.0	164.2	14.9
1.117	0.0	190.1	15.4
1.294	0.0	220.2	14.1
1.499	0.0	255.0	11.3
1.736	0.0	295.3	7.7
2.010	0.0	342.0	4.2
2.328	0.0	396.1	1.5
2.696	0.0	458.7	0.1
3.122	0.0	531.2	0.0
3.615	0.0	615.1	0.0
4.187	0.0	712.4	0.0
4.849	0.0	825.0	0.0
5.615	0.0	955.4	0.0
6.503	0.0	1106	0.0
7.531	0.0	1281	0.0
8.721	0.0	1484	0.0
10.10	0.0	1718	0.0
11.70	0.0	1990	0.0
13.54	0.0	2305	0.0
15.69	0.0	2669	0.0
18.17	0.0	3091	0.0
21.04	0.0	3580	0.0
24.36	0.0	4145	0.0
28.21	0.0	4801	0.0
32.67	0.0	5560	0.0
37.84	0.0	6439	0.0
43.82	0.0	7456	0.0
50.75	0.0	8635	0.0
58.77	0.0	1.000e4	0.0

Right click the table to change the displayed parameters and/or change the number of columns. Use the Edit - Copy Table command to copy the data for pasting into a third party spreadsheet or graphing package.

Trend Table

Rec #	Z-Avg d.nm	Pk 1 Avg (I) d.nm	Pk 2 Avg (I) d.nm	Pk 3 Avg (I) d.nm	DCR kcps
6	171.9	195.3	0.000	0.000	75769.9

Size Distribution Report by Intensity

Sample Details

Sample Name TiO2 in ethanol, 15min, 3500rpm, 15mL mark

SOP Name: TiO2 Size Ethanol Glass.sop

General Notes:

File Name:	Radius Comparison.dts	**Dispersant Name:**	Ethanol
Record Number:	8	**Dispersant RI:**	1.361
Material RI:	2.59	**Viscosity (cP):**	1.1170
Material Absorbtion:	0.10	**Measurement Date and Time:**	Wednesday, July 21, 2010 1..

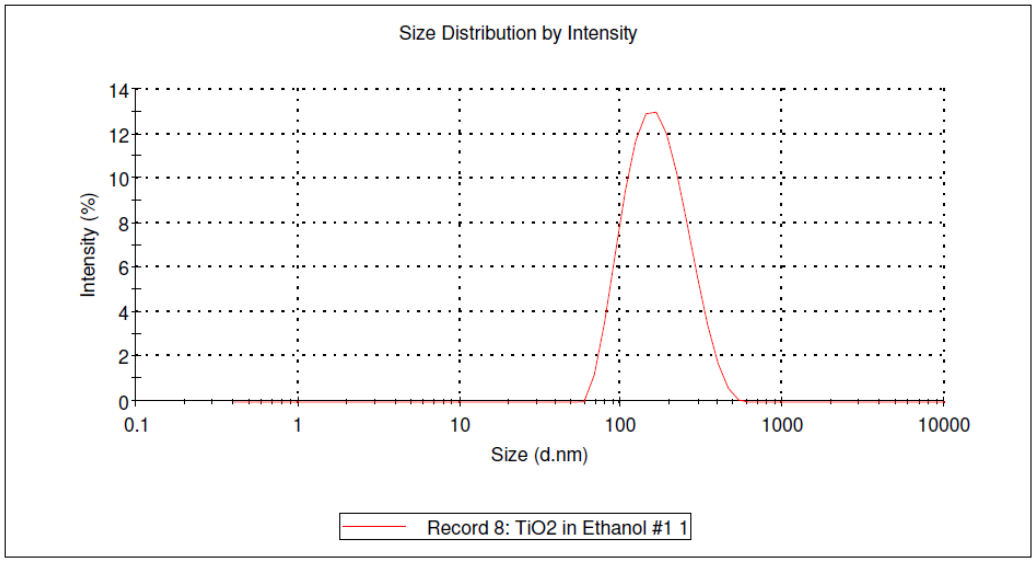
System

Temperature (°C):	20.0	**Duration Used (s):**	60
Count Rate (kcps):	362.3	**Measurement Position (mm):**	4.65
Cell Description:	Glass cuvette with round apert...	**Attenuator:**	7

Results

			Diam. (nm)	% Intensity	Width (nm)
Z-Average (d.nm):	151.2	**Peak 1:**	177.5	100.0	76.40
PdI:	0.146	**Peak 2:**	0.000	0.0	0.000
Intercept:	0.951	**Peak 3:**	0.000	0.0	0.000

Result quality : Good



Size Results Export Report

Sample Name TiO2 in ethanol, 15min, 3500rpm, 15mL mark
Record Number: 8

Results Table

Size d.nm	Intensity %	Size d.nm	Intensity %
0.4000	0.0	68.06	1.2
0.4632	0.0	78.82	3.6
0.5365	0.0	91.28	6.6
0.6213	0.0	105.7	9.4
0.7195	0.0	122.4	11.7
0.8332	0.0	141.8	12.9
0.9649	0.0	164.2	13.0
1.117	0.0	190.1	12.0
1.294	0.0	220.2	10.2
1.499	0.0	255.0	8.0
1.736	0.0	295.3	5.6
2.010	0.0	342.0	3.4
2.328	0.0	396.1	1.7
2.696	0.0	458.7	0.6
3.122	0.0	531.2	0.1
3.615	0.0	615.1	0.0
4.187	0.0	712.4	0.0
4.849	0.0	825.0	0.0
5.615	0.0	955.4	0.0
6.503	0.0	1106	0.0
7.531	0.0	1281	0.0
8.721	0.0	1484	0.0
10.10	0.0	1718	0.0
11.70	0.0	1990	0.0
13.54	0.0	2305	0.0
15.69	0.0	2669	0.0
18.17	0.0	3091	0.0
21.04	0.0	3580	0.0
24.36	0.0	4145	0.0
28.21	0.0	4801	0.0
32.67	0.0	5560	0.0
37.84	0.0	6439	0.0
43.82	0.0	7456	0.0
50.75	0.0	8635	0.0
58.77	0.0	1.000e4	0.0

Right click the table to change the displayed parameters and/or change the number of columns. Use the Edit - Copy Table command to copy the data for pasting into a third party spreadsheet or graphing package.

Trend Table

Rec #	Z-Avg d.nm	Pk 1 Avg (I) d.nm	Pk 2 Avg (I) d.nm	Pk 3 Avg (I) d.nm	DCR kcps
8	151.2	177.5	0.000	0.000	41975.7

APPENDIX C

SIZE DATA REPORTS FOR CENTRIFUGATION EXPERIMENTS EVALUATING THE EFFECTS OF ANGULAR VELOCITY ON PARTICLE DIAMETER

Size Distribution Report by Intensity

Sample Details

Sample Name: #2--1500RPM@15min D Glass 1
SOP Name: mansettings.dat
General Notes:

File Name: Radius Comparison.dts
Record Number: 69
Material RI: 2.59
Material Absorbtion: 0.10
Dispersant Name: Ethanol
Dispersant RI: 1.361
Viscosity (cP): 1.1170
Measurement Date and Time: Wednesday, August 04, 201..

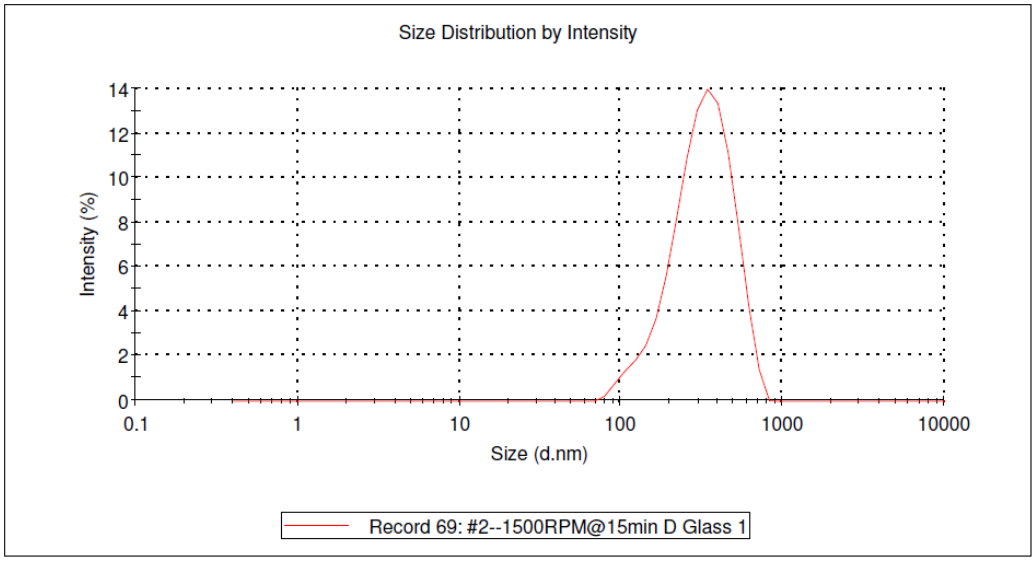
System

Temperature (°C): 19.9
Count Rate (kcps): 200.3
Cell Description: Glass cuvette with round apert...
Duration Used (s): 70
Measurement Position (mm): 4.65
Attenuator: 6

Results

			Diam. (nm)	% Intensity	Width (nm)
Z-Average (d.nm):	319.3	Peak 1:	338.4	100.0	134.0
PdI:	0.286	Peak 2:	0.000	0.0	0.000
Intercept:	0.942	Peak 3:	0.000	0.0	0.000

Result quality : Good



Size Results Export Report

Sample Name: #2--1500RPM@15min D Glass 1
Record Number: 69

Results Table

Size d.nm	Intensity %	Size d.nm	Intensity %
0.4000	0.0	68.06	0.0
0.4632	0.0	78.82	0.2
0.5365	0.0	91.28	0.8
0.6213	0.0	105.7	1.4
0.7195	0.0	122.4	1.8
0.8332	0.0	141.8	2.5
0.9649	0.0	164.2	3.7
1.117	0.0	190.1	5.6
1.294	0.0	220.2	8.2
1.499	0.0	255.0	10.9
1.736	0.0	295.3	13.0
2.010	0.0	342.0	14.0
2.328	0.0	396.1	13.3
2.696	0.0	458.7	11.1
3.122	0.0	531.2	7.8
3.615	0.0	615.1	4.3
4.187	0.0	712.4	1.4
4.849	0.0	825.0	0.0
5.615	0.0	955.4	0.0
6.503	0.0	1106	0.0
7.531	0.0	1281	0.0
8.721	0.0	1484	0.0
10.10	0.0	1718	0.0
11.70	0.0	1990	0.0
13.54	0.0	2305	0.0
15.69	0.0	2669	0.0
18.17	0.0	3091	0.0
21.04	0.0	3580	0.0
24.36	0.0	4145	0.0
28.21	0.0	4801	0.0
32.67	0.0	5560	0.0
37.84	0.0	6439	0.0
43.82	0.0	7456	0.0
50.75	0.0	8635	0.0
58.77	0.0	1.000e4	0.0

Right click the table to change the displayed parameters and/or change the number of columns. Use the Edit - Copy Table command to copy the data for pasting into a third party spreadsheet or graphing package.

Trend Table

Rec #	Z-Avg d.nm	Pk 1 Avg (I) d.nm	Pk 2 Avg (I) d.nm	Pk 3 Avg (I) d.nm	DCR kcps
69	319.3	338.4	0.000	0.000	64581.7

Size Distribution Report by Intensity

Sample Details

Sample Name: #4--2000@15min D Glass 1

SOP Name: mansettings.dat

General Notes:

File Name:	Radius Comparison.dts	**Dispersant Name:**	Ethanol
Record Number:	71	**Dispersant RI:**	1.361
Material RI:	2.59	**Viscosity (cP):**	1.1170
Material Absorbtion:	0.10	**Measurement Date and Time:**	Wednesday, August 04, 201..

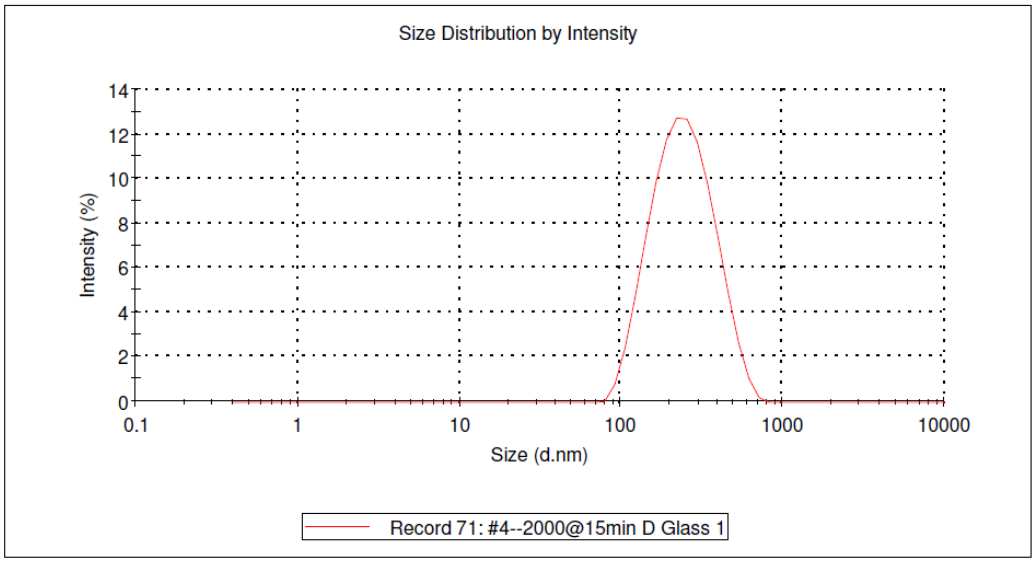
System

Temperature (°C):	20.0	**Duration Used (s):**	70
Count Rate (kcps):	231.8	**Measurement Position (mm):**	4.65
Cell Description:	Glass cuvette with round apert...	**Attenuator:**	6

Results

			Diam. (nm)	% Intensity	Width (nm)
Z-Average (d.nm):	220.0	**Peak 1:**	260.0	100.0	110.9
PdI:	0.145	**Peak 2:**	0.000	0.0	0.000
Intercept:	0.953	**Peak 3:**	0.000	0.0	0.000

Result quality : Good



Size Results Export Report

Sample Name: #4--2000@15min D Glass 1
Record Number: 71

Results Table

Size d.nm	Intensity %	Size d.nm	Intensity %
0.4000	0.0	68.06	0.0
0.4632	0.0	78.82	0.0
0.5365	0.0	91.28	0.8
0.6213	0.0	105.7	2.5
0.7195	0.0	122.4	4.8
0.8332	0.0	141.8	7.4
0.9649	0.0	164.2	9.8
1.117	0.0	190.1	11.7
1.294	0.0	220.2	12.7
1.499	0.0	255.0	12.7
1.736	0.0	295.3	11.6
2.010	0.0	342.0	9.7
2.328	0.0	396.1	7.4
2.696	0.0	458.7	4.9
3.122	0.0	531.2	2.7
3.615	0.0	615.1	1.1
4.187	0.0	712.4	0.2
4.849	0.0	825.0	0.0
5.615	0.0	955.4	0.0
6.503	0.0	1106	0.0
7.531	0.0	1281	0.0
8.721	0.0	1484	0.0
10.10	0.0	1718	0.0
11.70	0.0	1990	0.0
13.54	0.0	2305	0.0
15.69	0.0	2669	0.0
18.17	0.0	3091	0.0
21.04	0.0	3580	0.0
24.36	0.0	4145	0.0
28.21	0.0	4801	0.0
32.67	0.0	5560	0.0
37.84	0.0	6439	0.0
43.82	0.0	7456	0.0
50.75	0.0	8635	0.0
58.77	0.0	1.000e4	0.0

Right click the table to change the displayed parameters and/or change the number of columns. Use the Edit - Copy Table command to copy the data for pasting into a third party spreadsheet or graphing package.

Trend Table

Rec #	Z-Avg d.nm	Pk 1 Avg (I) d.nm	Pk 2 Avg (I) d.nm	Pk 3 Avg (I) d.nm	DCR kcps
71	220.0	260.0	0.000	0.000	74737.5

Size Distribution Report by Intensity

Sample Details

Sample Name: #6--2500@15min D Glass 1
SOP Name: mansettings.dat
General Notes:

File Name:	Radius Comparison.dts	**Dispersant Name:**	Ethanol
Record Number:	72	**Dispersant RI:**	1.361
Material RI:	2.59	**Viscosity (cP):**	1.1170
Material Absorbtion:	0.10	**Measurement Date and Time:**	Wednesday, August 04, 201...

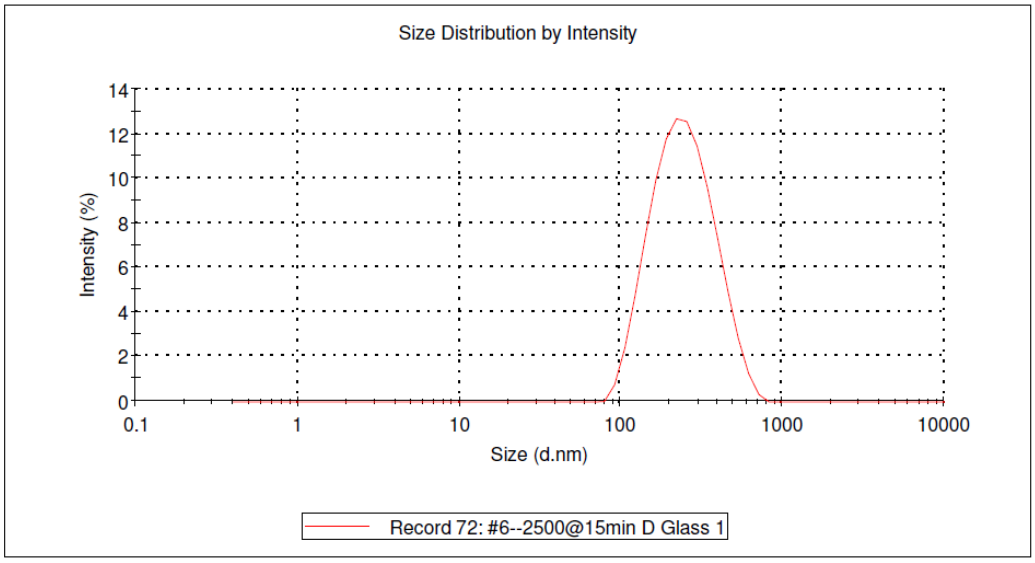
System

Temperature (°C):	20.0	**Duration Used (s):**	60
Count Rate (kcps):	366.6	**Measurement Position (mm):**	4.65
Cell Description:	Glass cuvette with round apert...	**Attenuator:**	6

Results

			Diam. (nm)	% Intensity	Width (nm)
Z-Average (d.nm):	218.4	**Peak 1:**	261.5	100.0	113.6
PdI:	0.191	**Peak 2:**	0.000	0.0	0.000
Intercept:	0.946	**Peak 3:**	0.000	0.0	0.000

Result quality : Good



Size Results Export Report

Sample Name: #6--2500@15min D Glass 1
Record Number: 72

Results Table

Size d.nm	Intensity %	Size d.nm	Intensity %
0.4000	0.0	68.06	0.0
0.4632	0.0	78.82	0.0
0.5365	0.0	91.28	0.8
0.6213	0.0	105.7	2.4
0.7195	0.0	122.4	4.8
0.8332	0.0	141.8	7.5
0.9649	0.0	164.2	9.9
1.117	0.0	190.1	11.7
1.294	0.0	220.2	12.7
1.499	0.0	255.0	12.5
1.736	0.0	295.3	11.4
2.010	0.0	342.0	9.6
2.328	0.0	396.1	7.3
2.696	0.0	458.7	4.9
3.122	0.0	531.2	2.8
3.615	0.0	615.1	1.3
4.187	0.0	712.4	0.3
4.849	0.0	825.0	0.0
5.615	0.0	955.4	0.0
6.503	0.0	1106	0.0
7.531	0.0	1281	0.0
8.721	0.0	1484	0.0
10.10	0.0	1718	0.0
11.70	0.0	1990	0.0
13.54	0.0	2305	0.0
15.69	0.0	2669	0.0
18.17	0.0	3091	0.0
21.04	0.0	3580	0.0
24.36	0.0	4145	0.0
28.21	0.0	4801	0.0
32.67	0.0	5560	0.0
37.84	0.0	6439	0.0
43.82	0.0	7456	0.0
50.75	0.0	8635	0.0
58.77	0.0	1.000e4	0.0

Right click the table to change the displayed parameters and/or change the number of columns. Use the Edit - Copy Table command to copy the data for pasting into a third party spreadsheet or graphing package.

Trend Table

Rec #	Z-Avg d.nm	Pk 1 Avg (I) d.nm	Pk 2 Avg (I) d.nm	Pk 3 Avg (I) d.nm	DCR kcps
72	218.4	261.5	0.000	0.000	118177.

Size Distribution Report by Intensity

Sample Details

Sample Name: #8--3000@15min D Glass 1
SOP Name: mansettings.dat
General Notes:

File Name:	Radius Comparison.dts	**Dispersant Name:**	Ethanol
Record Number:	75	**Dispersant RI:**	1.361
Material RI:	2.59	**Viscosity (cP):**	1.1170
Material Absorbtion:	0.10	**Measurement Date and Time:**	Wednesday, August 04, 201..

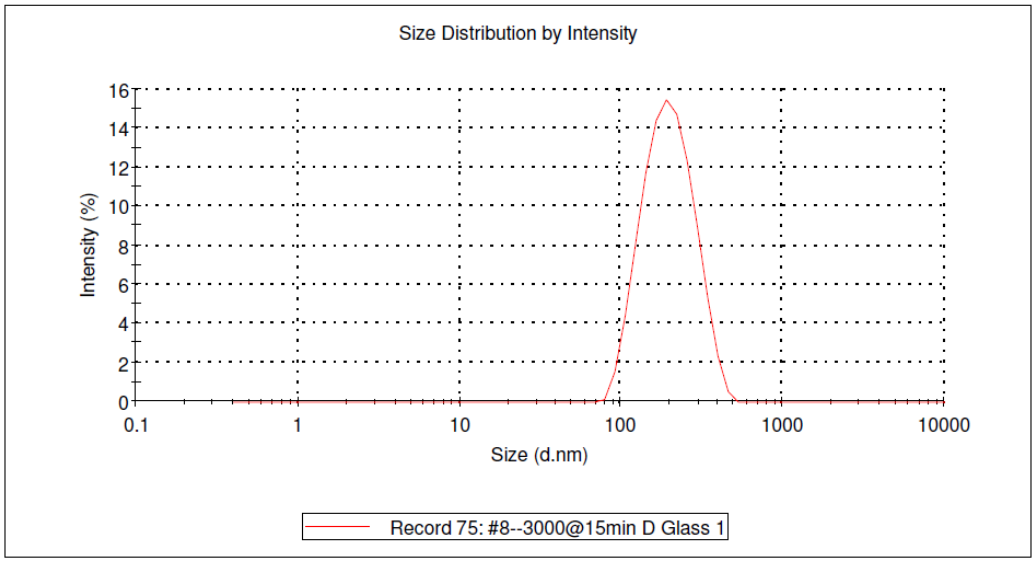
System

Temperature (°C):	20.0	**Duration Used (s):**	70
Count Rate (kcps):	219.7	**Measurement Position (mm):**	4.65
Cell Description:	Glass cuvette with round apert...	**Attenuator:**	5

Results

			Diam. (nm)	% Intensity	Width (nm)
Z-Average (d.nm):	178.2	**Peak 1:**	206.1	100.0	72.38
PdI:	0.131	**Peak 2:**	0.000	0.0	0.000
Intercept:	0.961	**Peak 3:**	0.000	0.0	0.000

Result quality : Good



Size Results Export Report

Sample Name: #8--3000@15min D Glass 1
Record Number: 75

Results Table

Size d.nm	Intensity %	Size d.nm	Intensity %
0.4000	0.0	68.06	0.0
0.4632	0.0	78.82	0.1
0.5365	0.0	91.28	1.6
0.6213	0.0	105.7	4.4
0.7195	0.0	122.4	8.1
0.8332	0.0	141.8	11.7
0.9649	0.0	164.2	14.4
1.117	0.0	190.1	15.4
1.294	0.0	220.2	14.7
1.499	0.0	255.0	12.3
1.736	0.0	295.3	9.0
2.010	0.0	342.0	5.4
2.328	0.0	396.1	2.4
2.696	0.0	458.7	0.5
3.122	0.0	531.2	0.0
3.615	0.0	615.1	0.0
4.187	0.0	712.4	0.0
4.849	0.0	825.0	0.0
5.615	0.0	955.4	0.0
6.503	0.0	1106	0.0
7.531	0.0	1281	0.0
8.721	0.0	1484	0.0
10.10	0.0	1718	0.0
11.70	0.0	1990	0.0
13.54	0.0	2305	0.0
15.69	0.0	2669	0.0
18.17	0.0	3091	0.0
21.04	0.0	3580	0.0
24.36	0.0	4145	0.0
28.21	0.0	4801	0.0
32.67	0.0	5560	0.0
37.84	0.0	6439	0.0
43.82	0.0	7456	0.0
50.75	0.0	8635	0.0
58.77	0.0	1.000e4	0.0

Right click the table to change the displayed parameters and/or change the number of columns. Use the Edit - Copy Table command to copy the data for pasting into a third party spreadsheet or graphing package.

Trend Table

Rec #	Z-Avg d.nm	Pk 1 Avg (I) d.nm	Pk 2 Avg (I) d.nm	Pk 3 Avg (I) d.nm	DCR kcps
75	178.2	206.1	0.000	0.000	190900.5

Size Distribution Report by Intensity

Sample Details

Sample Name: #10--3500@15min D Glass 1

SOP Name: mansettings.dat

General Notes:

File Name:	Radius Comparison.dts	**Dispersant Name:**	Ethanol
Record Number:	77	**Dispersant RI:**	1.361
Material RI:	2.59	**Viscosity (cP):**	1.1170
Material Absorbtion:	0.10	**Measurement Date and Time:**	Wednesday, August 04, 201..

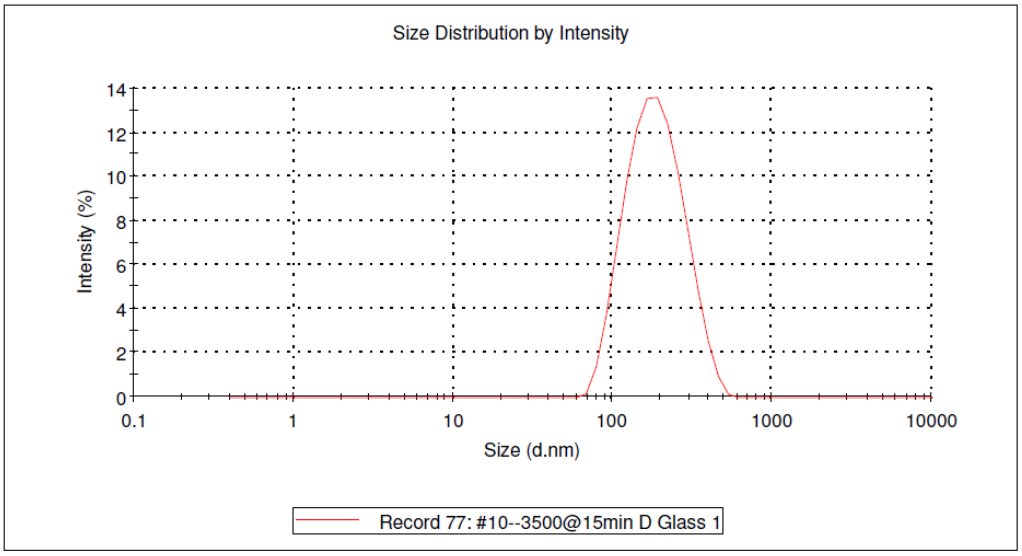
System

Temperature (°C):	20.0	**Duration Used (s):**	60
Count Rate (kcps):	405.5	**Measurement Position (mm):**	4.65
Cell Description:	Glass cuvette with round apert...	**Attenuator:**	6

Results

			Diam. (nm)	% Intensity	Width (nm)
Z-Average (d.nm):	172.1	**Peak 1:**	197.1	100.0	79.32
PdI:	0.153	**Peak 2:**	0.000	0.0	0.000
Intercept:	0.953	**Peak 3:**	0.000	0.0	0.000

Result quality : Good



Size Results Export Report

Sample Name: #10--3500@15min D Glass 1
Record Number: 77

Results Table

Size d.nm	Intensity %	Size d.nm	Intensity %
0.4000	0.0	68.06	0.2
0.4632	0.0	78.82	1.4
0.5365	0.0	91.28	3.8
0.6213	0.0	105.7	6.8
0.7195	0.0	122.4	9.8
0.8332	0.0	141.8	12.2
0.9649	0.0	164.2	13.5
1.117	0.0	190.1	13.6
1.294	0.0	220.2	12.4
1.499	0.0	255.0	10.3
1.736	0.0	295.3	7.6
2.010	0.0	342.0	4.9
2.328	0.0	396.1	2.6
2.696	0.0	458.7	1.0
3.122	0.0	531.2	0.2
3.615	0.0	615.1	0.0
4.187	0.0	712.4	0.0
4.849	0.0	825.0	0.0
5.615	0.0	955.4	0.0
6.503	0.0	1106	0.0
7.531	0.0	1281	0.0
8.721	0.0	1484	0.0
10.10	0.0	1718	0.0
11.70	0.0	1990	0.0
13.54	0.0	2305	0.0
15.69	0.0	2669	0.0
18.17	0.0	3091	0.0
21.04	0.0	3580	0.0
24.36	0.0	4145	0.0
28.21	0.0	4801	0.0
32.67	0.0	5560	0.0
37.84	0.0	6439	0.0
43.82	0.0	7456	0.0
50.75	0.0	8635	0.0
58.77	0.0	1.000e4	0.0

Right click the table to change the displayed parameters and/or change the number of columns. Use the Edit - Copy Table command to copy the data for pasting into a third party spreadsheet or graphing package.

Trend Table

Rec #	Z-Avg d.nm	Pk 1 Avg (I) d.nm	Pk 2 Avg (I) d.nm	Pk 3 Avg (I) d.nm	DCR kcps
77	172.1	197.1	0.000	0.000	130730.8

APPENDIX D

SIZE DATA REPORTS FOR CENTRIFUGATION EXPERIMENTS EVALUATING THE CENTRIFUGATION TIME ON PARTICLE DIAMETER

Size Distribution Report by Intensity

Sample Details

Sample Name: #4--3500 RPM@30min 1
SOP Name: mansettings.dat
General Notes:

File Name:	Radius Comparison.dts	**Dispersant Name:**	Ethanol
Record Number:	96	**Dispersant RI:**	1.361
Material RI:	2.59	**Viscosity (cP):**	1.1170
Material Absorbtion:	0.10	**Measurement Date and Time:**	Monday, August 09, 2010 3:1.

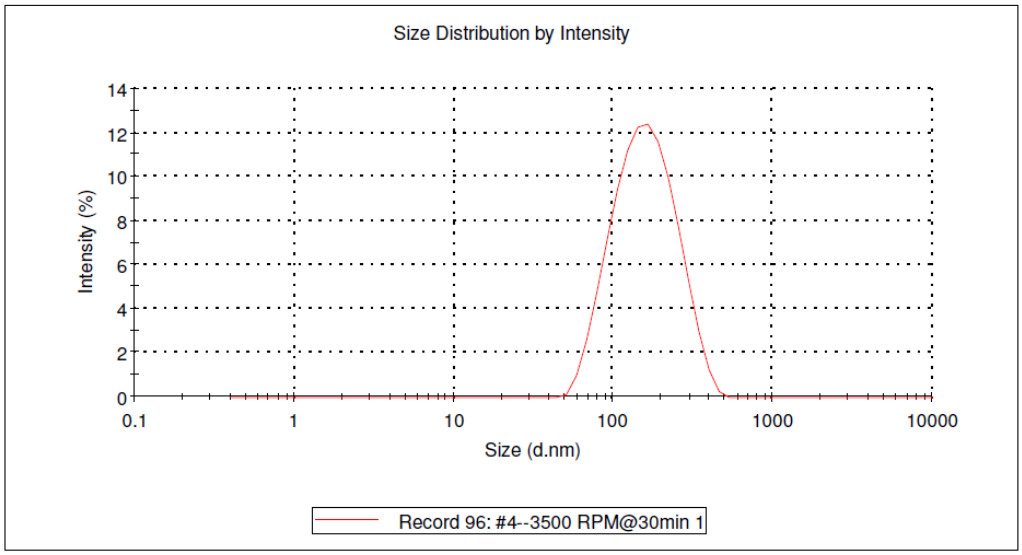
System

Temperature (°C):	20.0	**Duration Used (s):**	60
Count Rate (kcps):	455.0	**Measurement Position (mm):**	4.65
Cell Description:	Glass cuvette with round apert...	**Attenuator:**	6

Results

			Diam. (nm)	% Intensity	Width (nm)
Z-Average (d.nm):	142.9	**Peak 1:**	169.9	100.0	73.89
PdI:	0.142	**Peak 2:**	0.000	0.0	0.000
Intercept:	0.945	**Peak 3:**	0.000	0.0	0.000

Result quality : Good



Size Results Export Report

Sample Name: #4--3500 RPM@30min 1
Record Number: 96

Results Table

Size d.nm	Intensity %	Size d.nm	Intensity %
0.4000	0.0	68.06	2.6
0.4632	0.0	78.82	4.8
0.5365	0.0	91.28	7.1
0.6213	0.0	105.7	9.4
0.7195	0.0	122.4	11.2
0.8332	0.0	141.8	12.2
0.9649	0.0	164.2	12.4
1.117	0.0	190.1	11.6
1.294	0.0	220.2	10.0
1.499	0.0	255.0	7.7
1.736	0.0	295.3	5.3
2.010	0.0	342.0	3.0
2.328	0.0	396.1	1.3
2.696	0.0	458.7	0.3
3.122	0.0	531.2	0.0
3.615	0.0	615.1	0.0
4.187	0.0	712.4	0.0
4.849	0.0	825.0	0.0
5.615	0.0	955.4	0.0
6.503	0.0	1106	0.0
7.531	0.0	1281	0.0
8.721	0.0	1484	0.0
10.10	0.0	1718	0.0
11.70	0.0	1990	0.0
13.54	0.0	2305	0.0
15.69	0.0	2669	0.0
18.17	0.0	3091	0.0
21.04	0.0	3580	0.0
24.36	0.0	4145	0.0
28.21	0.0	4801	0.0
32.67	0.0	5560	0.0
37.84	0.0	6439	0.0
43.82	0.0	7456	0.0
50.75	0.1	8635	0.0
58.77	1.0	1.000e4	0.0

Right click the table to change the displayed parameters and/or change the number of columns. Use the Edit - Copy Table command to copy the data for pasting into a third party spreadsheet or graphing package.

Trend Table

Rec #	Z-Avg d.nm	Pk 1 Avg (I) d.nm	Pk 2 Avg (I) d.nm	Pk 3 Avg (I) d.nm	DCR kcps
96	142.9	169.9	0.000	0.000	146670.9

Size Distribution Report by Intensity

Sample Details

Sample Name: #7--3500 RPM@60min 1

SOP Name: mansettings.dat

General Notes:

File Name:	Radius Comparison.dts	**Dispersant Name:**	Ethanol
Record Number:	100	**Dispersant RI:**	1.361
Material RI:	2.59	**Viscosity (cP):**	1.1170
Material Absorbtion:	0.10	**Measurement Date and Time:**	Monday, August 09, 2010 3:3.

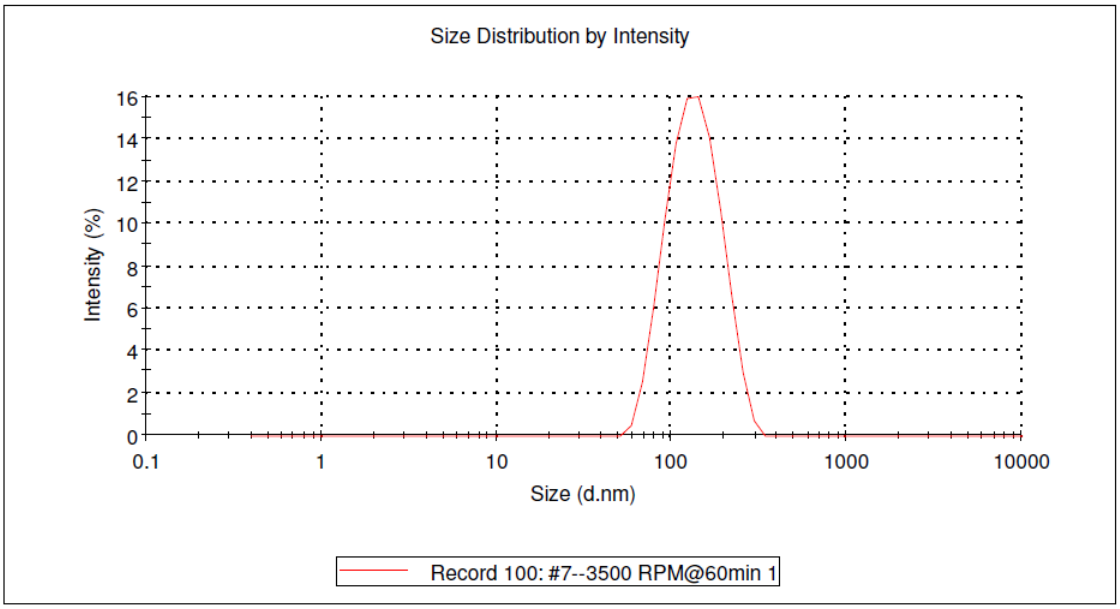
System

Temperature (°C):	20.0	**Duration Used (s):**	60
Count Rate (kcps):	276.9	**Measurement Position (mm):**	4.65
Cell Description:	Glass cuvette with round apert...	**Attenuator:**	6

Results

			Diam. (nm)	% Intensity	Width (nm)
Z-Average (d.nm):	124.5	**Peak 1:**	140.4	100.0	46.82
PdI:	0.109	**Peak 2:**	0.000	0.0	0.000
Intercept:	0.961	**Peak 3:**	0.000	0.0	0.000

Result quality : Good



Size Results Export Report

Sample Name: #7--3500 RPM@60min 1
Record Number: 100

Results Table

Size d.nm	Intensity %	Size d.nm	Intensity %
0.4000	0.0	68.06	2.6
0.4632	0.0	78.82	6.1
0.5365	0.0	91.28	10.2
0.6213	0.0	105.7	13.8
0.7195	0.0	122.4	15.9
0.8332	0.0	141.8	16.0
0.9649	0.0	164.2	14.1
1.117	0.0	190.1	10.6
1.294	0.0	220.2	6.6
1.499	0.0	255.0	3.0
1.736	0.0	295.3	0.7
2.010	0.0	342.0	0.0
2.328	0.0	396.1	0.0
2.696	0.0	458.7	0.0
3.122	0.0	531.2	0.0
3.615	0.0	615.1	0.0
4.187	0.0	712.4	0.0
4.849	0.0	825.0	0.0
5.615	0.0	955.4	0.0
6.503	0.0	1106	0.0
7.531	0.0	1281	0.0
8.721	0.0	1484	0.0
10.10	0.0	1718	0.0
11.70	0.0	1990	0.0
13.54	0.0	2305	0.0
15.69	0.0	2669	0.0
18.17	0.0	3091	0.0
21.04	0.0	3580	0.0
24.36	0.0	4145	0.0
28.21	0.0	4801	0.0
32.67	0.0	5560	0.0
37.84	0.0	6439	0.0
43.82	0.0	7456	0.0
50.75	0.0	8635	0.0
58.77	0.5	1.000e4	0.0

Right click the table to change the displayed parameters and/or change the number of columns. Use the Edit - Copy Table command to copy the data for pasting into a third party spreadsheet or graphing package.

Trend Table

Rec #	Z-Avg d.nm	Pk 1 Avg (I) d.nm	Pk 2 Avg (I) d.nm	Pk 3 Avg (I) d.nm	DCR kcps
100	124.5	140.4	0.000	0.000	89266.9

Size Distribution Report by Intensity

Sample Details

Sample Name: #9--3500 RPM@90min 1

SOP Name: mansettings.dat

General Notes:

File Name:	Radius Comparison.dts	**Dispersant Name:**	Ethanol
Record Number:	102	**Dispersant RI:**	1.361
Material RI:	2.59	**Viscosity (cP):**	1.1170
Material Absorbtion:	0.10	**Measurement Date and Time:**	Monday, August 09, 2010 3:5.

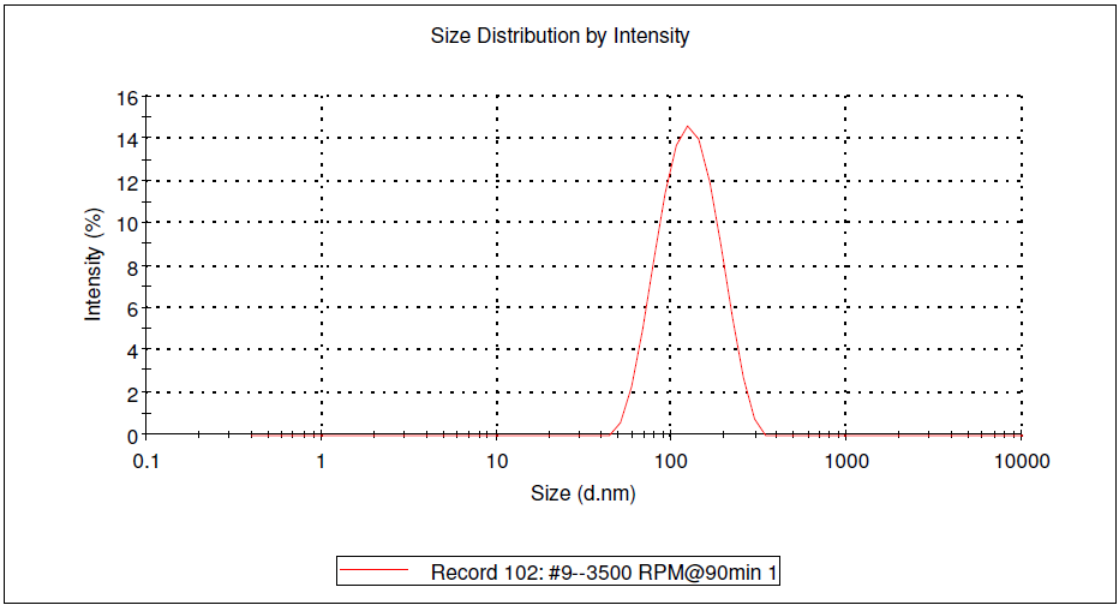
System

Temperature (°C):	20.0	**Duration Used (s):**	70
Count Rate (kcps):	184.5	**Measurement Position (mm):**	4.65
Cell Description:	Glass cuvette with round apert...	**Attenuator:**	6

Results

			Diam. (nm)	% Intensity	Width (nm)
Z-Average (d.nm):	116.1	**Peak 1:**	132.5	100.0	49.04
PdI:	0.116	**Peak 2:**	0.000	0.0	0.000
Intercept:	0.969	**Peak 3:**	0.000	0.0	0.000

Result quality : Good



Size Results Export Report

Sample Name: #9--3500 RPM@90min 1
Record Number: 102

Results Table

Size d.nm	Intensity %	Size d.nm	Intensity %
0.4000	0.0	68.06	5.1
0.4632	0.0	78.82	8.3
0.5365	0.0	91.28	11.4
0.6213	0.0	105.7	13.7
0.7195	0.0	122.4	14.6
0.8332	0.0	141.8	14.0
0.9649	0.0	164.2	11.9
1.117	0.0	190.1	8.9
1.294	0.0	220.2	5.6
1.499	0.0	255.0	2.7
1.736	0.0	295.3	0.8
2.010	0.0	342.0	0.0
2.328	0.0	396.1	0.0
2.696	0.0	458.7	0.0
3.122	0.0	531.2	0.0
3.615	0.0	615.1	0.0
4.187	0.0	712.4	0.0
4.849	0.0	825.0	0.0
5.615	0.0	955.4	0.0
6.503	0.0	1106	0.0
7.531	0.0	1281	0.0
8.721	0.0	1484	0.0
10.10	0.0	1718	0.0
11.70	0.0	1990	0.0
13.54	0.0	2305	0.0
15.69	0.0	2669	0.0
18.17	0.0	3091	0.0
21.04	0.0	3580	0.0
24.36	0.0	4145	0.0
28.21	0.0	4801	0.0
32.67	0.0	5560	0.0
37.84	0.0	6439	0.0
43.82	0.0	7456	0.0
50.75	0.6	8635	0.0
58.77	2.3	1.000e4	0.0

Right click the table to change the displayed parameters and/or change the number of columns. Use the Edit - Copy Table command to copy the data for pasting into a third party spreadsheet or graphing package.

Trend Table

Rec #	Z-Avg d.nm	Pk 1 Avg (I) d.nm	Pk 2 Avg (I) d.nm	Pk 3 Avg (I) d.nm	DCR kcps
102	116.1	132.5	0.000	0.000	59476.6

Size Distribution Report by Intensity

Sample Details

Sample Name: #11--3500 RPM@120min 1

SOP Name: mansettings.dat

General Notes:

File Name:	Radius Comparison.dts	**Dispersant Name:**	Ethanol
Record Number:	104	**Dispersant RI:**	1.361
Material RI:	2.59	**Viscosity (cP):**	1.1170
Material Absorbtion:	0.10	**Measurement Date and Time:**	Monday, August 09, 2010 4:1

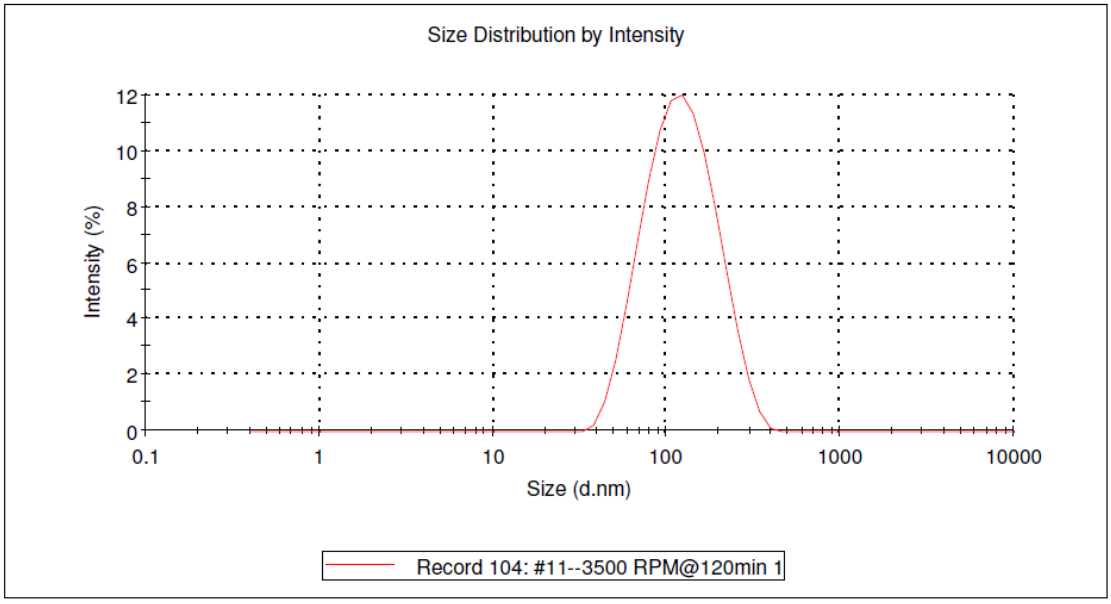
System

Temperature (°C):	20.0	**Duration Used (s):**	60
Count Rate (kcps):	345.0	**Measurement Position (mm):**	4.65
Cell Description:	Glass cuvette with round apert...	**Attenuator:**	7

Results

			Diam. (nm)	% Intensity	Width (nm)
Z-Average (d.nm):	109.7	**Peak 1:**	131.0	100.0	59.93
PdI:	0.179	**Peak 2:**	0.000	0.0	0.000
Intercept:	0.958	**Peak 3:**	0.000	0.0	0.000

Result quality : Good



Size Results Export Report

Sample Name: #11--3500 RPM@120min 1
Record Number: 104

Results Table

Size d.nm	Intensity %	Size d.nm	Intensity %
0.4000	0.0	68.06	6.8
0.4632	0.0	78.82	9.0
0.5365	0.0	91.28	10.7
0.6213	0.0	105.7	11.8
0.7195	0.0	122.4	12.0
0.8332	0.0	141.8	11.3
0.9649	0.0	164.2	9.9
1.117	0.0	190.1	8.0
1.294	0.0	220.2	5.8
1.499	0.0	255.0	3.6
1.736	0.0	295.3	1.9
2.010	0.0	342.0	0.7
2.328	0.0	396.1	0.1
2.696	0.0	458.7	0.0
3.122	0.0	531.2	0.0
3.615	0.0	615.1	0.0
4.187	0.0	712.4	0.0
4.849	0.0	825.0	0.0
5.615	0.0	955.4	0.0
6.503	0.0	1106	0.0
7.531	0.0	1281	0.0
8.721	0.0	1484	0.0
10.10	0.0	1718	0.0
11.70	0.0	1990	0.0
13.54	0.0	2305	0.0
15.69	0.0	2669	0.0
18.17	0.0	3091	0.0
21.04	0.0	3580	0.0
24.36	0.0	4145	0.0
28.21	0.0	4801	0.0
32.67	0.0	5560	0.0
37.84	0.2	6439	0.0
43.82	1.1	7456	0.0
50.75	2.5	8635	0.0
58.77	4.6	1.000e4	0.0

Right click the table to change the displayed parameters and/or change the number of columns. Use the Edit - Copy Table command to copy the data for pasting into a third party spreadsheet or graphing package.

Trend Table

Rec #	Z-Avg d.nm	Pk 1 Avg (I) d.nm	Pk 2 Avg (I) d.nm	Pk 3 Avg (I) d.nm	DCR kcps
104	109.7	131.0	0.000	0.000	39972.4

APPENDIX E

SIZE DATA REPORTS AND MOBILITY MEASUREMENTS FOR THE THREE SUSPENSIONS PREPARED WITH THE TIO_2 NANOPOWDERS FROM NANOAMOR AND ETHANOL

Size Distribution Report by Intensity

Sample Details

Sample Name TiO2 in ethanol, 30min, 2000rpm

SOP Name: mansettings.dat

General Notes:

File Name:	Radius Comparison.dts	**Dispersant Name:**	Ethanol
Record Number:	133	**Dispersant RI:**	1.361
Material RI:	2.59	**Viscosity (cP):**	1.1170
Material Absorbtion:	0.10	**Measurement Date and Time:**	Tuesday, August 24, 2010 12

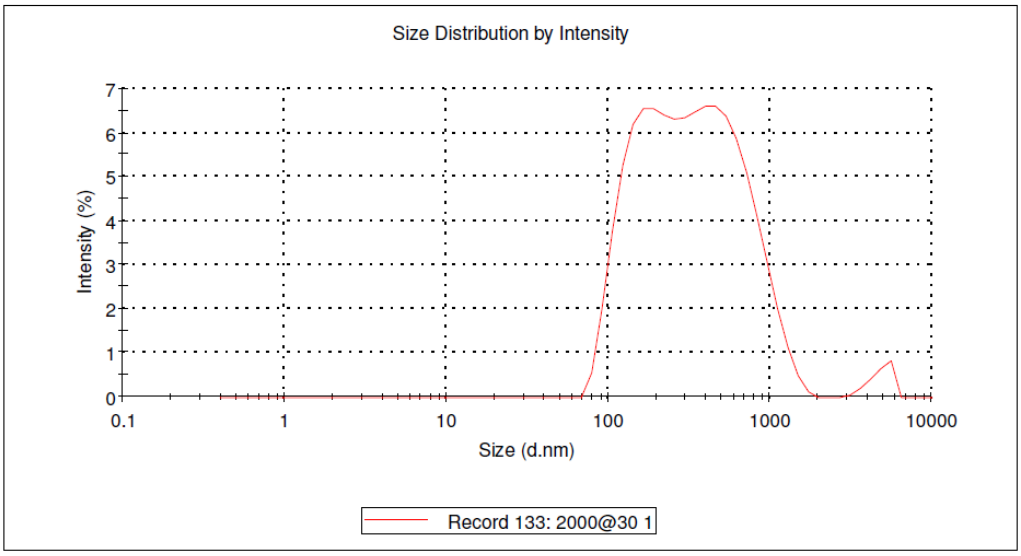
System

Temperature (°C):	20.0	**Duration Used (s):**	80
Count Rate (kcps):	139.4	**Measurement Position (mm):**	1.25
Cell Description:	Glass cuvette with round apert...	**Attenuator:**	3

Results

			Diam. (nm)	% Intensity	Width (nm)
Z-Average (d.nm):	268.0	**Peak 1:**	543.3	57.0	265.3
PdI:	0.352	**Peak 2:**	171.7	41.0	51.13
Intercept:	0.948	**Peak 3:**	4805	2.0	724.1

Result quality : Good



Size Results Export Report

Sample Name TiO2 in ethanol, 30min, 2000rpm
Record Number: 133

Results Table

Size d.nm	Intensity %	Size d.nm	Intensity %
0.4000	0.0	68.06	0.0
0.4632	0.0	78.82	0.6
0.5365	0.0	91.28	2.0
0.6213	0.0	105.7	3.8
0.7195	0.0	122.4	5.3
0.8332	0.0	141.8	6.2
0.9649	0.0	164.2	6.5
1.117	0.0	190.1	6.5
1.294	0.0	220.2	6.4
1.499	0.0	255.0	6.3
1.736	0.0	295.3	6.3
2.010	0.0	342.0	6.5
2.328	0.0	396.1	6.6
2.696	0.0	458.7	6.6
3.122	0.0	531.2	6.4
3.615	0.0	615.1	5.9
4.187	0.0	712.4	5.1
4.849	0.0	825.0	4.1
5.615	0.0	955.4	3.1
6.503	0.0	1106	2.0
7.531	0.0	1281	1.1
8.721	0.0	1484	0.5
10.10	0.0	1718	0.1
11.70	0.0	1990	0.0
13.54	0.0	2305	0.0
15.69	0.0	2669	0.0
18.17	0.0	3091	0.1
21.04	0.0	3580	0.2
24.36	0.0	4145	0.4
28.21	0.0	4801	0.7
32.67	0.0	5560	0.8
37.84	0.0	6439	0.0
43.82	0.0	7456	0.0
50.75	0.0	8635	0.0
58.77	0.0	1.000e4	0.0

Right click the table to change the displayed parameters and/or change the number of columns. Use the Edit - Copy Table command to copy the data for pasting into a third party spreadsheet or graphing package.

Trend Table

Rec #	Z-Avg d.nm	Pk 1 Avg (I) d.nm	Pk 2 Avg (I) d.nm	Pk 3 Avg (I) d.nm	DCR kcps
133	268.0	543.3	171.7	4805	980243.3

Zeta Potential Report

Sample Details

Sample Name TiO2 in ethanol, 30min, 2000rpm

SOP Name: mansettings.dat

General Notes:

File Name:	Radius Comparison.dts	**Dispersant Name:**	Ethanol
Record Number:	134	**Dispersant RI:**	1.361
Date and Time:	Tuesday, August 24, 2010 12:09:...	**Viscosity (cP):**	1.1170
		Dispersant Dielectric Constant:	25.2

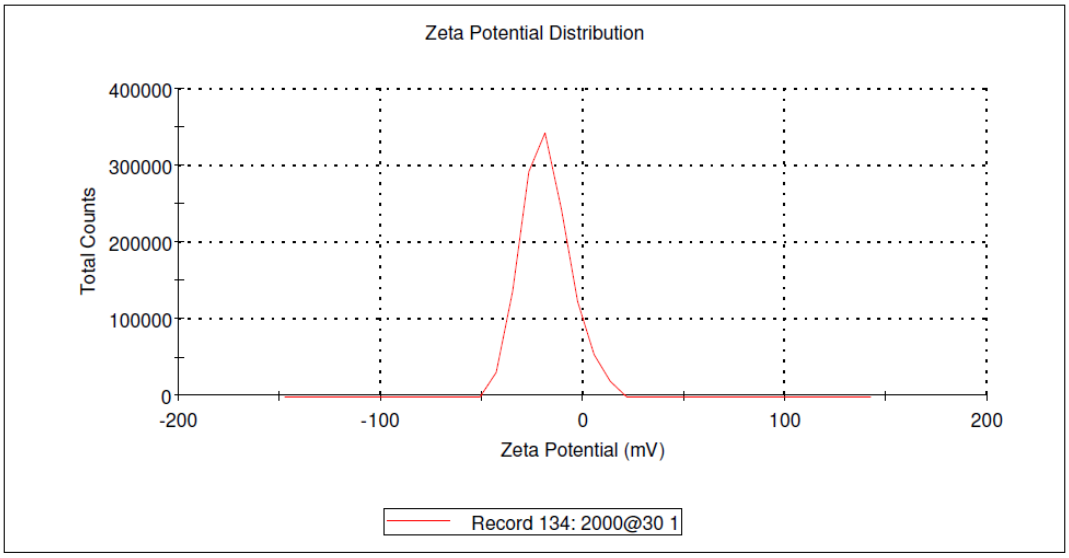
System

Temperature (°C):	20.0	**Zeta Runs:**	12
Count Rate (kcps):	71.4	**Measurement Position (mm):**	4.50
Cell Description:	Zeta dip cell	**Attenuator:**	7

Results

			Mean (mV)	Area (%)	Width (mV)
Zeta Potential (mV):	-18.4	**Peak 1:**	-18.4	100.0	11.8
Zeta Deviation (mV):	11.8	**Peak 2:**	0.00	0.0	0.00
Conductivity (mS/cm):	8.69e-4	**Peak 3:**	0.00	0.0	0.00

Result quality : Good



Electrophoretic Mobility Report

Sample Details

Sample Name TiO2 in ethanol, 30min, 2000rpm

SOP Name: mansettings.dat

General Notes:

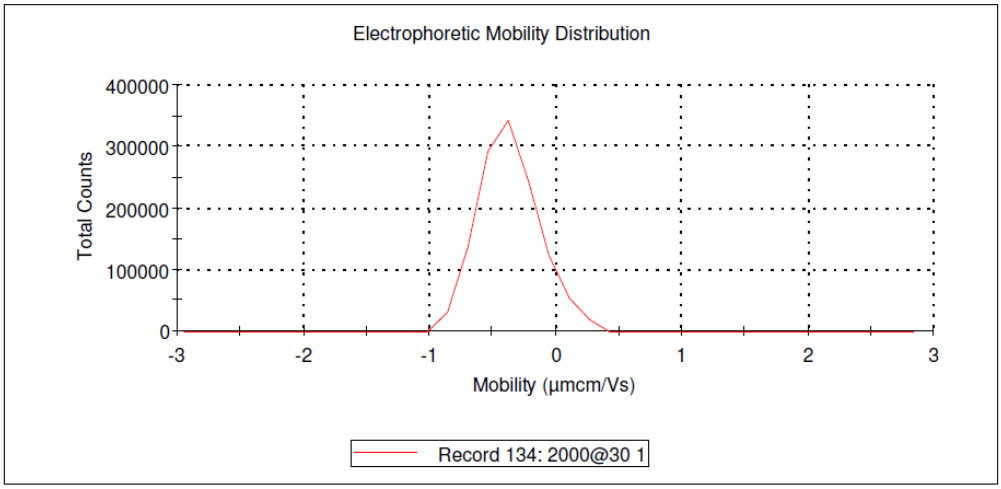
File Name:	Radius Comparison.dts	**Dispersant Name:**	Ethanol
Record Number:	134	**Dispersant RI:**	1.361
Date and Time:	Tuesday, August 24, 2010 12:0...	**Viscosity (cP):**	1.1170
		Dispersant Dielectric Constant:	25.2

System

Temperature (°C):	20.0	**Zeta Runs:**	12
Count Rate (kcps):	71.4	**Measurement Position (mm):**	4.50
Cell Description:	Zeta dip cell	**Attenuator:**	7

Results

			Mean (µmcm/Vs)	Area (%)	Width (mV)
Mobility (µmcm/Vs):	-0.3672	**Peak 1:**	-0.367	100.0	0.235
Mobility Dev. (µmcm/Vs):	0.2354	**Peak 2:**	0.00	0.0	0.00
Conductivity (mS/cm):	8.69e-4	**Peak 3:**	0.00	0.0	0.00
Result quality :	Good				



Size Distribution Report by Intensity

Sample Details

Sample Name TiO2 in ethanol, 90min, 3500rpm

SOP Name: mansettings.dat

General Notes:

File Name:	Radius Comparison.dts	**Dispersant Name:**	Ethanol
Record Number:	132	**Dispersant RI:**	1.361
Material RI:	2.59	**Viscosity (cP):**	1.1170
Material Absorbtion:	0.10	**Measurement Date and Time:**	Wednesday, August 18, 201..

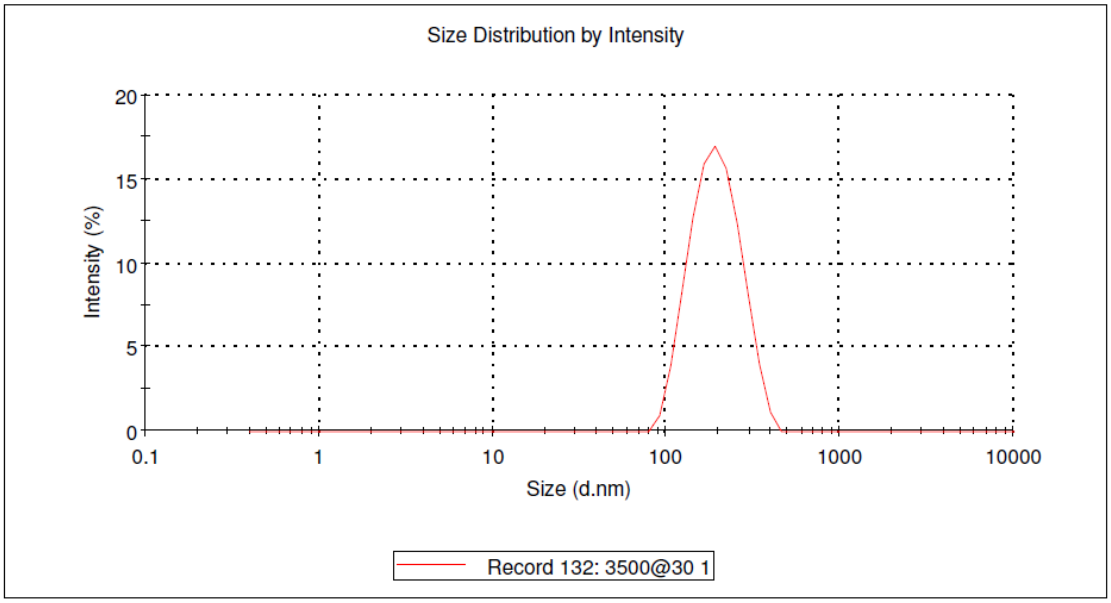
System

Temperature (°C):	20.0	**Duration Used (s):**	60
Count Rate (kcps):	462.8	**Measurement Position (mm):**	4.65
Cell Description:	Glass cuvette with round apert...	**Attenuator:**	6

Results

			Diam. (nm)	% Intensity	Width (nm)
Z-Average (d.nm):	179.1	**Peak 1:**	199.7	100.0	63.54
PdI:	0.104	**Peak 2:**	0.000	0.0	0.000
Intercept:	0.941	**Peak 3:**	0.000	0.0	0.000

Result quality : Good



Size Results Export Report

Sample Name TiO2 in ethanol, 90min, 3500rpm
Record Number: 132

Results Table

Size d.nm	Intensity %	Size d.nm	Intensity %
0.4000	0.0	68.06	0.0
0.4632	0.0	78.82	0.0
0.5365	0.0	91.28	1.0
0.6213	0.0	105.7	3.9
0.7195	0.0	122.4	8.3
0.8332	0.0	141.8	12.7
0.9649	0.0	164.2	15.9
1.117	0.0	190.1	17.0
1.294	0.0	220.2	15.6
1.499	0.0	255.0	12.4
1.736	0.0	295.3	8.1
2.010	0.0	342.0	4.0
2.328	0.0	396.1	1.2
2.696	0.0	458.7	0.0
3.122	0.0	531.2	0.0
3.615	0.0	615.1	0.0
4.187	0.0	712.4	0.0
4.849	0.0	825.0	0.0
5.615	0.0	955.4	0.0
6.503	0.0	1106	0.0
7.531	0.0	1281	0.0
8.721	0.0	1484	0.0
10.10	0.0	1718	0.0
11.70	0.0	1990	0.0
13.54	0.0	2305	0.0
15.69	0.0	2669	0.0
18.17	0.0	3091	0.0
21.04	0.0	3580	0.0
24.36	0.0	4145	0.0
28.21	0.0	4801	0.0
32.67	0.0	5560	0.0
37.84	0.0	6439	0.0
43.82	0.0	7456	0.0
50.75	0.0	8635	0.0
58.77	0.0	1.000e4	0.0

Right click the table to change the displayed parameters and/or change the number of columns. Use the Edit - Copy Table command to copy the data for pasting into a third party spreadsheet or graphing package.

Trend Table

Rec #	Z-Avg d.nm	Pk 1 Avg (I) d.nm	Pk 2 Avg (I) d.nm	Pk 3 Avg (I) d.nm	DCR kcps
132	179.1	199.7	0.000	0.000	149197.8

Zeta Potential Report

Sample Details

Sample Name TiO2 in ethanol, 90min, 3500rpm

SOP Name: mansettings.dat

General Notes:

File Name:	Radius Comparison.dts	**Dispersant Name:**	Ethanol
Record Number:	135	**Dispersant RI:**	1.361
Date and Time:	Tuesday, August 24, 2010 12:23:...	**Viscosity (cP):**	1.1170
		Dispersant Dielectric Constant:	25.2

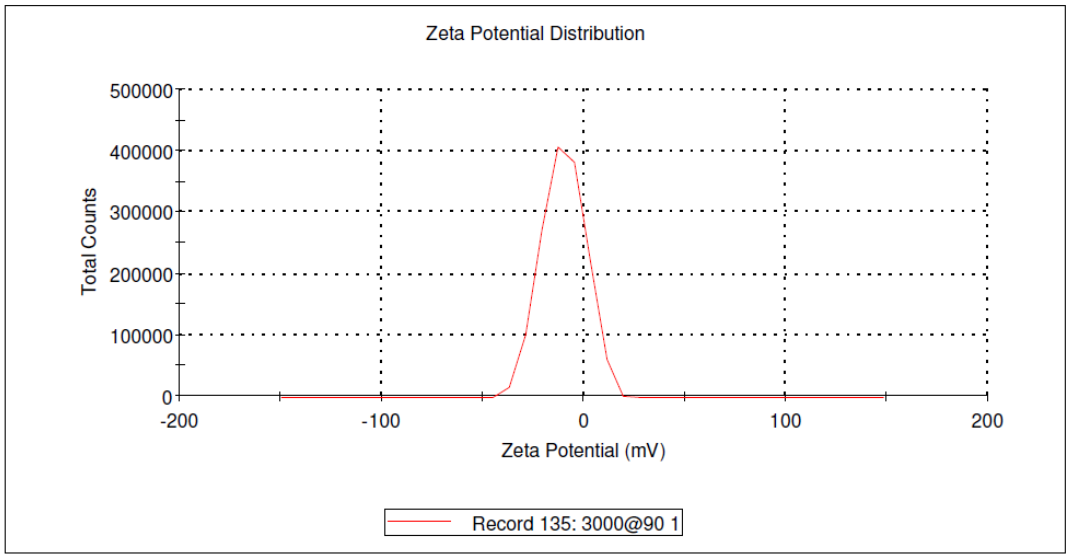
System

Temperature (°C):	20.0	**Zeta Runs:**	13
Count Rate (kcps):	233.3	**Measurement Position (mm):**	4.50
Cell Description:	Zeta dip cell	**Attenuator:**	6

Results

			Mean (mV)	Area (%)	Width (mV)
Zeta Potential (mV):	-10.1	**Peak 1:**	-10.1	100.0	10.5
Zeta Deviation (mV):	10.5	**Peak 2:**	0.00	0.0	0.00
Conductivity (mS/cm):	0.00111	**Peak 3:**	0.00	0.0	0.00

Result quality : Good



Electrophoretic Mobility Report

Sample Details

Sample Name TiO2 in ethanol, 90min, 3500rpm

SOP Name: mansettings.dat

General Notes:

File Name:	Radius Comparison.dts	**Dispersant Name:**	Ethanol
Record Number:	135	**Dispersant RI:**	1.361
Date and Time:	Tuesday, August 24, 2010 12:2...	**Viscosity (cP):**	1.1170
		Dispersant Dielectric Constant:	25.2

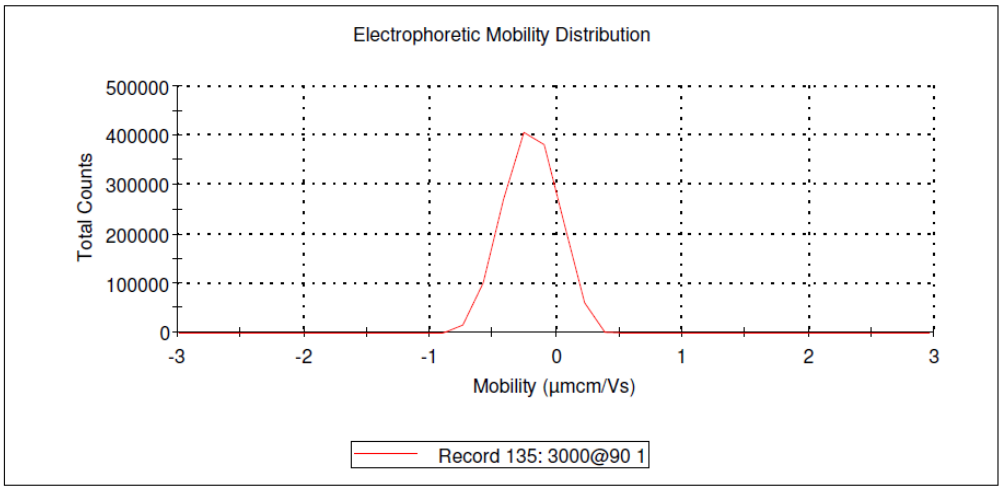
System

Temperature (°C):	20.0	**Zeta Runs:**	13
Count Rate (kcps):	233.3	**Measurement Position (mm):**	4.50
Cell Description:	Zeta dip cell	**Attenuator:**	6

Results

			Mean (µmcm/Vs)	Area (%)	Width (mV)
Mobility (µmcm/Vs):	-0.2023	**Peak 1:**	-0.202	100.0	0.210
Mobility Dev. (µmcm/Vs):	0.2101	**Peak 2:**	0.00	0.0	0.00
Conductivity (mS/cm):	0.00111	**Peak 3:**	0.00	0.0	0.00

Result quality : Good



Size Distribution Report by Intensity

Sample Details

Sample Name: TiO2 in ethanol, 90min 3500rpm, 30min 15000rpm

SOP Name: mansettings.dat

General Notes:

File Name:	Radius Comparison.dts	**Dispersant Name:**	Ethanol
Record Number:	131	**Dispersant RI:**	1.361
Material RI:	2.59	**Viscosity (cP):**	1.1170
Material Absorbtion:	0.10	**Measurement Date and Time:**	Wednesday, August 18, 201..

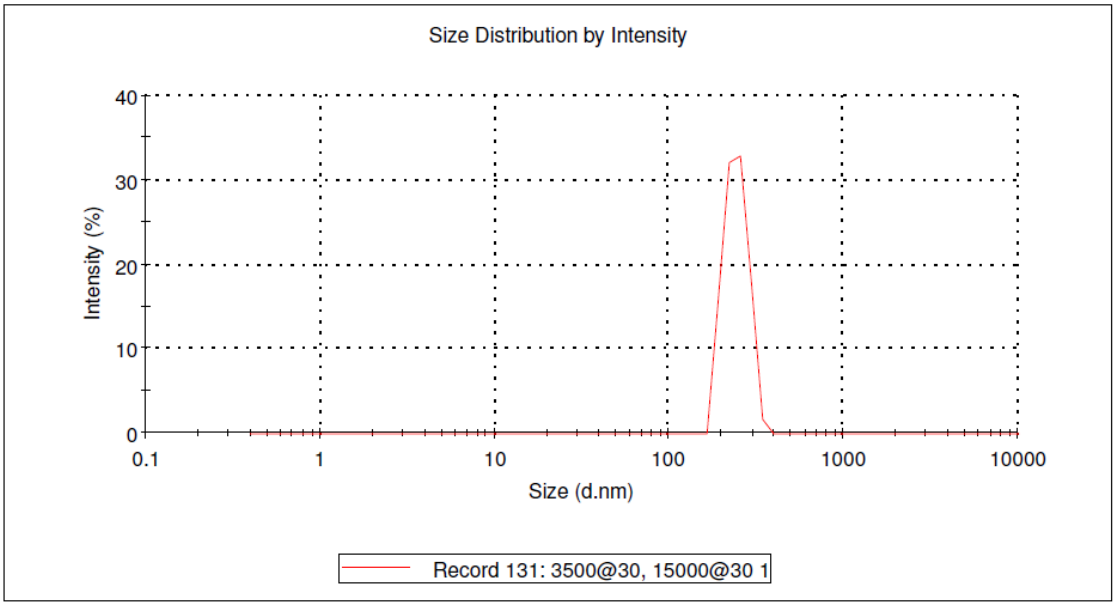
System

Temperature (°C):	20.0	**Duration Used (s):**	60
Count Rate (kcps):	336.2	**Measurement Position (mm):**	4.65
Cell Description:	Glass cuvette with round apert...	**Attenuator:**	10

Results

			Diam. (nm)	% Intensity	Width (nm)
Z-Average (d.nm):	379.4	**Peak 1:**	242.3	100.0	36.10
PdI:	0.465	**Peak 2:**	0.000	0.0	0.000
Intercept:	0.971	**Peak 3:**	0.000	0.0	0.000

Result quality : POOR - see result quality report



Size Results Export Report

Sample Name TiO2 in ethanol, 90min 3500rpm, 30min 15000rpm
Record Number: 131

Results Table

Size d.nm	Intensity %	Size d.nm	Intensity %
0.4000	0.0	68.06	0.0
0.4632	0.0	78.82	0.0
0.5365	0.0	91.28	0.0
0.6213	0.0	105.7	0.0
0.7195	0.0	122.4	0.0
0.8332	0.0	141.8	0.0
0.9649	0.0	164.2	0.0
1.117	0.0	190.1	15.7
1.294	0.0	220.2	32.1
1.499	0.0	255.0	32.8
1.736	0.0	295.3	17.7
2.010	0.0	342.0	1.7
2.328	0.0	396.1	0.0
2.696	0.0	458.7	0.0
3.122	0.0	531.2	0.0
3.615	0.0	615.1	0.0
4.187	0.0	712.4	0.0
4.849	0.0	825.0	0.0
5.615	0.0	955.4	0.0
6.503	0.0	1106	0.0
7.531	0.0	1281	0.0
8.721	0.0	1484	0.0
10.10	0.0	1718	0.0
11.70	0.0	1990	0.0
13.54	0.0	2305	0.0
15.69	0.0	2669	0.0
18.17	0.0	3091	0.0
21.04	0.0	3580	0.0
24.36	0.0	4145	0.0
28.21	0.0	4801	0.0
32.67	0.0	5560	0.0
37.84	0.0	6439	0.0
43.82	0.0	7456	0.0
50.75	0.0	8635	0.0
58.77	0.0	1.000e4	0.0

Right click the table to change the displayed parameters and/or change the number of columns. Use the Edit - Copy Table command to copy the data for pasting into a third party spreadsheet or graphing package.

Trend Table

Rec #	Z-Avg d.nm	Pk 1 Avg (I) d.nm	Pk 2 Avg (I) d.nm	Pk 3 Avg (I) d.nm	DCR kcps
131	379.4	242.3	0.000	0.000	1195.4

Zeta Potential Report

Sample Details

Sample Name: TiO2 in ethanol, 90min 3500rpm, 30min 15000rpm

SOP Name: mansettings.dat

General Notes:

File Name:	Radius Comparison.dts	**Dispersant Name:**	Ethanol
Record Number:	136	**Dispersant RI:**	1.361
Date and Time:	Tuesday, August 24, 2010 12:28:...	**Viscosity (cP):**	1.1170
		Dispersant Dielectric Constant:	25.2

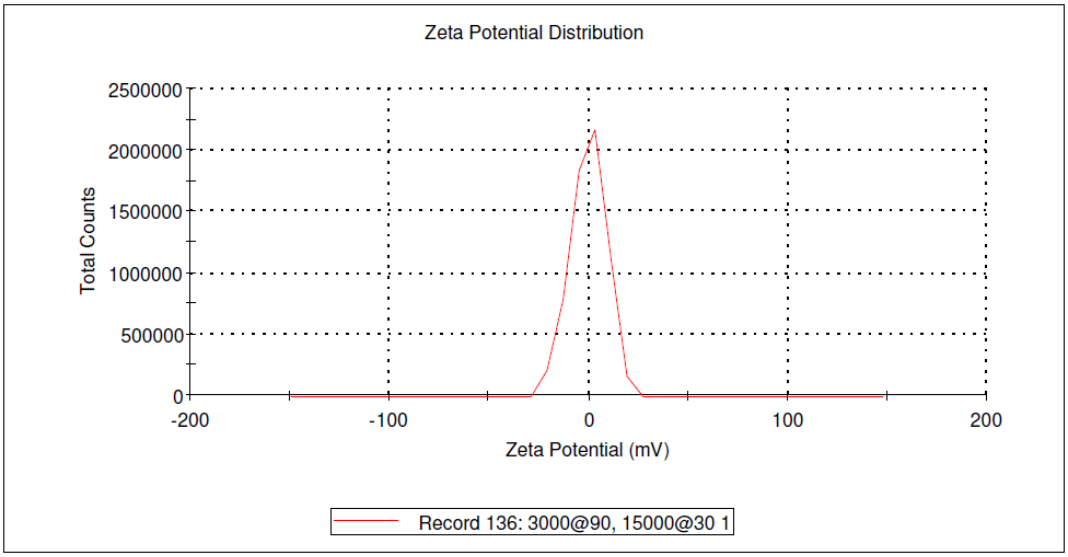
System

Temperature (°C):	20.0	**Zeta Runs:**	100
Count Rate (kcps):	49.1	**Measurement Position (mm):**	4.50
Cell Description:	Zeta dip cell	**Attenuator:**	10

Results

			Mean (mV)	Area (%)	Width (mV)
Zeta Potential (mV):	-0.266	**Peak 1:**	-0.266	100.0	8.84
Zeta Deviation (mV):	8.84	**Peak 2:**	0.00	0.0	0.00
Conductivity (mS/cm):	8.64e-4	**Peak 3:**	0.00	0.0	0.00

Result quality : POOR - See result quality report



Electrophoretic Mobility Report

Sample Details

Sample Name: TiO2 in ethanol, 90min 3500rpm, 30min 15000rpm

SOP Name: mansettings.dat

General Notes:

File Name:	Radius Comparison.dts	**Dispersant Name:**	Ethanol
Record Number:	136	**Dispersant RI:**	1.361
Date and Time:	Tuesday, August 24, 2010 12:2...	**Viscosity (cP):**	1.1170
		Dispersant Dielectric Constant:	25.2

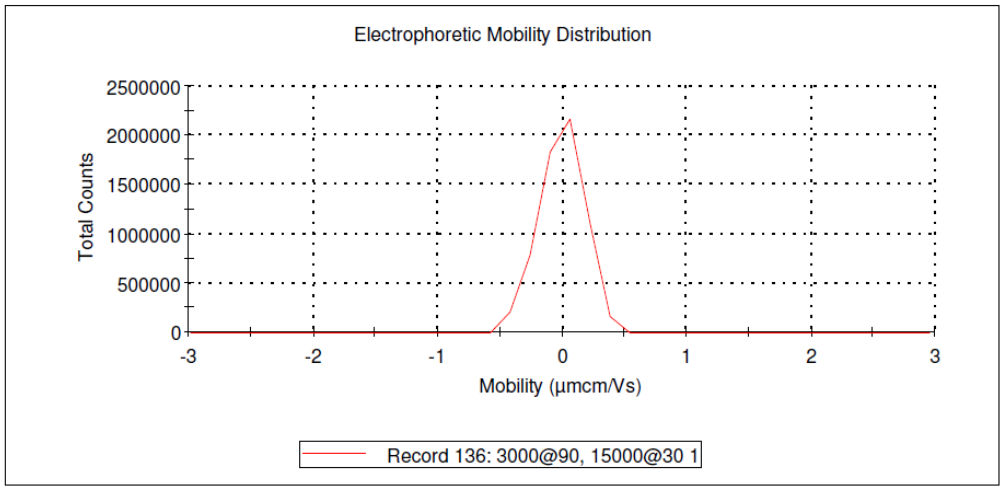
System

Temperature (°C):	20.0	**Zeta Runs:**	100
Count Rate (kcps):	49.1	**Measurement Position (mm):**	4.50
Cell Description:	Zeta dip cell	**Attenuator:**	10

Results

			Mean (µmcm/Vs)	Area (%)	Width (mV)
Mobility (µmcm/Vs):	-0.005304	**Peak 1:**	-0.00530	100.0	0.176
Mobility Dev. (µmcm/Vs):	0.1763	**Peak 2:**	0.00	0.0	0.00
Conductivity (mS/cm):	8.64e-4	**Peak 3:**	0.00	0.0	0.00

Result quality : POOR - See result quality report



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