

Particle Size Characterization in Turbid Colloidal Suspensions

Elliot Wainwright

Physics Department, The College of Wooster, Wooster, Ohio 44691, USA

(Dated: May 7, 2014)

This project aimed to refine an experimental method to measure the size of nano-scale polystyrene spheres suspended in a turbid medium. Turbidity is the murkiness or cloudiness of a liquid based off the presence of light scattering particles. By using static light scattering and the theory of Rayleigh scattering the size of the spheres can be determined by calculating the difference in light intensity due to single scattering in a turbid medium. Laser intensity measurements were performed at different number concentrations of particles. The relationship between the turbidity and number concentration allowed us to calculate the size of the spheres in the water. We calculated the approximate size of the 96 nm spheres to be 123 nm, and the size of the 107 nm to be 103 nm, with errors of 28% and 4%, respectively. These errors may be a result of aggregated and aged particle solutions, or from errors in initial number concentration estimations. A qualitative analysis on the turbidity suspension of powdered milk in distilled water was also reviewed, but concrete quantitative was not able to be acquired without a robust initial number concentration estimation, which was unobtainable for the samples used.

I. INTRODUCTION

Scattering of electromagnetic radiation can provide deep insight into the structure and shape of matter, especially in the characterization of micro or nano-scale size particles. Lord Rayleigh formulated the original theoretical groundwork in 1871 and 1881 describing light scattering off of arbitrary shape or size particle with refractive index near one [1]. Rayleigh famously explained that the reason that the sky was blue and sunsets were orange was due to wavelength dependent light scattering off of small particles in the atmosphere. Extensive work has been done on the topic by scientists such as Smoluchowski, Einstein, Debye, and Mie, where the final two independently solved the scattering solution for much larger spherical particles outside of the Rayleigh regime, now called Mie scattering [1]. Van de Hulst and Kerker's work was fundamental in the summarization and significant discussion of electromagnetic scattering in their popular books [2–4].

Static light scattering through a turbid medium, or a liquid solution filled with light scattering particles, is a relatively simple experimental method that can be used to test a myriad of physical properties about colloidal suspensions of small spheres. Static light scattering implements the simple principle of Rayleigh scattering in a straightforward experimental setup to help study a physical property of an object. By measuring the intensity of singly-scattered light transmitted through a liquid medium made murky by the presence of a highly concentrated nanosphere solution one can determine the size of the spherical objects, provided the spheres have a diameter much smaller than the wavelength of light being transmitted. By having *a priori* knowledge of the size of specific nano-scale spheres, we test the accuracy of the experimental method and refine it to improve results. Once the experimental method is accurate and robust we use it to determine the size of unknown spherical objects of a known initial number concentration within our calculated

certainty. Particle characterization through colloidal suspensions is particularly useful in the food industry and in environmental studies of water quality. Implemented correctly, the experimental apparatus can also serve to be extremely useful in multi-disciplinary research, including but not limited to virus sizing, polymer characterization, or granular material or dust sample research [4]. This experiment is particularly useful due to its simplicity in design and the availability of the apparatus pieces.

This project serves to improve and consolidate a relatively straightforward static light scattering experimental method. This particular experiment includes attempts at sizing 96 nm and 107 nm diameter polystyrene spheres suspended in distilled water by taking direct light intensity measurements using a lock-in amplifier. Extensions to characterize the size of the particles in everyday objects such as milk or food coloring solutions are also attempted.

II. THEORY

A. Rayleigh Scattering

Lord Rayleigh contributed significantly to a large range of light scattering applications and theory, the most predominately applicable to this research being his calculations on the scattering by small dielectric spheres. Lord Rayleigh developed his theory using dimensional analysis and on the basis of a few assumptions: that particles that were scattering the light were spheres and small to the order of a twentieth of that of the scattered light. Mie scattering takes over when the particles are on the same order as that of the wavelength of light. Rayleigh scattering is also limited to individual or single scattering instances, that is, one photon off of one particle. In Kerker's interpretation of the theory, the intensity of scattered light I must be proportional to the initial intensity I_0 modulated by some function f , which is dependent on the volume

of the spherical particle V , the distance to the observer r , the wavelength of the incident light λ , and the index of refraction of the particle and medium n_1 and n_2 , as described by

$$I = f(V, r, \lambda, n_1, n_2)I_0, \quad (1)$$

where $f(V, r, \lambda, n_1, n_2)$ is dimensionless [3]. Stratton's derivation of intensity of a scattered wave I off of a sphere includes all these terms

$$I = \frac{16\pi^4 a^6}{r^2 \lambda^4} \left(\frac{n^2 - 1}{n^2 + 2} \right)^2 \sin[\phi], \quad (2)$$

where a is the radius of the spherical particle, $n = n_1/n_2$ is the relative refractive index, and ϕ is the angle between the scattered wave and the spherical dipole [5].

The total energy scattered by a particle in all directions, called the coefficient of scattering C_{sca} , can be obtained by integrating Eq. 2 over the surface area of a sphere

$$C_{\text{sca}} = \int_0^\pi \int_0^{2\pi} I r^2 \sin[\phi] d\phi d\theta, \quad (3)$$

where we are integrating in spherical coordinates, resulting in

$$C_{\text{sca}} = \frac{128\pi^5 a^6}{3\lambda^4} \left(\frac{n^2 - 1}{n^2 + 2} \right), \quad (4)$$

for a Rayleigh scatterer [3]. We relate this scattering coefficient to the transmitted intensity of light through our colloidal sample, as outlined in the next section.

B. Turbidity Calculation

Turbidity is defined to be an extinction coefficient of a liquid or other light dispersing medium. One can think of turbidity as the ‘‘murkiness’’ of a liquid medium caused by the concentration and scattering ability of particles suspended in the medium; it is a factor that relates the transmitted light intensity to the incident light intensity. A simple sketch can lead to the turbidity derivation, as seen in Fig. 1, where light is scattered by the particles in the medium. From Fig. 1, we can see that each spatial step dx some light is scattered

$$dI = -\tau I_0 dx, \quad (5)$$

where τ is defined to be the turbidity, the positive constant of interest. Rearranging and integrating we obtain the final turbidity result of

$$\tau = -\frac{1}{L} \ln \left(\frac{I_t}{I_0} \right). \quad (6)$$

Kerker relates the transmitted intensity to this turbidity coefficient using the coefficient of scattering calculated in the previous section. That is,

$$\tau = -\frac{1}{L} \ln \left(\frac{I_t}{I_0} \right) = \left(\frac{N}{V} \right) C_{\text{sca}}, \quad (7)$$

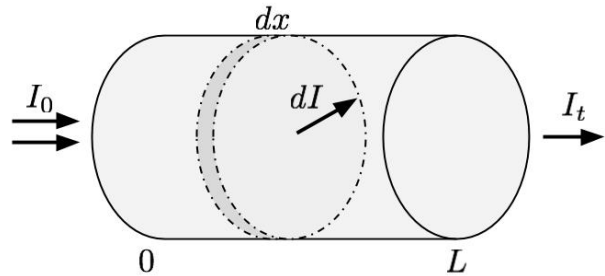


FIG. 1: Light incident on a cylinder of turbid fluid, where at each distance increment dx some light dI is scattered by the particles in the fluid, and L is the total length of the medium. Schematic idea courtesy of Dr. Don Jacobs.

where N/V is the number of particles per unit volume of the solution, which comprises the independent variable. C_{sca} is derived in Eq. 3. We can then solve for the radius a of the spheres in the solution

$$a = \left(\frac{\tau}{(N/V)} \frac{3\lambda^4}{128\pi^5} \frac{1}{n_{\text{eq}}} \right)^{1/6}, \quad (8)$$

where

$$n_{\text{eq}} = \left(\frac{n^2 - 1}{n^2 + 2} \right) \quad (9)$$

and $\tau/(N/V)$, will be experimentally determined by transmitted intensity measurements.

C. Concentration Calculation

One of the most difficult experimental processes for this project is changing the concentration of the particles in the liquid medium. This is generally achieved by increasing the amount of liquid in the solution without increasing the number of particles, but requires a technical calculation of the initial concentration, denoted by N_0/V_0 . The concentration of polystyrene spheres in a bottle differs in presentation depending on the manufacturer the spheres are acquired from. There are two primary forms of converting the percent concentration into the initial number of spheres per unit volume N_0/V_0 , which follow from given specifications of either weight per weight (W/W) of spheres in a bottle, or weight per volume (W/V), also called percent solids.

1. Weight per Weight to Number Concentration

If the concentration of spheres is given by a mass fraction W/W percentage, then we must calculate the volume fraction of the spheres relative to the total mixture

to obtain the number of particles. We calculate the volume fraction

$$\Phi = \frac{V_s}{V}, \quad (10)$$

where V_s is the volume of the spheres in the solution and V is the total volume. We can determine the total number of spheres N simply by dividing V_s by the volume of one sphere V_{1s} such that

$$\frac{N}{V} = \frac{1}{V} \cdot \frac{V_s}{V_{1s}} = \Phi \frac{1}{V_{1s}}. \quad (11)$$

To obtain a value for the Φ we use the given mass fraction $W/W \equiv f$ and relate through the relative density of the spheres ρ_s to that of the medium ρ_m , that is

$$\Phi = \frac{f}{f + (1-f) \frac{\rho_m}{\rho_s}}. \quad (12)$$

The volume of an individual sphere is trivially calculated to be

$$V_{1s} = \frac{4}{3} \pi a^3, \quad (13)$$

where we can now substitute into Eq. 11 to obtain our total number concentration of spheres for the entire bottle [6]. By simply multiplying by a specific initial volume, such as that in an individual drop from a syringe, we can obtain our initial number concentration N_0/V_0 .

2. Weight per Volume to Number Concentration

If the concentration of spheres is given by a percent solids, or weight per volume fraction (W/V), a more straightforward approach can be implemented for computing the initial number concentration. The W/V is simply the mass of the particles per total volume of the solution bottle, that is

$$\frac{W}{V} = \frac{M_s}{V} \implies \frac{N}{V} = \frac{1}{V} \cdot \frac{M_s}{M_{1s}}, \quad (14)$$

where M_s is the total mass of the spheres and M_{1s} is the mass of one sphere, expressed as

$$M_{1s} = \rho_s V_{1s}. \quad (15)$$

These terms combine into the final expression

$$\frac{N}{V} = \frac{W}{V} \cdot \frac{1}{\rho_s V_{1s}}, \quad (16)$$

where again we multiply by some arbitrary initial volume to reach our value of N_0/V_0 .

D. Uncertainty Analysis

When taking direct light intensity measurements, uncertainty must be propagated through calculations to obtain the turbidity uncertainty. The simplest way to do this is to use the general form of the uncertainty for a given function f

$$\sigma_f = \sqrt{\left(\frac{\partial f}{\partial x} \delta x\right)^2 + \left(\frac{\partial f}{\partial y} \delta y\right)^2 + \dots} \quad (17)$$

where x and y are parameters with their own associated uncertainty, which for our experiment with turbidity measurements becomes

$$\sigma_\tau = \sqrt{\left(\frac{\partial \tau}{\partial I_t} \delta I_t\right)^2 + \left(\frac{\partial \tau}{\partial I_0} \delta I_0\right)^2}, \quad (18)$$

where δI_t and δI_0 are the uncertainties of the transmitted and initial intensity, respectively. τ is of the form of Eq. 6. The uncertainty in the number concentration (N/V) is calculated using brute force, where theoretical maximum and minimum values of the volume are directly compared to actual concentration measurements based on situations that would cause the most error. Such situations include adding too much water volume into the concentration at each data iteration, or too little, which compounds uncertainty over measurements.

III. PROCEDURE

A. Experimental Apparatus

The experimental apparatus is made up of linearly aligned optical components including a Melles Griot intensity stabilized laser, a beam expander, an optical chopper, several pinhole apertures, a photo detector, a housing for the cuvette sample, and a Stanford Research System SR830 lock-in amplifier. The full apparatus layout can be seen in Fig. 2. We use a laser shone through each component as the incident light source which travels through the turbid sample and the transmitted intensity is recorded via an output current by the photo detector. The 632.8 nm laser is intensity stabilized ($I_0 \pm 0.1\%$) so that we can get accurate readings from the lock-in amplifier of the photo detector's output current. Once turned on, the laser takes approximately 20 minutes to stabilize to a steady value of intensity. The laser is designed to remain on for long periods of time and remained on for the duration of the experimentation.

The stabilized laser eliminates short term intensity drift from one moment to another, but it was discovered that the laser intensity has a tendency to drift over long periods of time. In future instances, it may be better to calculate the turbidity dependent on current I_0 values during a particular time of experimentation rather than

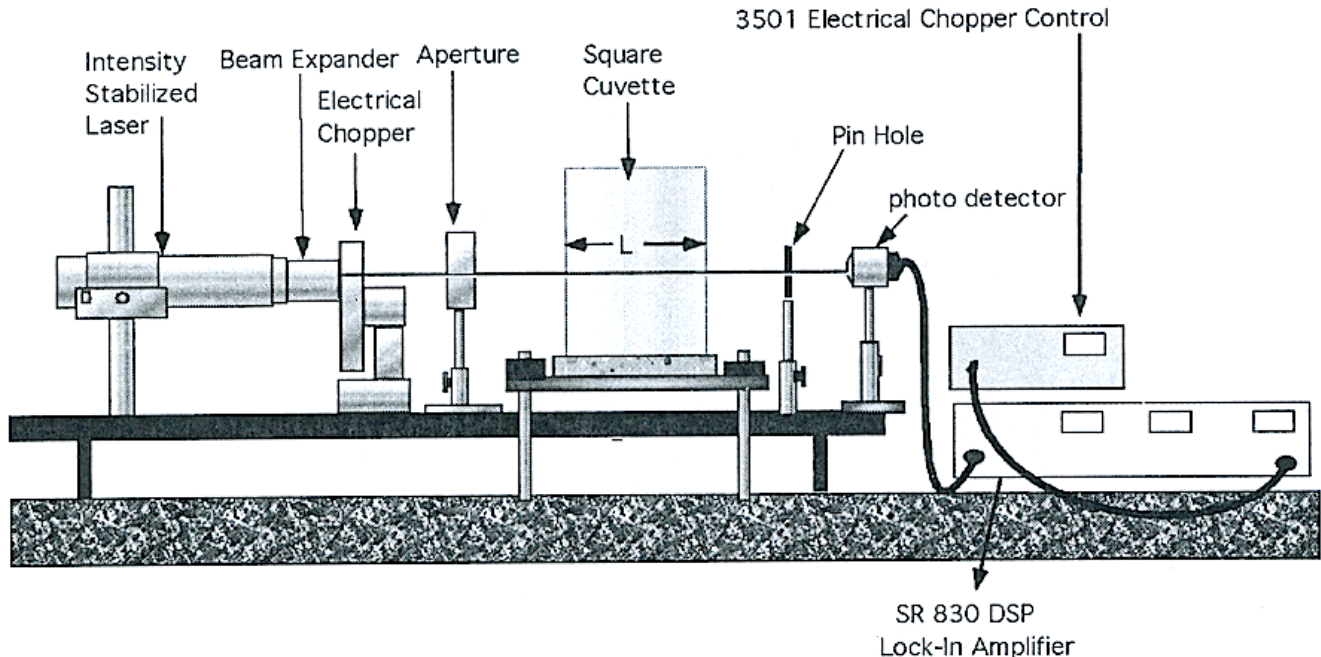


FIG. 2: A schematic of the experimental apparatus and optical arrangement.

macroscopically applying an initial intensity recorded before data was collected.

First, the beam traveled through a beam expander, which creates a uniform beam distribution over a much larger area. When focused properly the laser resulted in an Airy disk (i.e. bullseye) leaving from the expander, with backscattering directed away from the incoming stabilized laser. The beam expander's alignment can be adjusted on the scale of micrometers, so extreme care was taken to not bump the apparatus or make unnecessary modulations of the expander. The beam was chopped (i.e. periodically blocked) into an on-and-off square wave by the optical chopper, which was connected to the lock-in amplifier and serves as a reference signal so that light noise from the room can be eliminated from measurements. The beam was spatially filtered along the edges of the Airy pattern both before and after traveling through the sample cuvette by a pinhole aperture so that only the maximum, uniform intensity was incident on the photo detector.

B. Data Collection

Using a 10 mm, 3.4 mL rectangular optical cuvette, nanoscale polystyrene spheres from Duke Scientific of diameter 96 nm and Polysciences, Inc. of 107 nm were suspended into a deionized (distilled) water solution. According to online specifications the approximate concentration of the 96 nm spheres was 1% solids in a bottle,

which relates directly to a 1% weight per volume [7]. Similarly, the 107 nm had a reported value of 2.5% weight per volume [8]. Measurements of the light intensity without the cuvette in place and with only the cuvette and water in place were taken to serve as initial conditions, or I_0 . To create the initial concentration, 1.4 mL of water was placed into a cuvette, and a single drop of nanospheres was dispersed into the volume using a 2 mL disposable syringe. Each data point of transmitted intensity was recorded after an additional 0.25 mL of distilled water was added to the solution, up to 3.4 mL total in the cuvette. When the cuvette was full, 2.0 mL of the concentration in the cuvette was removed using the syringe, and the process was repeated. Three transmitted intensity data points for each level of concentration were taken since the perfect reproducibility of a specific concentration was difficult to achieve. That is, once water was added to the solution, it was very difficult to know how much of the sphere solutions should be added to return to the previous concentration.

The experiment was repeated using a 10 percent weight per weight solution of dried, non-fat, powdered milk solution. 1.2 grams of the milk powder was added to 12 grams of distilled water and was added into a 1.4 mL water solution to create an initial concentration using the syringe. Data were collected similarly to before.

IV. ANALYSIS

A. Polystyrene Sphere Characterization

In the first series of experiments 96 nm Duke Scientific and 107 nm Polysciences, Inc. polystyrene spheres were considered. Data was collected and entered into Excel for easy manipulation, then transferred into Igor Pro for plotting and fitting. The direct measurements of transmitted intensity for a given concentration of spheres can be seen in Fig. 3, where the intensity values follow an exponential increase as the concentration decreases. The transmitted intensity data is easily transformed into turbidity values seen in Fig. 4, using the conversion from Eq. 6. We used I_0 values corresponding to the intensity of the transmitted beam when the cuvette was present in the holder with just distilled water, which takes into account scattering from the glass and the water medium. For 96 nm, this was a value of $I_0 = 14.58$ nA while for the 107 nm a value of $I_0 = 11.26$ nA. Using the slope from Fig. 4 for the 107 nm, we can calculate the radius of the spheres using Eq. 19, where

$$C_{\text{sca}} = \frac{128\pi^5 a^6}{3\lambda^4} \left(\frac{n^2 - 1}{n^2 + 2} \right) = \frac{\tau}{(N/V)}, \quad (19)$$

with $\tau/(N/V)$ as the slope of the fit. Substituting our experimental values of $n = 1.2$ and $\lambda = 475 \times 10^{-5}$ nm and our slope value for 107 nm of $b = 7.76 \pm 0.08 \times 10^{-13}$ cm² into Eq. 8 results in

$$2 \cdot a = 2 \left((7.76 \times 10^{-13}) \frac{3(475 \times 10^{-5})^4}{128\pi^5} \frac{1}{0.0163} \right)^{1/6} = d, \quad (20)$$

with a final calculation of the diameter d of

$$d \approx 1.03 \times 10^{-5} \text{ cm} = 103 \text{ nm}. \quad (21)$$

A similar process follows for the 96 nm; we calculated the size of the 96 nm spheres to be approximately 123 nm, and the size of the 107 nm to be 103 nm, with errors compared to reported values of 28% and 4%, respectively. Larger error in the 96 nm solution could be a result of initial intensity shift over the data run or aggregation due to old age of the particles.

The uncertainty values seen on the plots are calculated by brute force. Uncertainty was found by taking the worst case scenario for the concentration, that is, when too much water has been added for each syringe and not enough solution withdrawn between each new syringe and visa versa. We assumed the maximum uncertainty in the volume per syringe added was 0.025 mL, where the uncertainty stacks per syringe added. This would compound with not enough solution being withdrawn, or a maximum of 1.425 mL remaining the cuvette. The uncertainty in the turbidity was calculated using propagation of error, where the uncertainty in a given transmitted intensity value varied approximately ± 0.2 nA.

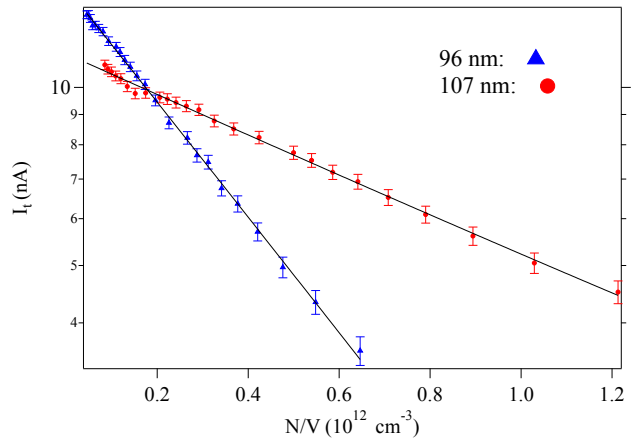


FIG. 3: Transmitted intensity data on a log scale for 96 nm and 107 nm polystyrene spheres. Each size is fit with a exponential function of the form $y = y_0 + Ae^{kx}$, where $k = 2.22 \pm 0.1 \times 10^{-12}$ cm³ for the 96 nm and $k = 8.24 \pm 0.09 \times 10^{-13}$ cm³ for the 107 nm. The initial condition A is 14.81 nA for the 96 nm and 10.99 nA for the 107 nm. In both cases y_0 has no particular significance.

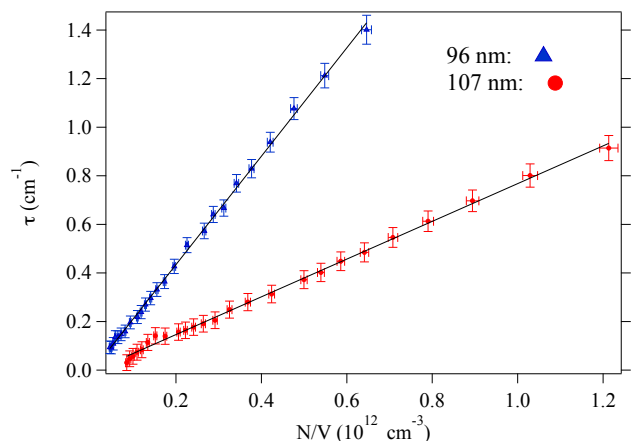


FIG. 4: Turbidity data for 96 nm and 107 nm polystyrene spheres. Each size fit with a linear function of the form $y = a + bx$, where $b = 2.23 \pm 0.02 \times 10^{-12}$ cm² for the 96 nm and $b = 7.76 \pm 0.08 \times 10^{-13}$ cm² for the 107 nm. a in both cases is within 0.01 of zero, and therefore is sufficient for omission for our purposes.

Eq. 18 is used to propagate uncertainty to turbidity values. The kink seen in both data sets in Fig. 4 below the 0.2×10^{12} cm⁻³ concentration mark is most likely from the withdrawal between syringes; fewer spheres may have been taken out than are accounted for in our concentration measurements.

Sizing of 51 nm and 30 nm spheres were also attempted in this experiment. In the case of the 30 nm spheres the spheres had long since passed their expiration date and had dried up. Attempts were made to revitalize the solution by reintroducing distilled water into the bottle.

Data was collected for the solution, but the solution had lost homogenous properties throughout the mixture, and gave unusable data. The spheres had become aggregated, or clumped together, and would drift in and out of the path of the laser, causing massive deviations in the transmitted intensity. The same problem occurred with the 51 nm solution. To successfully take data at these sizes, either rigorous and extensive mixing would be required, or the purchase of new spheres.

B. Qualitative Analysis of Milk

Data was collected for a 10% weight by weight concentration of a non-fat, dried milk and distilled water solution. The solution looked and behaved homogeneously, and created a turbid solution of similar appearance as the 96 nm and 107 nm spheres. Quantitative analysis of the size of the particles in the milk is beyond the time scope of this experiment; much more information about the mixture, including initial number of particles per volume, etc., would be required to make an estimate of the average sphere size in the mixture. Assumptions would need to be made about the index of refraction of milk, and subsequently the wavelength of the laser in the medium of milk. The data collected follows a similar trend of the 96 nm and 107 nm spheres, as seen in Fig. 5. The trend of the curve is not precisely linear however, and may be more accurately fit by a polynomial. This may be due to the polydispersity of the mixture, that is, there are particles of multiple different sizes within the milk powder solution.

Since we did not know the initial concentration of spheres in the milk, we analyzed the data by guessing a sphere size and checking to see if the calculated sphere size was self-consistent. By estimation of the ini-

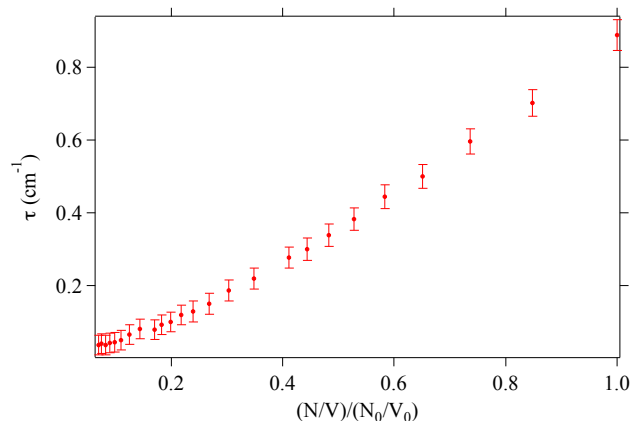


FIG. 5: Turbidity data for the powdered milk and distilled water sample. The concentration has been normalized to an arbitrary value of 1 since a value for the initial number concentration is presently unobtainable. Note that the curve is not exactly linear, and may follow a polynomial trend.

tial sphere size (and therefore the initial number concentration) we plotted the same turbidity data versus multiple different concentration values and recorded the estimated slope. By plugging the estimated slope back into Eq. 8 we could compare our calculated radius to our initial guess of radius, and find the percent difference between the two values to check for consistency. The closer the calculation of radius was to our initial guess, the stronger the estimation. A plot of the same turbidity data versus different initial concentrations (i.e. initial radius guess) can be seen in Fig. 6. Initial guesses for radii of 10 nm to 150 nm were tried, in steps of 10 nm. One can qualitatively use this method to estimate a range of possible sphere sizes in the mixture. Using the calculated slopes from Fig. 6 to calculate the diameter of the particles, we were able to compare to the initial guess of the diameter used to generate the initial concentrations for the turbidity data. The percent difference between the guess and the calculation for a given guess size can be seen in Fig. 7. As the guess approaches high values for the diameter, the guess becomes more and more attuned to the calculation based on raw data, with about 30% error at a guess of 300 nm diameter. This method has serious limitations, since the trend seems to imply the size of the spheres are well outside of the Rayleigh regime, which makes the turbidity measurements themselves meaningless when analyzed using our techniques.

Data collection was also attempted with McCormick's yellow food coloring, but similarly to the 51 nm and 30 nm spheres the data were inconclusive of any trend. Data collection for the one run attempted was completed with 1 drop of food coloring; higher concentrations may be needed to observe desired turbidity effects.

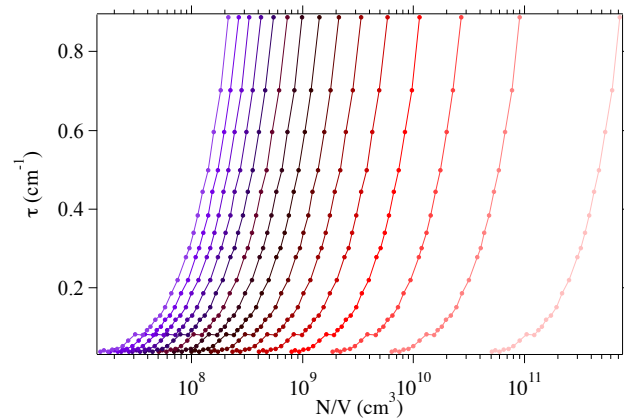


FIG. 6: The same turbidity data for the powdered milk and distilled water sample for multiple concentrations generated based off of initial sphere size guesses. The curves vary from the 20 nm diameter estimate on the far left to 300 nm on the far right in increments of 20 nm.

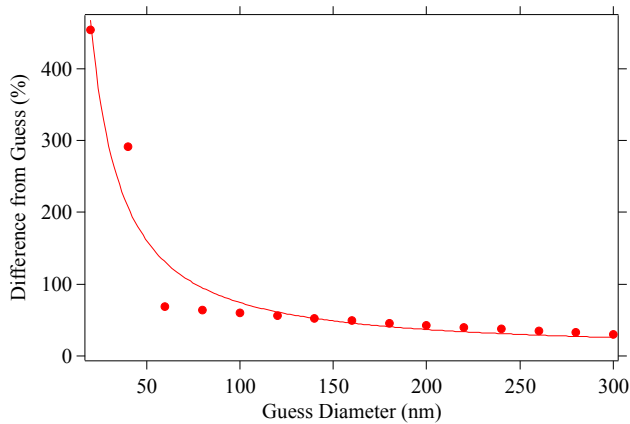


FIG. 7: Percent difference between the guess of the sphere size and the calculation of the sphere size based off the slope of the fit, for each sphere size guess. The plot was arbitrarily modeled by a power function to help guide the eye and lacks any particular significance.

V. CONCLUSION & FUTURE WORK

This experiment successfully demonstrated the ability to verify the diameter of a polystyrene sphere, given *a priori* knowledge about the particle size needed to make initial estimations on the concentration of spheres. We calculated the size of the 96 nm spheres to be approximately 123 nm, and the size of the 107 nm to be 103 nm, with errors of 28% and 4%, respectively. These errors may be a result of aggregated and aged particle solutions, or errors in initial number concentration estimations. Uncertainty in the number concentration of spheres was calculated by brute force, so in future instances a more sophisticated method may be preferable. These values were computed by comparing the slope of the turbidity and number concentration plots to a scattering coefficient determined by Rayleigh scattering theory. In future instances of this experiment, time allow-

ing, two full data runs for each sphere size should be completed to check for consistency of measurements and to avoid large over/under deviations of calculated values to reported values. In addition, new polystyrene spheres for smaller sizes of 20, 30, or 50 nm diameter should be acquired so data could be taken on spheres fitting well into the Rayleigh size regime. By acquiring new spheres from a company such as Polysciences Inc., manufacturer reported values of particles per milliliter come with the mixtures, and so more accurate concentration data may be achievable.

A qualitative analysis was completed on a powdered milk solution in distilled water. Without knowing the initial number concentration of particles in the solution it was difficult to quantitatively calculate the radius of the spheres in the milk. The turbidity plot for milk followed the same general trend as that of the polystyrene spheres, while self-consistency analysis implies the radius of the particles in the solution may be too large and outside that of the Rayleigh regime. Fittings to the turbidity plots were not precisely linear, which could imply the polydispersity of the milk solution. Future additions to this project should include a thorough literature review of the particle sizes present in milk so that an accurate estimation of the initial number concentration can be made. Once an initial concentration is established, future researchers can compare the milk solution to the polystyrene spheres directly, and if every different sphere size can be formulated into approximately the same initial number concentration the data could be acquired directly relating the sphere size to the slope of the turbidity curve.

Attempts were also made at characterizing the size of particles in yellow food coloring, but results were inconclusive. Higher starting concentrations may be needed, but as with the milk solution, more information is needed about the number concentration of particles within the liquid for a meaningful quantitative analysis.

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