

Dispersion Stability

Nicomp®

A common goal in formulating a two-phase dispersion is to maximize the stability, or shelf life, of the product. This is true for both suspensions and emulsions. Both particle size, and the surface charge (the zeta potential), are important physical characteristics that affect suspension stability. The application note explains how particle size and zeta potential measurements can be used to enhance dispersion stability.

INTRODUCTION

Suspensions (solid/liquid) and emulsions (liquid/liquid) are both examples of two-phase dispersions. The solvent is the continuous phase and the particles, or emulsions droplets, constitute the dispersed phase. In a stable dispersion, the dispersed phase maintains a consistent particle size over time and remains in suspension. Unstable emulsions can phase separate with the oil phase “creaming” to the top, or the emulsions droplets can increase with time due to aggregation, or Ostwald ripening. Unstable suspensions can flocculate/aggregate and then settle to the bottom as shown in Figure 1.

Creating stable dispersions involves controlling the chemistry and physical properties of both the continuous and dispersed phases. The chemistry of the continuous phase can be optimized by varying surfactant selection and concentration, changing the salt concentration, controlling the pH, and a combination of all these factors. The dispersed phase can be made more stable by either adding a coating of polymer to the surface (steric stabilization), by increasing the charge on the surface (electrostatic stabilization), by a combination of both.

When there is no charge on the particle surface, the particles can approach each other closely enough that there is no barrier keeping them from aggregating reducing dispersion stability. But if sufficient charge is present on the surface, the particles repel each other like magnets and never get close enough to aggregate—as shown in Figure 2.

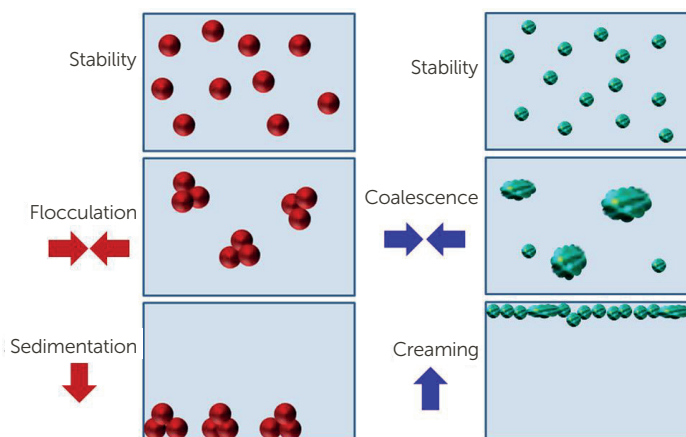


Figure 1. Stability vs. instability

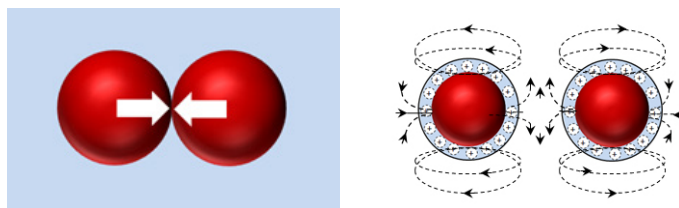


Figure 2. Attractive particles aggregate (left), repulsive particles separate (right)

ZETA POTENTIAL

From a measurement standpoint surface charge is characterized using zeta potential analysis. The zeta potential is the potential in mV a short distance from the particle, as shown in Figure 3. The measurement is performed by applying an electric field to the suspension and detecting the motion of the particles by electro-phoretic light scattering. The direction the particles move in determines the sign (+ or -) while the velocity determines the magnitude of the charge. The motion can be analyzed either by detecting the frequency shift (phase Doppler), or the phase shift (phase analysis light scattering, or PALS).

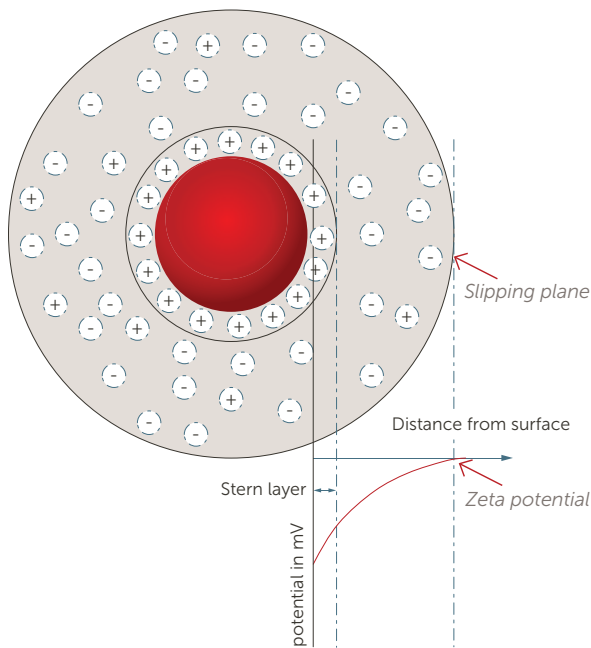


Figure 3. Zeta potential

The primary result from a zeta potential measurement is the electrophoretic mobility, μ , which is then used to calculate the zeta potential using the equation:

$$\xi = \eta\mu/\epsilon$$

Where:

- ξ = zeta potential
- η = viscosity of solvent
- μ = electrophoretic mobility
- ϵ = dielectric constant of solvent

It doesn't matter if the zeta potential is a positive or negative charge, just the absolute magnitude is important. A higher zeta potential value is an indicator of improved dispersion stability. Different kinds of dispersions require different charge values to enhance stability, as shown in Figure 4 (rough guidelines only).

SAMPLE TYPE	ZETA POTENTIAL
Metal sols	>40 mV
Metal oxides	>30 mV
Emulsions	>20 mV
Emulsions	>10 mV

Figure 4. Zeta potential guidelines for stability

Increasing stability by altering the zeta potential is typically important only for small particles—mean size under 1 μm , although for emulsions this size can be larger.

ISOELECTRIC POINT (IEP)

The isoelectric point (IEP) of a dispersion is the pH value at which the zeta potential equals zero¹. It is a common reason to make zeta potential measurements because the IEP indicates the surface chemistry conditions that may cause dispersion instability. This concept is depicted in Figure 5.

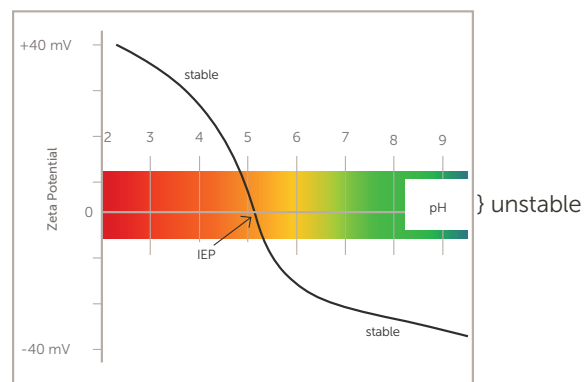


Figure 5. Isoelectric point

RESULTS: FOOD EMULSION

A pH was altered for a food emulsion by titrating with a weak acid from pH 6.8 to 3.2. Seven measurements using the PALS technique (applied electric field = 4 v/cm) were made at each pH value and the average is reported. A plot of zeta potential vs. pH is shown in Figure 6.

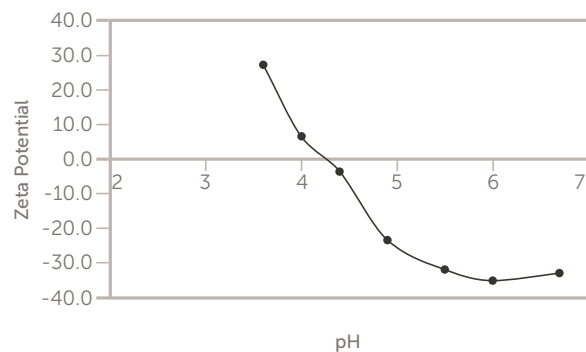


Figure 6. Zeta potential vs. pH

The size distribution for the emulsion sample at pH 6.5 is shown in Figure 7 and at pH 3.5 in Figure 8. Notice the dramatic increase in droplet size (302 – 496 nm) at the lower pH indicating that the emulsion is destabilizing.

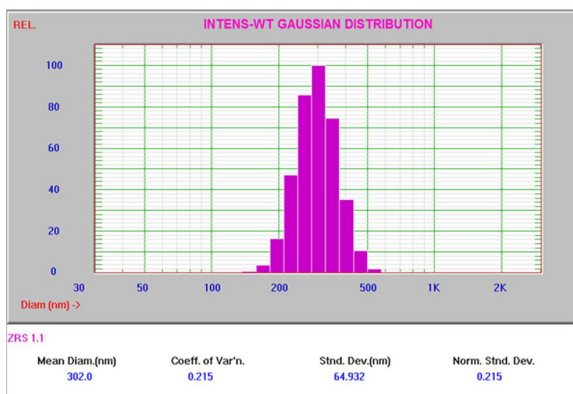


Figure 7. Particle size at pH 6.5, zeta potential = -34 mV

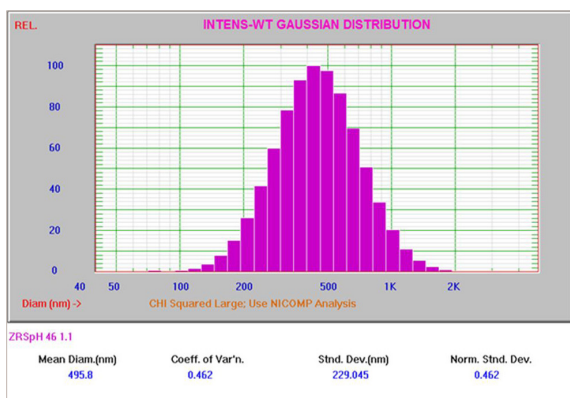


Figure 8. Particle size at pH 4.6, zeta potential = -9.8 mV

RESULTS: ZINC OXIDE SUSPENSION

A zinc oxide (ZnO) “nanopowder” was purchased (Sigma Aldrich, #544906) to study the effect of pH on zeta potential and particle size. All samples were prepared by dispersion with Igepal CA-630 non ionic surfactant and 3 minutes of ultrasound using an ultrasonic probe.² The zeta potential values for pH 6.9 and 11.2 are shown in Figures 9 and 10. Note the very stable zeta potential results even near zero.

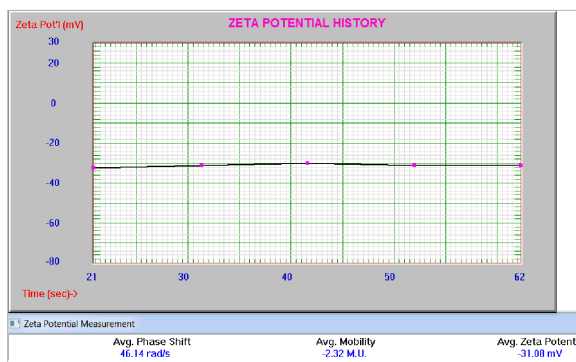


Figure 9. Zeta potential of ZnO at pH 6.9

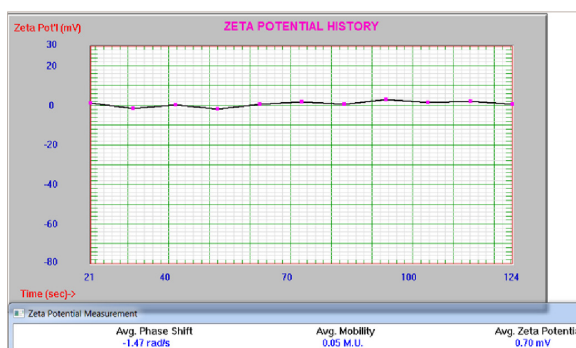


Figure 10. Zeta potential of ZnO at pH 11.2

Figure 11 shows the change in particle size from a monomodal, with peak at 198 nm at pH 6.9, to a bimodal distribution, with peaks at 204 and 573 nm at pH 11.2. As the zeta potential approached zero the suspension destabilized and began to aggregate.

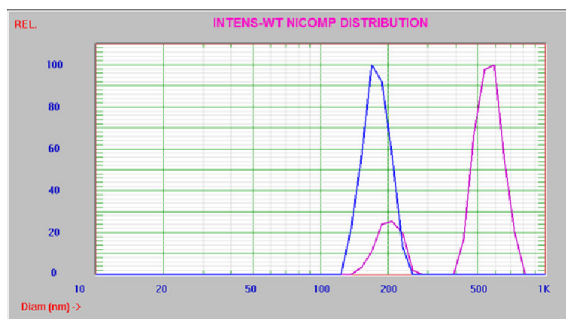


Figure 11. ZnO particle size at pH 6.9 (blue) and 11.2 (pink)

References

- ¹ Entegris Application Note, Isoelectric Point (IEP)
- ² Entegris Technical Note, DLS Sample Preparation

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