

Particle Size of Pigments for Soy Water-Based Inks

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Keywords: water-based, soy protein, particle size distribution, pigment dispersion, milling time

Abstract

The purpose of this study was to investigate the influence of particle size of pigments for Soy water-based inks on the pigment dispersion quality. The information can be used to obtain a standard laboratory condition at which a wide range of pigments may be dispersed to formulate water-based flexographic packaging inks. The influence of pigment concentration and shear forces on the pigment dispersion were also studied. Also, it is to provide basic grounds for understanding of the pigment's particle size characterization for soy water-based ink with the use of three different types of laboratory scale pigment dispersion and milling equipment used for grinding the pigments and ink formulation components to achieve the desired particle size for the flexographic packaging ink formulation. The procedure consisted of dispersing the pigment using a three different types of laboratory mills at various milling times with two different rotation speeds and measuring the particle size and particle size distribution of the milled samples. This pigment was dispersed at standard laboratory condition with different pigment concentrations.

Introduction

Most of the commercially available water-based inks are formulated and manufactured using acrylic chemistry-based resins. Excluding from being nonrenewable, they are also non-biodegradable raw materials. Besides being used as resins for water-based printing inks, they are used in various different applications, such as in prosthetic dentistry, automotive industry, medical devices, paints, storage tanks, metal buildings, rail car coatings, bridges, pipes, sealant/adhesive, paper and printing industries. Transparent coatings made of acrylics contain of about 20-25 weight % solids and do not meet the requirements of VOC regulations anymore (US EPA, 1999). Therefore, these types of materials need to be replaced by some

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other alternatives that are sustainable natural water borne polymer systems, which was the motivation behind the present research. The aim of this work is to develop soy-based pigment dispersions for water-based inks to replace petrochemical acrylic ones.

Soy proteins are obtained through the extraction of soybean oil (Zang, 2108; Chen, 2018; Wu, 2018). Soy protein is mainly used as a food ingredient, and industrially it is implemented in adhesives, asphalt additives, resins, cleaning materials, cosmetics, paints, plastics, polyesters and textile fibers (Browner, 1992; Erhan, 1995; Smith, 1996). The basic application of industrial-grade soy protein is as a binder in paper coatings (Ma, 2013). In our previous work, we used soy polymer in the let-down portion of the water-based ink (Pingale, 2019; Pekarovicova, 2019). In the current work, soy-based resin was used as a replacement for acrylic resin to grind the pigment and also as a let-down vehicle to make fully soy-based ink.

The raw materials used in ink production are pigments, binders, solvents and additives. The most obvious role of pigment is to color the ink. However, pigments can also provide gloss, abrasiveness and resistance to attack by light, heat, solvents etc. Resins are primarily binders; they bind the other ingredients of the ink together so that they form a film and bind the ink to the substrate. They also contribute to such a property as gloss and resistance to heat, chemicals and water.

The Pigment Particle Size:

At the most basic level, we can define a particle as being a discrete sub-portion of a substance with physical dimensions ranging from sub-nanometer to several millimeters in size. Measuring the pigment particle size while doing the ink formulation is so important in order to better control product quality, which delivers real economic benefits and achieve the highest printability results (Thompson, 1998).

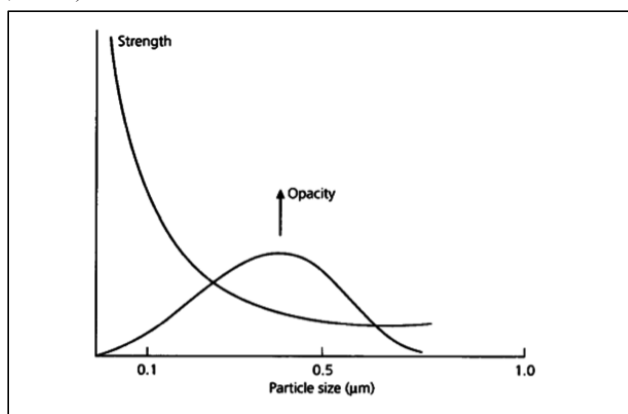


Figure 1: Dependence of Color Strength and Opacity of Pigments on Particle Size (Thompson, 1998)

In addition to controlling product quality, a better understanding of how particle properties affect product, ingredients and processes in terms to improve product performance and optimize the efficiency of ink manufacturing processes. In addition to the ink formulation ingredients, the behavior of particulate materials is often dominated by the physical properties of the constituent particles such as pigment particles. From the manufacturing perspective some of the most important physical properties needed to be measured are particle size, particle shape, surface properties, mechanical properties, charge properties and microstructures (Malvern, 2015).

Technically, pigment particles are 3-dimensional objects and unless they are perfect spheres e.g., emulsions or bubbles, they can't be fully described by a single dimension such as radius or diameter. In order to simplify the measurement process, it is often convenient to define the particle size using the concept of equivalent spheres. In this case the particle size is defined by the diameter of an equivalent sphere having the same property as the actual particle such as volume or mass. For example, it is important to realize that different measurement techniques use different equivalent sphere models and therefore it will not necessarily give the same exact results for particle diameter.

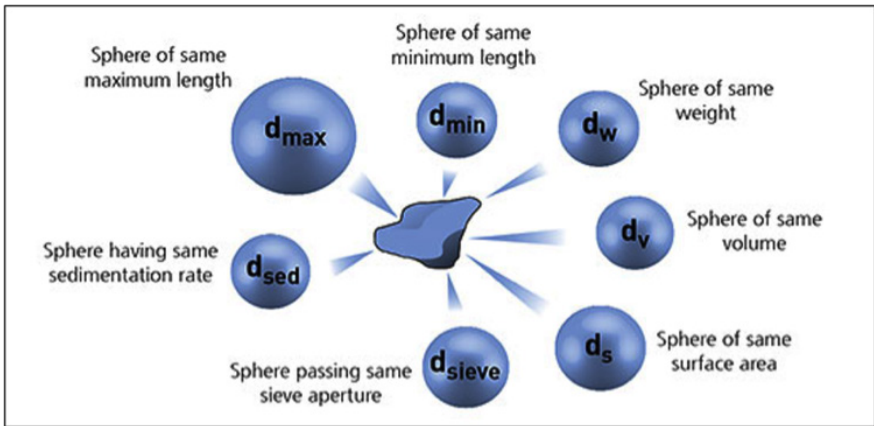


Figure 2: Illustration of the concept of equivalent spheres for regular shape particles (Malvern, 2015)

The equivalent sphere concept works very well for regular shaped particles. However, it may not always be appropriate for irregular shaped particles.

Particle Size Distributions:

Unless the pigment particle sample is perfectly mono disperse that is, every single particle has exactly the same dimensions, it will consist of statistical distribution of particles of different sizes. It is also common practice to represent this distribution in the form of a frequency distribution curve, or cumulative (undersize) distribution curve. A particle size distribution can be represented in different ways with respect to the weighting of individual particles. The weighting mechanism will depend

upon three different measuring principle being used such as: Number, Volume and Intensity weighted distributions.

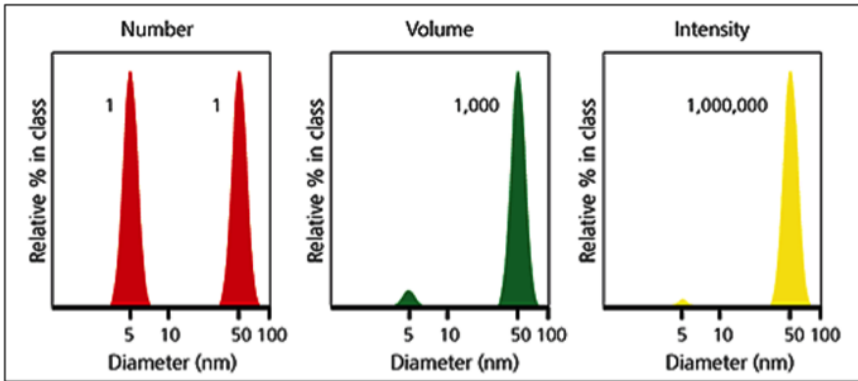


Figure 3: Example of number, volume and intensity weighted particle size distribution for the same sample (Malvern, 2015)

It is possible to convert particle size data from one type of distribution to another, however this requires certain assumptions about the form of the particle and its physical properties. One should not necessarily expect, for example a volume weighted particle size distribution measured using image analysis to agree exactly with a particle size distribution measured by laser diffraction.

Distribution Statistics:

In order to simplify the interpretation of pigment particle size distribution data, a range of statistical parameters can be calculated and reported. The choice of the most appropriate statistical parameter for any given sample will depend upon how that data will be used and what they will be compared with. For example, if we can report the most common pigment particle size from our ink formulation samples that could be choose between the following parameters:

- Mean - 'average' size of population.
- Median - size where 50% of the population is below/above.
- Mode - size with highest frequency.

If the shape of the particle size distribution is asymmetric, as is often the case in many pigment particle dispersion samples, we would not expect these three values to be exactly equivalent, as illustrated below (Malvern, 2015).

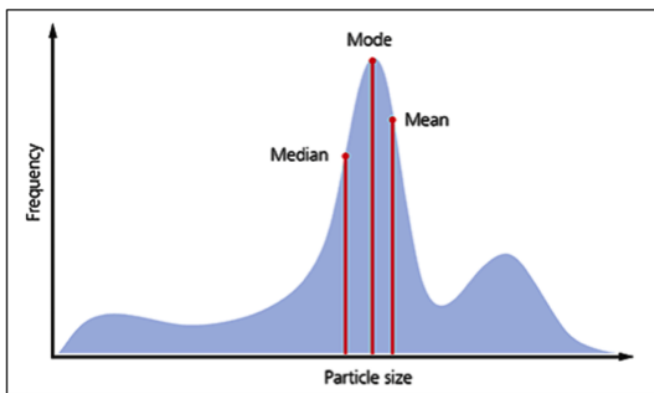


Figure 4: Illustration of the Median, Mode and Mean for a Pigment Particle Size Distribution (Malvern, 2015)

Experimental

Soy Water-Based Flexographic Packaging Ink Formulation:

Formulating a flexographic soy water-based packaging printing ink formulation is a complex process to satisfy the requirements of printing ink. Printing ink formulation is a combination of the basic ingredients such as pigments, vehicles and additives.

The physical properties of pigments, such as particle size and particle surface are of great influence in the dispersion process. Primary dispersion for ink production demands that the pigment particles are thoroughly wetted by the liquid phase. The pigment particle size strongly affects the color strength since the smaller particle size has the higher surface area and thus the stronger the color. If this dispersion level is not achieved, printing problems will arise. To achieve the optimum benefits of pigment, it is necessary to obtain as full a reduction as possible to the primary particle size.

In this ink formulation process, we have completed the two-step mixing/dispersing process using a high shear mixer to produce ink with acceptable dispersion. In the first stage of mixing varnish (Soy Protein Resin) has been made, and then pigments are mixed into it.

Step 1: Soy Water-Based Varnish Formulation:

Soy based varnish is a clear liquid that solidifies as a thin film. It binds the pigment to the printed surface, provides the printability of the ink, and wets the pigment particles. In this experiment we have used the Soy Protein Resins to make the varnish (which incorporates water and NH_4OH). This soy protein resin varnish was cooked at 65°C to 70°C and much more rigorous conditions were employed. The small portion of NH_4OH was used to control the pH of this soy protein resin varnish.

In the production of a typical soy protein resin varnish, first we add the deionized water with soy protein resin powder, and later on NH_4OH and other foaming and buffering agents were added to the vessel. After cooking more hard resins were added when the correct temperature was attained. The cooking process continued until the reactants were either totally consumed in the cooking process or achieve adequate solubility in the solvent. Finally, the varnish mixture was reheated to obtain targeted rheological properties. During this varnish production pH and viscosity, were closely monitored and tested before proceeding to mixing/dispersing or milling with pigments.

Step 2: Pigment Dispersion:

The primary purpose of the dispersion process is to break down pigment aggregates and agglomerates to their optimum pigmentary particulate size and distributes these pigment particles evenly throughout the similar medium, i.e., the carrier. Once the varnish (containing the soy protein resin) was produced, the pigment was mixed into it. At this point, the pigment particles clumped together. These clumps must be broken up and the pigment dispersed evenly through the resin with the use of the following three different types of laboratory scale pigment grinding/dispersing mills.

With the use of Speed Demon Paint Mixer or Red Devil Paint Shaker – these two grinding mixers have fixed speed, but time cycles are different. Also, we have used a third equipment, which is a Three Roll Mill – with variable speed control for milling the ink. Fine grinding was done with an ultrasonic probe Vortex-Genie 2 vortex mixer. Pigment dispersions were formulated with soy polymer with the aid of several different surfactants and wetting agents. Soy protein resin varnish made with APS Grupa's ProSoy 7475 Soy powder was used for grinding the pigment with Clariant's Hansa Brill Yellow 5GX 03 pigment. Then, the pigment particle size was measured using Nicomp 380 laser scattering particle sizing instrument.

Pigment Dispersing/Mixing Process, and Equipment's used for Soy Water-Based Ink Formulation:

A three-roll mill consists of a series of cambered rollers rotating in opposite directions (Fig.5). The pigment particles are fed into a hopper above the two rear-most rollers and are dispersed by the shear forces between the rollers. A doctor blade is fitted to the front roller to remove the dispersed product (Torrey Hills, 2017).

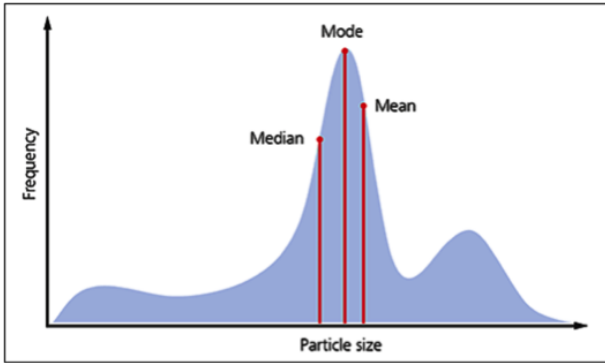


Figure 5: Pigment Dispersing/Milling Process (Exakt, 2021)

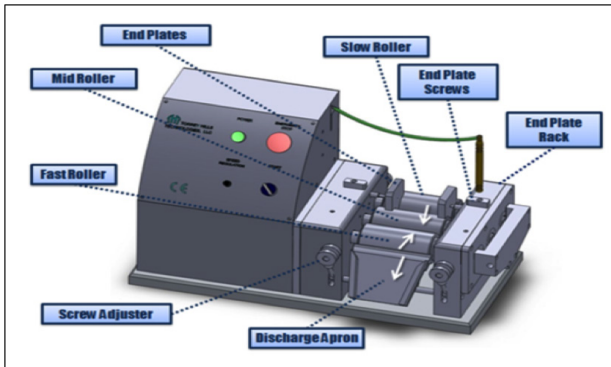


Figure 6: Three-Roll Mill Structure (Torrey Hills, 2017)

The Speed Demon Mixer has a 1-gallon capacity for mixing the pigments and varnish ink ingredients together. It also offers the increased dispersion rate and reduced noise level, while dispersing the materials together. Also, the Speed Demon’s stackable technology offers the easy-to-use and quite operations to entire mixing process.



Figure 7: Speed Demon Mixer (Radia, 2017)

The Red Devil Paint Shaker Mixer offers a similar mixing solution that is capable of handling a broad range of materials for applications in various types of lab settings.

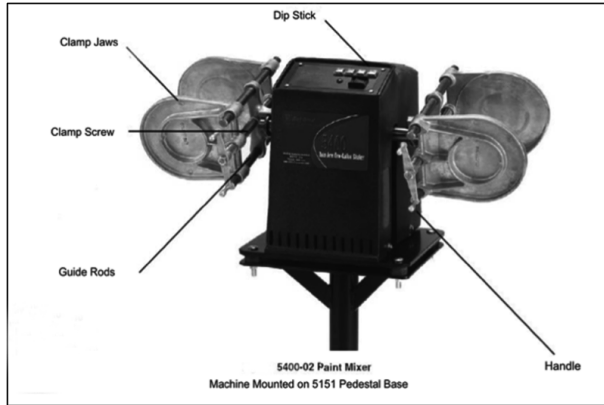


Figure 8: Red Devil Paint Shaker (Radia, 2017)

Analytical

Step 3: Pigment Particle Size Measurement

The Particle Sizer Submicron 380 NICOMP analyzer based on DLS (Dynamic Light Scattering) principle was employed in the measurement of this soy water-based pigment ink particle size (Figure 8).

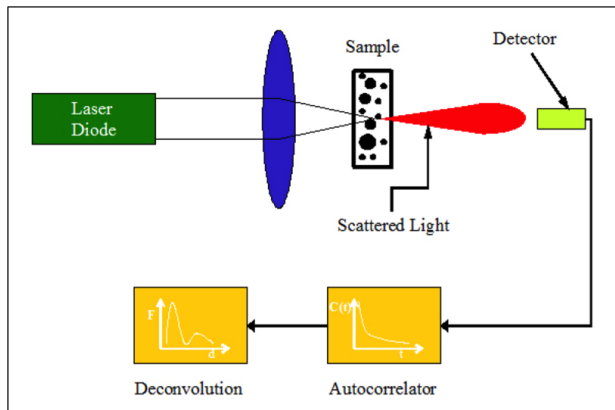


Figure 9: Simplified Block Diagram – NICOMP DLS Instrument (Particle Sizing Systems, 2006)

The operating principle of dynamic light scattering is illustrated in Figure 8. Incident light from the laser is focused into a glass tube or cuvette containing a diluted suspension of particles. The temperature of this scattering cell is held constant, for reasons, which will soon become apparent. Each of the particles illuminated by the laser beam scatters light in all directions. The intensity of light scattered by a single isolated particle depends on its molecular weight and overall size and shape, and also difference in diffractive indices of the particle and the surrounding solvent.

The incident light wave can be thought of as consisting of a very rapidly oscillating electric and magnetic fields, of amplitudes E_0 and B_0 . The arrival of this alternating fields in the vicinity of a particle causes all of the electrons, which are free to be influenced (polarizable electrons) to oscillate at the same frequency. These oscillating electrons give rise to a new oscillating electric field, which radiates in all directions - the scattered light wave. The quantity of interest in a scattering measurement is the intensity of the scattered wave, rather than amplitudes. The intensity is proportional to the square of the amplitude.

The dependence of the scattered light intensity I_s on the molecular weight (MW) or volume (V) of the particle is particularly simple when the particle diameter is much smaller than the laser wavelength λ (Rayleigh region). In this case, all of polarizable electrons within a particle oscillate together in phase, because at any given time they all experience the same incident electric field. Hence, the scattered wave amplitude is simply proportional to the number of polarizable electrons, times the incident wave amplitude.

The former quantity is essentially proportional to the overall molecular weight of the particle or its volume. The constants of proportionality, which connect these various physical quantities, depend on the indices of refraction of the particle n_p and solvent n_s . That is, how well a given particle scatters light depends not only on MW or V but also on the polarizability of the particle (related to n_p) relative to that of the solvent (related to n_s). For the very small particles in the Rayleigh region, we arrive at simple expressions for the scattered intensity I_s :

$$I_s = f(n_p, n_s) * (MW)^2 * I_0 \quad (1)$$

$$I_s = g(n_p, n_s) * V^2 * I_0 \quad (2)$$

Where I_0 is the incident laser intensity, $f(n_p, n_s)$ and $g(n_p, n_s)$ are functions of the indices of refraction of the particle and solvent, which are fixed for a given system composition. For these small particles in the Rayleigh region, here is negligible angular dependence in the scattered intensity I_s (Malvern, 2015). After setting up the appropriate conditions, for water-based inks, the particle size of each sample was measured. The following conditions were set for the measurements:

- *Refractive Index:* 1.333 (Water)
This establishes the index of refraction of the solvent, in which the particles are suspended, assuming a dilute dispersion. The ink particle sizes were measured in this case using water as solvent.

- *Viscosity: 1.002 cP (Water)*
The viscosity of the sample suspension is expressed in units of centipoises (cP). The particle suspension must be very dilute for measurements based on dynamic light scattering, in order to avoid errors due to interparticle interactions and multiple scattering. Therefore, the viscosity is by default as a viscosity of the pure solvent in which the sample particles are suspended.
- *Intensity: 200-300 kHz.*
The average scattered intensity or photo pulse rate, expressed in kHz, which is desired for a measurement, can be established by setting this parameter. The default value was set at 200-300 kHz. This value is typically recommended for most samples, which scatter adequately. It is designed to optimize the efficiency of the autocorrelation process and thereby minimize the time needed to obtain reliable, accurate results for most samples.
- *Temperature: 20°C.*
After measurements, the results obtained from NICOMP Particle Sizing Systems - CW380 Software.

Ingredient		Amount (%)	Amount (grams)	Purpose
Pigment	Hansa Brill Yellow	35	210	Colorant
Vehicle	Soy Polymer Resin (ProSoy 7475)	25.7	154.2	Dispersing Agent
	Surfactant (Surfynol 104 H)	5	30	Wetting Agent
	Antifoam	0.5	3	Antifoam Agent
	Water	33.8	202.8	Diluent

Table 1: Soy Water-Based Flexographic Packaging Ink Pigment Dispersion Formulation

Dispersion Equipment	Shear Force Classification	Premix Requirement	Dispersion Time
Red Devil Paint Shaker	High shear	Required	60 Minutes
Red Devil Paint Shaker	High shear	Required	120 Minutes
Speed Demon Mixer	High shear	Required	60 Minutes
Speed Demon Mixer	High shear	Required	120 Minutes
Three-Roll Mill	High shear	Required	60 Minutes
Three-Roll Mill	High shear	Required	120 Minutes

Table 2: List of Pigment Dispersion Equipment's used and their Mechanical Properties

Results and Discussion

The pigment was dispersed/milled under standardized laboratory conditions at three different types of equipment. Particle size and particle distribution were measured by using NICOMP 380 DLS particle sizing systems submicron analyzer.

Table 1 shows the ingredients of soy water-based flexographic packaging ink pigment dispersion formulation. For this ink formulation, we have used the organic pigments and soy protein resin as vehicles for the purpose of dispersion and Surfactant (Surfynol 104 H) was used as wetting agent with water as diluent.

Table 2 shows that, the list of pigment dispersion/mixing equipment's and their mechanical properties. There are three different types of dispersion equipment used such as Red Devil and Speed Demon Mixers constant speed mixing equipment and Three Roll Mill as a variable speed pigment dispersion/mixing equipment. All these three equipment rely on high shear force mixing and multi pass dispersion time used for each at 60 and 120 minutes respectively.

Dispersion Equipment	Pigment Grinding Time (Minutes)	Pigment Particle Size (nm) – Peak I	Intensity (%) of Mixture – Peak I	Pigment Particle Size (nm) – Peak II	Intensity (%) of Mixture – Peak II
Red Devil Mixer	60 Minutes	61.3	42.6	912.3	57.4
Red Devil Mixer	120 Minutes	175.1	46.2	897.8	53.8
Speed Demon Mixer	60 Minutes	151.1	51.2	682.2	48.1
Speed Demon Mixer	120 Minutes	388.3	51.3	899.9	48.4
Three-Roll Mill	60 Minutes	128.0	25.7	466.2	74.3
Three-Roll Mill	120 Minutes	135.8	34.8	347.5	65.1

Table 3: Pigment Grinding Results with Equipment, Time and Particle Size Parameters (Mean Diameter)

Dispersion Equipment		Red Devil	Red Devil	Speed Demon	Speed Demon	Three-Roll Mill	Three-Roll Mill
Milling Time (Minutes)		60.00	120.00	60.00	120.00	60.00	120.00
Peak I	Mean Diameter (nm)	61.3	175.1	151.1	388.3	128.0	135.8
	Std. Deviation (nm)	6.2	19.5	17.4	41.7	13.7	14.0
	Std. Deviation (%)	10.1	11.2	11.5	10.7	10.7	10.3
	Intensity (%)	42.6	46.2	51.2	51.3	25.7	34.8
Peak II	Mean Diameter (nm)	912.3	897.8	682.2	899.9	466.2	347.5
	Std. Deviation (nm)	79.6	58.0	91.4	57.2	52.6	28.7
	Std. Deviation (%)	8.7	6.5	13.4	6.6	11.3	8.7
	Intensity (%)	57.4	53.8	48.1	48.4	74.3	65.1

Table 4: INTENSITY-Weighted NICOMP DISTRIBUTION Analysis (Solid Pigment Particle)

The Gaussian Chi squared value is very high ranging from 7.13 to 37.86 and therefore the multimodal distribution is appropriate. Hence, based on the NICOMP distribution analysis, a comparison of particle size from Table 3 and Figure 10 shows the intensity mean diameter of the pigment Hansa Brill Yellow decreasing with variable speed milling equipment such as Three Roll Mill used for pigment grinding/dispersion.

The experiment allowed a particle size reduction from 912.30 nm to 466.20 nm at variable speed dispersing equipment at first 60 minutes of grinding time after 2nd pass of 120 minutes of grinding time it is resulted from 897.80 nm to 347.50 nm. A comparison of particle size shows larger reduction at variable speed grinding/milling equipment with increasing milling time. The lesser decrease in particle size for the constant speed mixing equipment such as Red Devil and Speed Demon Mixer's. These milled samples probably result from rheological properties of the dispersion. This needs to be confirmed with rheological measurements.

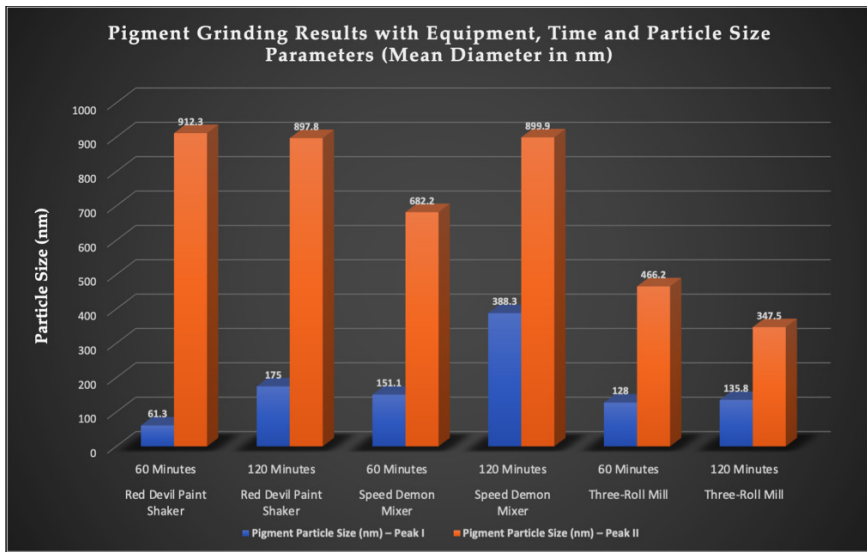


Figure 10: Pigment Grinding Results with Equipment, Time and Particle Size Parameters (Mean Diameter in nm)

From the Tables 3 and 4 and following Figure 11 shows the Intensity-Weighted NICOMP Distribution analysis for solid pigment particles. In these tables it is clearly shown as the different types of dispersion equipment used with two different milling times such as 60 minutes and 120 minutes for each type of equipment respectively. Also, from the data obtained from NICOMP Particle Sizer Analysis software there are Peak 1 and Peak II values recorded and mean diameter values are varying from 61.3 nm to 388.3 nm for the intensity lies in 42.3% to 51.3% for Peak I readings and for the Peak II mean diameter values resulted as 347.5 nm to 912.3 nm and intensity values are lies in 48.8% to 74.3%. This shows the particle size distribution narrowing with an increase in milling time.

In-addition to that, from the Table 4 data for Peak I and Peak II values for standard deviation in (nm) and standard deviation in (%) were recorded from Intensity-Weighted NICOMP Distribution analysis for solid pigment particles. In this statistical data analysis, the standard deviation is a measure of the amount of variation or dispersion of a set of values. A low standard deviation indicates that the values tend to be close to the mean of the set, while a high standard deviation indicates that the values are spread out over a wider range.

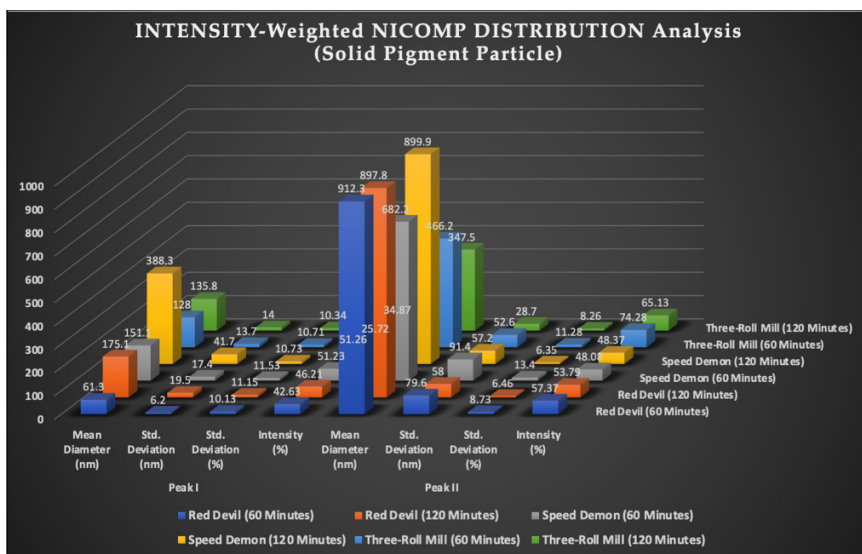


Figure 11: INTENSITY-Weighted NICOMP DISTRIBUTION Analysis (Solid Pigment Particle)

A dramatic change in the dispersibility of a given water-based soy ink formulation and solid pigment particle dispersion size, it is observed that the particle size decreases in Peak 1 and Peak 2 readings with the use of variable speed dispersion/milling equipment such as Three-Roll Mill. This is clearly shown in the above Figure 10 and 11, where we use the high shear variable speed dispersion/milling equipment helps to achieve the target values of pigment particles. In this whole experiment, it is clearly noted that in Peak 1 and Peak II the largest particle size reduced from 912.3 nm to 61.3 nm, whereas the intensity of mixtures in Peak 1 and Peak 2 varies from 25.7% to 74.3 % respectively.

The particle size of the pigment is one factor that influences the rheological properties of a given pigment dispersion. Also, we can say that based on above experimental result and findings, the high shear dispersion/milling equipment helps to decreasing the size of the pigment will led to an increase in the strength of any particle-particle interactions. As well as increasing the likelihood of agglomeration with the use of Three-Roll Mill is the best method was found for this Flexographic packaging soy water-based ink formulation and pigment dispersion process.

Conclusion

With the use of three different types of dispersing/mixing equipment for pigment dispersion in soy water-based ink formulation allowed the substantial amount of particle size reduction in various stages of ink milling operations. Regardless of speed, there is significant reduction in particle size after the use of three different types of dispersing/mixing equipment for ink milling. A Three Roll Mill used at variable speed for pigment dispersion seems to achieve the targeted pigment

particle size. It is observed that, this is the most effective equipment helped to achieve the ideal pigment particle size 347.50 nm for Hansa Brill Yellow pigment.

From both the tables and figures it is shown that there is a significant reduction in particle size after the use of variable speed dispersion equipment such as Three Roll Mill for 120 minutes of grinding time at variable speed, since that the particle size is reduced at every pass. In this experiment, a comparison of particle size results at standardized laboratory condition from Table 4 and Figure 11 shows the intensity mean diameter of the Hansa Brill Yellow pigment increasing with pigment concentration as agglomerates can be formed by collisions between particles, and as the probability of such collisions is proportional to the square of the particle dispersion methods or equipment's used for milling the ink. Also, the mechanical properties of the high shear pigment dispersion equipment are substantially affecting to achieve the ideal particle size due to their variable speed configuration properties.

In summary, we can say that the impact of particle size on pigment properties is both complex and far reaching. Particle size optimization and the control of any undesirable effects of selecting a given particle size are key steps in the development of effective competitive products specifically in the ink formulation context. Fully understanding the influence of particle size on the rheological and optical properties and the major impact from mechanical properties of dispersing/mixing equipment used for milling the ink pigment dispersion allows optimization to be carried out in a scientific and systematic manner.

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