

## Particle Size and Shape Analysis of Powder Coatings

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### Introduction

Today, powder coating is largely preferred than liquid coating and widely applied in various industries because of its high solid content, negligible VOC (volatile organic compound) emission and low energy consumption. There are multiple factors that would affect the performance of powder coatings, such as the molecular weight distribution of the resin particles, surface modifications of different types of fillers, and purity and chemical properties of the ingredients.

Besides these factors, the particle size and shape of the powder are also key parameters that contribute to the determination of powder coating's final mechanical properties. In particular, the gloss, flowability, utilization rate, and adhesion properties of powder coating will be altered by changing the powder's size and shape. Therefore, it is crucial to control the particle's size and shape in optimal ranges throughout the whole production process in order to maximize the powder coating's final mechanical performances.

This application note will focus on key production processes of powder coating and explore how these processes affect the size and shape of powder particles and eventually the final product performance.

### 1. The Melting and Mixing of Fillers and Resin

Being the first step of the powder coating production process, the melting and mixing of fillers and resin is the foundation of the coating's mechanical properties. One of the biggest challenges in this step is to make a uniformly dispersed mixture of fillers and resin, which greatly depends on the particle size distribution. To investigate the relationship between the particle size and the mixing

quality, two types of titanium dioxide applied as fillers are characterized.

As can be seen from Figure 1, Titanium Dioxide B's particle size was significantly smaller than that of Titanium Dioxide A, and thus should be, theoretically, more uniformly mixed than Titanium Dioxide A. However, in practice, it was more difficult to uniformly mix B compared to A. To resolve the reason for this discrepancy, the Bettersizer 2600 laser particle size analyzer (Bettersize Instruments Ltd.) was utilized to measure the particle size distributions of Titanium Dioxide A and B powders. And results were obtained as follows:

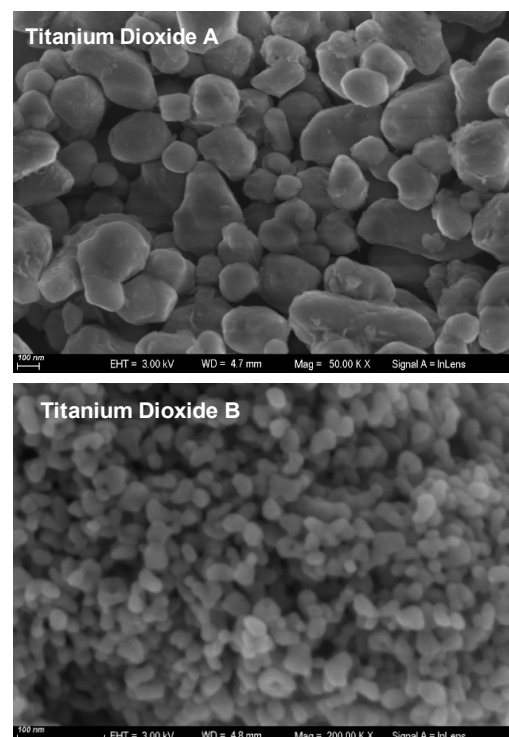


Figure 1. SEM (Scanning Electron Microscopy) images of Titanium Dioxide A (x 50K) and Titanium Dioxide B (x 200K)

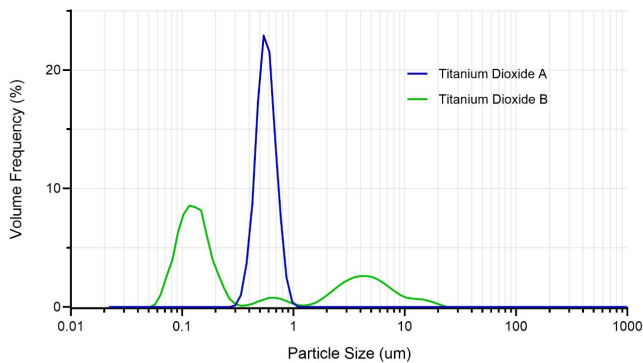


Figure 2. Particle size distributions of Titanium Dioxide A and Titanium Dioxide B

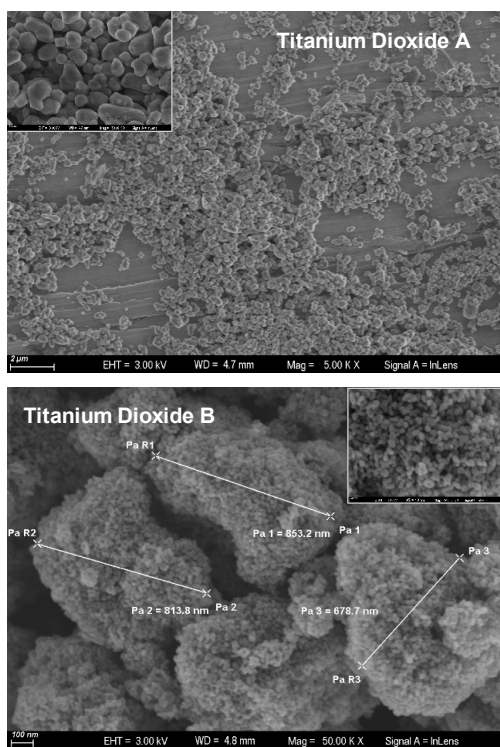


Figure 3. SEM images of Titanium Dioxide A (x5K) and Titanium Dioxide B (x50K)

The particle size distribution of Titanium Dioxide B suggested that there were large micro-sized particles present in the sample, and the sample size was wide-distributed. Moreover, by observing the samples under lower magnification SEM (Figure 3), it was found that small particles in Titanium Dioxide B formed larger aggregates, by increasing the surface area and intermolecular interactions between the particles. Therefore, in this case, fillers with smaller particle sizes had larger aggregates and thus were harder to be mixed.

After sizing two types of titanium dioxide powders with the Bettersizer 2600, we arrive at two conclusions: on one hand, if the fillers are oversized, the large particles will form agglomerates in the paint during extrusion and

decrease the product's mechanical properties; on the other hand, the interface energy increases as particle size decreases, resulting in larger aggregates that are harder to be uniformly mixed, which also affects the product's mechanical properties.

## 2. Grinding and Classifying Powder Coatings

Followed by the melting and mixing of fillers and resin, the next production process is the classification of powder crushing. Powder coating's size is greatly influenced by set parameters during the crushing process, for example, different types and speeds of the mills, speeds of feeding, airflow condition, and duration of crushing. All these parameters have to be considered comprehensively such that the maximum particle size of the powder can be controlled, and the narrow particle size distribution can be achieved. The figures below show the difference in mass fractions of samples when different production processes (A and B) are applied during the powder crushing:

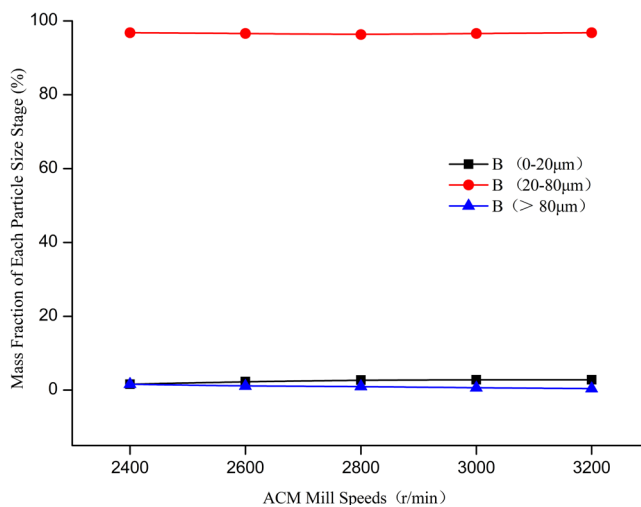
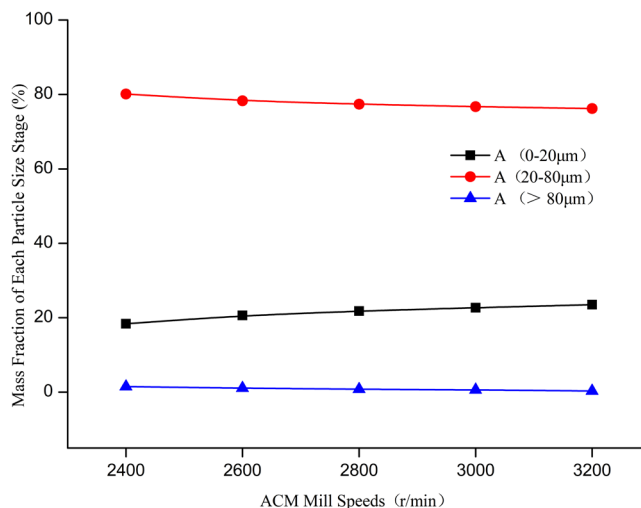


Figure 4. Size of coating samples manufactured by Process A and B

In Process B, there is an additional secondary classification while Process A does not. As the figure indicates, the secondary classification effectively decreases the percentage of coarse particles ( $>80\mu\text{m}$ ) as well as small particles ( $<20\mu\text{m}$ ). In other words, the particle size distribution has been much narrower than that without the secondary classification system.

On the other hand, within the same process (A or B), when the mill speed increases, the percentage of particles greater than  $80\mu\text{m}$  decreases while that of particles greater than  $20\mu\text{m}$  increases. It should be noted that the energy consumption required for the operation of mills with higher speed also increases, which might cause undesirable overheating.

### 3. Performance of Final Product of Powder Coating

The particle size affects the efficiency and the mechanical properties of powder coating. During the powder spraying process, smaller particles will move in the airflow path, and eventually fall and stay at the higher position of the workpiece; larger particles will fall at the bottom of the workpiece, resulting in lower outflow quality.

However, undersized particles lower the spraying efficiency due to the decrease in feeding speeds. Undersized particles also accelerate the absorption of moisture that further leads to the aggregations of bigger particles which clog the spraying gun up. Hence, by measuring and monitoring the particle size distribution of powder coating, its mechanical properties and performance can be guaranteed and maximized.

#### Particle Sizing of Powder Coating Particles

As mentioned above, the particle size is a very important property for powder coatings. As we know, there are many particle sizing technologies available, from the traditional sieving and microscopy to the widely used laser diffraction. Each technology has its own advantages and disadvantages. For example, the principle of the sieving method is simple and the cost is low. But the operation is time-consuming and the tests repeatability is not satisfying. The microscopic imaging can visualize each particle, and the size and morphological parameters can be given, but the sampling may not be representative. The laser diffraction technology has rapid measurement speed, good statistics and representativeness, but the laser analyzer is

relatively expensive. The main measurement methods are discussed below.

#### a) Laser Diffraction Method

The laser diffraction method usually has two dispersion methods, namely dry test and wet test. The dry test mainly uses air as a dispersion medium, transfers particles through compressed air, and disperses powder particles through shearing, particle-particle collision and particle-wall collision. The dry test has advantages including fast operation, ease of use, and fewer factors affecting the test results. Therefore, it is widely used in particle size measurements of powder coating.

In the dry test measurements, the compressed air pressure is very important. If the air pressure is too low, the sample dispersion may be unstable and result in poor repeatability of results. However, if the dispersion pressure is too high, some secondary particles (agglomerates) may be broken into unwanted small particles. Consequently, for dry tests, it is necessary to perform a pressure titration in advance, that is, to monitor the change and stability of the particle size distributions of powder coatings under different pressures, such that an appropriate dispersion pressure can be determined.

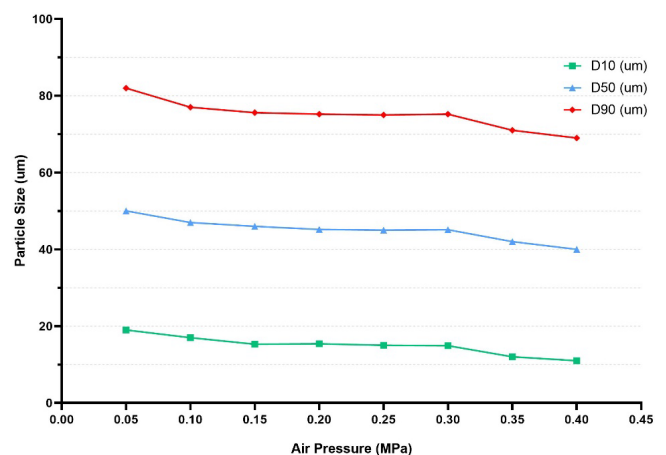


Figure 5. Example of a pressure titration curve by dry test

Especially for agglomerated or fragile samples, the pressure titration of the dry measurement is especially important. An example of a pressure titration curve is shown in Figure 5. When the pressure is less than  $0.15\text{MPa}$ , the sample dispersion is unstable and therefore the repeatability of test results is poor under this pressure. In other words, If the air pressure is too low, it is difficult to disperse some particles thoroughly.

However, when the pressure is greater than  $0.3\text{MPa}$ ,

the particle size will gradually decrease as the pressure becomes larger. That is because some aggregated particles, the fractions that are considered very important for a powder coating, may be broken into individual particles under high pressure. Thus, 0.25MPa is an appropriate dispersion pressure for measuring this sample.

It should be noted that for most powder coating samples, the particle size measurement results are not readily influenced by the dispersion pressure (0.1 - 0.4 MPa) due to their good flowability, less agglomeration and non-fragility. The measurement risk caused by inappropriate pressure is low and therefore the dry test is usually the best and most convenient choice for powder coatings.

Figure 6 shows the particle size distributions of different polyurethane coatings provided by dry tests. It can be seen from Table 1 that the D10 of the three coating samples are very close, which means that their fine-end particles are similar in size, but the coarse-end particles (D90) have a completely different particle size distribution. This also proves that the dry dispersion method is effective in measuring powder coating samples. In addition to a high resolution for large particles, the dry measurement can effectively monitor the particle size distribution of coatings during the production process, ensure the stability of product performance, and improve product quality.

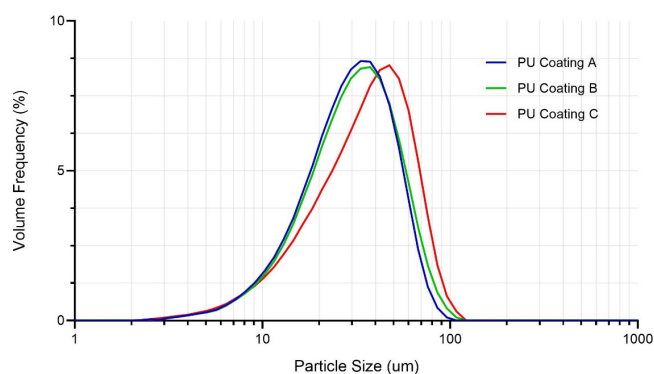


Figure 6. Particle size distributions of polyurethane (PU) coatings from different manufacturers measured by dry tests

Table 1. Critical particle size values of PU coatings from different manufacturers measured by dry tests

Sample	D10 (μm)	D50 (μm)	D90 (μm)
PU Coating A	12.34	28.19	50.87
PU Coating B	12.67	29.28	54.59
PU Coating C	12.72	34.13	62.24

Although the dry method has advantages including fast operation, high efficiency, and excellent resolution of large particles, it has disadvantages unsurprisingly. For example, because it mainly relies on compressed air to disperse particles, fine particles (particles below 1μm) may not be dispersed completely by air, especially when there is a significant amount of them. Under this circumstance, the wet test will be a better choice as it has a higher resolution for fine particles. Since the wet method uses water or organic solvents as the dispersion medium, the strong dispersion and measuring of ultrafine particles can be achieved by adding surfactants, ultrasonication, stirring and other means. As shown in Figure 7, two epoxy ester coatings with slightly different proportions of fine particles were successfully distinguished, by wet dispersion method, thereby verifying wet test's excellent resolution for fine particles of powder coatings.

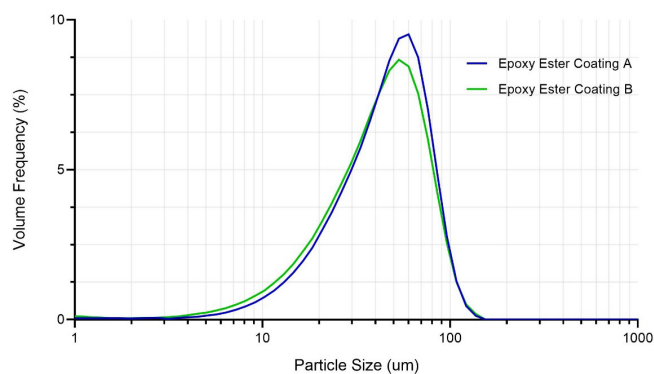


Figure 7. Particle size distributions of epoxy ester coatings from different manufacturers measured by wet tests

## b) Dynamic Image Analysis

The morphological information may also affect the spraying and performance of powder coatings. And the most important feature of the imaging analysis method is that it can visualize the particles. The morphology information of the particles can be given, such as the circularity and aspect ratio of the particles. In particular, the Bettersizer S3 Plus with the combined system of dynamic imaging and laser diffraction method is capable of providing comprehensive information of powder coatings.

Here the dynamic image analysis system of the Bettersizer S3 Plus was used to measure two polyurethane coatings offered by two powder coating manufacturers. The circularity distributions are shown in Figure 8. It can be seen that the two powder coatings have distinct differences in the circularity as well as the shape. The average circularity of PU Coating A is 0.717 with a circularity span

of 0.486 whereas the average circularity of PU Coating B is 0.654 with a circularity span of 0.386.

In short, the image analysis system is able to monitor the particle size, particle size distribution and circularity effectively during the production process of coatings. In addition, it has a higher resolution of coarse particles and can quickly capture large particles, which is especially helpful to indicate the presence of oversized particles in production to help control product quality, thereby improving the performance of powder coating products.

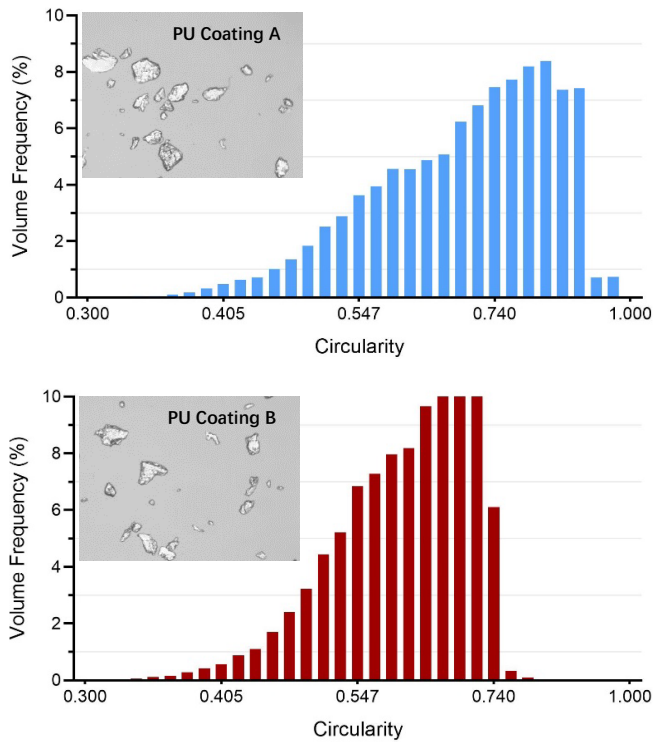


Figure 8. Circularity distributions of PU coating A and B obtained by dynamic image analysis

## Conclusion

In the production process of powder coatings, particle size is one of the most important physical properties, which not only affects the spraying performance of finished coatings, but is also closely related to the entire production process of coatings, such as the selection and dispersion of fillers, the operating parameters of mills and classifiers, and the spraying of finished coatings.

Laser diffraction analyzers have replaced conventional methods to a large extent mainly due to the advantages of the technology including ease of use, fast operation and high reproducibility, which are powerful tools for the powder coating industry. However, the laser particle size analyzer itself cannot provide information about the shape and the particle dispersion state. Therefore, with the dynamic imaging analysis, not only the particle size can be given, but also the critical shape information such as circularity and aspect ratio can be provided, which is an ideal supplementary tool to the laser analyzer.

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Further information can be found at

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