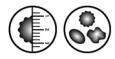


Toner size and shape characterization using FPIA-2100



Toner production overview

Toners are used in photocopiers and laser printers to produce copies or printed images. The process depends on the transfer of the toner to charged areas of a photoconductive drum or belt to produce an image of the document to be copied or printed.

The process begins with the generation of an electrostatic image on the surface of a drum. The electrostatic image causes a powder toner to adhere to the surface of the drum, and then the adhering toner is transferred onto a copying medium such as paper and subjected to heat fusion to complete the process (see Figure 1).

Early toner manufacturing involved the pulverizing and sorting of black, charged graphite. However, toner technology has become gradually more sophisticated in order to meet the ever-increasing quality and technical demands of the industry. The mobility of the toner in the supply reservoir mechanism, the transferability performance to paper, and the property of peeling from the drum are all affected by toner particle size, shape and material properties. In addition to colorants (predominantly pigments), resins, electric charge control agents and releasing agents have been added to toners as blend components. Fluidizing agents, lubricants and electric charge control agents have also been applied to the exterior of the toner particles.

Additionally, the use of carriers such as ferrite has been adopted to further improve the flow properties of toners (see Table 1 for a summary of additives).

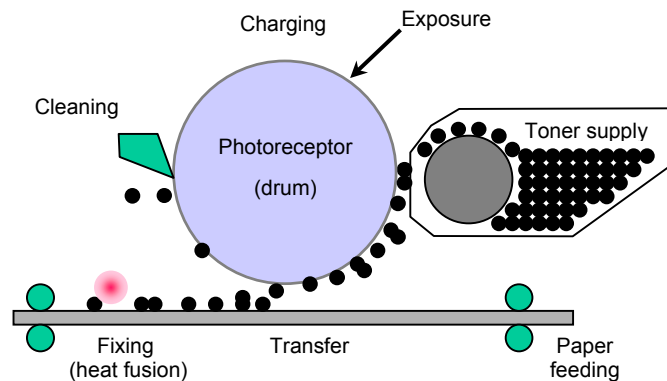


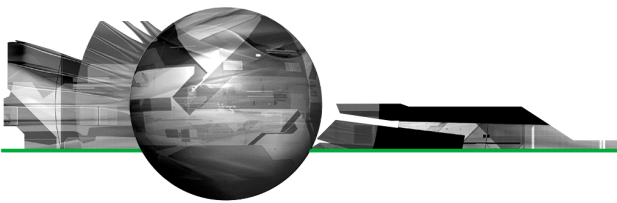
Figure 1. Outline of Copying Process

Description of agent and function	Typical components
Colorants	Pigments
Resin for adhesion and charging	Polyester
Electric charge control agents	Nitrogen containing compounds and metal compound coatings
Releasing agent for preventing adhesion to the fixing roller	Wax
Fluidizing agent - provides powder fluidity	Silica
Lubricant coatings to reduce adhesion to photoreceptor (drum)	Zinc Stearate
Magnetic carriers to aid transferability	Ferrites (Fe_xO_y)

Table 1: Common toner additives and their functions

Toner production methods have also continued to develop. There are two fundamental technologies applied to toner production – the pulverization method and the polymerization method. The conventional pulverization method, where an ingot (or a film) as raw toner material is pulverized and sorted, is slowly being superseded by the polymerization

method, which is capable of yielding toner particles closer to a spherical shape. The polymerization method, which is also referred to as the chemical toner method is a technique in which granulation is conducted by utilizing an aqueous medium. This method can be further broken down into three classifications: the suspension polymerization



Suspension polymerization method	Emulsion polymerization method	Dissolved suspension method
Process steps: 1. Mixing of resin monomer and colorant. 2. Suspension in water 3. Polymerization 4. Filtration/rinsing/drying Features: the toner is close to perfectly spherical, but tends to adhere to the drum.	Process steps: 1. Suspension of dispersed particles of colorant in water (particle: 100 nm) and preparation of resin particle (100 nm) by emulsion 2. Polymerization 3. Mixing 4. Agglomeration/fusion bonding 5. Filtration/rinsing/drying Features: the method is capable of controlling the particle shape through agglomeration/fusion bonding process.	Process steps: 1. Mixing resin and colorant in solvent 2. Oil droplet formation 3. Drying Features: Polyester resin can be used because no polymerization is needed.

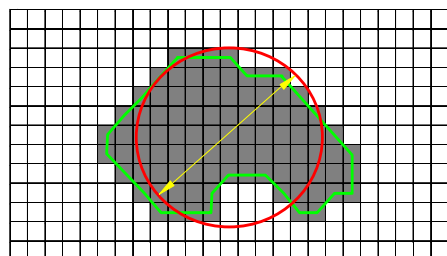
Table 2: production methods of the toner based on polymerization

method, the emulsion association method and the dissolved suspension method (see Table 2).

Characterization method using the FPIA-2100 instrument

The Sysmex FPIA-2100 (Flow Particle Image Analyzer) analyzes a particle image using two parameters. Size is characterized using the **circle-equivalent diameter** – which is defined as the diameter of a circle that has the same area as the projected particle image. With this diameter, various irregularly shaped particles can be evaluated on the basis of a single consistent measure. Shape is characterized using circularity - a parameter that compares the perimeter of the projected particle image with the circumference of the area-equivalent circle thus permitting a numerical representation of complex particle shapes (see Figure 2). Both these parameters can be plotted on a scattergram as shown in Figure 3, so producing a unique 'finger-print' of the sample being analyzed.

In order to gain further insight, both these parameters can be classified into three fractions (hi, medium and lo) and ratios of one fraction to another calculated. In addition, other statistical parameters such as



- Projected area
- Equivalent circle of projected area
- Diameter of the equivalent circle
- Particle perimeter

$$\text{Circularity} = \frac{\text{Equivalent circle circumference}}{\text{Particle perimeter}}$$

Figure 2: Calculation of circularity parameter in FPIA-2100

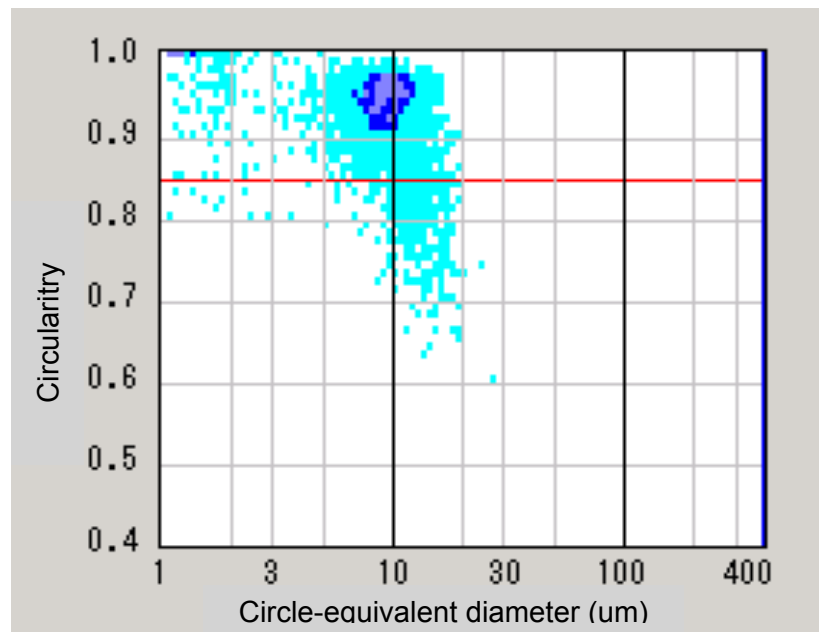


Figure 3: Scattergram showing typical toner distribution with a mean diameter of 10µm and a mean circularity of 0.96

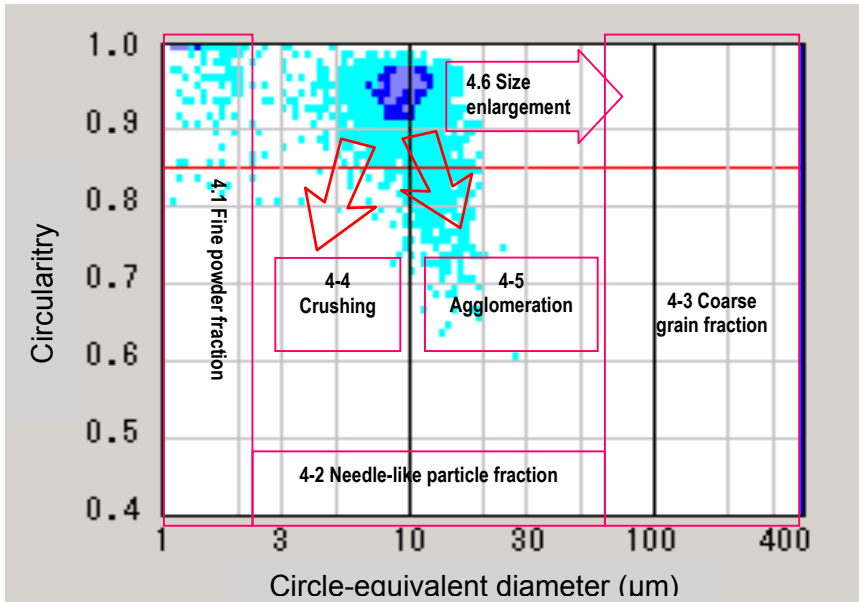
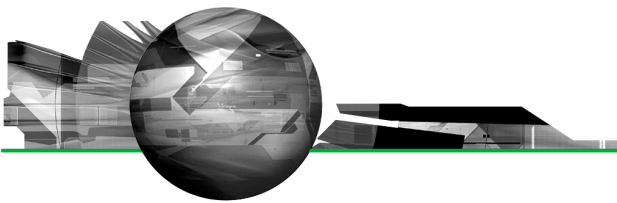


Figure 4. Typical fractional analysis of toner particle distribution

- 4.1 to 4.3 Fine, needle-like and coarse grain fractions
- 4.4 Smaller size and lower circularity indicates crushing
- 4.5 Larger size and lower circularity indicates agglomeration
- 4.6 Larger size with no reduction in circularity indicates chemical size enlargement

standard deviations, D10, D50 and D90 can be calculated. All this data combines to give users a comprehensive package of

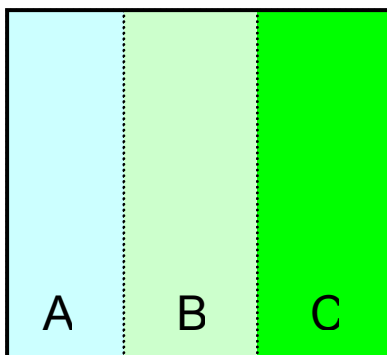
information that they can use to better understand and control their toner production process.

A typical toner analysis method is as follows. Three steps of sophistication are available depending on the purpose of the analysis:

Step 1: From the average value/SD/CV value, the particle size and dispersion degree are evaluated. This alone may be enough to characterize the toner sample. Alternatively further analysis can be carried out as follows:

Step 2: By separating a small particle size fraction, a fine powder ratio can be derived. By separating a large particle size fraction, a coarse grain ratio can be derived. By setting a particular fraction, agglomerated particles or crushed particles can be detected and a mixing ratio derived (see Figure 4).

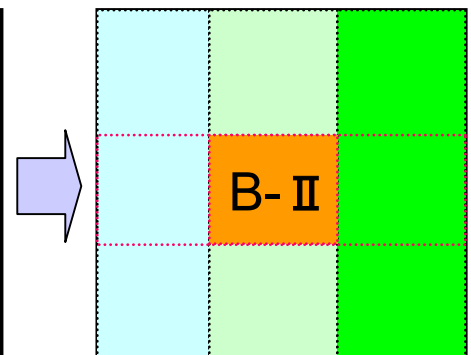
Step 3: If the above analysis is still not sensitive enough, analyses based on combining particle size and circularity can be carried out. (see Figure 5). By setting limits on both diameter and circularity it is possible to accurately define the specification of a material. A 'fingerprint' specification can be defined from a sample of known good material that performs well in final –product form. This analysis can then be applied to incoming toner raw material batches to in order to make decisions much earlier and reduce manufacturing cycle times and costs.



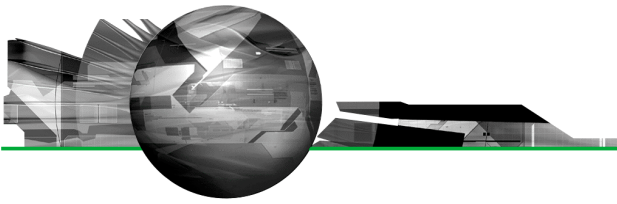
5-1 Analysis based on circle-equivalent diameter fractionation



5-2 Analysis based on circularity fractionation



5-3 Analysis by combining diameter and circularity



Toner characterization of 4 batches showing fractionation technique

Toners A, B, C and D produced by the polymerization technique were subject to characterization on the FPIA-2100. The results obtained revealed that the particle size distributions in terms of circle-equivalent diameter showed few differences, and agglomeration tendencies examined in the two-dimensional scattergrams yielded only slight differences (Table 3-1).

In order to sensitize the analysis towards the areas furthest away from the mean and reduce the influence of

the large number of particles around the mean, fraction bands were created. The threshold diameter was set at 10µm (see figure 7). Remarkable differences were then found in the contents of the particles larger than 10µm. Table 3-2 shows that batch A has a much larger percentage in the middle size band (particles above 10µm) than batches B, C or D

Of these particles in the >10µm size band, far fewer of them had circularity less than 0.9. Hence this analysis identified significant differences between batches that on paper and

from a microscopy point of view would appear identical.

The ability to include and exclude parts of the size and shape distributions is an important capability for toner analysis. This is particularly so when the mean statistics of the total range are not sensitive enough to identify differences.

Conclusions

Recent years have seen the development of wet chemical toner processes such as suspension polymerization and emulsion polymerization, which do not involve a

Table 3. Comparisons Conducted Before and After Fractionation Analysis

Table 3.1. Total Range

Sample	Mean particle size	Particle size SD	Mode diameter	50% Diameter	Low size ratio	Middle size ratio	Mean circularity	Limited circularity ratio
A	8.50	2.33	9.09	8.84	0	100	0.978	1.77
B	5.73	1.42	5.74	5.74	0	100	0.958	0.75
C	6.49	1.47	6.43	6.43	0	100	0.978	1.77
D	6.97	1.49	6.82	6.82	0	100	0.973	0.69

Table 3.2. Range excluding particles less than 10 µm

Sample	Mean particle size	Particle size SD	Mode diameter	50% Diameter	Low size ratio	Middle size ratio	Mean circularity	Limited circularity ratio
A	11.15	1.27	10.2	10.81	79.88	20.12	0.983	2.11
B	13.66	3.53	10.2	12.13	99.51	0.49	0.866	57.69
C	12.03	2.65	10.2	10.81	98.65	1.35	0.898	47.46
D	11.69	3.07	10.2	10.50	98.06	1.94	0.929	19.72

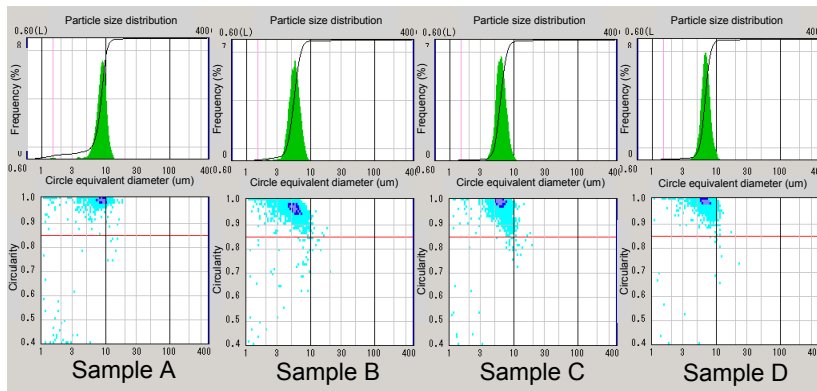


Figure 6: Scattergram comparison of four toner samples

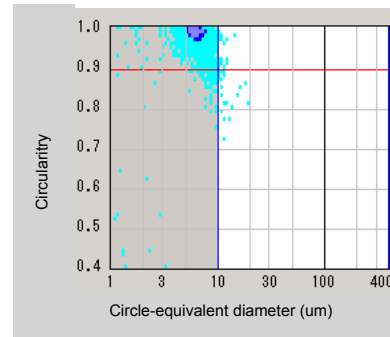
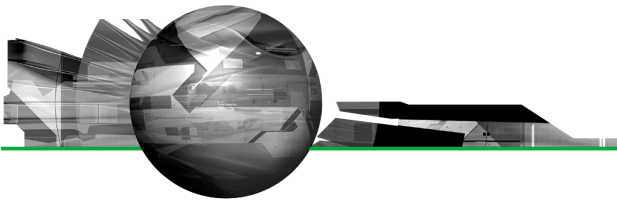


Figure 7: Toner scattergram showing exclusion of particles under 10µm



milling or classification stage and which have the merit of allowing much greater control of the size distribution, shape and material properties of the toner particles produced. Compared to pulverized toner the shape is far more regular.

Many companies are now producing both monochrome and color toners using a wet polymerization process. The toners produced using this process produce material with a narrower particle size and particle shape distribution; this offers improved powder flowability, improved transfer ratio from the photoconductor to the paper and improved image quality.

A number of companies have patented chemical processes for growing toner particles of well-defined shapes. With the ability to produce toners with more precise shape and size distributions comes the need to characterize such materials. Most particle size analysis instruments are not able to measure shape but one exception is the Sysmex FPIA-2100. This instrument has wide use in the toner industry and many patents have been produced based on the optimization of particle shape. Methods have been developed using circularity as the key parameter. A circularity of 0.95-0.96 is optimum, lower than this the toner particles act as an abrasive, higher than this and they act as a lubricant.

The Sysmex FPIA-2100 uses sheath flow and patented high speed image analysis for rapid particle size and shape characterization. Analysis typically takes 5 minutes compared with the 2-3 hours necessary using traditional techniques such as conventional microscopy.

Toner manufacturers invest heavily in novel production techniques in order to develop processes that will maximize

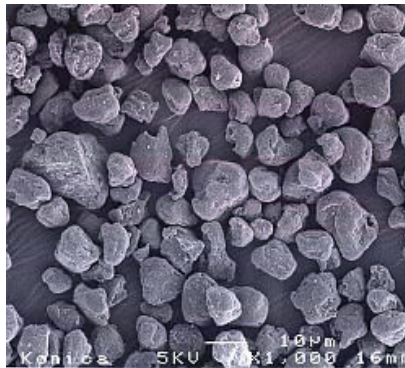
the proportion of particles with high circularity.

The main problem has always been to find a simple way of monitoring this parameter.

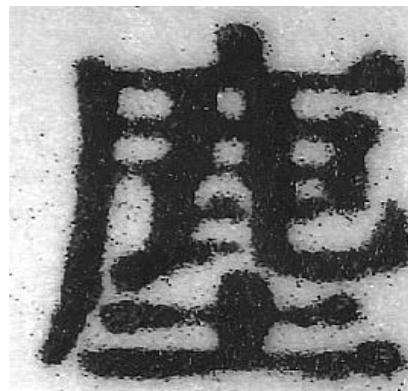
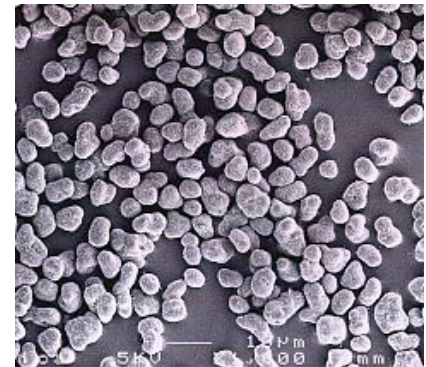
The Sysmex FPIA-2100 offers a rapid way for routine shape characterization. In addition to particle

size data it displays images of the particles and also displays a circularity diagram to the analyst who then has all the necessary data for informed decision-making.

Typical low quality toner – low circularity and heterogeneous



Typical high quality toner – high circularity and homogeneous



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