Industrial Applications of Particle Size Analysis

The following are summaries of two of the papers presented at a Joint Meeting of the Western Region and the Particle Size Analysis Group held on May 20th, 1981, at the University of Bristol.

Particle Size Effects in Electrophotographic Toners—A Review

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The concept of the office photocopier can be traced back to a young American patent lawyer, Chester Carlson, who in the 1930s had the idea of forming a latent electrostatic image on a photoconductor and making it visible by attracting small ink (toner) particles to the charged areas.[1] Three main types of toner are used to make visible images on photoconductors: two component powder toners (triboelectric mixtures); single component, magnetic powder toners; and liquid electrophoretic toners.

Two Component Powder Toners (Triboelectric Developer Mixes)

These developers are created by mixing a thermoplastic toner powder with a carrier, such as steel shot, steel filings or glass beads. Careful selection of the raw materials ensures a uniform triboelectric or frictional electrostatic charge between the carrier and the toner such that the toner can be selectively attracted to the latent electrostatic charge on the photoconductor.

Typical powder toner particle size distributions (PSD) centre around 15–20 μm, with sharp cut-offs below 5 and above 30 μm. The carriers are normally considerably larger than the toner; steel filings range between 50–150 μm and shots between 250 and 1000 μm.

A powder toner's PSD plays an important role in determining a photocopier's ultimate print quality by influencing the following properties: print resolution; toner charge - mass ratio and print density; exposure latitude; and particle size changes caused by selective development. A typical 15 μm $\bar{d}_{50}$ powder toner is not normally capable of resolving more than approximately 10 lines mm^{-1}. Reducing the toner's particle size can increase the resolving power but other size related effects then become noticeable.

The amount of toner laid down to neutralise the latent electrostatic image charge is governed by the toner particle's charge-to-mass ratio (Q/M). It can be shown that

$$Q/M \propto \frac{1}{\text{toner radius}}$$

which implies that an oversize toner will deposit an excessively dense image, whereas an undersize toner will give a weak image. Normal Q/M limits for 15 μm toners are between 5 and 20 μm C g^{-1} for optimum print quality. However, undersize toners with very high specific charges, which would normally imply a very weakly developed image with a very low background density, are, in practice, observed to give weak images with "foggy" backgrounds. It is believed this is due to image-forces created by highly charged, small toner particles approaching and attracting the discharged, background areas of the photoconductor. The image force effect increases with the particles' charge - radius ratio.[2]

The breadth of the toner's PSD has an effect on the exposure latitude or response of the system. In a mono disperse distribution, each toner particle will tend to experience forces similar to every other particle, whereas a broad distribution will contain particles of varying Q/M values, which can develop a range of electrostatic charge values. Thus a narrow PSD toner gives a "threshold effect," which leads to a high contrast print, whereas a broader PSD tends to be more suitable for developing continuous tones.

Selective development effects are observed in two component developers[3] when the developed toner's PSD shifts with development time to small particles as the larger diameter, lower Q/M value particles are selectively scavenged from the photoconductor's surface. Over a long period, selective development effects can alter the balance of the toner sizes in the photocopier's development unit, with consequential changes in the print quality.

These competing effects are all a function of particle size and to maintain print quality, the toner's PSD must be maintained within close specifications. Useful particle sizing techniques include optical and electron microscopy, sieve analysis and sedimentation, but the most widely used method is the orifice (Coulter) technique.

Single Component Magnetic Toners

These powder toners consist of 10–30 μm particles containing a magnetic pigment. The uncharged powder, held on a magnetic applicator, is brought into the close vicinity of a latent electrostatic image. An image charge force is induced into the conductive toner, which is sufficient to overcome the retarding magnetic force, and the latent image is thus developed.

It has been shown[2,4] that the retarding magnetic forces are given by

$$F_{\text{m}} = 2\pi\, a^3\, B_0 \nabla B_0$$

(B_0 is the applied field and a the particle radius) and the attractive electrostatic force as

$$F_1 = \frac{q^2}{16\pi\, e_0\, a^2} f(Kd)$$

where q is the induced charge, a the radius, K the dielectric constant and d the distance to the photoconductor surface. These equations imply that the magnetic retarding forces become larger for larger toner particles, whereas the electrostatic development forces become weaker. Conversely, as the size is reduced the development forces predominate and the background density increases. Thus, the print quality is controlled by a sensitive balance between magnetic and electrostatic forces, which are themselves strong functions of particle size. In practice, most magnetic toners have narrow size distributions centred around 20 μm. The narrow distribution implies a development system that exhibits a strong threshold effect and therefore gives high contrast prints.

Liquid Electrophoretic Toners

Liquid toners are suspensions of pigment particles in insulating, inert, low dielectric constant organic liquids. Typical average particles are less than 1 μm in size, which can be compared with the 15–20 μm of the powder toners.

The dispersions are normally stabilised by the addition of charge control resins, which coat the toner particles to give electrostatic and steric stabilisation effects.[5] The charge control resins are also required to ensure that the particles acquire a uniform electrical polarity to respond to the electrophoretic forces of the development process. The development stage of a liquid toner photocopier takes place in a tank containing the suspension. The charged photoconductor is immersed and the particles migrate by electrophoresis to form an image. Many of the newer machines then transfer the toner to plain paper, whereas older equipment produced copy directly on to photoconductive zinc oxide coated paper.

Among the size effects that are observed with liquid toners can be mentioned: suspension stability; transfer efficiency; optical density effects; Q/M ratios; and resolution. It can be appreciated that, generally, finer suspensions tend to be more stable. Suspension stability is a prime factor in ensuring uniform copy quality in a copying machine cycle which could involve heavy usage followed by long standby periods or periods of not being in use at all. Contrasting with the requirement for fine particles for optimum stability is the requirement for large particles for transfer efficiency.

Many modern liquid photocopiers employ an optical sensing device in the tank to control the toner concentration. Because light absorption is a function of particle size, it is obviously essential to maintain a uniform particle size in the liquid by avoiding selective development that would upset this balance.

In an analogous manner to powder toners, the charge - mass ratio of liquid toners becomes greater as the particle size is reduced. If the toner is over-ground, the print density is significantly reduced owing to the low toner mass deposition. However, the fine particle size of liquid toners makes them suitable for high resolution, high quality processes and up to 500 lines mm^{-1} can be achieved.

Particle sizing of liquid toners has received considerable attention, with a wide variety of techniques being used. These include auto-correlation spectroscopy, disc centrifuges, electron microscopy and, in particular, measurements based on the electrophoretic mobility of toner particles under the influence of electric fields.[6] This last takes the form of plotting the current decay between electrodes immersed in the toner solution. From these measurements, the mobility, Q/M, zeta potential and average particle size can be calculated and compared against direct measurements.

Concentrations

It has been shown that micrometre sized powder toners and sub-micrometre sized liquid toners each exhibit size effects that have considerable effects on a photocopier's print quality. A range of techniques can be used to measure and to control the particle size distributions of the toners.

References

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Grading Grains in the Flour Milling and Baking Industries

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Separations on the basis of particle size constitute an essential part of the process by which white flour is produced from wheat kernels. The industrial process provides a means of achieving a bulk dissection as a result of which near-quantitative separations of the anatomical components can be achieved. Thus, from a grain consisting of approximately 80% endosperm, 3% germ and 15% pericarp and associated tissues, the miller can produce about 75% white flour and 1% separated embryo, the remaining bran and wheatfeed products being composed mainly of the kernels' outer coats of pericarp, testa and aleurone layer.

Even before milling begins, particle size is a property on which are based separations designed to purge the wheat of impurities such as field debris and seeds of other cereal and weed species. In addition to simple screening equipment more sophisticated machines such as disc separators are used. In such machines, a series of discs, spaced along a horizontal axis, rotate through a hopper of uncleaned grain. On both sides of each disc are uniformly sized indentations in which suitably sized seeds, etc., can be accommodated. Such seeds are lifted out of the hopper and conveyed to a chute. Rejected seeds pass through the hopper to be streamed separately. By use of disc separators with indentations greater or smaller than wheat seeds contaminants greater or smaller than wheat seeds can be removed. In one instance the contaminants are accommodated in the discs, in the other they are excluded.

The milling system itself includes many sieving operations on wire and silk or nylon covers. These alternate with the grinding stages performed on roller mills. The first series of grinding stages, known collectively as the "break system," uses fluted rollers rotating with a speed differential within a pair of 2.5:1. In the "first break" the grain is sheared open and the outer coats spread into flat sheets, which are separated by sieving from finer stocks and which pass to further break treatments whose function is to scrape off the endosperm increasingly closer to the bran coat. The coats remaining after the full break system are concentrated over sieves with apertures of 1000 μm and constitute the bran. Stocks finer than bran are graded after each grinding treatment; they include semolina (250–1000 μm), middlings (132–250 μm) and flour (0–132 μm). The semolina and middlings are large particles of endosperm with some bran and embryo attached to many of them. These pass to the "reduction system," consisting of grinding stages with smooth surfaced rolls rotating at almost the same speed (1.25:1), alternating with the sieving operations. Endosperm particles become increasingly fragmented during the roller milling, while bran particles remain intact as flattened sheets. The embryo, being soapy in consistency, becomes squashed into large flakes that then overtail sieves through which, before flattening, they were capable of passing. The flours produced during all grinding and sieving operations of the milling system are blended to produce a "straight-run" flour.

Further separations on a compositional basis can be made by discrimination according to particle size at a sub-sieve level. This is possible as a result of the special relationships between starch granules and the protein matrix in which they are embedded in the endosperm. Starch granules occur in two distinct size ranges. Until recently it was thought that the granules of the smaller population (less than 10 μm) accounted for only 10% of the total mass of starch. However, it has now been demonstrated, by micro-sieving, Coulter Counter and Quantimet assessments, that they contribute 30% or more. The interface between the larger starch granules (10–30 μm) and the protein matrix is a relatively weak structure. Fine grinding of flour in, for example, a pinned disc mill breaks many particles at such interfaces, releasing many wedge shaped fragments of protein from the interstices among the larger granules. The protein fragments have a diameter of about half that of the starch granules, hence a separation at a cut size of 15 μm concentrates protein in the fine fraction. A further cut of the coarse fraction at 30 μm concentrates free starch granules into its finer fraction. The coarse fraction consists of composite particles that have remained in spite of fine grinding. The protein contents of the fractions of a flour with an initial protein content of 8% might be 20% for the 0–15 μm fraction, 5% for the 15–30 μm and 10% for the over 30-μm fraction. Between 40 and 55 μm lies a fraction with a higher protein content (about 12%). This fraction contains a concentration of cells originating in the outer or subaleurane layer of endosperm. They contain no large starch granules and consequently break down much less readily than other endosperm cells. Individual cells may have a protein content of 40%.

Flours are produced for many specialised purposes for which different types of wheat are suitable. One source of variation is the hardness of the endosperm. This characteristic profoundly affects milling properties and influences the ease with which a good yield of white flour can be obtained by milling. On a standard milling system, soft wheats break down more readily than hard ones and hence the proportion of a flour below a certain size can be used as an indication of the hardness of the wheat variety from which it was milled. For indicating hardness to millers, breeders and agencies responsible for publicising information on newly introduced wheat varieties, the F.M.B.R.A. sieves a flour produced by a standard laboratory milling system on an air jet sieve, using a 75-μm screen. Soft wheats are defined as those producing a flour of which at least 50% passes through the screen. Recent tests have shown that hardness may be one of the many characteristics that can be determined by near infrared reflectance (NIR) analyses.

Particle size distribution of flour is becoming recognised as an important quality factor in flour - confectionery production. Although it is influential in biscuit and wafer manufacture its importance is probably greatest in those cakes, with a high sugar content, in which the use of chlorinated flour imparts a fine soft crumb. The importance of particle size was strikingly brought to the attention of bakers in 1976, when problems of the collapse of sponge cakes occurred widely. In that year weather conditions had led to a wheat crop with endosperm that fragmented less readily than usual and it was thought possible that abnormal particle size distribution was the reason for the flours substandard behaviour. In a series of experiments flours were graded before chlorination into four fractions: <125 μm, 90–125 μm, 55–90 μm and 0–55 μm. Collapse was marked in sponges baked from the coarsest fraction and evident in those baked from intermediate fractions, though less so for the 55–90 μm fraction. The best sponge was made from the finest fraction. Marked improvements also resulted from fine grinding of ungraded flours both in sponges and madeira cakes. Industrial experience confirmed experimental results and cake manufacturers now include a particle size requirement in their specifications for high-ratio cake flours.

Measurement of sub-sieve particle size in the industry is performed most widely by simple sedimentometry. This provides a quick means of assessing the proportion of undesirably large particles as these sediment very rapidly in the toluene that is used. A complete analysis can take over 12 h where a large proportion of very fine particles are present. The Coulter Counter is used only in the larger quality-control laboratories. This method has recently been shown to be capable of good reproducibility among laboratories. In a recent collaborative test, agreement among 4 participants on four flours was within 5%. In the same test, similar encouraging results were obtained by Coulter Counter, air jet sieve and a Microtrac particle size analyser used with the dry flour.