# The First Measurement of Cohesive Force Distributions in Powder Flow:

New Insights into the Effect of Metal Oxide Surface Additives on Xerographic Toner

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## Abstract

The Hosokawa powder tester has been used for years to determine cohesion of bulk powders, including toner. For the first time a method is demonstrated to measure cohesive force distributions, rather than just average values, using the Hosokawa tester. The method is demonstrated for toner, varying surface additive composition and loading. Force distributions are shown to be generally log-normal. Both the mean and width of the distributions generally decrease with increased surface additive loading. Thus, better flow with additives decreases peak cohesion and distribution width.

## Introduction

Surface oxide particles control xerographic toner flow, charge<sup>1-5,6</sup> and transfer.<sup>7</sup> While there are many methods to measure flow of powder, including toner,<sup>8</sup> all give only an average value. This paper shows a novel Hosokawa tester method that measures particle cohesive force *distributions*.

### **Experimental**

Cohesion was measured on a Hosokawa Micron Powder tester, with an added vibration sensor<sup>6</sup> (300 mV output per mm vibration). A standard Hosokawa test places a 2 g sample on nested screens of varying size (150, 75, 45  $\mu$ m) at fixed vibration, usually 1 mm, for 90 s, giving:

$$\% \ cohesion = 50 \bullet A + 30 \bullet B + 10 \bullet C \tag{1}$$

A, B and C are toner weights left on each screen, respectively. The weighting factor in Eq 1 increases with screen size: toner left on a larger screen contributes more. Cohesion for most toners here was too low to be measured.

The new method uses only one screen, testing sequential 10 g samples at 6 different vibrations, from 1/10 to 1 mm for 90 s, weighing toner remaining on the screen.

Base spherical toner particles<sup>9</sup> were blended with additives at 16 Krpm on a sample mill.

# **Results and Discussion**

#### **Powder Cohesion and Inter-particle Forces**

Experimental toner cohesion data can be explained if inter-particle forces are linear with % Hosokawa cohesion.<sup>6</sup> Briefly, toner is an ensemble of agglomerated particles whose cohesion follows a log-normal distribution (Eq 2 and Fig 1a).  $N_F$  is the number of particles experiencing an interparticle cohesive force F, while  $N_o$ , is the total number of particles, and  $\sigma$  is the geometric standard deviation.

$$N_F = \frac{N_0}{\sqrt{2\pi} \sigma} \exp\left(\frac{(\log F - \overline{\log F})^2}{2\sigma^2}\right)$$
(2)



Figure 1. Log cohesion distribution: a) number b) cumulative.

Fig 1a shows log-normal distribution for samples ① and ② with different peak cohesion, but equal distribution widths. Fig 1b is the cumulative log normal distribution, the integral of Fig 1a. Also shown is the applied vibration force,  $F_{vib}$ . Particles of cohesion  $\langle F_{vib}$  (to the left in the Figure) pass through the screen, those  $\rangle F_{vib}$  will remain. If ① is replaced by the less cohesive ③, more toner passes (the  $F_{vib}$  line "moves" from A to B on the distribution curve in Fig 1b). If the vibration is decreased for ①, less toner passes ( $F_{vib}$ "moves" along the distribution curve from A to D).

Hosokawa % cohesion then has an associated  $F_{vib}$  that is approximately linear with the cohesion force, as shown by the fitted line in Fig 1b. The linear fit is a good, with  $\pm 1-2$  $\sigma$  from the peak. Only if the applied force goes into the distribution tails, does the test become insensitive.

To keep the Hosokawa in the linear range over a range of cohesion force, Carr<sup>10</sup> uses different size screens. It is observed that a small screen requires a larger force (vibration) to pass toner than a large screen. If all the toner passes the largest screen, a smaller screen below still can partitions toner linearly. By proper choice of screen sizes and weighting factors the linear range can be maximized.

To produce a cohesive force distribution like Figure 1a, data is plotted as the natural log of  $F_{vib}$  (Eq 2.) Data is collected in discrete intervals. The x-axis will plot the natural log of the vibration on an interval. Vibration was left in the measured mV. (Neither mm, nor mV, are force units, the maximum vibration force applied, according to basic physics, is proportional to vibration amplitude.) The y-axis will then transform the data as  $\Delta w / \Delta ln(v_{i+1} - v_i)$ , where  $v_i$  is the vibration at data point *i*, and  $\Delta w = (w_{i+1} - w_i)$ , is the toner passed over the interval. Completing the y-axis transformation, the data is normalized by dividing  $\Delta w$  by  $\Sigma \Delta w$ , the total over all the toner that passes. This is necessary to ensure that the curve areas normalize to 1. The y-axis is thus transformed to a fractional population, which enables observed curves to be fitted directly by Eq 2.

A suitable screen size was chosen to measure the full cohesive distribution within the vibration range. For poorly flowing toner, without flow additive, this was not possible. With additives, all toners had sufficient flow to enable full measurement of the force distribution.



Figure 2. Cohesion distribution with 1 wt% TS530

Figure 2 shows data for a toner with 1 wt% Cabot TS530 silica on a 38  $\mu$ m screen, capturing the full force distribution. The fit to a log-normal distribution is outstanding: only the width,  $\sigma$ , and mean,  $\mu$ , of the distribution are adjustable parameters. Generally, with all three surface additives studied, there was a good fit to a log-normal distribution. Data at two larger screen sizes is not shown: while force distributions are cut off with these screens it is possible to estimate a peak and width.

Figure 3 shows the effect of screen size on distribution mean and width, showing a 1/(screen size) fit to the mean, and a linear fit to the width. Increased screen size passes more toner, as expected. Thus, smaller screens enable the measurement of toners with wider cohesion distributions.



Figure 3. Effect of screen size on the force distribution.



Figure 4. Cohesive force distribution at 0.5 wt% TS530

Figure 4 shows lower 0.5% TS530. The distribution is log-normal, shifted to a higher mean and width. Fig 5 summarizes results for 0%, 0.5% and 1% TS530. As expected, more TS530 reduces peak cohesion. Broadening at low silica is ascribed to inhomogeneous additive distribution: some toner has more silica (flowing better) and some less (flowing worse). At optimal loading all toners are effectively coated with silica, and all flow well.



Figure 5. Force distributions (fitted curves) with % TS530.



*Figure 6. Peak cohesive force with additive loading.* 



Figure 7. Cohesive distribution width with additive loading.

Flow with two different titania additives, coded Titania A and B, was also studied. The data is not shown, but a summary for all additives is shown in Figs. 6 and 7.

Figure 6 shows peak cohesion with additive loading. TS530 and Titania B show equivalent cohesion, reaching an asymptote of  $\approx 80$  mV at the highest 1% loading. Titania B is less effective: at 2.1% the asymptote is  $\approx 120$  mV, and thus requires over 2x more additive to reach optimal flow, still 50% worse than the other additives studied.

Figure 7 shows distribution widths with loading. Silica and Titania B decrease monotonically with loading. Titania A shows an initial increase in width, but an overall decrease. The initial width increase is likely due to an inhomogeneous distribution of additive, which improves at higher optimal loading.

## Conclusions

A novel method has been developed that measures cohesive force distributions with a Hosokawa tester. The method was demonstrated for varying surface additive type and loading. Force distributions are log-normal. The mean cohesion decreases with increased additive loading. At optimal additive loading there is a concomitant decrease in distribution width. "Best" flow with additives is a combination of both reduced distribution peak and width.

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#### **Biography**

Rick Veregin has a B.Sc., M.Sc. and Ph.D. in Chemistry. He has worked at the Xerox Research Centre of Canada for the last 17 years, where he manages the Developer Physics group. His interests focus on the interface of chemistry and physics in xerographic materials. He is an author of 54 papers, an inventor on 52 US patents, and a recipient of the American Chemical Society A.K. Doolittle Award.