Effect of Additive Blending Temperature on Toner Properties

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Abstract

Surface additive blending is one of the most important processes in toner manufacturing, which determines the toner's xerographic performance. The blending conditions as well as additive composition can affect several important toner properties such as triboelectric charge, charging rate, free flow, cohesivity and cleanabiltiy. An understanding of blending process and its effects to toner performance is of considerable use in designing additive blending and choosing optimal blending conditions.

So far effects of many blending conditions, such as blending rpm and blending time have been extensively studied. However the blending temperatures are normally kept at low temperature to remove the heat generated during high rpm blending and its effect has not been studied much.

Since toner's mechanical properties changes with temperatures as illustrated by the fact that storage modulus of DMA analysis changes with temperature, the adhesion of additives on toner surface may changes. In this study, we try to address the effect of blending temperature and cooling rate on toner properties.

Introduction

Additive-blending is one of the major toner manufacturing processes and this process yields toner the required physical properties, such as triboelectric charge and free flow. In chemical toners, where toner particles are smaller and toner shape is more controlled, additive-blending process plays a much more important role. The benefits of using additives include improved toner flow, better triboelectric charge, increased charge stability, better transfer from developer unit to OPC and from OPC to paper, and easier cleaning of toners from OPC. Additives are generally inorganic particles, such as silica, titania, or alumina. They are smaller than toner particles and the size normally ranges from few nanometers to hundreds of nanometers. Additives are normally surface-treated with hexamethyldisilazane, polydimethylsilicone, and octyltriethoxysilane to make hydrophobic surface for increased environmental stability.

In additive-blending process, the key factors are blending rpm, blending time, and sequence of blending. For a given toner and additive formulation, the blending conditions significantly impact the toner properties. In most cases, additive-blending is carried out under controlled temperature environment, i.e. cooling the blender.

When toner is used in electrophotographic systems, toner is normally agitated and a lot of stress is applied on toner. This stress can cause additives either to fall off from toner or to be buried into toner surface. These phenomena decrease the effective amount of additives on the toner surface and degrade toner performance. The additive adhesion to toner surface can be calculated by measuring the amount of additives still remaining on toner surface after extensive sonication, which reproduces the stress toner experience.

The study attempts to address the effect of blending temperatures and cooling rates on toner properties, such as triboelectric charge, free flow and additive adhesion.

Experiments

Toner formulation – Samsung Fine Chemicals' ACE toner

Samsung Fine Chemicals' (SFC's) ACE toner is an eco-friendly polyester-based chemical toner with low level of total VOCs. The TVOC level is lower than 300 ppm measured using thermal desorption GC-MS [1]. This value is lower than that of color toner used in leading brand laser printing system. Using a proprietary process, SFC produces toner particles with narrow size distribution and uniform shape. This method can consistently produce toner shape from potato shape to spherical shape, depending on the application [2].

The polyester resin, which has a proprietary composition, is dispersed using a neutralization emulsion method with minimal amount of an organic solvent. The solvent is then completely removed once the sub-micrometer resin particles are stabilized in the dispersion medium. The colorant and lubricant are individually dispersed in water-based systems using a high pressure homogenizer and the resulting size is sub-micrometer. These dispersions of polyester resin, colorant and lubricant are mixed together to produce toner particles through a size and shape control process.

The Table 1 lists the toner particle size and distribution, as measured using Multisizer 3 Coulter counter. The size ranges from 6.0 μm to 6.5μm, however it can be easily modified as needed without compromising the geometric size distribution.

Table 1. Particle Characteristics

D50,v[1)]	GSD p[2)]	GSD v[3)]	>10μm[4)]	Circularity[5)]
6.0~6.5	≤ 1.300	≤ 1.250	<2.0	≥ 0.985

1) Average volume diameter (μm)
2) Geometric Size Distribution of number data, which is

$$GSD_P = \sqrt{D_{84,n} / D_{16,n}}$$

3) Geometric Size Distribution of volume data, which is

$$GSD_V = \sqrt{D_{84,v} / D_{16,v}}$$

4) Percentage of particles of volume data whose diameter is larger than 10μm
5) Ratio of circle circumference to perimeter of projected particle image

Additive-Blending

The aforementioned ACE toner was blended with surface treated nano-size silica particles. The blended toner was prepared by blending 200g of toner and 3g of additive using a powder blender. The toner, additive and blender were N/N conditioned at 25°C and 50% RH. To clearly see the effect on blending condition,

one additive was used to avoid the possible interaction between different types of additives. Instead of the conventional fumed silica with irregular shape and wide distribution and aggregations, newly developed sol-gel type silica was used. The characteristics of the additive are listed in Table 2. It has a very narrow size distribution and uniform spherical shape.

The blending was performed under controlled temperature of 15°C, 25°C, 35°C, 45°C, 55°C for total of 10 minutes. The blending temperature was accurately controlled by circulating the water, whose temperature was controlled. During the first 6 minutes, the blending rpm was 1000 and during the last 4 minutes, the blending rpm increased to 8000. After blending was finished, the blended toner was moved into a metal container with rapid air cooling system using liquid nitrogen. The blended toner was stirred during cooling and the temperature reached the set temperature (0°C) within 15 seconds. After cooling, the blended toner was sieved to remove possible aggregates.

This procedure was repeated and the blending was performed at 15°C, 25°C, 35°C, 45°C, 55°C again. Then the blended toner was left in the ambient temperature, instead of being rapidly air-cooled.

Table 2. Additive Characteristics

Size (nm)	Purity (%)	B.E.T (m^2/g)	Moisture (%)
100	>99.9	25	0.26

Triboelectric charge measurement

Triboelectric charging property was measured by blow off method using q/m-meter, Epping GMBH. Dual component measurement sample was prepared by mixing toner sample with ceramic surface treated Ferrite carrier whose average particle size is 40μm (SY-248, Kanto Denka Kogyo Co. Ltd). Toner and carrier were mixed at 96rpm for 10minutes to sufficiently saturate the triboelectric charge and mixing was done using Turbular Mixer, WAB. The charge to mass ratio (q/m) of the toner is calculated as follows:-

$$q/m = C \times V/m = 10 \times V/m$$

where m is mass of toner in mg, C is capacity constant (10 nF) and V is the voltage of the remaining carrier after blow-off. (Voltage of the toner has the same value with the opposite sign). The average result of three repeated runs was used for reliability.

Cohesion measurement

Cohesion was used as a measure of flowability of toner. It was measured using Hosokawa powder tester PT-S. 2grams of blended toner was allowed to pass through three sieves whose mesh-sizes are 53μ m, 45μ m and 38μ m in order by vibration (set at level 3) and gravity. The sieves were vibrated for 40 seconds, after which the amount of toner left on each sieve was weighed to calculate the cohesion by the following relationship:-

$$Carr\ Cohesion = \frac{100 \times m_{53} + 60 \times m_{45} + 20 \times m_{38}}{toner\ load\ \ weight}$$

where m_{53}, m_{43} and m_{38} are the weights of toner left on each subscript corresponding sieve.

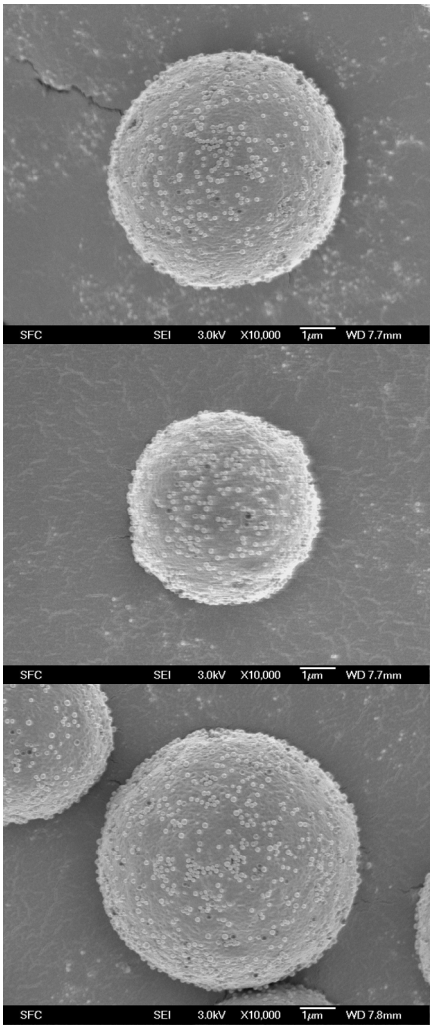

Figure 1. SEM image of toner blended at 15℃ with rapid air cooling after blending (top.a). . SEM image of toner blended at 15℃ without air cooling (middle.b) . SEM image of toner blended at 55℃ with rapid air cooling after blending (bottom.c)

Additive adhesion measurement

The additive adhesion was calculated by measuring the amount of additives in blended toner before and after the toner was exposed to ultrasonic blast. This measurement technique was proposed by Deluca et. al [3] and called Additive Adhesion Force Distribution (AAFD). It can quantify how strongly additives adhere to toner even after exposed to intense sonic energy.

The amount of additives in blended toner was measured using XRF (X-Ray Fluorescence). The intensity of XRF measurement was compared to the reference line, which was generated by measuring the intensity of toners with known amount of additives, and the amount of additives could be calculated.

The blended toner was dispersed with a presence of surfactant and stirred while sonicated in ultrasonic bath (Branson 3510) for 30 minutes. The ultrasonic bath was kept at 15°C. The toner was filtered and dried for XRF measurement. The ratio of additives before and after the sonication was referred as additive adhesion

value and was used to determine how strongly additives adhere to base toner.

Results and Discussions

The SEM images of the blended toners are shown in Figure 1. Figure 1(a) is the SEM image of toner blended at 15°C with rapid air cooling, Figure 1(b) is the SEM image of toner blended at 15°C without rapid air cooling and Figure 1(c) is the SEM image of toner blended at 55°C without rapid air cooling. Any sign of deformation due to the high blending temperature was found in Figure (c). From those images, we could not find any differences due to different blending temperature or cooling method.

The physical properties of toner are shown in Figure 2. The cohesion data is shown in Figure 2(a). The toners without rapid air cooling showed similar cohesion (24~25.3) regardless of blending temperature. The toners with air cooling shows higher cohesion, or less free flow, than toners blended at same temperature but without cooling except 15°C. The toners with air cooling shows a wide range of cohesion (23.6~27.6), but no relationship between cohesion and blending temperature could be found.

The triboelectric charge is shown in Figure 2(b). The triboelectric charge remains same (-49.28 μC/mg ~-48.63 μC/mg) regardless of blending temperature when samples are cooled in ambient. For rapidly cooled toner, the triboelectric charge is less than that without cooling. The triboelectric charge slightly increases as blending temperature increase. However we could not find any major impact of blending temperature and cooling rate on toner flow and triboelectric charge.

The amount of additives on the blended toner is shown in Figure 3(c). In the toner blended below 25°C has more additives than the toner blended above 35°C. This phenomenon is same both with and without rapid air cooling.

The additive adhesion is shown in Figure 3(d). For toners blended above 35°C and rapidly cooled, the additive adhesion is higher than toner blended below 25°C. For toners without air cooling, toners blended above 35°C also have higher additive adhesion than toners blended below 25°C.

Figure 3 shows Dynamic Mechanical Analysis (DMA) of base toner and Storage modulus (M'), loss modulus (M'') and tangent delta data is plotted. The analysis was performed using Mettler-Toledo SDTA8621 and the temperature ranges from 0°C to 200°C with the rate of 3°C/min temperature increase. The analysis was performed at 10Hz and 0.1N under nitrogen atmosphere. 1.75mm long, 1.800mm wide, 1.92mm thick sample was measured and the geometry factor is 304.7/m.

The storage modulus was kept at constant value below 30°C and starts to decrease at 30°C.

The temperature where additive amounts changes falls within 25°C ~35°C, where the DAM analysis shows the storage modulus (M') starts to decreases.

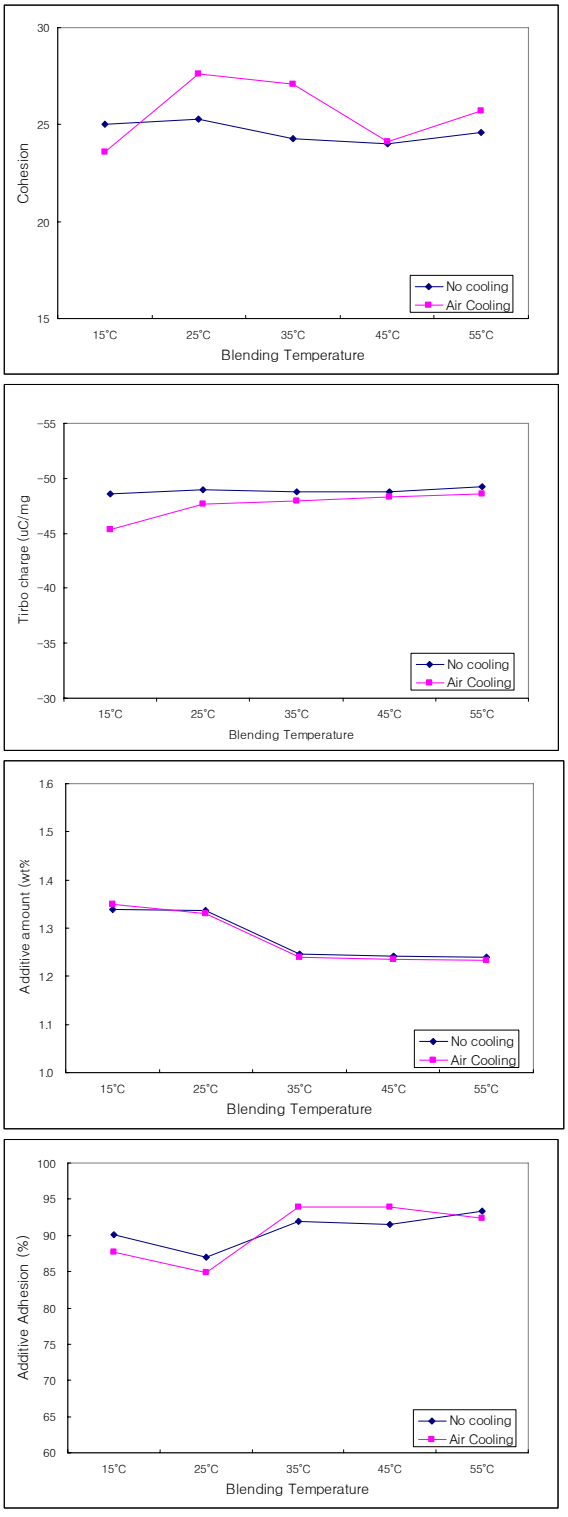

Figure 2. The cohesion data of toner samples blended under different conditions (top, a). The triboelectric charge data of toner samples blended under different conditions (middle, b). The amount of additives on blended toner measured by XRF (middle, c). The additive adhesion data of toner samples blended under different conditions (bottom, d)

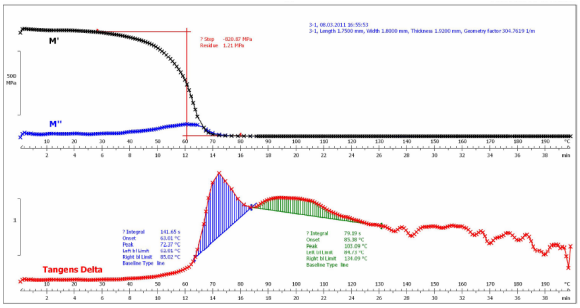

Figure 3. DMA analysis of base toner. The storage modulus and loss modulus are illustrated in top. The tangent delta is illustrated in bottom. The temperature was increased from 0℃ to 200℃ at the rate of 3℃/min. The analysis was done at 10kHz under nitrogen atmosphere. The storage modulus starts to decrease at 30℃.

Conclusion and Future work

The blending temperature is found to have no effect on toner's physical properties such as triboelectric charge and cohesion. Also the rapid cooling after blending has no effect on those properties either.

However, when blending temperature is above 35°C, the additive adhesion to toner increase for both naturally cooled or rapidly cooled toner.

References

[1] Boyoung Kim et. al., "Polyester-based Chemical Toner with Low Level of Total Volatile Organic Compounds", NIP26 and Digital Fabrication 2010, p 77.

[2] Eui-Hyun Ryu et. al., "Eco-Friendly Prepared Chemical Toner with Mixed Polyester Resin", NIP26 and Digital Fabrication 2010, p 73.

[3] US Patents 6,508,104 B1

Author Biography

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