# Effects of Coalescence Agent in Toner Particle Shape Factor Control

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## Abstract

*Shape factor control is important in chemically produced toners (CPT) to achieve higher toner transfer efficiency in the machine and better image quality. Among all the CPT processes, emulsion aggregation process offers more tunability on particle design, especially on particle composition, structure, and morphology and shape factor. The coalescing agents have been widely used in painting/coating industrial applications to increase the easiness of latex particles to coalescence through softening the latex particles and lowering the minimum film-formation temperature (MFFT) of the latex.*

*In this paper, criteria of good coalescence agent in CPT particle shape control will be discussed. Examples of non-reactive and reactive coalescence agents will be provided. Their effects on the toner particle shape factors control will be discussed in emulsion aggregation process.*

## Introduction

Chemically produced toners (CPT) have attracted more and more attention both in academic studies and industrial application for digital printing. Depending on the chemical process used for the preparation of toner, CPT can be made through suspension or dispersion polymerization, emulsion aggregation, and chemically milling process [1-3]. Compared with conventional toner process (mechanical grinding process), CPT approaches to toner offers advantages such as smaller toner particle size and narrower size distribution, higher toner transfer efficiency in the machine and better image quality [4]. Among all the CPT processes, emulsion aggregation (EA) process offers more tunability on particle design, especially on particle composition, structure, and morphology and shape factor. EA process consists of aggregating polymeric particles prepared by emulsion polymerization (latex) with pigment and other toner components. Typically, in EA process, the toner particles are produced directly from submicron polymer particles dispersions, by mixing latex prepared by emulsion polymerization with pigments and other ingredients (also dispersed in aqueous phase).[5, 6] All the latex and pigments dispersion are negatively charged. Then a flocculated agent was added into the mixture (typically positively charged) and aggregate particles ranged from 0.8 to 2 micron can be formed through the aggregation of latex and pigment particles. This is referred as primary aggregation. The primary aggregates can the further grow to the 5 to 10 micron in diameter through secondary aggregation under carefully controlled operating conditions. Finally, those aggregates went through a coalescence step under which the particles are fused into homogeneous toner particles of controlled shape by heating above the glass transition temperature. Toner particle shape and size distribution are important factors in determining the electrophotographic printing machines performance and the final print image quality, especially for advanced high-resolution and color printings. A lot of development efforts have focused on the control and manipulation of shape factors in toner production.

The coalescing agents have been widely used in painting/coating industrial applications [7, 8]. They play most important roles in the film formation of water-based coatings, by increasing the easiness of latex particles to coalescence through softening the latex particles and lowering the minimum film-formation temperature (MFFT) of the latex.

Coalescence process in current EA technology typically consists three different stages: (1) water release brings sub-micron particles (latex, pigment and wax) into closer contact, formed even more denser aggregate particles; (2) Sub-micron particles undergo deformation to form a void-free solid structure, while still maintain its identifications; (3) fusion (coalescence) occurs among adjacent particles through polymer chain diffusion across interfaces [9, 10]. A drawing depicting this process is shown in Figure 1.

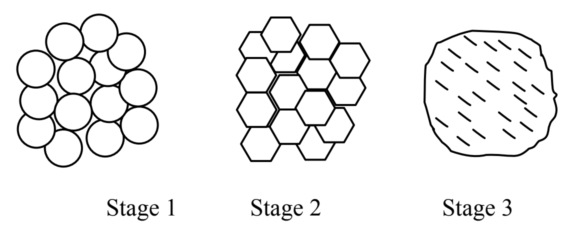


***Figure 1**. Pictorial view of CPT particle coalescence process*

An ideal coalescing agent for EA toner application should be able to fulfill the following criteria:

(1) Sufficiently soften the polymer during the temperature ramp or the early stage of coalescence, so that the individual particles will combine to form a continuous film easily;

(2) The coalescing agent must then evaporate during later stages; thereby it won't increase total VOC in the final toner product, and minimizing any impact on the machine performance;

(3) It is highly desirable that the coalescing agent has very low solubility in water, so that most of them can concentrate inside or on the surfaces of the particles. In this paper, we will discuss the effects of coalescence aid agent on the particle spheridization rate during the process and the final particle shape.

## Experimental

**EA toner particle preparation**

All the toner particles in this paper were prepared in a 2L glass reactor using the following process. In a 2L beaker, 258 grams styrene-butyl acrylate latex (particle size of 220 nm), 80.0 grams pigment dispersion (magenta, or black), and de-ionized water 670 grams were mixed by a homogenizer for 15 minutes at 20 ºC. Then a flocculate agent was added dropwise in 5 minutes. The resulted viscous mixture was continuously mixed by a homogenizer for another 20 minutes to form primary aggregates with particle size of about 2.0 micron. Then the homogenizer was removed and the mixture was transfer into a 2L glass reactor. And the temperature of the mixture was raised to 50ºC in about 35 minutes under a mechanical stirrer mixing at 550 rpm. After the particles reach at 5.8 micron, 140 grams shell latexes (the same as the core latex) were added dropwise in 10 minutes. After particle size reached at 6.5 microns, a freeze agent was added to stop the particle growth. In the case of coalescent agents used a certain amount of coalescence aid agents will be added in, and hold for another 20 minutes. Then, the temperature of the mixture was raised to 90 to 96$^o$C in 35 minutes and hold for certain time. The mixture was cooled down to 35ºC. After washing de-ionized water, acid and DI-water, and dried at 45ºC, the final toner product has a volume median particle size of 6.0 microns.

## Results and Discussions

**Toner particle preparation with non-reactive coalescence Aid Agent**

Magenta Particles Sample #1: A magenta toner particle was prepared using above mentioned process conditions with and without any coalescence aid agent (Velta® 262, isodecyl benzoate) added. Table 1 summarizes the comparisons between the two runs for some key parameters.

***Table 1: Comparison of Magenta Parent Particle Properties with and without Coalescence Aid Agent***

| Samples | Sample #1 | Comparative sample #1 |
|---|---|---|
| Coalescent | Velta® 262 | No |
| Particle Size (μm) | 5.84 | 5.81 |
| GSDv | 1.232 | 1.251 |
| Circularity | 0.964 | 0.963 |
| Coalescence Temp. ($^o$C) | 94.0 | 96.0 |
| Coalescence time (minute) | 60 | 240 |
| MFI (g/10 min) | 27.93 | 28.25 |
| BET (multi poit) ($m^2$/g) | 1.68 | 2.42 |
| Tg ($^o$C) | 51.3 | 51.7 |

Compared with the sample made without coalescence agent, the sample A made with small amount of coalescence agent, The coalescence time needed was significantly reduced (only 1 hour coalescence for 0.964 magenta particle circularity, compared with current 4 ~ 5 hours) at even lower coalescence temperature (94 $^o$C). The resulted toner particles also show improved GSD.

**Sample # 2:** Black toner particles were prepared with or without coalescent aid agent Velta® 368 (2-ethylhexyl benzoate). The comparison results were summarized in Table 2.

***Table 2: Comparison of Black Parent Particle Properties with and without Coalescence Aid Agent***

| Samples | Sample #2 | Comparative sample#2 |
|---|---|---|
| Coalescent | Velta® 368 | No |
| Particle Size (μm) | 5.98 | 5.95 |
| GSDv | 1.22 | 1.231 |
| Circularity | 0.976 | 0.960 |
| Coalescence Temp. ($^o$C) | 94.0 | 96.0 |
| Coalescence time (minutes) | 60 | 150 |
| MFI (g/10 min) | 29.21 | 28.95 |
| BET (multi poit) ($m^2$/g) | 1.42 | 1.49 |
| Tg ($^o$C) | 50.2 | 50.4 |

Similar to sample #1, compared with standard EA process (without coalescence agent), the process with small amount of coalescence aid agent, The coalescence time needed was significantly reduced at even lower coalescence temperature (94 $^o$C). The resulted toner particles also show improved GSD and BET. And the impact of the small amount of the coalescence agent added during the AC process is minimal on the toner properties, as suggested by the MFI and Tg data.

**Toner particle preparation with reactive coalescence Aid Agent**

Reactive coalescent Archer RC (propylene glycol monoester, nonvolatile coalescent) was selected as the reactive coalescent. Compared with regular (non-reactive volatile coalescent), this reactive coalescent has a head group similar to the industry standard trimethylpentanediol monoisobytyrate, but the long fatty acid chain makes it reactive and nonvolatile, significantly improve the coalescing aid functionality and reducing VOC emissions.

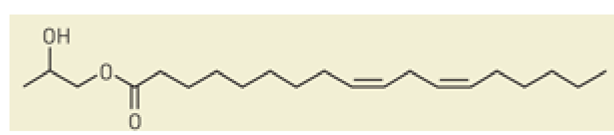


***Figure 2:*** *Chemical structure for reactive coalescent Archer RC: Propylene Glycol Monoester*

In a 2L stainless steel reactor, 258 grams styrene-butyl acrylate latex, 80.0 grams pigment dispersion PR122, 20.1 grams PR185 pigment dispersion, Wax Dispersion 60 grams, and de-ionized water 670 grams were mixed by a homogenizer for 15 minutes at 20ºC. A flocculate agent was added dropwise in eight minutes. The resulted viscous mixture was continuously mixed by homogenizer for another 20 minutes. Then the mixture was stirred by a mechanical stirrer at 550 rpm, and the temperature of the mixture was raised to 50ºC in about 35 minutes. After the particles reach at 6.2 micron, 140 grams shell latexes (same as the core latex) were added dropwise in 10 minutes. After particle size reached at 7.2 microns, a freeze agent was introduced into the reactor to stop the particle growth. Then certain amount of reactive coalescent Archer RC was added in, and hold for another 20 minutes. The temperature of the mixture was raised to 94ºC in 35 minutes, and hold for 60 minutes before cooled down to 35ºC. Then the particles were washing with DI water and dried. The final toner product has a volume median particle size of 7.06 microns, with circularity 0.978, GSDv 1.193.

Comparative Sample #3: A magenta toner particle was prepared using the same formulation and process conditions as the above sample, except that no coalescent added, higher coalescence temperature (96ºC) and longer coalescence time (4.5 hr). The same circularity 0.971 was achieved after 4.5 hours, with a particle size of 7.04 microns, GSDv 1.255. More comparisons are listed in Table 3.

***Table 3: Comparison of Magenta Toner Particle Properties with and without Reactive Coalescence Aid Agent***

| Samples | Sample #3 | Comparative sample #3 |
|---|---|---|
| Coalescent | Archer RC | No |
| Particle Size (μm) | 7.06 | 7.04 |
| GSDv | 1.193 | 1.255 |
| Circularity | 0.978 | 0.971 |
| Coalescence Temp. ($^{o}$C) | 94.0 | 96.0 |
| Coalescence time (minutes) | 60 | 270 |
| MFI (g/10 min) | 8.9 | 8.99 |
| BET (multi poit) ($m^2$/g) | 1.45 | 2.38 |
| Tg ($^{o}$C) | 41.2 | 34.6 |

Compared with standard EA process (without coalescence agent), with small amount of reactive coalescence agent, the coalescence time needed was significantly reduced (only 1 hour coalescence for 0.978 magenta particle circularity, compared with current 4 ~ 5 hours) at even lower coalescence temperature (94 $^{o}$C). The resulted toner particles also show improved GSD and BET. And, the MFI are almost the same as the toner particle prepared without coalescence agent, indicating that the impact of the small amount of the coalescence agent added during the AC process is minimal on the toner properties.

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## Author Biography

*Dr. Jerry Lai holds Bachelor, Master and Ph.D degree in Chemical Engineering. He is current work for Xerox Corporation in developing and delivering chemically Produced Toners for digital printing application. Jerry has extensive background and experience in polymer colloids synthesis and characterization, colloidal surface and engineering. Dr. Lai is Certificated Black Belt for Design for Lean Six Sigma, who has more than 15 patents pending in chemical toners.*