# Encapsulated Emulsion Aggregation Toner for High Quality Color Printing

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## Abstract

Emulsion aggregation toner (E/A Toner) has been developed for digital color copiers & printers.

Emulsion aggregation toner has been prepared by using the following process steps: pigment/wax dispersion preparation, emulsion polymerization, homogenization, aggregation, and coalescence. Shape optimized toner particles are obtained by the control of temperature, pH and time.

The emulsion aggregation process with stepwise aggregation in particle formation provides a capsule structure and it also exhibits narrow particle size distribution.

Narrow size distribution can be obtained by optimization of the latex size and Zeta potential, controlled in the emulsion polymerization step.

This study shows the surface controlled toner can provide 2 important advantages for high quality color printing compared to conventional pulverized toner. The first is a method to control the charging of different colors by uniform surface composition. The same level of charge is obtained for each color as well as stable print quality under various conditions. Charging properties can also be controlled by metal ion treatment in the toner-washing step. The second advantage is the prevention of surface wax, which enables high toner flow, even for small particle sizes about (5-micron particle). This process also enables compatibility with an oil-less fusing system and excellent image quality with lower toner mass per unit area even for rough paper surfaces.

## Introduction

Digital color Xerography requires small and narrow size distribution for toner to achieve high image quality. The importance of chemical toner technology has been increasing as a method to provide above characteristics to Xerography. In addition, the capability of structure control realized by chemical toner processing provides more improved basic performance compared to conventional toner.

As a chemical toner technology, this paper reports the features and evaluation results of emulsion aggregation toner that has encapsulated structure for color printing.

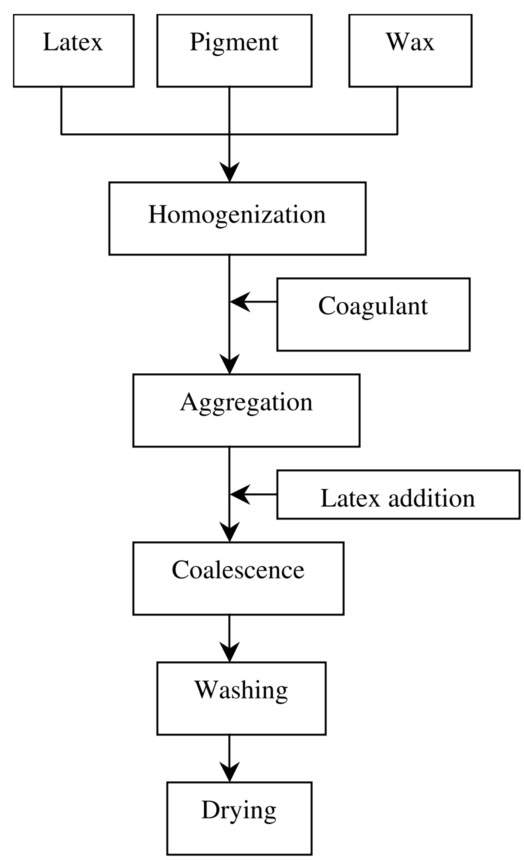



*Figure 1. Process flow of emulsion aggregation toner*

## Preparation Method of Emulsion Aggregation Toner

### 1. Emulsion Polymerization

Styrene, Acrylate ester and acid monomer are added into hot de-ionized water with anionic surfactant, initiator and chain transfer agent and polymerized. The latex obtained is about 200 nm in size and anionicly charged.

Latex size can be controlled by changing the surfactant concentration.

### 2-2. Pigment Dispersion

Black, cyan, magenta and yellow pigments are dispersed in de-ionized water with small amounts of anionic surfactant.

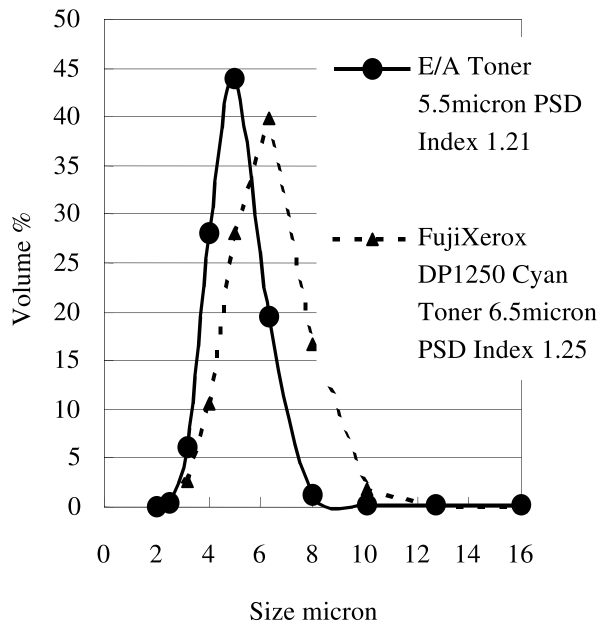



*Figure 2. Particle Size Distribution*

### 2-3.Wax Dispersion

Low molecular weight polyethylene wax is dispersed in de-ionized water with small amounts of anionic surfactant by heating under high shear mixing.

### 2-4.Toner Particle Preparation

Latex, pigment and wax are mixed in de-ionized water and a metal compound ascoagulant is added into the mixture. The mixture is homogenized with a high shear mixer and the aggregated average size is 2.5 micron after the homogenization.[1]

The homogenized mixture is heated with continuous mixing in the reactor and with temperature ramping, the aggregated particles are observed to grow in size. When the aggregated particle grows to 4.8 micron, latex is added to the mixture for shell formation. After the latex addition, the aggregated size is about 5.5 micron.[2]

The pH of the slurry is controlled to freeze the aggregated size and then the mixture is ramped to 90C and the temperature held for 4hrs with mixing. After cool down, the filtered coalesced particle is washed with de-ionized water and dried. Based on the above procedure, 4 colors of semi-spherical shaped 5.5 micron toners were obtained. Fig.1 shows the schematic flow of toner preparation.

## Particle Size Distribution

Emulsion aggregation toner shows narrow particle size distribution, compared to conventional pulverized toner, without a classification process step. (Figure 2) Particle size distribution (P.S.D) strongly depends on the latex particle size. Figure 3 and figure 4. show the relationship between P.S.D index (root of D16/D84), latex size and Zeta potential of latex. In the case of smaller latex size and lower Zeta potential, addition of coagulant tends to generate the high viscosity state and prevents uniform temperature conditions under mixing. Larger latex size is better to obtain narrower particle size distribution. Zeta potential can be controlled by the acid monomer and surfactant content.[3]

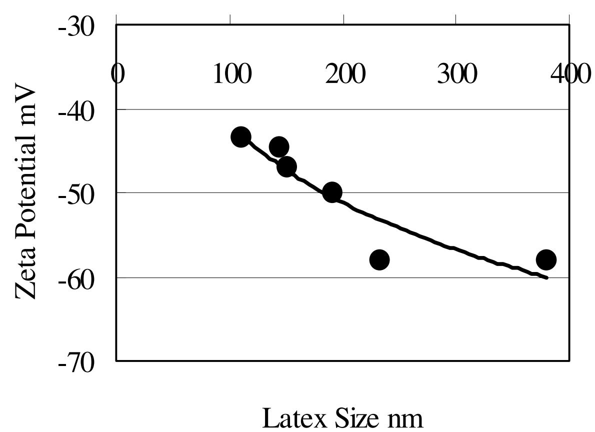



*Figure 3. Relation between Zeta potential and Latex size*

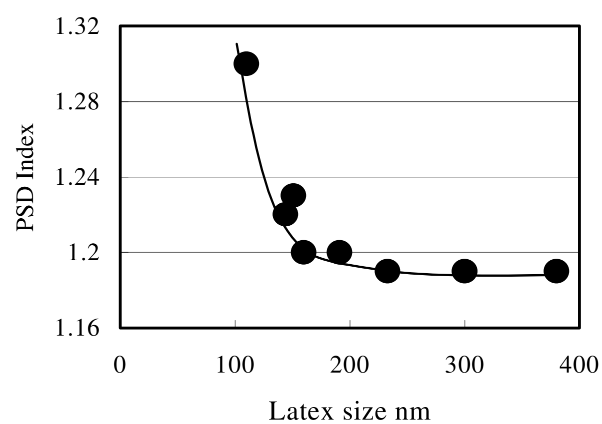



*Figure 4. Relation between PSD Index and Latex size*

## Particle Observation of Encapsulated Toner

Based on the above preparation method, 4 kinds of black toner were prepared with different level of shell latex content. Fig. 5 is the transparent electron microscope (TEM) photograph of toners with 0% and 40% shell latex content.

Carbon black can be observed in the cross-sectional image and in the case of 40% shell latex, a thick surface layer without pigment is observed. Fig. 6 shows the relationship between free carbon content quantified by the sonification of toner in water and optical density measurement of the supernatant after centrifuging. Shell formation by the step-wise aggregation method prevents the surface pigment or free pigment in the toner. By theoretical calculation, over 20% shell content can form over 2 layers of latex particles on the toner surface.

Surface carbon has a large impact on charging and electrical properties of the toner. Shell formation can minimize the difference of these properties between different colors.

In the scanning electron microscope photograph of a toner with greater than 28% shell content, surface pigment is not observed and a considerably smoothed surface is obtained.

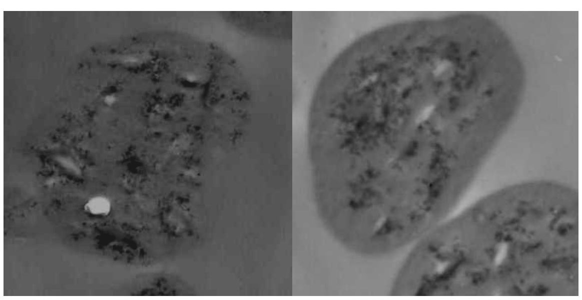



*Figure 5. TEM Photograph of Emulsion Aggregation Toner*

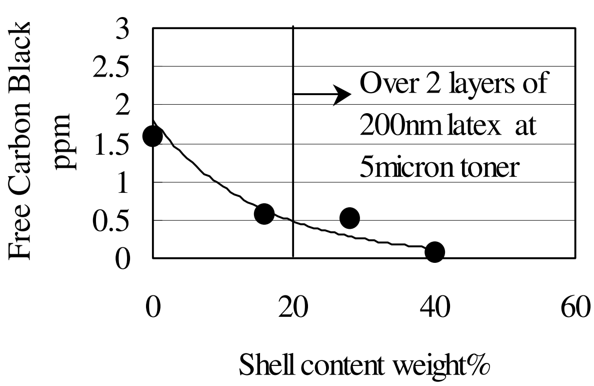



*Figure 6. Relation between Shell content and Free Carbon Black on Toner surface*

## Charging Properties and Control Method

In conventional toner, the charging property is dependent on each color because of the presence of surface pigment. Fig. 7 shows the comparison of blow off tribo charge value between conventional color tones for Fuji Xerox DP1250 (6.5um D50 about 6.5 micron) and encapsulated emulsion aggregation color toners in 2 environmental conditions(H/H means High temperature & High RH,L/L means Low temperature Low RH) Encapsulated E/A toner showed considerably higher charge and small differences in charge value for each color compared to conventional toners.

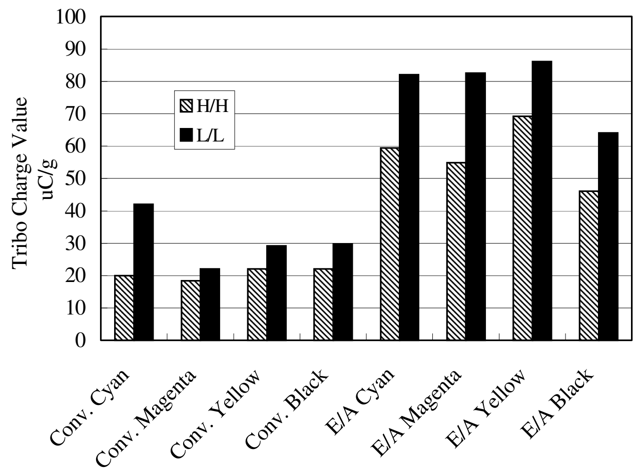



*Figure 7. Charging properties of conventional Toner and E/A Toner without surface additive*

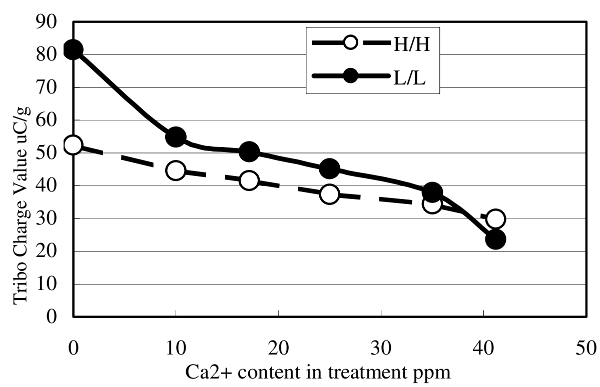



*Figure 8. Relation between Tribo Charge Value and Ca2+ Treatment - Magenta Toner-*

Use of metal ion addition before filtration in the washing procedure can control the charge level of E/A toners. Fig. 8 shows the relationship between the tribo charge value and added amount of Calcium salt to toner slurry before filtration. Charging of this toner depends on the surface acid group but the calcium ion can form a low soluble salt in water with the acid groups of the toner surface. Therefore addition of similar multi valent metal ions ($Mg^{3+}$, $Fe^{2+}$..) show similar effects.[4]

It can be assumed that Encapsulated E/A toner has the possibility to provide ease of use in hardware designs and to achieve stable print quality.

## Wax Structure and Oil-less Color Fusing

The encapsulation process can also prevent surface wax on the toner and this provides better flow for small size toners. Even if this chemical toner has smaller particle size and higher wax content, flow is better than that of large size conventional toner with wax. In the case of toner with additives, this toner shows considerably better flow because of its smooth surface and spherical or semi-spherical shape.[5]

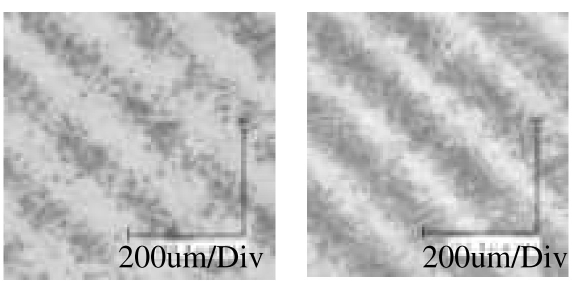
Generally, high molecular weight polymer designs can easily provide oil-less fusing because of the lower release force from the coated heat roll in fusing due to the high visco- elasticity.

But in that case, gloss of the image is necessarily decreased and lower gloss impacts the color image quality, specifically, poor color gamut.

By the reduction of the releasing force with a high content (about 10%) of low molecular weight polyethylene wax incorporation, emulsion aggregation toner can achieve the compatibility between oil-less performance and optimized image gloss.

## Image Quality Evaluation

Small particle size, narrow size distribution and spherical or semi-spherical shape can achieve high quality transfer performance in the Xerographic process. Figure 9 shows the photograph of magnified fine line image on rough paper:Xerox 4024 paper. Compared to conventional toner:DP1250, Emulsion Aggregation Toner shows considerably better reproduction.




Conventional Toner E/A Toner

*Figure 9. Line image reproduction on rough paper*

Fused gloss behavior of conventional polyester based toner strongly depends on paper roughness and toner mass per unit area. By optimization of the polymer latex design without being restricted to the resin pulverizability restriction like conventional toner, an optimized glossy image can be achieved. In that case, emulsion aggregation toner provides a uniform gloss image with little paper quality dependence.

Figure 10 shows the comparison of color gamut. Fusing property and pigment optimization enables the compatibility of wider color gamut and better reproduction performance.

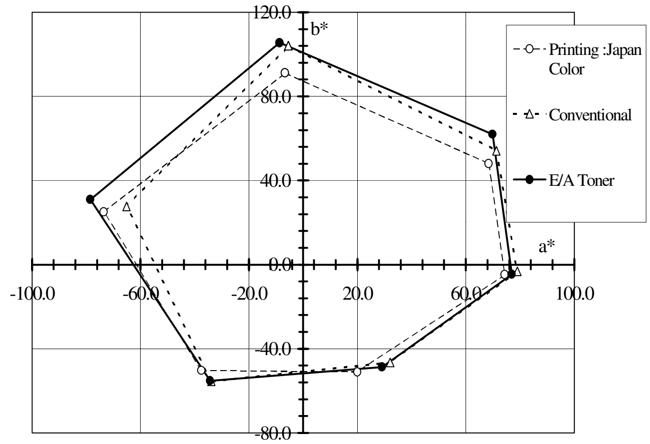



*Figure 10. Color gamut of conventional & emulsion aggregation toner FujiXerox JC Paper*

## Conclusion

Encapsulated Emulsion Aggregation Toner has been developed. Features of this toner are as follows,

1. Narrower size distribution achieved by the latex size and Zeta potential control.
2. Encapsulated structure can control surface morphology of the toner and minimizes the influence of pigment type.
3. Use of multi-valent metal cation addition can easily control the charging level of the toner.
4. High color image quality could be achieved by fine image reproduction and wide color gamut based on small particle size, narrow distribution and the optimization of fusing properties with low molecular weight polyethylene wax incorporation.

## References

1) USP 6,153,346
2) USP 5,849,456
3) Patent pending
4) Patent pending
5) N. Yanagida, T. Takasaki and Jun Hasegawa, *Proceedings of PPIC/JH'98*, 46(1998)

## Biography

Yasuo Matsumura received his Master's degree in polymer chemistry from Kyoto university in 1981. Since 1981 he has worked in the marking material development division at FujiXerox Co.,Ltd. His work has mainly focused on conventional and chemical toner technology development and evaluation. He is a member of the Imaging Society of Japan.