

New Characteristic Additives designed for Imaging Toner

- Fine Metallic Soaps & High-Purified Solid Esters -

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Abstract

Fine metallic soap particles and high-purified solid esters are the first products in the world. These products are produced by the latest production technologies, and give various interesting features to toner. Fine metallic soap particles are thin and smooth, and have extremely narrow particle size distribution. Further, average particle size of fine metallic soap particles is controlled 0.1~3 μm (in the case of zinc stearate or calcium stearate). These particles can uniformly coat resin particles as toner, and give high fluidity and anti-blocking characteristic. On the other hand, high-purified solid esters have 60~85°C melting point and quite narrow melting temperature range. Accordingly, these esters give low-temperature fixing and good keeping stability for toner. Further, these esters include little impurities. So starting temperatures of decomposition are very high. Accordingly, These esters are difficult to product decomposition gas at fixing temperature of toner.

I. Introduction

In recent year, much higher performance is required for copying apparatus such as laser beam printer with high-speed, high-quality, low-energy, and full color printing. For these requirements, improvement of functions as low-temperature fixability and anti-high temperature offset characteristic have been in demand for imaging toner. Further, miniaturization of imaging toner particle is increasing in recent years. These fine toner particles are suited for high quality imaging and full color printing. But these particles agglutinate easily. Therefore, it is necessary to add high performance anti-blocking characteristic. Many solid wax additives (polyethylene wax, polypropylene wax, natural wax, metallic soaps, Fatty alcohol *etc.*) are used for imaging toners as release agent and anti-blocking agent. These additives give good fixability and fluidity for imaging toner. However, these general solid waxes don't sufficiently satisfy of high performance anti-blocking characteristic, low-temperature fixability and anti-high temperature offset characteristic to high performance toner. Fine metallic soap particles and high-purified solid esters

were developed for high performance toner. For example, fine metallic soap particles have characteristic average particle size, particle size distribution and particle configuration. Furthermore, high-purified solid esters have high melting point, very narrow melting range, high hardness, quit low volatile, and high heat-resistance. These products are manufactured by w production process technologies of oleo chemical derivatives, and provide various interesting functions (extremely high lubricity, fluidity, mold releasably, and anti-blocking characteristics) for micro particles as high performance toner.¹

II. Fine Metallic Soap Particles

Metallic soaps are fatty acid metal salt except alkaline metal salt. Metallic soaps have a combination of organic function and inorganic function, characterized by fatty acid chain site and metal salt site. The fine metallic soap particles have a very fine particle sizes, narrow particle size distribution, and characteristic figures. The fine metallic soap particles are the first products in the world on the basis of new production process technology (Jet-mixing process; Figure 1). The jet-mixing method doesn't use pulverizing and classifying.^{2,3}

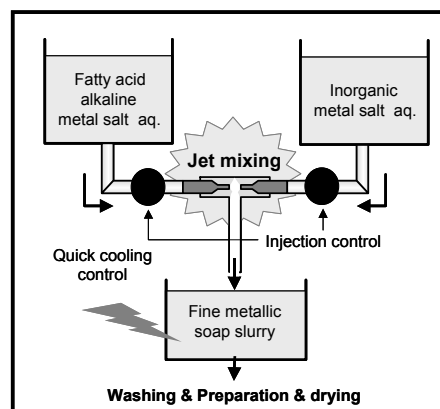


Figure. 1. The image showing new process for producing the fine metallic soap particles (Jet-mixing process).

In this respect, the fine metallic soaps are available for high functional materials.

Particle Size Distribution

Particle size distributions of zinc stearate fine particles and general zinc stearate particles are shown in Figure 2. Compared with general zinc stearate particles, Zinc stearate fine particles are extremely fine and have narrow particle distribution. The average size of zinc stearate fine particles in Figure 2 is about $1\mu\text{m}$ and maximum particle size is about $2\mu\text{m}$. If Jet-mixing process is used, other fine metallic soaps (calcium salt, magnesium salt *etc.*) can be produced and the average particle size can be controlled from $0.1\mu\text{m}$ to $3\mu\text{m}$ (zinc stearate).

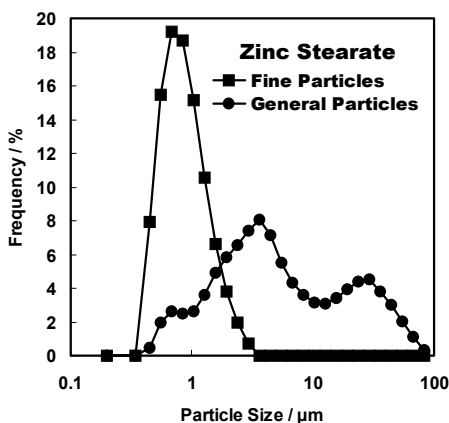


Figure 2. Particle size distributions of fine zinc stearate particles and general zinc stearate particles.

Figure of Fine Metallic Soap Particles

General metallic soaps consist of large and coarse particles by pulverizing and classifying. On the other hand, fine metallic soap particles are very fine and have smooth surface compared with general metallic soaps. The scanning electron micrographs of fine metallic soap particles (zinc stearate and magnesium stearate) at magnification of $10,000\times$ are shown in Figure 3.

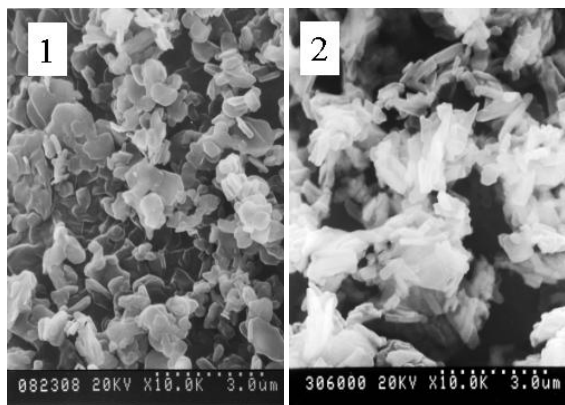


Figure. 3 Scanning electron micrograph images for fine zinc stearate particles (1) and fine magnesium stearate particles (2) at a magnification of $10,000\times$

From these figures, shapes for particles of fine metallic soap particles are very thin and smooth respectively. Furthermore, fine particles of calcium stearate have same figure of zinc stearate fine particles and fine magnesium stearate is finger-shaped and thin particles having high aspect ratio.

Usefulness of Fine Metallic Soap Particles

Fine metallic soap particles have very fine particle size and disk-shape figure. Then these particles have wide surface area, so these particles have high absorption for non-polar substance dramatically. Consequently, fine metallic soap particles are attached and uniformly coated to surfaces of particle as micro toner. The scanning electron micrographs of poly-MMA particles (average particle size: $5\mu\text{m}$) mixed with fine zinc stearate particles (average particle size: $0.1\mu\text{m}$) at magnification of $10,000\times$ are shown in Figure 4. These poly-MMA samples are mixed by ball-mill at 300min. The metallic soaps are contained 10% by weight to poly-MMA particles.

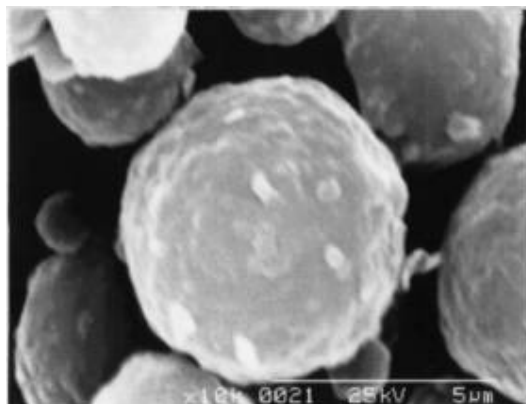


Figure 4. Scanning electron micrograph images for Poly MMA particles containing 10% zinc stearate particles ($0.1\mu\text{m}$) by weight at a magnification of $10,000\times$

When Poly-MMA particles and fine metallic soap particles are mixed, Poly-MMA particles are coated by metallic soap particles. If general metallic soaps are used, this phenomenon cannot create. Especially, if $0.1\mu\text{m}$ zinc stearate particles and Poly-MMA particles are mixed, Poly-MMA particles surfaces are uniformly coated by zinc stearate particles in small amount, and the resin particles surfaces are extremely smooth.

These coated Particles have high fluidity, anti-blocking characteristic, and generate high electrostatic property. These characteristics are very effective functions for toner. Furthermore, fine metallic soap particles give other many characteristics to solvent, film, metal particles *etc.* For example, fine metallic soap particles give high viscosity for non-polar solvent, and give fluidity for inorganic particles.

III. High-Purified Solid Esters

Solid esters are fatty acid derivatives formed by long chain fatty acid and alcohol. They have been used as a lubricant for resin, a mold-releasing agent, a lubricant for the surface of sheets, *etc.* High-purified solid esters are produced with new production and purification process technologies of solid esters. Accordingly, high-purified solid esters have various interesting properties characterized by quite narrow melting range, low endothermic energy at melting, high thermostability, low quantity of thermolysis sublimate *etc.* In recent year, the lower temperature fixing for copying apparatus is increasing according with the lower power consumption, and the simultaneous pursuit of low temperature fixing and good keeping stability is required to high quality toner now. But, traditional common toner waxes aren't use for future generation toner, because, these waxes have high melting point, and wide melting range, so these waxes cannot combine the lower temperature fixing and high keeping stability. High-purified solid esters have a sharp-melting characteristic and melting point are 60~85°C. Furthermore, these ester are colorless and have low viscosity at melting state. So high-purified solid esters give quick fixability and high transparency to future generation toner. These behaviors are caused by high purity of solid esters. These esters include very little impurities like fatty acid, alcohol, and reaction solvent. Accordingly, these esters are chemically stable, and give reproducibility to emulsions and suspension that is using chemical toner.

Differential Scanning Calorimetry (DSC) Analysis

Thermal absorption curves of High-purified solid esters 1~5 measured by Differential Scanning Calorimetry (DSC) are showed in Figure 5. High-purified solid esters 1~5 have narrow thermal absorption range and have single thermal absorption peak between 60°C and 85°C respectively.

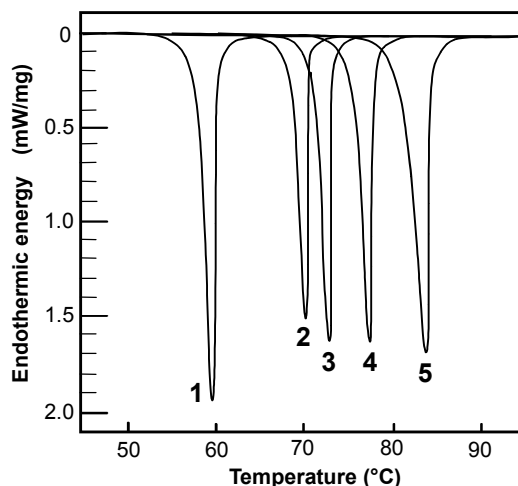


Figure 5. Thermal absorption curves of High-purified solid ester 1~5 measured by Differential Scanning Calorimetry (DSC). The increasing Speed in temperature is 2 °C/min

Next, Thermal absorption curves of High-purified solid esters 5 and general solid ester 5', which has same molecular structure of esters 5, measured by DSC are showed in Figure 6. Acid value and hydroxyl Value of solid ester 5 are 0.1 mgKOH/g and 2.3 mgKOH/g respectively. On the other, these values of solid ester 5' are 10 mgKOH/g and 6.0 mgKOH/g respectively. Then, the solid esters 5 is purified higher than 5', and has more sharp-melting characteristic compared with solid ester 5'.

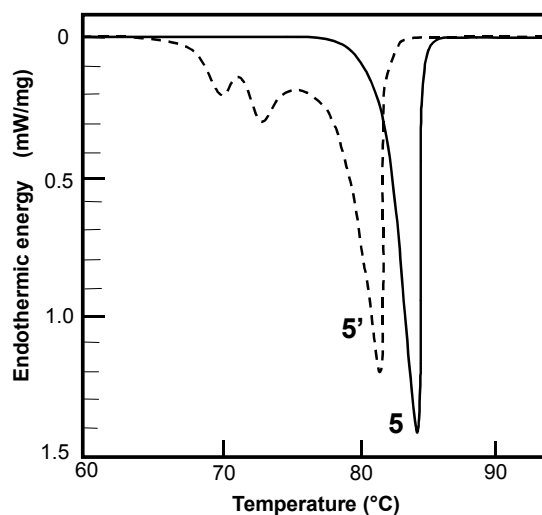


Figure 6. Thermal absorption curves of High-purified solid ester 5 and general solid ester 5', which has same molecular structure of esters 5, measured by Differential Scanning Calorimetry (DSC). The increasing Speed in temperature is 2 °C/min

High-purified solid esters have sharp-melting characteristic, and melting temperatures of these ester are between 60°C and 80°C. These characteristics result from high purities of esters. Accordingly, toners including these esters combine the low temperature fixing and the high keeping stability.

Thermal Gravimetric Analysis (TGA)

Thermal gravimetric curves of High-purified solid ester 2 and general solid esters 2', which has same molecular structure of esters 2, measured by differential gravimetric analysis (TGA) are showed in Figure 7. Acid value and hydroxyl Value of solid ester 2 are 0.1 mgKOH/g and 1.9 mgKOH/g respectively. On the other, these values of solid ester 2' are 12 mgKOH/g and 4.9 mgKOH/g respectively. So the solid ester 2 is purified higher and has higher decomposition starting temperature (300°C). Further, solid esters 2 has simple decomposition characteristic.

Starting temperatures of decomposition for high-purified solid esters 1~5 are higher than general ester which have same structures. Accordingly, If the toner including esters 1~5 are fixed by printer, these purified esters in toner don't produce a decomposition gas.

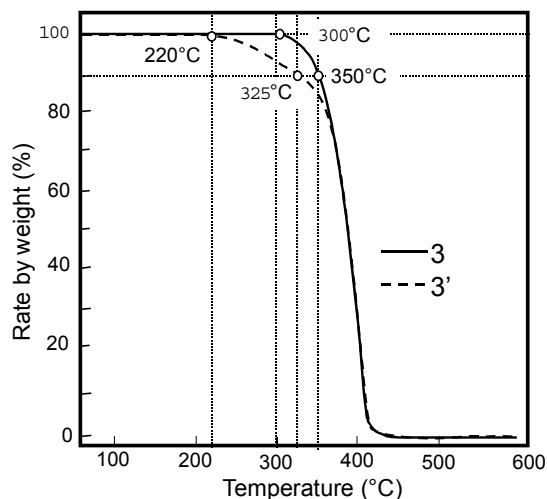


Figure. 7 Thermal gravimetric curves of High-purified solid ester 2 and general solid esters 2', which has same molecular structure of esters 2, measured by differential gravimetric analysis (TGA). The increasing Speed in temperature is 5°C/min

Usefulness of High-Purified Solid Esters

High-purified solid esters have very sharp melting range and higher decomposition point. Furthermore, These solid esters have higher hardness, colorless, and lower viscosity when melted. These characteristics of purified ester give very effective functions to future generation toner.

Conclusion

1. New functional materials (Fine metallic soap particles and high-purified solid ester) were created by new production process technologies.
2. Fine metallic soap particles are very fine, thin, and have extremely narrow particle size distribution. The average particle size of these particles can control, for example, Zinc stearate fine particles are controlled from 0.1 to 3 μ m.
3. Fine metallic soap particles can uniformly coat for surface of resin and inorganic particles, and give very effective functions as fluidity, anti-blocking characteristic, generate electrostatic charges.

4. High-purified solid esters have very narrow melting range and melting points of these esters are 60~85°C. Accordingly, toners including these esters combine the low temperature fixing and the high keeping stability.

5. High-purified solid esters include little impurities. So starting temperature of decomposition for these esters are very high. Accordingly, these esters don't product a decomposition gas at fixing temperature of toner.

References

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Biography

Kouhei Sawada graduated from the Faculty of Science, Ehime University with a degree of master in science and joined NOF Corporation in 1992. Then, He was re-admitted to the Faculty of Science, Ehime University in 1997, and graduated with a degree of Doctor in science in 2000. He has been researching about the structure design for fatty acid derivatives with new functions, and about the relationship between functions and structures. Most recently, He is studying about applications to the metallic soaps and solid esters with new functions.

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