



CHAPTER I

INTRODUCTION

1.1 Scientific Background and Rationale

Polymerized toners have attracted much attention recently because the demand for fine image and high resolution with their uniformity in electrophotography has increased ever since full-color copiers and printers employing an electrophotographic system have been introduced. To achieve high resolution images in electrophotography, processes and materials involved in electrophotography must be improved. Enhancement of image quality requires that toners have a very small particle size within a narrow size distribution.

Since toners were introduced in the mid of 1950, there has been a steady demanding trend toward the smaller particle size. The average particle size of toner before 1980 was over 13 μm . The particle size was reduced to approximately 11 μm in 1990s, and if prediction holds, by the end of the 1990 decade, the technology for producing toner should have been improved, most toners should have particle sizes from 3-8 μm .¹ The smaller toner particles are necessary to take advantage of image quality because they have given better print images with higher resolution and less roughness. Its smaller particle size together with its uniform size distribution makes it possible to impart a much more even charge on the particles and the particles become easier to control within the machine as they are transferred onto the paper. So, the particle size and

size distribution are important parameters strongly influencing the quality of toners.

The size of currently used toner particles generally ranges from 7 to 10 μm . Conventionally, most toners are currently produced by a pulverization method from which charge controlling agents, colorants, modifiers, and other additives are dispersed in a toner resin matrix, followed by cooling, crushing, pulverizing and classifying the pulverized material to separate toner particles having an intended particle size. Such a production process produces a toner of sufficiently high quality but accompanied with potential problems such that the selection of the material therefore is rather limited. For example, the colorant-containing resin composition should be brittle to be pulverizable economically by a pulverizing machine. However, the colorant-containing resin composition, which has been made brittle tends disadvantageously to have broader particle size distribution after a high speed pulverization to contain relatively larger particles. Moreover, such a brittle toner material tends to be further crushed or pulverized during image development. In this pulverization process, it is extremely difficult to uniformly disperse solid fine particles such as magnetic powder or colorant in a resin. The insufficient dispersion can cause increased fogging, lower image density, low toner color mixing characteristics, or lower transparency. A colorant, which is uncovered on the cleavage surface of the toner, may cause variation of development characteristics of the toner.²

The conventional toner manufacturing process results in toners with a relatively wide size distribution. Many toner particles will be significantly larger or smaller than average. These toner particles will also be irregular in shape. The irregular size and

shape of the toner particles make it difficult to put a uniform charge on them. As printer resolution increases, smaller toner particles are necessary to take advantage of increased resolution. However, small toner particles are specially difficult to charge evenly. Without a uniform charge, toner particles become very difficult to control in the machine and dusting can be problematic. In addition to dusting, another problem that results from non-uniform charging can be low toner yield, in either the development or transfer stage. At the extreme, this can result in void areas when printed. Also, toner particles, when fall outside of the size specification must often be discarded, increasing waste and reducing manufacturing yields. Finally, conventional toner manufacturing has a sharp increase in production costs. The reasons for the sharp rise in cost are the exponential increase in energy needed for pulverization, and the decrease in collection efficiency during classification.

To solve the problems involved in the toner produced by the aforementioned pulverization, the production of toner using a polymerization method is attractive. Generally, polymerization methods such as a suspension polymerization, an emulsion polymerization and a dispersion polymerization are known as the appropriate methods for producing toner.³ Each of these polymerization methods has its own advantages and disadvantages, therefore, selection of the polymerization method depends on the performance expected for the toner particles. In the dispersion polymerization method, it is easy to synthesize particles having an average particle size of $7\ \mu\text{m}$ or less and a narrow particle size distribution, but it is difficult to retain inner additives inside each particle. Toner particles produced through the emulsion polymerization method are generally having the particle size of submicrometers or less. In addition, polymer particles

from the emulsion polymerization method must be increased to the actual size of toner commercially used, because the emulsion type particles are in the range of hundred nanometers. In the emulsion polymerization method, there are two methods for growing the particles. One is a seed polymerization method and the other is a coagulation method, both of which are rather complicated and require numerous time-consuming reaction steps and procedures.

Suspension polymerization is the process overcoming aforementioned problems because of the simplicity of the reaction process and equipment. The advantages of suspension polymerization including, low viscosity throughout, easily thermal control, a variety of choices of vinyl monomers. Additionally, the polymer particles can be modified by functional groups with various rheological properties. This system consisted of a water-insoluble monomer as a raw material of a fixing resin, a monomer-soluble initiator, a crosslinking agent, a charge controlling agent, and other additives, is mixed to form monomer droplets to be polymerized in an aqueous medium containing a dispersion stabilizer with agitation by a suitable mixer to form toner particles having a desired particle diameter.

In this research, the preparation of polymerized toner by suspension polymerization for electrophotographic printers is described. To fulfil the interest in micrometer-sized particles with a narrow particle size distribution, a typical by mechanical homogenizer is used for suspension polymerization to generate small, uniform, and well controlled droplets of less than 10 μm . In addition, to achieve the polymerized toners with desired properties, the influence of different reaction parameters on particle sizes, particle size distribution and thermal properties is investigated.

1.2 Objectives of the Research Work

1.2.1 To prepare the micrometer-sized polymerized toner by suspension polymerization with the following properties: average particle size distribution of 4-10 micrometers, glass transition temperature 50-70°C, and average molecular weight 40,000-200,000.

1.2.2 To characterize properties of the micrometer-sized polymerized toner by suspension polymerization method by physical methods and imaging test.

1.3 Scope of the Research Work

In this research work, the focus is to synthesis and characterization of the micrometer-sized polymerized toner by suspension polymerization for electrophotographic printers. To achieve the goal, the necessary procedures may be as follows:

1.3.1 Literature survey and in-depth study for this research work.

1.3.2 Synthesizing the copolymer of styrene and normal butyl acrylate with controlled average particle size in the range of 4-10 micrometers with a narrower size distribution using the suspension polymerization technique by varying the following parameters:

- a) The influence of the initiator concentration on the particle size, size distribution, average molecular weights and molecular weight distribution.
- b) The influence of the reaction temperature on the particle size, size distribution, average molecular weights and molecular weight distribution.

- c) The influence of the monomer feed ratio on the particle size, size distribution, average molecular weights, molecular weight distribution and glass transition temperature.
- d) The influence of the dispersant concentration on the particle size, size distribution, average molecular weights and molecular weight distribution.
- e) The influence of the mechanical homogenizing speed on the particle size, size distribution, average molecular weights and molecular weight distribution.
- f) The influence of the agitation rate on the particle size, size distribution, average molecular weights and molecular weight distribution.
- g) The influence of the reaction time on the particle growth, conversion, average molecular weights and molecular weight distribution.
- h) The influence of the crosslinking agent on the particle morphology.

Then, the resulting poly(styrene-*co*-normal butyl acrylate) particles are characterized for the following properties.

- a) The functional groups are determined by fourier transform infrared spectroscopy (FT-IR).
- b) The average molecular weights and molecular weight distribution are measured by gel permeation chromatography (GPC).
- c) The average particle size and size distribution of poly(styrene-*co*- normal butyl acrylate) particles are evaluated by scanning electron microscopy (SEM).
- d) Thermal properties of poly(styrene-*co*-normal butyl acrylate are studied by differential scanning calorimetry (DSC).

1.3.3 Synthesizing the micron-sized polymerized toner by suspension polymerization. The above experiments in 1.3.2 will give an appropriate experimental condition for the preparation of polymerized toner. The various parameters were investigated as follows:

- a) The influence of the carbon black on the particle size, size distribution, average molecular weights, molecular weight distribution, glass transition temperature, the distribution of carbon black of sample bead.

Then, the resulting polymerized toners are characterized for the following properties.

- a) The average particle size and size distribution of polymerized toners are studied by scanning electron microscopy (SEM).
- b) The average molecular weights and molecular weight distribution are measured by gel permeation chromatography (GPC).
- c) Thermal properties of polymerized toners are studied by differential scanning calorimetry (DSC).
- d) The carbon black distribution of a sample bead in the polymerized toner is obtained by transmission electron microscopy (TEM).

1.3.4 Triboelectrification measurements of various polymerized toners prepared by suspension polymerization technique is carried out by varying the amount of the charge controlling agent, and the triboelectric charging properties are measured by blow-off method.

1.3.5 Evaluation of print quality with various toner conditions was prepared by a specially designed test form. The print-outs of a test form on the paper, were printed by OKI 400 micro line CL printer to elucidate the print quality on each paper. The quality of the print-out papers was evaluated as follows:

- a) The solid density at a solid area was measured by a reflection densitometer.
- b) The background density at a non-image area was measured by a reflection densitometer.

1.4 Content of the Thesis

Chapter II deals with the theoretical background and literature reviews. Chapter III gives the description on chemicals, glassware and equipments, apparatus and experimental procedures. Chapter IV contains the results and discussion in polymerizing efficiency, charging properties, and image qualities of the resulting polymerized toner. Finally, the results are conclude in Chapter V along with some possible suggestions.