Composites: Part B 64 (2014) 78-83

Contents lists available at ScienceDirect

**Composites:** Part B

journal homepage: www.elsevier.com/locate/compositesb

# Production of carbon black acrylic composite as an electrophotographic toner using emulsion aggregation method: Investigation the effect of agitation rate

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### ARTICLE INFO

Article history: Received 3 April 2013 Received in revised form 7 July 2013 Accepted 19 August 2013 Available online 5 September 2013

Keywords: A. Printing A. Polymer–matrix composites (PMCs) D. Optical microscopy

- D. Physical methods of analysis
- E. Powder processing

#### ABSTRACT

Toner as a composite of colorant, polymer and additives is a primary component of electrophotographic printing and copying processes. It can be produced by conventional mechanical milling or by more recently developed chemical polymerization methods. The emulsion aggregation is a popular chemical method that allows tight control on the size, size distribution and shape of the synthesized toner particles. These characteristics are important for producing high quality, high-resolution printed images. This research investigated the effect of agitation speed on toner synthesis using emulsion aggregation method. Particle size analysis, scanning electron microscopy, and field emission scanning electron microscopy were used to study the size, shape, and morphology of toner particles. It was found that toner particles synthesized using emulsion aggregation method is spherical in shape and decrease in size as agitation speed increases. Differential scanning calorimetry and spectrophotometric analyses results showed that the toner synthesized by this method had appropriate thermal and colorimetric characteristics as compared to an industrial toner.

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## 1. Introduction

A typical standard laser printer works using the basic principle of electrophotography [1,2]. In this process, polymeric toner powder is picked up and precisely deposited on a substrate, usually paper, by electrostatic forces [3,4]. The toner is a dry pigmented composite powder with a particle size of  $8-12 \mu m$  composed of [2]:

- Polymer to enable rapid thermal fusing.
- Colorant (carbon black) to provide the desired toner color.
- Charge control agent (CCA) to provide charge characteristics of the toner [5].
- Flow control additives (fumed silica) to prevent the toner from caking.
- Wax to prevent the toner from sticking to the heated fuser rollers [3].

Four basic color toners, black, yellow, cyan, and magenta, are normally used in a standard color laser printer [1]. The laser-based electrophotography process requires five steps to complete one printing cycle:

http://dx.doi.org/10.1016/j.compositesb.2013.08.076 1359-8368/© 2014 Elsevier Ltd. All rights reserved.

- 1. The photoreceptor surface is charged using a corona discharge device [6].
- 2. The charged surface is exposed to a light source of suitable wavelength, selectively discharging areas of the photoreceptor.
- 3. Toner is brought close to the photoconductor and adheres selectively to the discharged areas of the surface.
- 4. Toner is transferred from the photoconductor drum to the paper by the application of an electrostatic field.
- 5. Toner is fused thermally to fix it to the paper [2-4].

The toner is formulated to be compatible with the properties of the particular printer, including toner cartridge configuration, image development, transfer mechanism, and fusing technique [2,7]. The toner can be produced either by conventional mechanical milling (pulverization) or by more recent chemical polymerization techniques [1,3,4].

Chemically produced toner (CPT) has attracted a great deal of attention in both academic studies and industrial applications in the field of digital printing [3,4]. Depending on the type of the chemical process utilized for preparation of the toner, CPT can be produced via suspension polymerization [8], dispersion polymerization [9], emulsion aggregation [10], or chemical milling [11]. Chemical methods offer advantages over conventional toner production techniques, such as small toner particle size, narrower





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particle size distribution, higher toner transfer efficiency in the machine, and better image quality [12]. Environmental issues are also a consideration in the development and improvement of chemical methods [4].

One of the biggest limitations of conventional mechanical milling is the minimum particle size. Particle size is important because it limits the device resolution and mechanical milling limits the minimum particle size to about 7  $\mu$ m. Theoretically, for the perfect reproduction of dots and print features at 600 dpi, a particle size of about 5  $\mu$ m is required and, at 1200 dpi, about 3  $\mu$ m is required [11]. In addition to the limitation of mean particle size, mechanically milled toners exhibit a wide distribution of particle sizes and shapes that adversely affects image quality. One solution is the use of chemically prepared toner [12].

Of the chemical production processes, emulsion aggregation (EA) results in better-controlled toner particle design, especially the tight control over morphology, particle size, and particle size distribution [1,13]. This method can deliver the desired narrow particle size distribution required for excellent color image quality [14]. Attaining a toner with small, uniform particles using EA is much more likely than with conventional mechanical processes and other chemical techniques [15].

The term "emulsion" refers to the step in which the polymer latex (usually styrene-acrylic) is formed via a chemical synthesis reaction and the term "aggregation" refers to bringing the toner components together to form spherical particles of the desired particle size [16,17]. In this process, polymer and other ingredients are mixed at elevated temperature in a mixer for a given number of hours to form the primary particles. The secondary particles are formed by agglomeration of the solid primary particles in an aqueous medium. These particles contain polymer, pigment, and/or a charge control agent. The secondary particles agglomerate further to form associated particles [1,10,14].

Shape adjustment of associated particles is mainly accomplished by controlling the temperature. Increasing the temperature to above the glass transition temperature ( $T_g$ ) controls the viscosity of the polymer and allows interfacial forces and surface tension to be used to change the particle shape. The particle shape can be changed from completely irregular to perfectly spherical. The mixture is then filtered, washed, and dried, yielding a toner ready for blending with the required additives [15,18].

The quality of digital printing with laser printers and photocopiers is determined by the properties of the toner particles (size, size distribution, shape, composition) and with physical properties of the toner, such as fixability on paper, chargeability, color, and gloss [19,20]. All of these characteristics in EA are directly related to the dispersion of the toner components and producing parameters.

Previous studies have focused on key parameters in other chemical methods, such as the concentrations of the aqueous and organic phases, coagulation agents [18,21,22], and time [10]. The present study looks, for the first time, at optimization of the agitation rate as a parameter in toner production using EA method. In this work, the agitation rate was adjusted during all phases of the synthesis process to investigate the effect of agitation speed on the structural and physical properties of the resultant toner.

#### 2. Experimental

#### 2.1. Materials

The polymer used in this study was a styrene-acrylic resin (NS88; Simab Resin Co., Tehran, Iran) with a medium pH value and  $T_g$  of 51.12 °C. A polyethylene emulsion wax (EE 95, Kala Kar Co., Tehran, Iran) and a carbon black pigment (Printex U, Degussa-Evonik, Germany) were also employed. Polyaluminum chloride was used as a coagulation agent.

#### 2.2. Preparation of toner composite

All toners in this study were prepared using the same procedure [18,10]. In step A, a 1 L beaker was filled with 24.5 g styreneacrylic latex (particle size: 220 nm), 2 g carbon black, 3 g wax, and 120 g deionized water and the contents were mixed manually at room temperature for 15 min. In step B, the contents were mixed using a homogenizer for 5 min. The mixture was continuously mixed for 60 min at room temperature in step C and a solution of 0.6 g coagulation agent in nitric acid was added drop wise over 10 min until the pH value of the mixture was adjusted to 2. A gel formed during this process, changing the viscosity of the suspension from a Newtonian water-like fluid to a shear thinning paste-like gel. In step D, the temperature of the mixture was increased to 50 °C for about 30 min while the gel was continually mixed. The mixture was held at this temperature for another 60 min in step E. In step F. the temperature of the mixture was increased to 96 °C for 30 min and, in step G, it was held at this temperature for a further 60 min.

The mixture was neutralized with sodium hydroxide solution after the temperature was increased. Finally, the mixture was cooled to 25 °C, after which the produced microparticles were isolated from the water, washed to remove divalent ions, filtered, and dried with a frizzed dryer. Fig. 1 shows the variation of pH and temperature over the course of the synthesis process of the toner.

A set of experiments were undertaken using steps a-g to explore the effect of agitation rate during all steps of the process on particle size and particle size distribution. The effect of processing conditions on morphological and other properties of the toner was also examined. Table 1 shows the defined experimental sets with variations in the agitation rate.

#### 2.3. Characterization of toner composite

The size and size distribution of the toner particles was determined using a MAL100229 particle size analyzer (Malvern, UK). In order to investigate the thermal behavior of the toner, a melting point meter (BUCHI, Switzerland) and a Pyris 6 differential scanning calorimeter (Perkin Elmer, Germany) were employed. A LEO-1530FE field emission scanning electron microscope (FE-SEM) (Carl Zeiss NTS GmbH, Germany) and a Leo 1455VP scanning electron microscope (SEM) (Oxford, UK) were used to investigate the shape and morphology of the toner particles. Color measurement of the samples was done using a SP64 spectrophotometer (Gretag Macbeth, USA). The spectral reflectance factor of

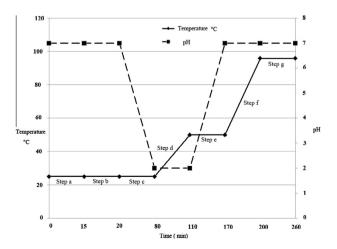


Fig. 1. The variation of pH and temperature during the synthesis process of the toner composites.

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