



# Mobility of black pigments for electrophoretic display depending on the characteristics of carbon sphere



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## ABSTRACT

A carbon sphere can be used as a black pigment in an electrophoretic display, substituting the conventional carbon black. The carbon sphere is synthesized through a hydrothermal reaction of glucose or fructose, and its characteristics are strongly dependent on the reaction parameters. The size of the produced carbon sphere simply increases with the reaction time, while the surface state related to the outermost functional group is not linearly changed. Since the electrophoretic motion of the carbon sphere is determined by the surface zeta potential of the black pigment, the impact of both size and surface chemistry on the surface zeta potential should be investigated. In this study, the changes in the size and surface state of carbon sphere according to the reaction time are examined, and their influences on the surface zeta potential before and after the polymerization step are clarified. It is confirmed that the surface zeta potential and the electrophoretic motion of black pigment under the electric field are obviously affected by the surface chemistry as well as the size of the carbon sphere. The results suggest that the accurate control of the size and surface state of the carbon sphere during the hydrothermal reaction is important to improve the performance of the electrophoretic display.

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## 1. Introduction

An electronic paper has many advantages compared to LCD in terms of high brightness, high contrast, wide viewing angle, and low energy consumption. Since the electronic paper is a non-emissive display, high contrast can be realized under sunlight, while using this display does not lead to eye fatigue caused by backlight illumination. The electronic paper is energy-efficient because the energy consumption required to keep the images on the display is almost zero. Also, ultra-thin and flexible devices can be realized using the electronic paper [1–3].

Various types of electronic paper can be used, and could be categorized according to the coloring materials, partitions for arranging the independent pixels, and medium species. Currently, the electrophoretic display (EPD) is the most prevalent type because of its brief structure for mass production [4]. The basic composition of EPD is microcapsules which contain colloidal pigments dispersed in a dielectric fluid. At the initial stage of

development, a colored particle with a dye solution was used for the monochromic display [5]. Recently, more than two types of particles have commonly been applied for the enhancement of contrast ratio and response time [4], the color realization [6] and the delicate control of display. Black and white particles with opposite sign of charge are used for the monochrome display. The charged particles migrates through the dielectric fluid by an applied electric field. Due to the low permittivity ( $\epsilon \approx 2$ ) of the dielectric fluid, most electric power is used for particle migration. The microcapsule, acting as a partition, hinders the lateral movement of particles and adhesion of particles to the electrodes. The layer of microcapsules is coated between two parallel indium tin oxide (ITO) electrodes and an electric field is applied through the electrodes. Under the electric field, the positively charged pigment migrates to the negative electrode and the negatively charged pigment migrates to the positive electrode. The light passing through the transparent electrode reflects on the surface of the particles near the electrode, and the reflected light then reaches the human eye. According to the direction of electric field, white and black images can be realized. Under the equivalent electric field strength and fluid solution, the switching time is determined by the properties of particles such as shape, size, and

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surface charge. Accordingly, the basic characteristics of the particle should be controlled to improve the performance of EPD.

Carbon-based materials such as carbon black [7,8], carbon sphere [9,10], and iron oxide-carbon composite [11,12] have been researched for creating a black color on displays. Carbon black is the most common black pigment in the application of EPD, which is attributed to reflectivity, thermal stability, chemical inertness, and availability. To enhance the movement of carbon black under the electric field, the uniform zeta potential and size of pigments are required. However, it is difficult to control these properties of carbon black due to the aggregation between primary particles, resulting in an irregular shape and size. Therefore, spherical particles with uniform size, such carbon sphere, are more beneficial in terms of EPD performance. Carbon spheres with narrow size distribution were previously obtained from hydrothermal reaction under a controlled experimental condition, and it was reported that the carbon sphere reveals high mobility and fast response as a black pigment in EPD [9].

However, the relation between the properties of the carbon sphere, such as size and surface chemistry and the electrophoretic motion is not clear. The size and the surface chemistry of the carbon sphere definitely change according to the time of hydrothermal reaction. The particle size can influence the amount of absorbable charge control agent. Also, the surface chemistry can affect the adsorption affinity of the charge control agent, as well as the subsequent polymerization step after the synthesis. It is highly possible that these factors significantly change the electrophoretic motion of black pigments.

In this paper, the change in size and surface chemistry of a carbon sphere according to the reaction time was examined, followed by an investigation of the effect of these changes on the electrophoretic motion of the particle after the polymerization step. Finally, using an electrophoretic display cell which is similar to the actual EPD device, we compared the response times of the black pigments under an electric field.

## 2. Experimental

### 2.1. Preparation of the carbon sphere

All reagents were purchased from Sigma–Aldrich as analytical grade and used without further treatment. A 25 ml aqueous solution containing 1.5 M fructose was filled in a 100 ml autoclave reactor. The reactor was placed into a heated oven (160 °C) for the hydrothermal reaction. After the reaction, the reactor was immediately cooled using 18 °C running water for 10 min. The synthesized particles were collected using centrifugation with 10,000 rpm for 20 min and washed with ethanol 2 times.

### 2.2. Grafting polymerization on the carbon sphere

0.3 g of an as-prepared carbon sphere was dispersed in 50 ml de-ionized water. The temperature of the solution was maintained at 45 °C with a thermostat. 0.2 ml of hydrochloride (37 wt% aqueous solution) and 0.1 ml of 4-vinylaniline were added, then 0.1 ml of water containing 0.174 g of sodium nitrite ( $\text{NaNO}_3$ ) was added dropwise. The solution was stirred for 16 h and rinsed with de-ionized water and ethanol. The rinsed particles were dried for 24 h in a vacuum oven. The dried carbon sphere was poured in 30 ml of toluene and 20 ml of 2-ethylhexyl acrylate (EHA) was added. The solution was purged with nitrogen gas (99.9%) for 20 min. The polymerization reaction started with the addition of 0.106 g of azobisisobutyronitrile (AIBN) at 70 °C for 1 h. The non-reacted reactant was cleaned with tetrahydrofuran (THF) and

acetone. The produced polymer grafted particle was dried in a vacuum oven at 60 °C for 16 h.

### 2.3. Characterization of pigments

The surface morphology of synthesized carbon spheres was observed through a field emission-scanning electron microscope (FE-SEM, JSM-6701F, JEOL). The chemical state of the synthesized carbon spheres was inspected through a Fourier transform infrared spectroscopy (FT-IR, Nicolet 6700, THERMO SCIENTIFIC). FT-IR spectra were obtained by attenuated total reflectance (ATR) mode with the corresponding transformation to absorbance spectra. After removing the baselines automatically, the relative surface composition was compared with the area ratio of the deconvoluted peaks. The zeta potential of particles was analyzed using a dynamic laser scattering instrument (zeta potential analyzer, ELS-Z2, OTSUKA ELECTRONICS) before and after the polymerization. Because the surface of carbon spheres is hydrophilic, the mobility of carbon spheres was measured using a flow cell in deionized water and the measurement was then converted to zeta potential using the Smoluchowski equation [13]. On the other hand, the mobility of polymer grafted particles was measured with a low conductive cell in tetrachloroethylene (TCE) and the zeta potential was derived from the Hückel equation [13]. Surface charge variation with the addition of an acidic charge control agent (Sorbitan monooleate, SPAN 80) was also investigated with a low conductive cell in TCE. The density of particles was measured through gas pycnometry (AccuPyc 1340, MICROMERITICS).

### 2.4. Black/white conversion test

Black and white inks were prepared before mixing. The carbon sphere, previously prepared by hydrothermal reaction and grafting polymerization, was dispersed in TCE with SPAN 80, and the concentrations of the carbon sphere and SPAN 80 were 0.3 g/ml and 0.7 M, respectively. The sol–gel synthesized  $\text{TiO}_2$  was used as a white pigment, of which the concentration was 0.4 g/ml. 0.1, 0.2, and 0.3 g/ml of a basic charge control agent (polyisobutylene succinimide, OLOA 1200) were added to  $\text{TiO}_2$  containing a solution to control the surface charge of  $\text{TiO}_2$ . The prepared black and white inks were sonicated separately for 1 h. As-prepared black and white inks were mixed with the volume ratio of 1:5 and then sonicated for 1 h. The mixed ink was dropped between parallel ITO coated electrodes, and the gap between the electrodes was controlled to 0.06 mm. 30 V of electric potential was applied by a DC power supply (PWS-3005D, JEILMI) for investigating the movements of white and black pigments. The black/white conversion was recorded using a video microscope (SV-35, SOMETECH) and the response time was measured from the video clips.

## 3. Results and discussion

The carbon sphere was synthesized via hydrothermal reaction under high temperature and pressure. The dissolved fructose was converted to various intermediates during the reaction, and these intermediates were polymerized and formed the spherical carbon material [14]. As the reaction progressed, the size of the carbon sphere was clearly increased as shown in Fig. 1. Due to the radial growth of the carbon sphere without aggregation, between 120 and 180 min of reaction time, the particle size showed linear dependency on the reaction time. The primary particle size of the carbon sphere from 200 min reaction time also follows the linear increment, as described in Fig. 1(E). However, severe aggregation arising in 200 min reaction time led to the abrupt increase of particle size to 2.6 times larger than the size at 180 min reaction time,

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