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Surface modification of organic pigment particles for microencapsulated electrophoretic displays

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ABSTRACT

The surface modification of Pigment Yellow 13, Pigment Red 254 and Pigment Blue 15 to improve their electrophoretic properties in electrophoretic suspension were studied. The particle size distribution and the surface morphology of the modified pigments were determined, the presence of the functional groups on the surface of pigments was confirmed, and the dispersion stability and chargeability of pigments suspended in electrophoretic slurry were characterized. For the application of microencapsulated electrophoretic displays, the colored microcapsules containing the modified organic pigments were prepared and spread on an ITO coated conducting film. A working prototype of electrophoretic display was successfully fabricated in this approach.

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1. Introduction

Microencapsulated electrophoretic display (EPD), which is also called electronic ink display, is made of microcapsules including charged particles and fluid medium. It has been of great interest due to its advantages of ink-on-paper appearance, high reflectance, good contrast ratio, wide-viewing angle, state bistability and low power consumption [1,2]. Currently, black–white electronic books (e-books) based on microencapsulated EPD have been commercialized and have achieved mass market success. The paper-like displays designed by the E-Ink corp., are used by the majority of today's e-book readers. However, full-color e-books or displays are not realized, so the colorization will be the next target of e-reader market [3].

The black–white EDPs comprise of a flat, two-dimensional array of microcapsules containing a dual-particle system of titanium oxide white particles and black carbon pigments with opposite charges. When the backplane electronics apply voltage to the microcapsules, one set of particles is driven to the top of the microcapsules and the other set goes to the bottom to show images and texts. Based on this principle, several techniques of colorization are explored. The conventional method of color filtering will bring on the loss of light and resolution, leaving unsaturated colors as a result. The genuine realization of full-color display needs a subpixel comprising of tricolor unites such as red, green and blue or cyan, magenta and yellow.

Pigment particles in electrophoretic fluid must have properties such as an appropriate density, low solubility, good disperse stability and high chargeability. Many kinds of pigments can be used as colored electrophoretic particles. Tang et al. doped the hollow titania with Cr^{3+}, Fe^{3+} and Co^{2+}/Al^{3+} to fabricate colored inorganic pigment particles [4]. Kang et al. prepared polymer nanoparticles by free-radical polymerization of styrene and 4-vinylpyridine, encapsulating organic dyes such as Acid Blue 25, Acid Red 8, and Acid Yellow 76 [5]. Kim et al. used laser printer toner particles modified with wax materials to fabricate colored microcapsules containing cyan–white, magenta–white and yellow–white pigment pairs [6]. However, the weakness of inorganic [7,8] and polymer particles [9] in chroma and light reflectance was known to influence the color saturation of display. The organic pigments, on the other hand, have merit of excellent optical properties such as good brilliance, color strength and transparence, and therefore can offer great potential in the context of electronic, optical and photoelectric devices. Tang et al. prepared pigment-based RGB tricolor ink particles via mini-emulsion polymerization, but the polystyrene coating in pigments decreased the reflex light saturation and made the ink particles look albescent [10].

In this article, we report the use of the typical organic pigments such as C. I. Pigment Yellow 13, C. I. Pigment Red 254 and C. I.

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Pigment Blue 15 as colored electrophoretic particles. Organic pigment particles with smooth surface, small particle size and narrow size distribution were obtained after surface modification. Good dispersion stability and surface charges of the modified particles in solvent were achieved in the presence of hyperdispersants and charge control agents, and the modified particles offered good electrophoretic behavior in electrophoretic slurry. The microcapsules of dual-particle systems containing yellow–white, red–white and blue–white pigments were prepared via coacervation of gelatin and gum arabic [11,12]. The resulting particles exhibited both good electrophoretic movement and ideal bistability in the microcapsules. Therefore, these microcapsules containing modified organic pigment particles could be used as a good candidate for the colored EPDs.

2. Experimental section

2.1. Materials

The organic pigments of yellow, red and blue (commercial C. I. name: Pigment Yellow 13, Pigment Red 254, Pigment Blue 15) were obtained from Clariant Tianjin Co. Ltd (Tianjin, China), and their chemical structures were shown in Fig. 1. Sodium silicate, tetrachloroethylene, polyethylene, stearic acid, cetyltrimethylammonium bromide, gelatin and gum arabic were purchased from Guangfu Chemicals Co. Ltd (Tianjin, China). Silane coupling agent 3-aminopropyltriethoxysilane was obtained from Tianyang Assistant Co. Ltd (Yangzhou, China). Hyperdispersants CH-5 and CH-11B were purchased from Sanzheng Polymer Co. Ltd (Shanghai, China). All the chemicals used in this study were of reagent grade.

2.2. Modification of yellow particles and preparation of yellow electrophoretic fluid

Pigment Yellow 13 (PY13) was modified using the following process. PY13 (1.0 g) was emulsified in 150 mL deionized water with 0.18 g cetyltrimethylammonium bromide (CTAB). The mixture was then transferred into a 500 mL round bottom flask and heated to 95 °C. Sodium silicate solution (20 mL, 1 M) was dripped in 2 h and the pH was adjusted to 9–10 by addition of 2 M sulfate acid solution. The reactions were maintained for 5 h at pH = 10, and continued for another 2 h at pH = 3. The solid product was collected by centrifuge, and then dispersed into 200 mL mixture of water and ethanol (10:90, weight ratio). To the mixture, 3-aminopropyltriethoxysilane (0.2 g) was added and pH was adjusted to 3. The mixture was heated to reflux with moderate stirring and the reaction was maintained for 10 h at 80 °C. The resulting product was collected by centrifuge and washed several times with deionized water and absolute ethanol respectively.

The yellow electrophoretic fluid was prepared by ball milling method. The suspension was prepared by mixing 0.25 g modified PY13 particles, 10 mL tetrachloroethylene, 0.025 g CH-5 as emulsifier, and a certain amount of CTAB as charge control additive. The mixture was milled for 24 h on a bead miller with 0.5 mm ZrO_2 bead as milling medium at a stirring speed of 2000 rpm to obtain yellow electrophoretic slurry.

2.3. Modification of red particles and preparation of red electrophoretic fluid

Pigment Red 254 (PR254) was modified using the following process. Polyethylene (PE) 0.5 g was dissolved in 250 mL cyclohexane with stirring at 80 °C. PR254 (1.0 g) was ultrasonicly dispersed in the above solution for 30 min. After the suspension was placed for 24 h, the product was centrifuged and washed by absolute ethanol. Red pigment scarlet powders modified by PE were obtained.

The red electrophoretic slurry was prepared by the same ball milling method as that described for the yellow pigments. The red electrophoretic fluid contained 0.25 g modified PR254 particles, 10 mL tetrachloroethylene and 0.025 g CH-5 as stabilizer.

2.4. Modification of blue particles and preparation of blue electrophoretic fluid

Pigment Blue 15 (PB15) 0.25 g, assistant-hyperdispersant CH-11B 0.01 g and hyperdispersant CH-5 0.025 g were dispersed in 10 mL tetrachloroethylene, with a certain amount of stearic acid as charge control additive. The blue electrophoretic slurry was prepared by the ball milling method as described in Section 2.2.

2.5. Preparation of dual-particle systems electrophoretic fluid

White electrophoretic slurry (0.1 M) containing TiO_2/PMMA composite particles (made in our laboratory) [13,14] was also prepared by ball milling. Yellow–white or red–white or blue–white electrophoretic fluid of dual-particle system was obtained by mixing two kinds of electrophoretic fluid and ultrasonicly dispersing for 0.5 h. The electrophoretic slurry volume ratio of yellow/white, red/white and blue/white was set at 1/1, 1/10 and 1/2, respectively.

2.6. Preparation and coating of colored microcapsules

The microcapsules were prepared by coacervation of gelatin and gum arabic used as wall materials. First gum Arabic (0.4 g) was dissolved in 20 mL deionized water with stirring at 50 °C for 30 min. Colored electrophoretic slurry (3 mL), prepared as described in Section 2.5, was introduced into the aqueous phase.

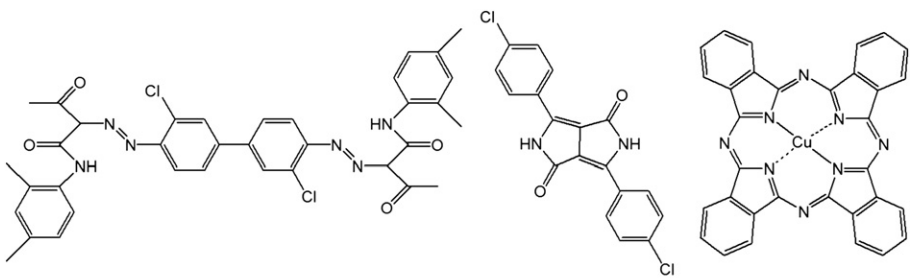

Fig. 1. Chemical structures of pigment raw materials.

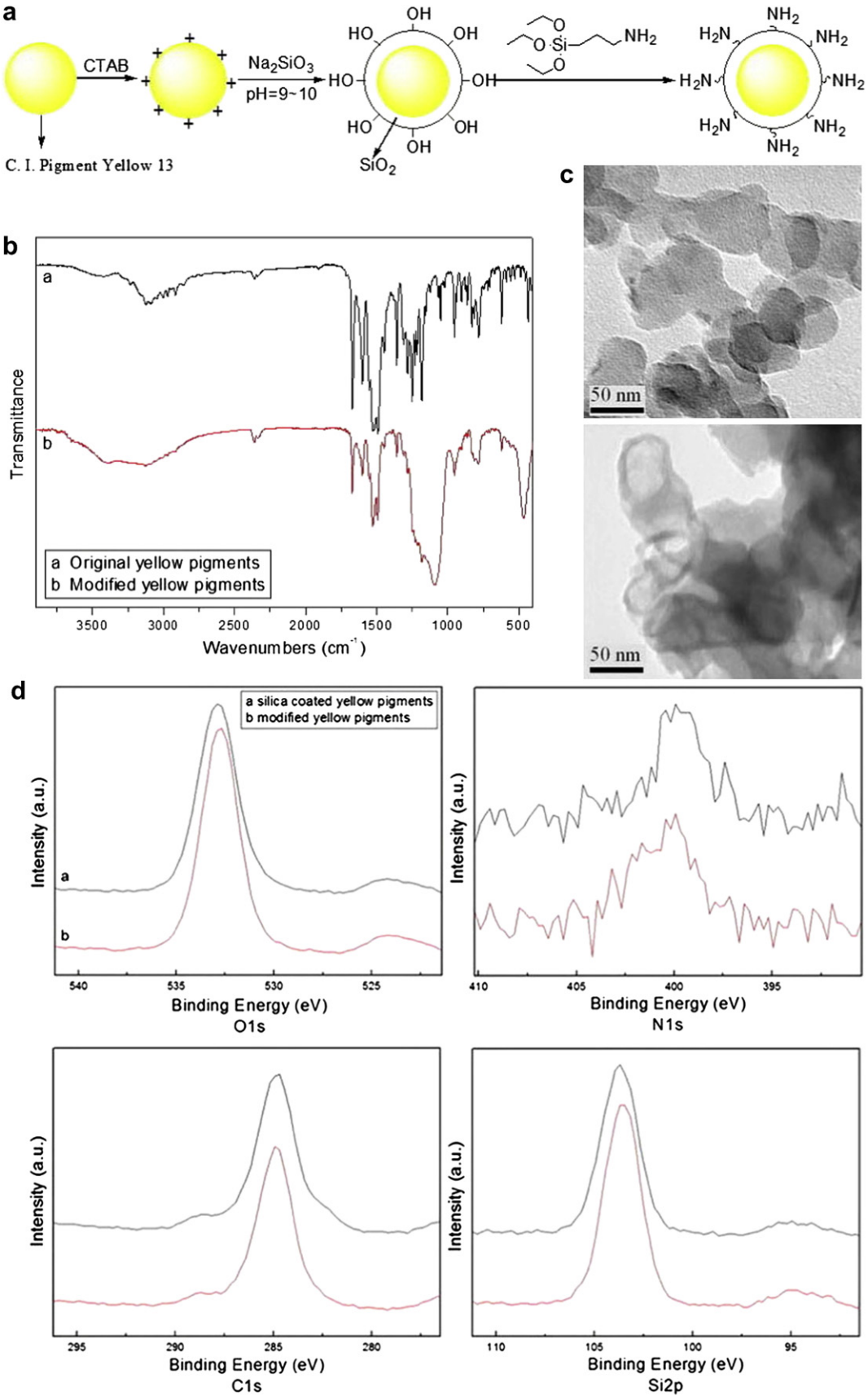

Fig. 2. (a) Schematic representation of preparation of core–shell composite particles, (b) FT-IR spectra and (c) TEM micrographs of original and modified yellow pigments, (d) XPS spectra of silica coating and modified yellow pigments. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

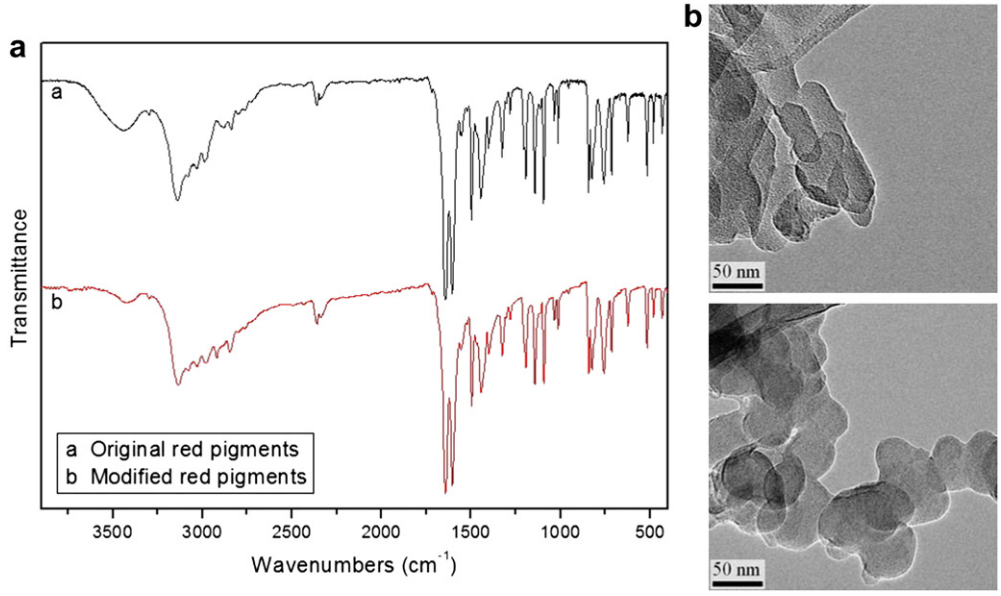

Fig. 3. (a) FT-IR spectra and (b) TEM micrographs of original and modified red pigments.

The emulsion was agitated at 900 rpm for about 2 min, and then the agitation was reduced to 300 rpm. Gelatin water solution (20 mL, 0.2 M) was drop-wise added in 5 min. The pH was then regulated to 3.8 by addition of 10 wt.% acetic acid solution. The emulsion was slowly cooled to 10 °C and maintained for 1 h. After adding 5 wt.% glutaricdialdehyde solution (5 mL) as crosslinker for wall material, the polymerization was held for another 1 h below 0 °C. The precipitated microcapsules were then rinsed off with deionized water several times, and the colored electric ink was obtained.

The colored microcapsules were coated on indium tin oxide (ITO) film with 5 wt.% polyvinyl alcohol (PVA) as binder by using a home-made coating machine. The weight ratio of microcapsule/PVA was 3/1, the coating rate was 60 mm/min and the coating height was 0.75 mm. After natural drying, the coating microcapsules were bonded with polyurethane adhesive L758 on a patterned electrode. The prototype of colored microencapsulated EPDs was obtained.

2.7. Characterization

Chemical constitution of the product particles was identified by fourier transform infrared spectroscopy (FT-IR, BID-RAD3000, scan 400–4000 cm^{-1}) and X-ray photoelectron spectroscopy (XPS, PHI-1600, Perkin–Elmer, USA). The surface morphology and structures were examined by transmission electron microscopy (TEM, JEM-2001F, Jeol, Japan) and scanning electron microscopy (SEM, S4800, Hitachi, Japan). The particle size distribution, zeta potential and electrophoretic mobility of pigment particles were measured by zeta potential and submicron particle size analyzer (Delsa Nano C, Beckman Coulter, USA). In addition, the microcapsules were

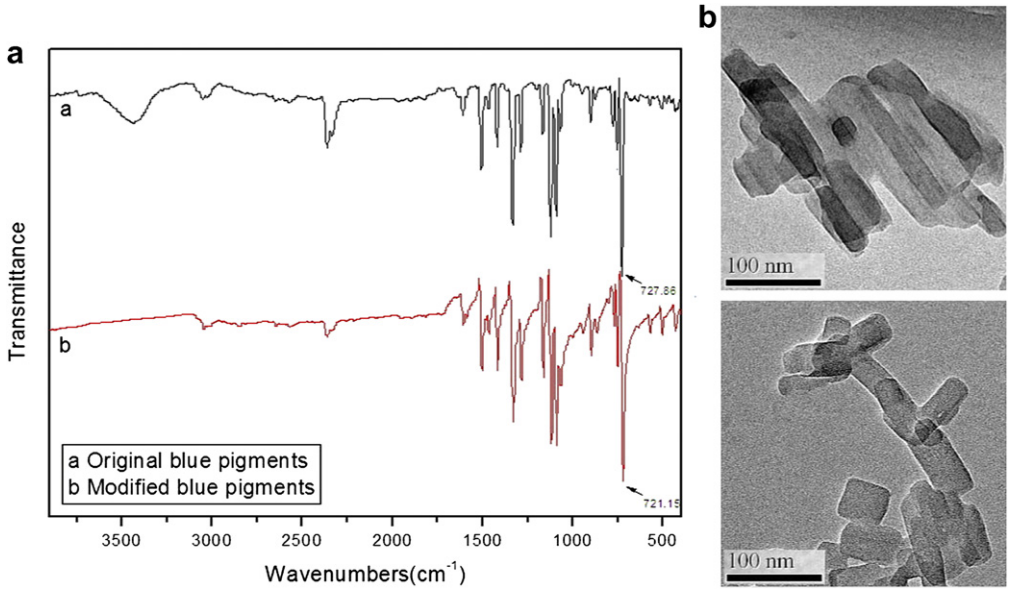

Fig. 4. (a) FT-IR spectra and (b) TEM micrographs of original and modified blue pigments.

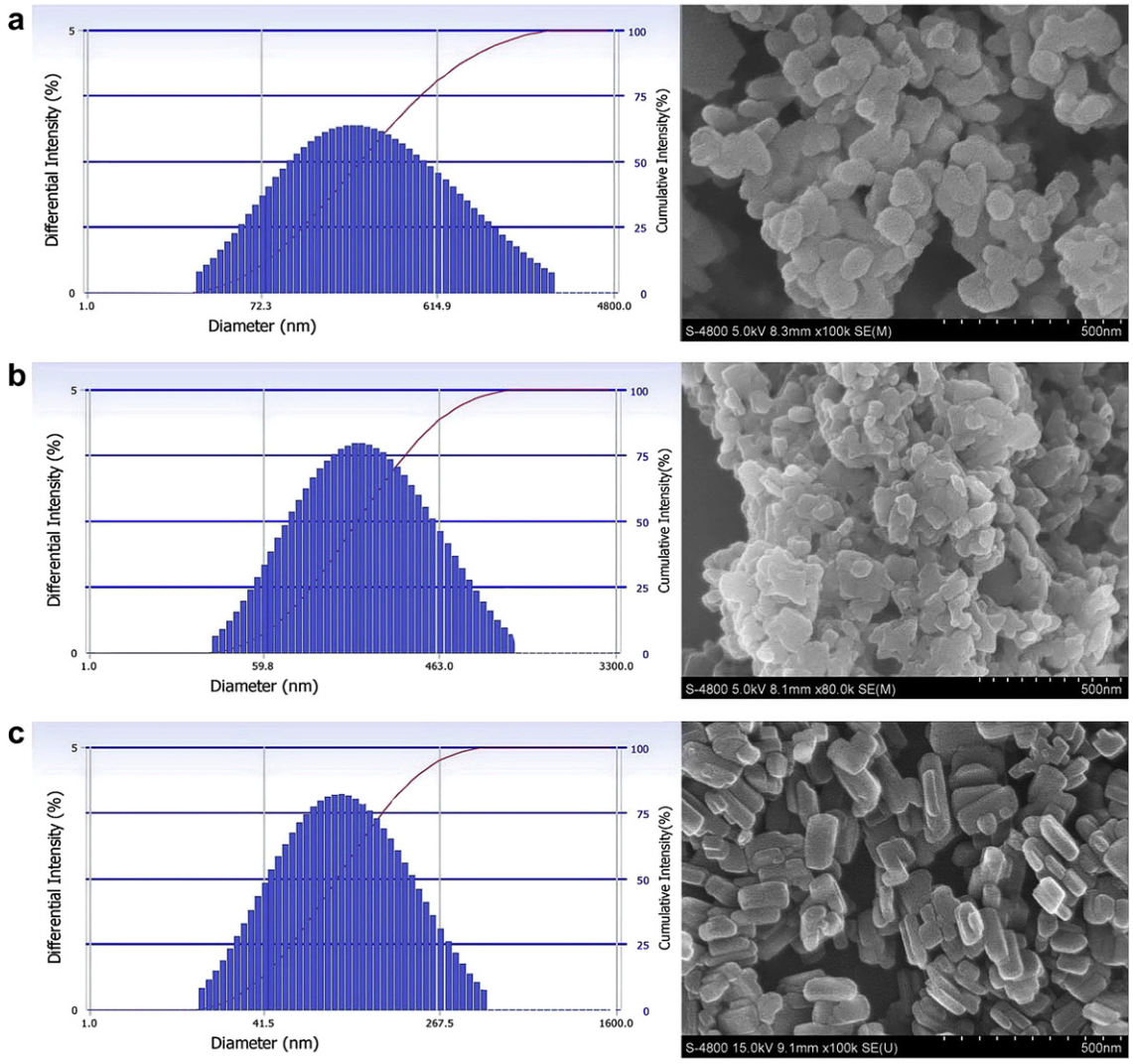

Fig. 5. Particle size distributions and SEM microphotographs of modified (a) yellow, (b) red and (c) blue pigment particles in electrophoretic slurry.

observed under an optical microscope with CCD (BX51, Olympus, Japan).

3. Result and discussion

3.1. Modification of organic pigment particles

Pigment Yellow 13 is a traditional disazo pigment. However, the poor solvent resistance and light fastness limit its applications in EPDs [15]. A nano-silica layer coating on the surfaces of the organic pigment particles to improve the solvent resistance and weatherability [16], was reported in our previous work [17]. Fig. 2a is the schematic representation of the preparation of core–shell composite particles. First, the yellow pigments were dispersed in CTAB/water solution, and SiO_2 shell was precipitated onto the surface of the pigments via the hydrolysis of sodium silicate at 92 °C and at pH 9–10. The silane coupling agent 3-aminopropyltriethoxysilane was then grafted onto the surface of the composite particles, and a lipophilic and highly-charged surface was resulted. Fig. 2b shows the FT-IR spectra of the pigment before and after modification. The Si–O stretching vibration at 1080–1100 cm^{-1} and Si–O–Si bending at 460–480 cm^{-1} of the modified pigment confirmed the presence of the silica. The N–H stretching vibration due to the amino group can be found at 3420–3440 cm^{-1}. In order to confirm the grafting of 3-aminopropyltriethoxysilane, XPS analysis was carried out. Fig. 2d shows the XPS spectra of silica coating and modified yellow pigments. A comparison of the silica coating pigments and modified pigments indicates the shift of the Si2p XP signal from 103.68 eV to 103.40 eV and the shift of the O1s from 532.93 eV to 532.65 eV, corresponding to the conversion of the Si–O–H on the surface to Si–O–Si after grafting with 3-aminopropyltriethoxysilane, which increases the shielding effect of electrons. The N1s XP signal appearing at 401.65 eV is characteristic for NH_3^+ which confirms that the amino groups are introduced on the surface of composite particles. Fig. 2c is the TEM photographs of the original and modified yellow pigments. The

Table 1
Zeta potentials and electrophoretic mobilities of modified organic pigment particles in tetrachloroethylene.

Electrophoretic particles	Zeta potential (mV)	Electrophoretic mobility (cm^2/V s)
Modified yellow particles	−3.45	−8.269e-7
Modified red particles	−31.83	−7.631e-6
Modified blue particles	−0.45	−1.076e-7

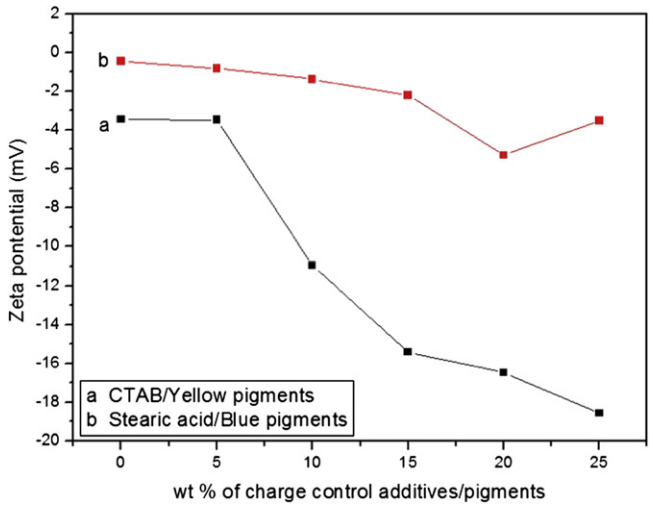

Fig. 6. Zeta potentials of pigments with different contents of charge control additives in electrophoretic slurry.

strong contrast between the gray edge and white center of the modified pigments confirmed the core–shell structure, and the composite form was found to enhance the solvent resistance and light fastness.

Many red organic pigments have been used in EPD application [18,19]. In this paper, we chose Pigment Red 254, a high-performance pigment of diketopyrrolopyrrole (DPP), to function as the red electrophoretic particles. Despite the low molecular masses, the DPP pigments are highly insoluble, and very chemically and thermally stable. Such behavior may be attributed to the presence of strong intermolecular forces in the pigment crystal lattice, for instance, π–π interaction between two planar molecules and H-bonds between the –NH group and oxygen [20,21]. To improve their affinity to the solvent, the pigments were coated with polyethylene. The PE membrane covered on pigments presents a chemically modified surface for the purposes of charging and providing a steric barrier between pigment particles to prevent agglomeration. Fig. 3a presents the FT-IR spectra of the pigment scarlet powders. The peaks of C–H stretching vibration at 2918 cm^{-1} and 2849 cm^{-1} of the modified particles confirmed that the red pigments were coated with PE. TEM micrographs in Fig. 3b show that the powders modified with PE have a spheroidal shape and ideal dispersity in solvent.

Copper phthalocyanine, which has excellent all-round fastness properties, is known to occur in no less than five crystal modifications [22]. Pigment Blue 15 exists as the α-form copper phthalocyanine. It is known that the α-form is not stable and tends to convert to β-form. Therefore, in this paper, we used assistant-dispersant CH-11B (4 wt.% relative to the pigments) to improve the stability of the α-form copper phthalocyanine in solvent. In the preparation of blue electrophoretic fluid, the solubility of copper phthalocyanine in tetrachloroethylene played a decisive role in the phase transformation. As a derivative of phthalocyanine, CH-11B has physical and chemical properties similar to those of copper phthalocyanine [23]. It participates in the crystal of copper phthalocyanine, which can be effectively stabilized against solubility and recrystallization to restrain the phase conversion process of α-form to β-form copper phthalocyanine. This can be confirmed from the FT-IR result as shown in Fig. 4a. The original blue pigments which were milled for 24 h in tetrachloroethylene, have the β-form characteristic absorption at 727.88 cm^{-1}. The FT-IR spectra of the CH-11B modified pigments milled for 24 h show the α-form's characteristic absorption at 721.15 cm^{-1}. CH-11B is absorbed easily on the surface of PB15 to provide polar anchoring positions for stabilizers, and improves PB15 dispersibility in tetrachloroethylene. Fig. 4b is the TEM micrographs of the original and modified blue pigments. It can be seen that the original particles without CH-11B have stronger flocculation effect than those of modified particles. This is probably because CH-11B enhances the absorption of stabilizer on the particle surface, and inhibits the aggregation of particles.

3.2. Dispersion stability and chargeability of particles in electrophoretic slurry

Normally, the low molecule-weight surfactants are regarded as the dispersants in the preparation of electric ink for homogenizing and stabilizing the pigment particles in organic medium. However,

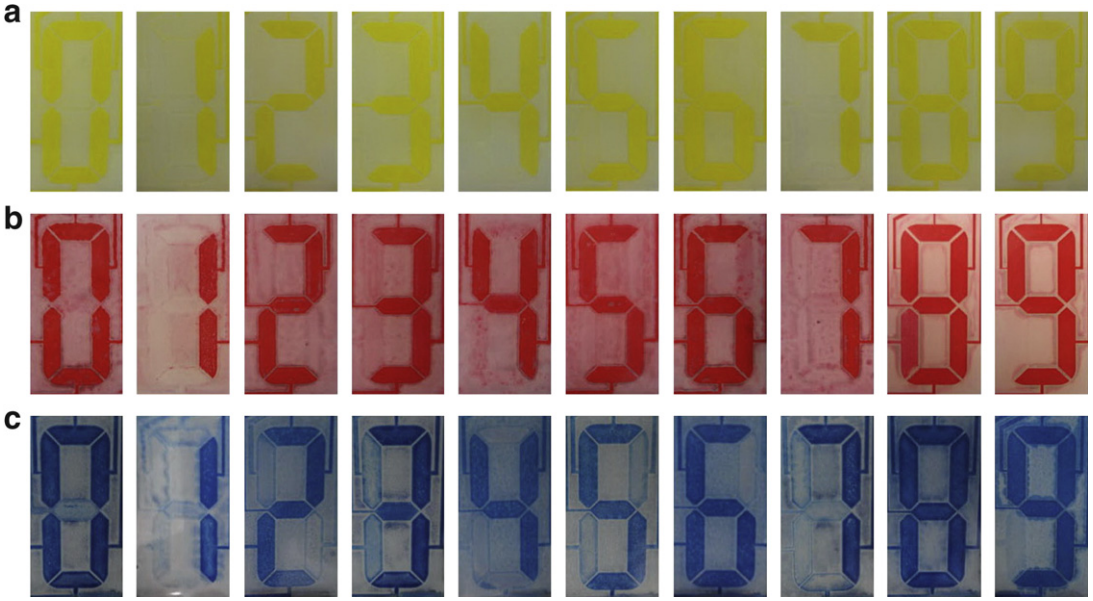

Fig. 7. Electrophoretic display patterns of (a) yellow–white, (b) red–white, (c) blue–white electrophoretic slurry in an electric field (E = 100 V/mm). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

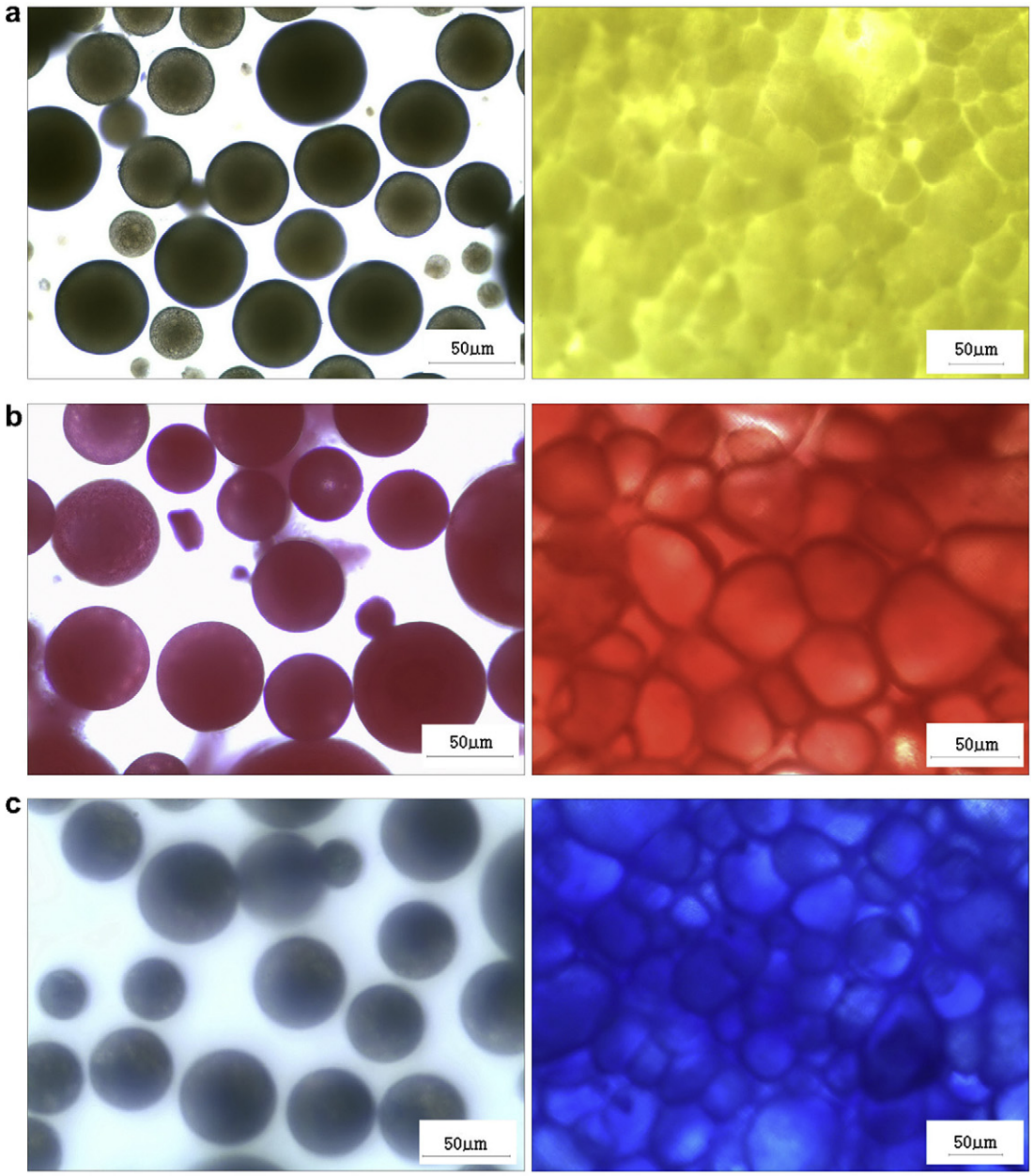

Fig. 8. Optical microscopy images of (a) yellow–white, (b) red–white and (c) blue–white microcapsules before and after coating in ITO film. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

it is known that the particles modified by these surfactants easily reflocculate and recoagulate in the nonpolar solvent medium. In order to obtain more stable dispersion liquids including electrophoretic particles, it is necessary that a more effective stabilizing layer is absorbed onto the particle surface. We used hyperdispersant CH-5 (10 wt.% relative to the pigments) in the preparation of electrophoretic slurry to homogenize the pigment particles dispersion. CH-5 is an aliphatic acid polymer derivative with multiple anchoring groups and soluble polymeric chains. Colored electrophoretic fluid was fabricated by the modified pigments suspended in tetrachloroethylene with CH-5 after ball milling. When CH-5 was added to the suspension, its anchoring carboxyl group would bond to the surface of pigments, and its polypropylene chain would enclose the spheres to keep them from agglomeration [24]. The colored electrophoretic slurry with good dispersion stability was obtained. Particle size distributions and SEM micrographs of the color-modified pigment particles in the electrophoretic slurry are shown in Fig. 5. It is found that the modified pigments have smooth surface, sphere-like shape and small particle size with narrow size distributions. Through the test results of size distributions, we can see that the average size of the yellow, red and blue modified particles is 217.7 nm, 155.2 nm and 80.3 nm respectively, and the sample polydispersity index is 0.374, 0.330 and 0.280 respectively.

To be applied in EPDs, the particles in the suspension are required to repeat the electrophoretic migration after changing the polarities of the applied voltage, so the chargeability of the particles is the key factor in determining the speed or efficiency of EPDs. In the case of a suspension with a nonaqueous liquid, the particles are charged mainly as a result of the dissociation of surface groups or the adsorption of ionic surfactants. The hyperdispersant CH-5, which is used in the suspending of the modified

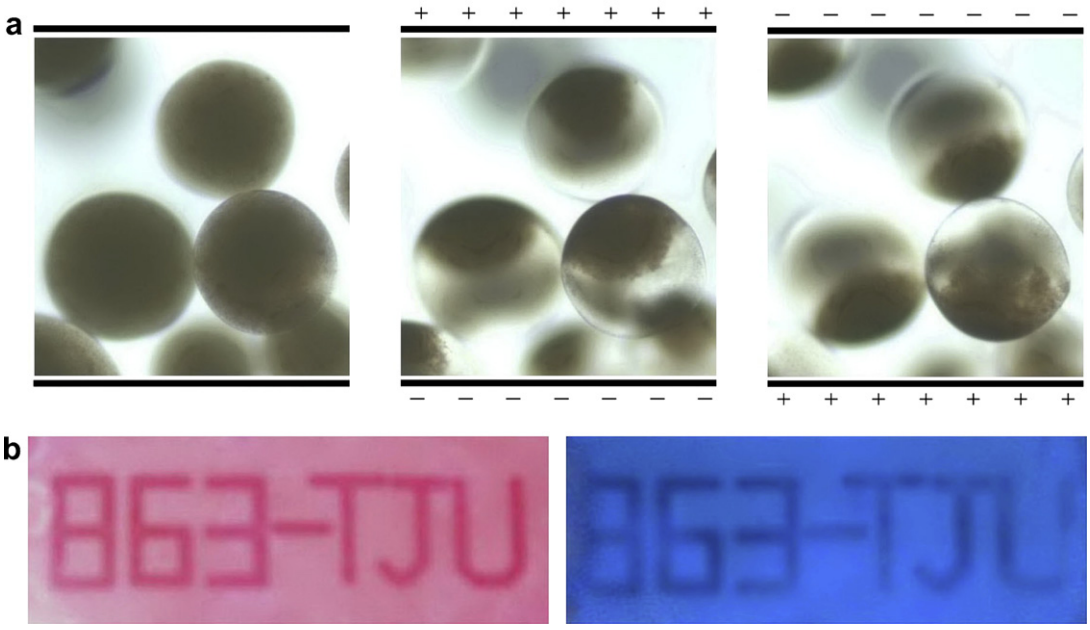

Fig. 9. (a) Reversible electric response of yellow–white particles in microcapsules in an electric field, (b) the front electrode of ITO film coating with microcapsules of red–white and blue–white systems under an applied voltage. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

particles, is an aliphatic acid polymer derivative with many carboxyl groups. The carboxyl groups of CH-5 reacted with the active groups of the modified particles, and the particles are charged mainly due to the remaining carboxyl groups of CH-5. Therefore, the resulting particles usually have the negatively charged surface. Zeta potential and electrophoretic mobility of the modified yellow, red and blue organic pigment particles in tetrachloroethylene measured by zeta potential and submicron particle size analyzer are exhibited in Table 1. We can see that the red pigments have a good chargeability to migrate. However, the electrophoretic mobility of yellow and blue pigments is not sufficient, so the charge control additives are needed in the fluid to provide more charges. CTAB in yellow electrophoretic fluid and stearic acid in blue electrophoretic fluid were used as charge control additives. Fig. 6 shows the effect of different amounts of charge control additives on the zeta potential of pigment particles in electrophoretic slurries. With the increasing of the charge control additives, the particles have the increased negative charges, and the optimum dosage is 15 wt.% CTAB relative to the yellow pigments and 20 wt.% stearic acid relative to the blue pigments.

Electrophoretic slurries of dual-particle systems were prepared by blending the colored electric ink with white electric ink. In order to get good contrast ratio, the optimum volume ratio of yellow/white, red/white, blue/white was found to be 1/1, 1/10, 1/2, respectively. The electrophoretic motions of the ink particles were studied in a simple 7-sections electrophoretic cell. The test cell was fabricated by placing the electrophoretic ink slurries between two ITO-covered glass slides with 100 μm spacing. A reversal between the colors of the displayed pattern and the background could be obtained by changing the polarities of the applied DC voltage. Fig. 7 presents the 0–9 text displays in yellow, red and blue color with white background in the electrophoretic cell under the electric field of 100 V/mm. The responding time of the yellow, red and blue electrophoretic fluid is 1 s, 0.8 s and 0.5 s, respectively. The device was found to have memory function, therefore the images showed by the pigment particles on the surface of the ITO still remain for a considerable length of time after the applied voltage was removed.

3.3. Response behavior of modified particles in colored microcapsules

To inspect the feasibility of modified organic pigment particles as electrophoretic particles, the microcapsules of dual-particle systems were prepared using the complex coacervation by the mutual neutralization of two oppositely charged polymers, gelatin and arabic gum. Fig. 8 is the optical microscopy images of colored microcapsules before and after spreading on the ITO film. It is found that the gelatin–arabic gum microcapsules have smooth surface, good sphericity and excellent transparence, and their average size is about 30–50 μm. After coating on the ITO film, the microcapsules keep flattened and closely linked to each other, which is due to the good flexibility and mechanical strength of gelatin–arabic gum walls.

To investigate the response behavior of the pigment particles in microcapsules under an applied voltage, the dried microcapsules were fixed into the middle of two parallel copper electrodes with a DC voltage applied between them. Fig. 9(a) is the microphotograph of microcapsules containing both yellow and white particles. Under no electric field, the pigment particles distributed randomly in capsule. Positive DC electric field ($E = 100$ V/mm) was applied to the upper electrode, the yellow particles moved upward fast and the white particles were pulled back, so we can see the yellow color at the front electrode. When the field was reversed, the particles migrated reversibly, and the front electrode showed the color of the white particles. This result confirmed that the modified particles have a reversible electric response in microcapsules under an applied voltage. A display prototype has been prepared by coating the microcapsules on the patterned ITO film substrate. Fig. 9(b) is the display showing red and blue patterns with white background under an applied voltage. The change of the pattern color, when the voltage is applied, demonstrates the principle of the image formation process in microencapsulated EPDs. In the future, more efforts should be done to improve the performance of the display.

4. Conclusion

In this paper, we described the successful surface modification of yellow, red and blue organic pigment particles, their dispersion in

solvent medium, and use in microcapsules of electrophoretic display. To improve the electrophoretic properties, the yellow particles were coated with a shell of SiO_2, which was then grafted with silane coupling agent. The red pigments were coated with polyethylene and the blue pigments were modified with assistant-dispersant CH-11B. The stable dispersion liquid containing ink particles were fabricated with the stabilizer CH-5, and the surface charge density of the ink particles could be controlled by varying the concentration of the charge control additives. Stable, elastic, and optically transparent gelatin–gun arabic microcapsules containing modified particles and dielectric media as core materials were prepared by a coacervation process for electrophoretic displays. The modified organic pigment particles had good electrophoretic properties in the electric ink microcapsules.

Acknowledgments

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