

Partially Crosslinked P(SMA-DMA-St) Copolymer *In Situ* Modified RGB Tricolor Pigment Particles for Chromatic Electrophoretic Display

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ABSTRACT: RGB tricolor organic pigments were *in situ* modified by stearyl methacrylate-2-(dimethylamino)ethyl methacrylate-styrene random copolymer [P(SMA-DMA-St)] through solution polymerization. The modified particles were charged in tetrachloroethylene (TCE) with the aid of suitable charge control agent. The structure and electrophoretic property of ink particles were characterized by FTIR, TEM, PALS, et al. P(SMA-DMA-St) modification decreased the particles size and enhanced the suspending stability of the pigments in tetrachloroethylene due to the repulsive forces among the treated pigment particles resulting from the extending polyacrylate chain in the media. Partial crosslinking the P(SMA-DMA-St) on the pigment surface with ethylene glycol dimethacrylate (EGDMA) helped to increase the Zeta potential and electrophoresis mobility of RGB electrophoretic particles. The maximum values of the Zeta potential for red, green, and blue electrophoretic particles reached -36.2 , -57.7 , and -42.7 mV, respectively. The obtained RGB electrophoretic particles could be applied in the color/white dual-particle electronic ink together with charged TiO_2 to show vivid color and white images under a DC electric field. © 2013 Wiley Periodicals, Inc. *J. Appl. Polym. Sci.* 130: 645–653, 2013

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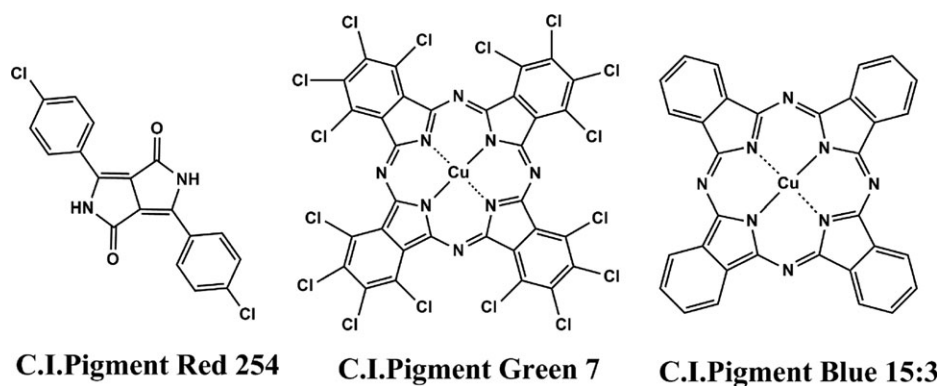
INTRODUCTION

Since the pioneering work of Jacobson,¹ acting as one of the most promising technique in fields of electronic paper, electrophoretic displays (EPD) stepped into a great development because of its advantages like light-weight, wide viewing angles, and low-power consumption, et al.^{2–6} In the past years, though the white-black monochrome EPD has been commercialized by some corporations, efforts to make full color display with rapid response were never stopped. E-ink Corporation developed a useful technology for fabricating chromatic electrophoretic display (CEPD) by combining color filter arrays with white-black monochrome EPD.^{7,8} Nevertheless, the color filter absorbed a great deal of reflected light which resulted in low contrast and brightness. Another CEPD composed of tricolor electronic ink units containing tricolor electrophoretic particles and corresponding suspension may be a reliable avenue to achieve CEPD with high brightness and contrast.^{9–11}

Studies were focused on the preparation of tricolor electrophoretic particles for CEPD recently. Both of the encapsulated-dye and modified-pigment were applied for synthesis of electrophoretic particles. Dyes are used to preparing chromatic ink by doping into polymers such as polystyrene, poly(*N*-vinyl

pyrrolidone), poly(methyl methacrylate) and some copolymers.^{11–13} However, some shortcomings like low brightness and bad optical stability limit the application of dyes in CEPD. By comparison, organic pigments with superior light resistance, better stability, and higher color strength are suitable for CEPD.¹⁴ Various methods were used to apply modified-pigment as colorants to prepare chromatic electrophoretic particles. Lee et al. synthesized red electrophoretic particles via dispersion polymerization.¹⁵ Tang et al. prepared polystyrene-encapsulated RGB pigment via mini-emulsion polymerization for CEPD.¹⁶ Silica enwrapping was also used to prepare color electrophoretic particles.^{17,18} Le et al. modified ionic polymer onto green pigment particles via dissolving-recrystallizing process.¹⁹ Up to now, the preparation of RGB tricolor electrophoretic particles with high surface charge, fine dispersion stability, and good light resistance is still required.

In this study, RGB tricolor organic pigments were *in situ* modified by the solution polymerized styrene-acrylate copolymer. Kinds of acrylate including stearyl methacrylate (SMA), 2-(dimethylamino)ethyl methacrylate (DMA) and ethylene glycol dimethacrylate (EGDMA) were applied as monomer. DMA unit can be anchored onto the surface of pigments through polar



Scheme 1. Chemical structures of RGB pigments.

bonding. The polyacrylate chain was expected to extend in media to prevent pigment particles from agglomeration and sedimentation. EGDMA was applied to crosslink the copolymer attached on the pigment surface because crosslinking could bind the copolymers on the pigment surface.²⁰ Furthermore, it is reported that crosslinking polymer/copolymer encapsulated pigment particles own excellent optical property and high stability in emulsion paints and ink jet printing.^{21–25} Characteristics of the modified pigment particles such as chemical structure, morphology, particle size as well as the electrophoretic properties were examined by FIIR, TEM and dynamic light scattering, respectively. It was found that the modified pigments possess characteristics of high surface charge and mobility, fine suspension stability in tetrachloroethylene (TCE), and good light resistance as well. The mobility of the RGB electrophoretic particles in TCE is in the range of $-4.5 \sim 9.0 \times 10^{-6} \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$. The highest mobility of RGB electrophoretic particles could be obtained with suitable amount of EGDMA addition. The partially crosslinked P(SMA-DMA-St) treated RGB tricolor pigment particles were applied as color electrophoretic particle in the dual-particle electrophoretic system together with the charged poly[N-(p-vinyl benzyl)phthalimide]-grafted TiO_2 particles. Under the DC electric field of 45 V/mm, the positive/negative charged electrophoretic particles can move towards opposite direction and color/white image can display.

EXPERIMENTAL

Materials

The red pigment (C.I. pigment red 254, PR254), green pigment (C.I. pigment green 7, PG7), blue pigment (C.I. pigment blue 15 : 3, PB15:3) were offered by DIC. The monomers styryl methacrylate (SMA), 2-(dimethylamino)ethyl methacrylate (DMA), ethylene glycol dimethacrylate (EGDMA) and initiator 2,2'-azobis(2-methylpropionitrile) (AIBN) were purchased from Aladdin reagent. AIBN was purified before use and others were used as received. Toluene, styrene (St), and tetrachloroethylene (TCE) were purchased from Sinopharm Group Chemical Reagent. Styrene was distilled to remove the inhibitor. The self-made poly[N-(p-vinyl benzyl)phthalimide]-grafted TiO_2 was applied as white electrophoretic particle.²⁶

Preparation of RGB Electrophoretic Particles and Electronic Ink

Pigment (1.0 g) was dispersed in 40 mL toluene in a three-neck flask together with ZrO_2 ball ($\Phi 1$ mm) and ultrasonic dispersed for 30 min. Then 6.792 g SMA (0.02 mol), 3.143 g DMA (0.02 mol) and 2.083 g St (0.02 mol) were added into the flask. The mixture was mechanically stirred at 600 rpm for 2 h with ball milling. Then 20 mL toluene solved with 0.100 g AIBN was added under N_2 atmosphere. The mixture was heated to 70°C under a stirring rate of 300 rpm and kept for 2 h. Then the mixture was kept for another 24 h after the addition of different amount (0.003, 0.006 mol) of crosslinking monomer EGDMA. The product was centrifuged at 9000 rpm for 15 min, washed thrice with toluene and then dried in vacuum oven under 55°C .

Totally, 0.10 g treated color pigment was added in 10 g TCE containing 0.1 g Span 85 as charge control agent. The color electronic ink forms after 30 min of ultrasonication. The white/color dual-particle electronic ink was prepared by mixing 0.1 g treated color pigment, 0.3 g poly[N-(p-vinyl benzyl)phthalimide]-grafted TiO_2 particles and 0.2 g Span 85 together in 20 g TCE under ultrasonication for 30 min.

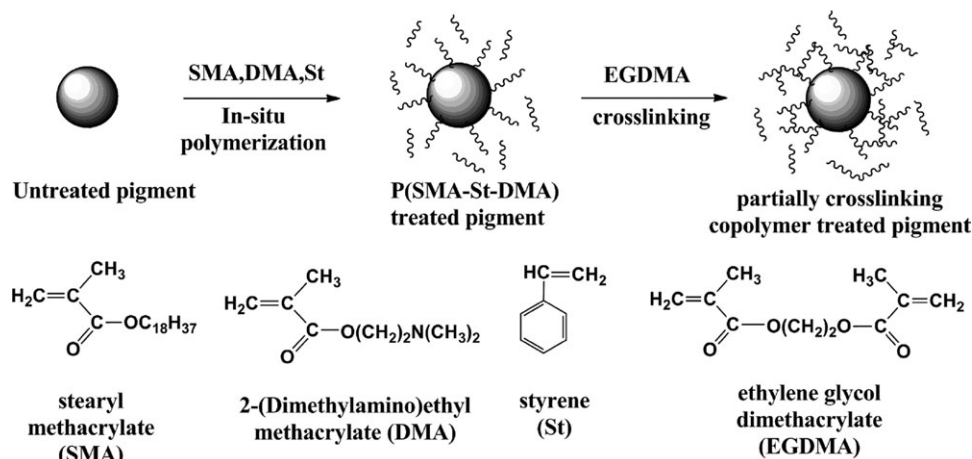
Characterization

Fourier-transform infrared spectra (FTIR) were recorded over the range of $400\text{--}4000 \text{ cm}^{-1}$ with potassium bromide pellet on a Bruker Vector 22 Fourier transform infrared spectrometer. The morphology of nanoparticles were observed on a transmission electron microscope (TEM, JEOL 200CX). Particle size distribution and Zeta potential were monitored by the Zeta-PALS from Brookhaven Instruments, USA. The reflectance spectra of the electronic ink were detected by a UV-vis spectrophotometer (UV-2450, Shimadzu).

RESULTS AND DISCUSSION

Preparation of the RGB Electrophoretic Particles

The electrophoretic particles are prepared by *in situ* treating the color pigments with the P(SMA-DMA-St) copolymer slightly crosslinked by EGDMA. The chemical structure of the RGB pigments is shown in Scheme 1. The schematic representation of the *in situ* modification of the pigment is given in Scheme 2. First, the pigment and monomer are mixed in toluene, and solution polymerization is initiated by AIBN. Parts of the



Scheme 2. Schematic representation of the *in situ* modification of the color pigments.

copolymer can be anchored onto the surface of the pigments because of the interaction between pigments and DMA. Then, a small quantity of EGDMA acting as crosslinking agent is added to bind the copolymer on the pigment surface.

Figure 1 gives the FTIR spectra of the pigment particles with and without surface modification. Compared with untreated red

pigments, new peaks at 2925 cm^{-1} , 2850 cm^{-1} , and 1730 cm^{-1} in Figure 1(a) can be detected in the spectra of the treated pigments. These peaks can be attributed to the $\text{sp}^3\text{ C-H}$ stretching and C=O vibration in polyacrylate segment of the P(SMA-DMA-St) copolymer. The peaks, 1450 cm^{-1} and 1143 cm^{-1} , assigned to the absorption of $=\text{CH}_2=$, $=\text{CH}_3$, and C=O=C groups in the copolymer can also be detected in the spectra of

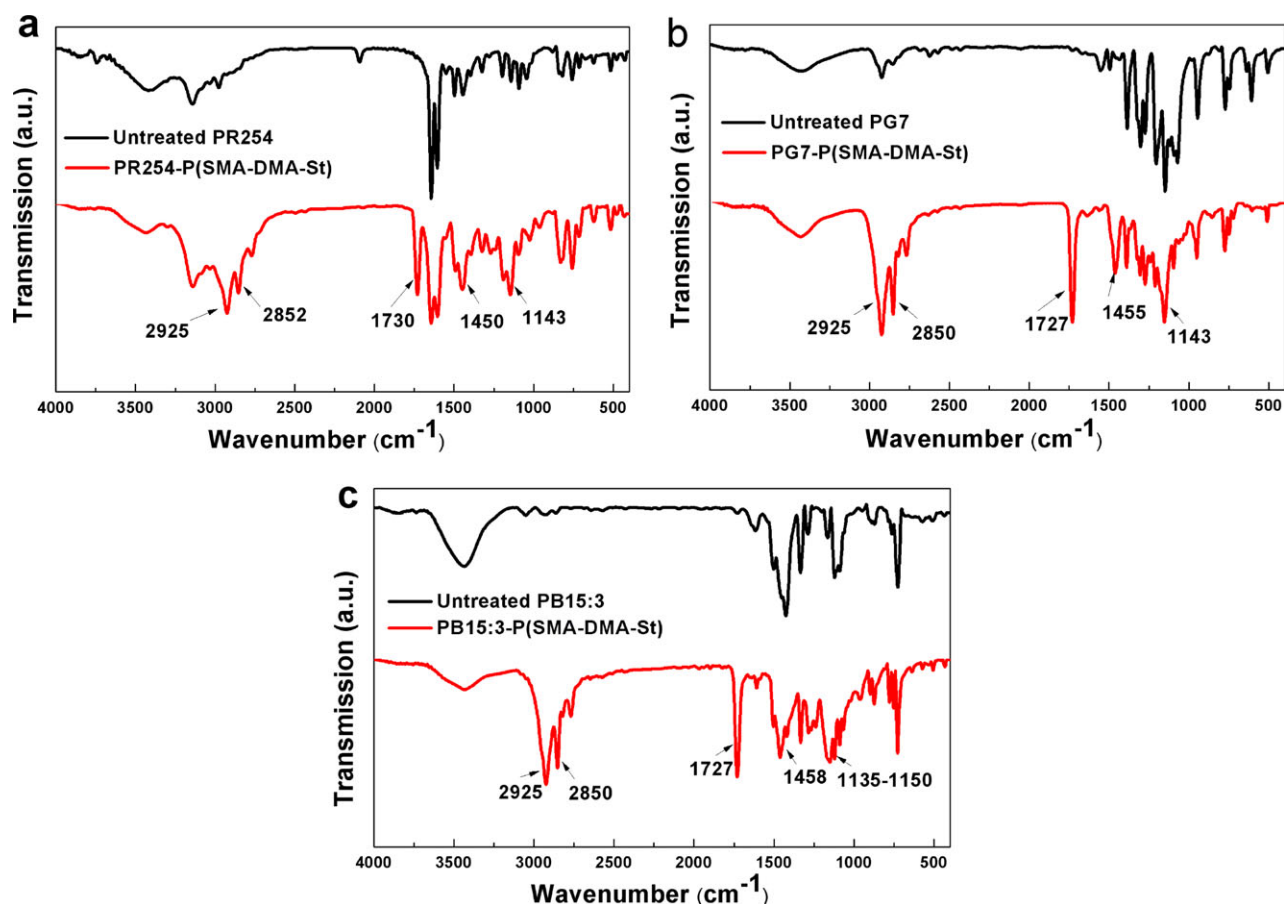


Figure 1. FTIR spectra of the color pigment with and without *in situ* modification. (a) red pigment PR254, (b) green pigment PG7, (c) blue pigment PB15:3. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

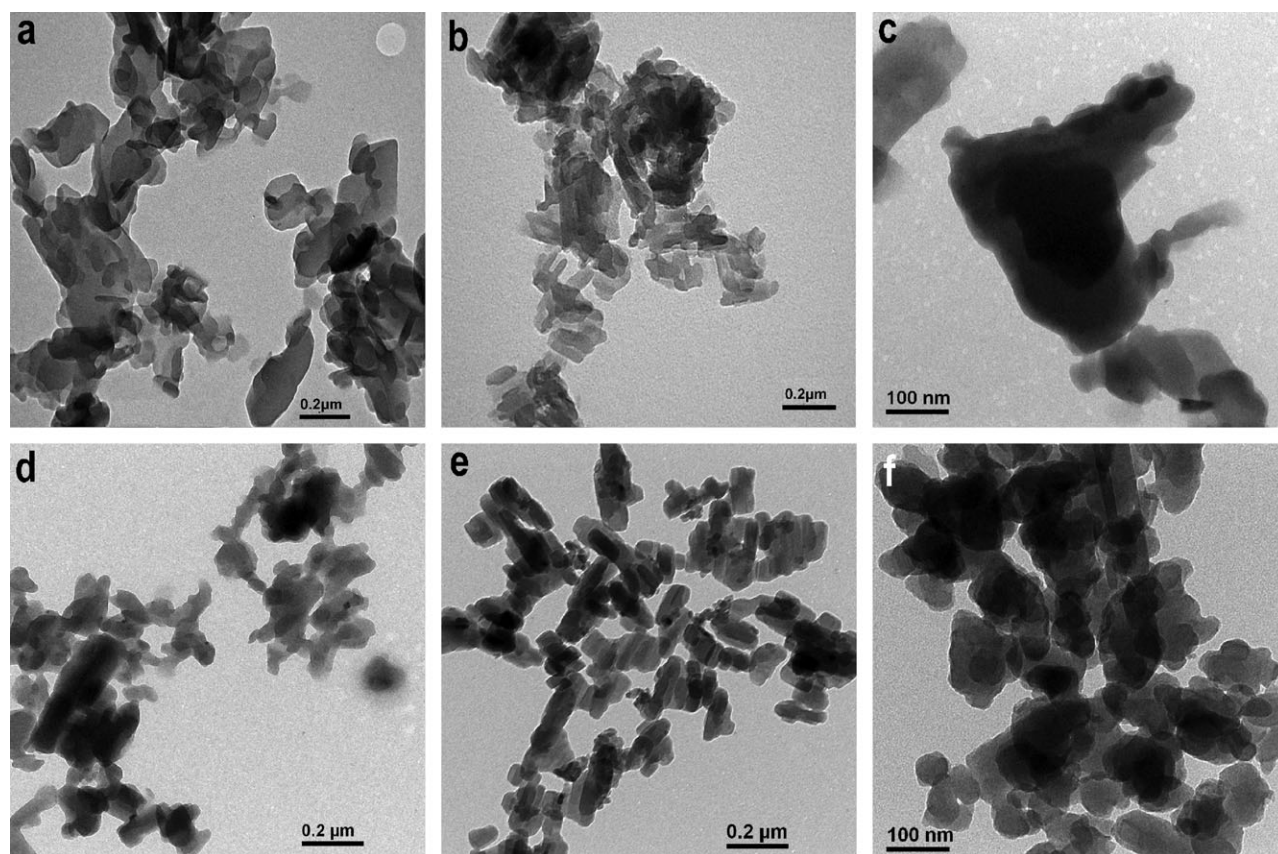


Figure 2. TEM images of the untreated red (a), green (b), blue (c) pigment particles and partially crosslinked copolymer modified red (d), green (e) and blue (f) pigment particles.

the treated particles. Figure 1(b,c) are similar. The characteristic peaks mentioned above can also be detected in the FTIR spectra of the modified green and blue pigments.

The TEM images of the RGB pigments with and without treatment are shown in Figure 2. To reduce the aggregation of the particles, the pigments were dispersed in TCE under ultrasonication. Sampling was done immediately after ultrasonic treatment. The appearance of the untreated pigment particles is irregular, as shown in Figure 2(a–c). From Figure 2(d–f), the morphology of the pigment particles after *in situ* modification seems more uniform than that of the untreated pigments. With the anchoring of copolymer onto the pigment surface, the dispersibility of the pigment in the nonpolar media can be improved because of the repulsive forces between the treated pigment particles resulting from the extending polyacrylate chain in the media.

The reflectance spectra of the RGB pigment powder with and without surface modification were given in Figure 3. Except for the red pigment, the peak reflectivity of the green and blue pigment decrease with surface modification. However, the peak position does not change much, which indicates P(SMA-DMA-St) modification does not bring large change to the color of the RGB pigments.

Electrophoretic Properties of the RGB Electrophoretic Particles

For the reason that the interaction between the P(SMA-DMA-St) copolymer and the pigment particles is not covalent bond,

the balance of absorption and desorption of the copolymer on the pigment surface could be established in the suspension system. This is not good for improving the suspension stability or electrophoretic properties of the pigment particles in the media. In this study, a small quantity of EGDMA monomer is applied to crosslink the P(SMA-DMA-St) copolymer, which helps to bind the copolymer on the pigment. Table I gives the average particle size, Zeta potential and the mobility of the RGB pigment particles treated by the copolymer with different amount of EGDMA addition. When EGDMA is added to crosslink the copolymer, the average diameter of treated pigment particles decreases obviously. If no EGDMA is added, the average diameter of the P(SMA-DMA-St) treated red pigment particle is 808 nm, which decreases to the value smaller than 500 nm with the addition of 0.003 mol EGDMA monomer. For the blue and green pigment, similar phenomena can also be detected, which can be attributed to the result of EGDMA addition. Crosslinking leads to the formation of network structure and restricts the extension of the copolymer chain in the nonpolar media on the one hand, and hinders the aggregation of the pigments on the other hand. Increasing the amount of EGDMA from 0.003 mol to 0.006 mol cannot make obvious influence on the size of the treated pigment particles. More important, the Zeta potential and the mobility of the treated particles are improved much after the addition of EGDMA. The Zeta potential of the uncrosslinked P(SMA-DMA-St) treated RGB pigment is -24.5 , -28.9 and -35.3 mV respectively. When 0.003 mol EGDMA is added,

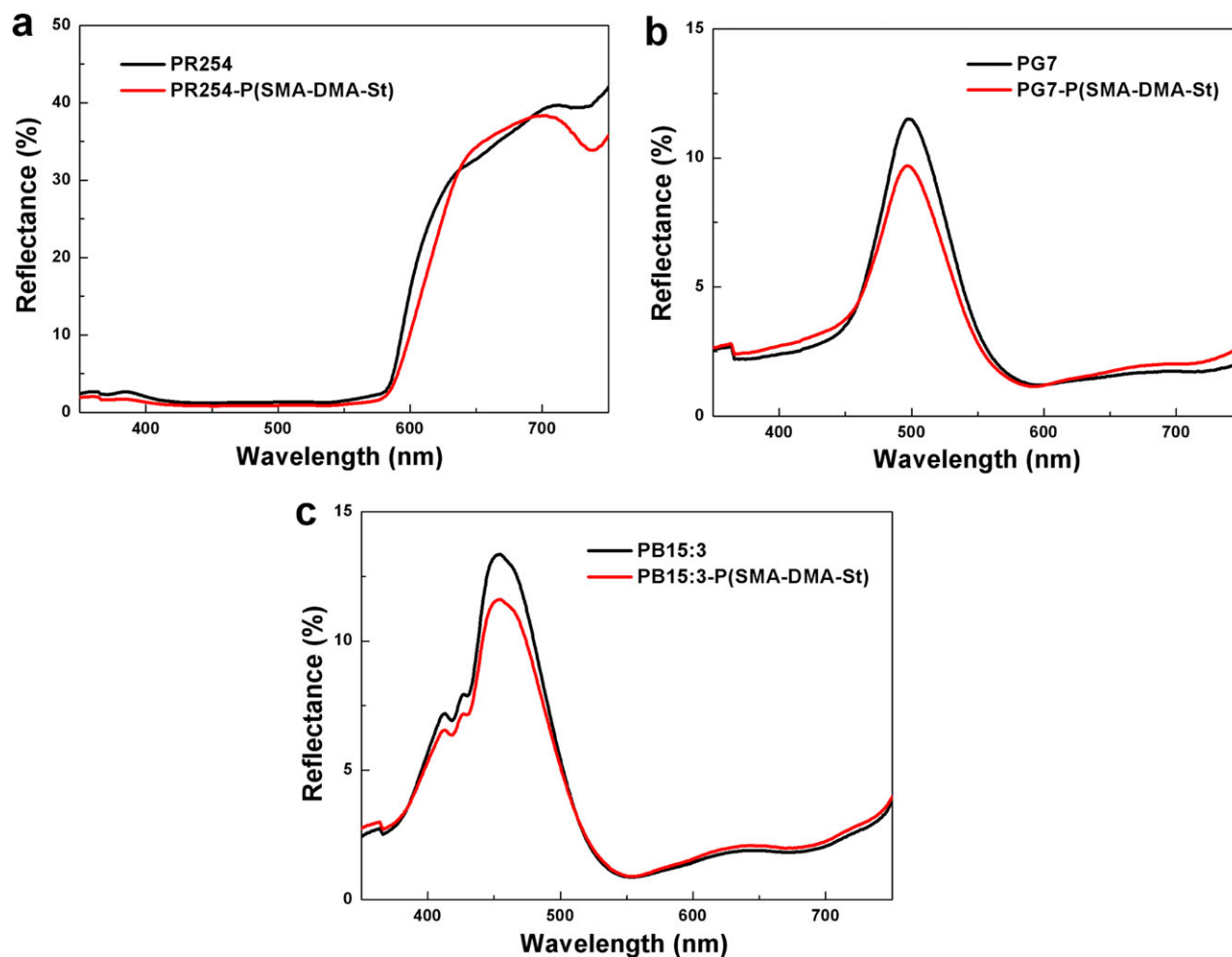


Figure 3. Reflectivity spectra of the color pigment with and without *in situ* modification. (a) red pigment PR254, (b) green pigment PG7, (c) blue pigment PB15:3. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

the Zeta potential of the RGB electrophoretic particles increases to -36.2 , -57.7 , and -40.5 mV respectively, and the corresponding electrophoresis mobility is -5.69×10^{-6} , -9.05×10^{-6} , and $-6.35 \times 10^{-6} \text{cm}^2 \text{V}^{-1} \text{s}^{-1}$. Higher Zeta potential and mobility of the electrophoretic particles are favorable to increase the performance of the EPD device. Further increasing the

amount of crosslinking agent brings little improvement to the electrophoretic properties of the particles.

Suspension Stability of the RGB Electrophoretic Particles

The dispersion and suspension stability of the electrophoretic particles in the media is very important for its application in

Table I. Mean Size, Zeta Potential, and Electrophoresis Mobility of RGB Electrophoretic Particles in Tetrachloroethylene

Particles	Crosslinking monomer (10^{-3}mol)	Mean size (nm)	Zeta potential (mV)	Mobility ($10^{-6} \text{cm}^2 \text{V}^{-1} \text{s}^{-1}$)
PR254-P(SMA-DMA-St)	0	808	-24.5	-3.83
	3	429	-36.2	-5.69
	6	491	-31.0	-4.87
PG7-P(SMA-DMA-St)	0	405	-28.9	-4.54
	3	192	-57.7	-9.05
	6	208	-45.4	-7.12
B15:3-P(SMA-DMA-St)	0	207	-35.3	-5.54
	3	182	-40.5	-6.35
	6	183	-42.7	-6.86

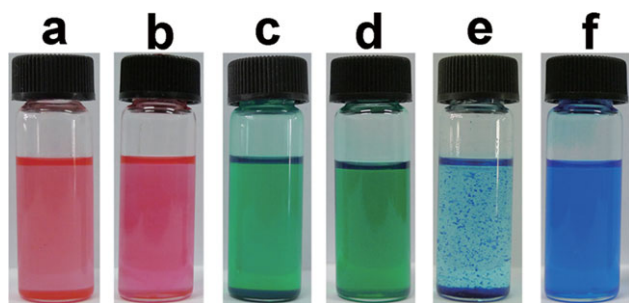


Figure 4. Suspension behavior of the untreated red (a), green (c), blue (e) pigment particles and *in-situ* treated red (b), green (d) and blue (f) pigment particles in TCE. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

the area of EPD. The images in Figure 4 give the comparison of the suspension stability of the RGB pigments with/without *in situ* modification after 24 h storage. Obviously, as shown in Figure 4(a,c,e), sediment can be found on the bottom of the bottle because of the aggregation of the untreated color pigments. Besides, agglomerates of the pigment particles can be found suspending in the blue bottle [Figure 4(e)]. On the contrary, in

Figure 4(b,d,f), the treated pigments can disperse in TCE for days and the precipitate of pigments can hardly be observed by naked eyes. This result indicates that *in-situ* modification of the color pigment introduced here is an effective way to improve the dispersibility of color pigments in TCE.

The suspension stability of the treated RGB pigment particles in TCE was studied by monitoring the variation of the particle size in the media. The particle size and particle size distribution for the partially crosslinked P(SMA-DMA-St) treated RGB pigments in TCE are given in Figure 5(a–c). The testing time selected was 0.5, 2, 6, and 24 h after storage (line A, B, C, D in Figure 5). The particle size and particle size distribution of the untreated pigment after 0.5 and 24 h storage are also given for comparison (line E, F in Figure 5). The increase of the particle size of the untreated color pigment can easily be detected because of the aggregation of the particles. The average size after 24 h storage is several times larger than the original value and the size distribution also varies much. For the *in-situ* treated RGB pigments, the particle size distribution is much narrower than that of the untreated one. The suspending stability is also much more stable. Figure 5(d) summarized the storage time dependence of the particle size and particle size distribution for the *in-*

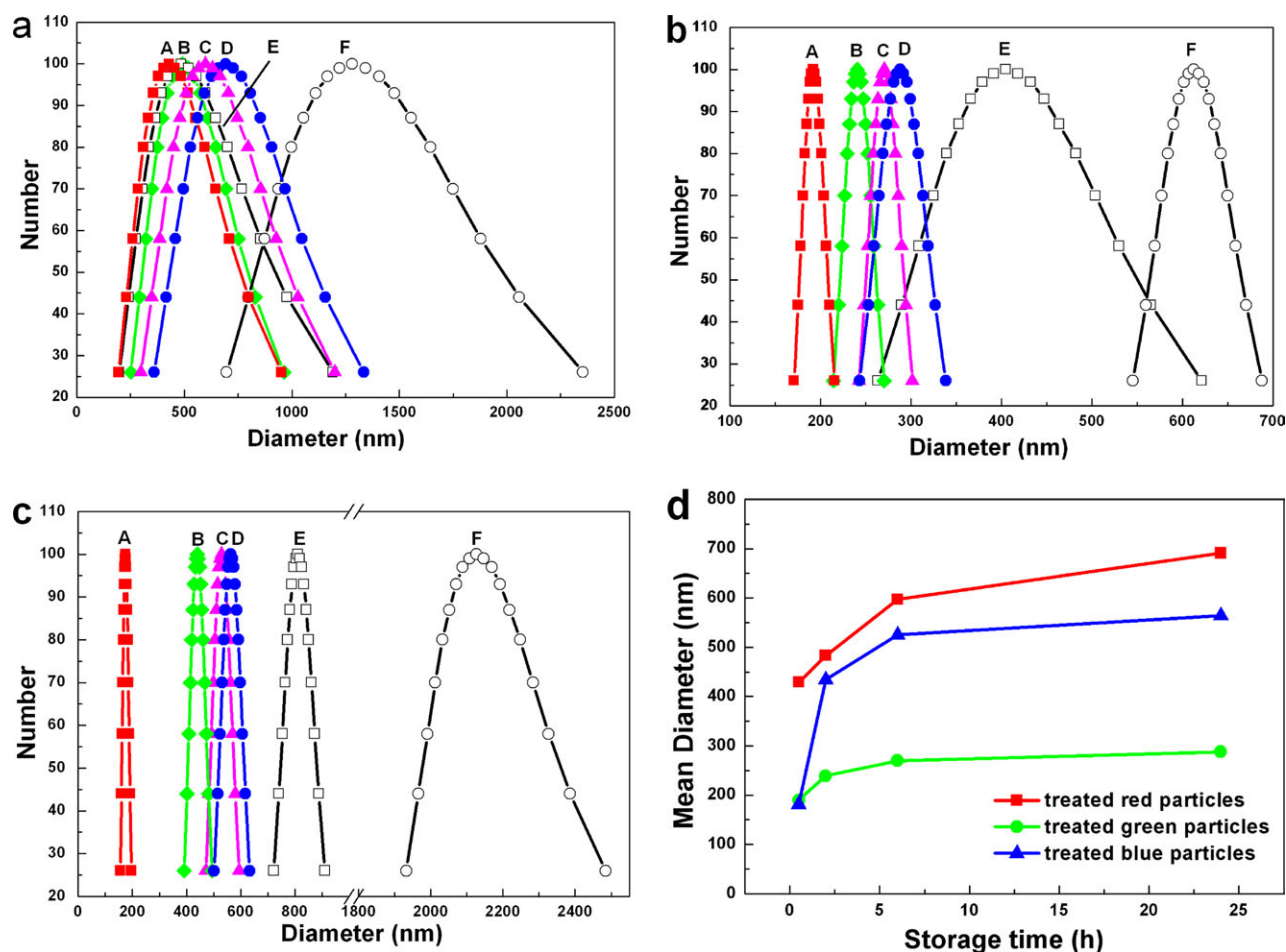


Figure 5. Particles size of the red (a), green (b) and blue (c) pigments in TCE with different time of storage. 0.5h (A, E), 2h (B), 6h (C), 24h (D, F). Treated pigments (A, B, C, D), untreated pigments (E, F). Storage time dependence of the average size for the treated RGB pigment particles (d). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

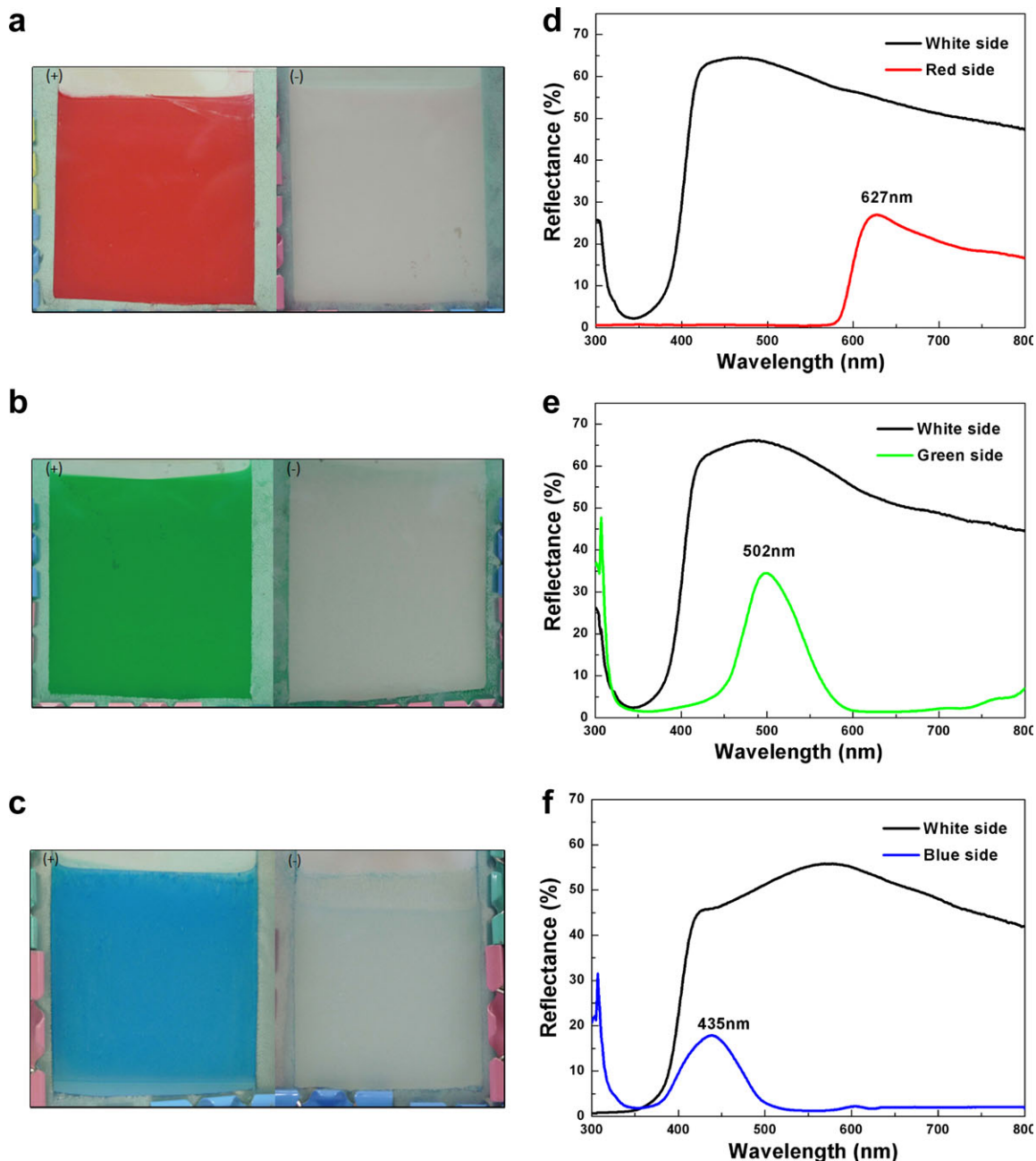


Figure 6. Images for the red/white (a), green/white (b), blue/white (c) dual-particle electrophoretic system under DC field of 45V/mm; the reflection spectra of the white and color side of the device prototype (d, e, f). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

situ treated RGB pigments. The particle size of the treated RGB pigments after 0.5 h storage is about 428.9, 191.8, 181.6 nm, respectively. After 24 h storage, the size respectively increase to about 692.2, 288.0, 563.7 nm. The growth of the particle size tends to be stable along with the extension of the storage time, which is in favor of the stabilization of the electrophoretic properties of the particles.

Fabrication and Character of the Electrophoretic Device Prototype

A self-made simple electrophoretic cell is applied to examining the performance of the dual-particle electronic ink. The electro-

phoretic cell is composed of two parallel ITO glass slices (5 cm × 5 cm) separated by a 600 μm spacer. The partially crosslinked P(SMA-DMA-St) modified RGB pigments is applied as color electrophoretic particles in the dual-particle electrophoretic system together with the charged poly[N-(p-vinyl benzyl)phthalimide]-grafted TiO₂ particles with average particles size of 433.6nm, Zeta potential of +41.5mV, mobility of $+6.52 \times 10^{-6} \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$. Under the DC field of 45 V mm⁻¹, as shown in Figure 6, the white particles migrate to the cathode side and the white image can be seen because TiO₂ can reflect most of the incident light. The maximum reflectivity is about 60–70%. For the reflective display, the contrast is the ratio of

Table II. The Peak Position of the Reflectance of the Color Side of the Color/White Display Under Irradiation of Different Time

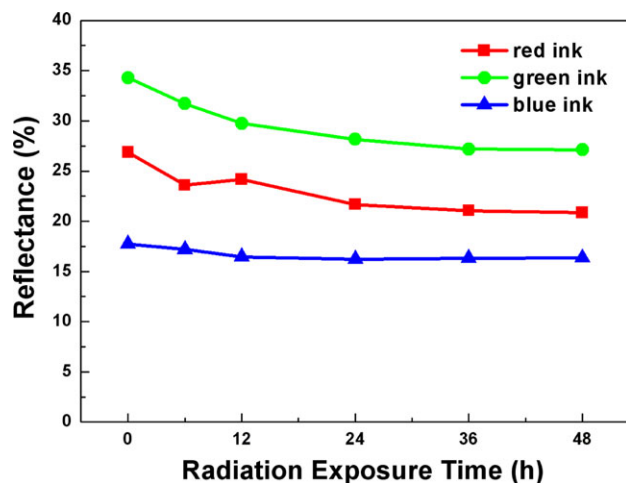
Dual-particle display prototype	Peak position of the color side under different irradiation time (nm)					
	0 h	6 h	12 h	24 h	36 h	48 h
Red/White	627	628	630	632	632	632
Green/White	502	500	499	499	498	498
Blue/White	435	441	442	443	444	443

the reflectivity of the white state to that of the black state. Higher reflectivity of the white state commonly results in higher contrast. On the anode side, the positively charged colored particles aggregate and the images with vivid color can be observed. The reflectance spectra of the color size were also given here. The response time of RGB device prototype is about 0.7~1 s, which should be further improved.

The light resistance of RGB electronic ink is important to the performance of the CEPD device. To examine the optical stability of the RGB electrophoretic particles in TCE, the self-made EPD device prototype was exposed to the irradiation of a Xenon lamp (500W). The radiation intensity of 9860 W m⁻², 14.5 times of the intensity of the midday sunlight (680 W/m²), was used accelerate the fading of the color pigments. Table II summarizes the peak position of the color-side reflectance of the device prototype under irradiation of different time. Obviously, the peak position is almost independent of the exposure time. The variation of the reflectivity at the reflectance peak with different irradiation time was shown in Figure 7. It is found that in the given 48 h, the fading of the green side is most obvious and the maximum decline of the reflectance is below 30%. The fading of the blue side is as slight as 10%. After 36-h exposure, the reflectance of the RGB side tends to stable, which is favor of the stabilization of the performance of the CEPD device.

CONCLUSIONS

In summary, partially crosslinked P(SMA-DMA-St) *in-situ* modified RGB electrophoretic particles are prepared via solution polymerization. Owing to the repulsive forces among the treated pigment particles resulting from the extending polyacrylate chain in the media, the surface modification hinders the aggregation of the particles and increases the dispersion and suspension stability of the pigment particles in TCE. Partial crosslinking helps to bind the copolymer on the pigment surface and improves the electrophoretic properties of the particles obviously. The maximum values of the Zeta potential for RGB electrophoretic particles reach -36.2, -57.7, and -42.7 mV, respectively. The treated RGB pigment particles can be applied in the color/white dual-particle electronic ink together with charged TiO₂. Under the DC field of 45 V mm⁻¹, the vivid color image can be detected. Fine light resistance of the chromatic electronic ink is advantageous to the long-time performance of the CEPD device.

**Figure 7.** Exposure time dependence of the reflectance of the color side of the color/white display. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

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