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ORIGINAL CONTRIBUTION

Self-organization of polymer particles on hydrophobic solid substrates in aqueous media. II. Self-organization of cationic polymer particles on polymer films and wettability control with particle monolayers

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Abstract Self-organization of cationic polymer particles through hydrophobic interaction on polymer films in aqueous system and characteristic properties of the resulting particle monolayers were investigated. Cationic polymer particles bearing quaternary ammonium groups on their surfaces effectively self-organized on polymer films. With an increase of the particle surface charge density, the surface coverage and average aggregate size (N_a) decreased. The surface coverage control was accomplished by tuning the ionic strength of the media. The wettability of polymer films for water was imparted by the formation of particle monolayers on them. Annealing of the particle monolayers resulted in the increase of the adhesive strength, while the wettability for water was lost. Further improvements of both wettability and adhesive strength of particle monolayers were achieved by the immobilization of silica colloids on the particle monolayers. This method would be effective for the hydrophilization of polymer films.

Keywords Cationic polymer particle · Self-organization · Particle monolayer · Hydrophobic interaction · Hydrophilization

Introduction

Several methods for preparing polymer particles that have monodispersity and various surface characteristics over a wide range of diameter have been established. Fabrication and application of particle assemblies using such monodisperse polymer particles have received much attention in recent years. For the fabrication methods of particle assemblies in two dimensions, there are physical and chemical methods such as the solvent evaporation method [1], Langmuir–Blodgett method [2], electrophoretic deposition method [3], and methods through chemical reaction and interaction between particles and substrates [4, 5]. The particle assemblies have many potential applications such as lithographic masks [6], anti-reflection surfaces [7], sensors [8], and microlens arrays [9, 10]. Furthermore, wettability control of substrates using particles has been also reported recently. Zhang et al. [11] fabricated wettability-gradient surfaces by changing polystyrene microsphere topography in a temperature-gradient field. Takeshita et al. [12] reported the wettability control in a rational manner by individually and simultaneously manipulating surface topography with particles and surface chemical structure.

Focusing on the surface characteristics of polymer particles, they have charges or hydrophilic groups on their surfaces that provide dispersion stability in aqueous system. On the other hand, surfactants adsorb even on polymer particles having similar kind of charges, and furthermore, proteins also adsorb on polymer particles [13]. It is well known that these adsorptions are caused through hydrophobic interaction, which indicates that polymer particles have hydrophobicity on their surfaces. Thus, the surfaces of polymer particles have both hydrophilic and hydrophobic natures. The amphiphilicity of particle surfaces is dependent on the polarity of the particle core, functional groups on the particle, the kind of charges, and the surface charge densities. Polymer particles resemble surfactants in many respects such as surface tension reduction of water by

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adsorption at air/water interface, stabilization of emulsions by adsorption at oil/water interface, and adsorption at charged solid surfaces [14–17]. Furthermore, surfactants are known to adsorb on hydrophobic solid substrates through hydrophobic interaction in aqueous media. However, to our knowledge, only a little has been known about the self-organization of polymer particles on hydrophobic solid surfaces.

We have reported that cationic polymer particles bearing sulfonium and quaternary ammonium groups self-organized on alkylated glass plates through hydrophobic interaction only over a certain concentration range to form aggregated or dispersed type of particle monolayers, depending on the hydrophobicity of substrates and hydrophilic–hydrohobic balance of particles [18–22]. In this work, we demonstrated that cationic polymer particles having different surface charge densities self-organized on polymer films and that the surface coverage was controlled by changing the ionic strength of the media. Furthermore, we investigated the wettability and adhesive strength of the resulting particle monolayers. These results would suggest new methods to fabricate particle monolayers on polymer films through interaction and to control over the wettability of hydrophobic materials for water.

Experimental

Materials and methods

Styrene (ST) and butyl acrylate (BA) were purified by distillation under reduced pressure in a nitrogen atmosphere. Water-soluble, cationic monomer, methacryloyloxyethylbutyldimethylammonium bromide (C_4Br) was prepared by the reaction of 2-(dimethylamino)ethyl methacrylate with *n*-bromobutane in acetone and purified by recrystallization from a mixture of acetone and acetonitrile (3:1 by volume). 2,2’-Azobis(2-amidinopropane) dihydrochloride (V-50) was used as radical initiator for the emulsifier-free emulsion copolymerization. N/400 potassium poly(vinyl sulfate) solution [N/400 potassium poly (vinyl sulfate) (PVSK)] was used with a dilution to N/20000. All reagents were purchased from Wako Pure Chemical Industries, unless otherwise noted. Water was distilled and deionized by a Millipore system before use. Silica 300 colloids (300 nm in diameter) were obtained from Nissan Chemical Industries and used as received. Silica 7 colloids (7 nm in diameter) purchased from Aldrich were dispersed in water by ultrasonic irradiation for 30 min at an output power of 6 and duty cycle of 50% (Branson, Model 250). Polymer films of polypropylene (PP), polyethylene (PE), and polycarbonate (PC) were received from Idemitsu Kosan, Chisso, and Asahi Glass, respectively.

Synthesis of cationic polymer particles

Poly(ST-*co*-C_4Br) particles bearing quaternary ammonium groups (latex code; CN-0.1, CN-0.5, CN-1.0, CN-2.0) were prepared by emulsifier-free emulsion copolymerization of ST with different amounts of C_4Br, as described elsewhere [23]. Poly(ST-*co*-BA-*co*-C_4Br) particles (latex code; CBN) having a lower T_g were synthesized by emulsifier-free emulsion copolymerization of ST (320 mmol) and BA (160 mmol) with C_4Br (3.2 mmol) at 60 °C using V-50 (3.2 mmol) as initiator. Those cationic polymer particles were purified by centrifugation and washed with deionized water several times. The number average diameter, D_n, and the coefficient of variation, C_v, were determined using a scanning electron microscope (SEM; JEOL, JSM-5310).

A colloid titration method was employed to determine the surface charge density of the polymer particles. The purified latex dispersion of the cationic polymer particles was titrated with N/20000 PVSK solution using toluidine blue as indicator. The charge density on the particle surface was expressed as equivalent mole of charge groups per unit area. The extent of flocculation in CN and CBN latex dispersions was examined according to the turbidity method [24]. CN latex dispersions containing NaCl as electrolyte at varying concentrations were allowed to stand at 25 °C for 24 h. The extent of flocculation in the latex dispersion was evaluated from *n* values, which can be determined from the variation of absorbance from 400 to 600 nm by ultraviolet-visible (UV-vis) spectroscopy (Shimadzu, UV-1600), according to the Eq. 1:

$$n \text{ value} = -\frac{d\,\log(\text{absorbance})}{d\,\log(\text{wavelength})}\,. \qquad (1)$$

The *n* value is constant for the latexes without flocculation and decreases upon the formation of secondary particles. As the electrolyte concentration is increased, the *n* value begins to decrease above a certain concentration that is defined as critical flocculation concentration (cfc). The characteristics of the resulting cationic polymer particles were summarized in Table 1.

Characterization of polymer films

The surface hydrophobicity of the films was characterized by contact angle measurements (Erma, goniometer type, model G-I) of deionized water (3.0 µl) at 25 °C.

Self-organization of polymer particles on polymer films

Self-organization experiments were conducted as follows: Polymer films (1.0×2.5×0.5 mm) of polycarbonate (PC), polyethylene (PE), and polypropylene (PP) were immersed

Table 1 Characteristics of cationic polymer particles produced by emulsifier-free emulsion copolymerization[a]

Latex code	C_4Br (mol% to ST)	Particle size[b]		Surface charge density[c] (μeq/m^2)	A_p^d (Å^2)	cfc[e] (mM)
		D_n(nm)	C_v(%)			
CN-0.1	0.1	336	3.1	1.30	125	50
CN-0.5	0.5	212	2.6	1.46	114	150
CN-1.0	1.0	203	2.4	1.62	103	250
CN-2.0	2.0	138	19.1	1.94	86	300
CBN	1.0	251	6.3	1.61	103	300

[a] Styrene, 320 mmol and 160 mmol (for CBN); *V*-50, 3.2 mmol; water, 160 g; temperature, 60 °C; stirring rate, 200 rpm; time, 12 h

[b] Determined by SEM. D_n, number average diameter; C_v, coefficient of variation of particle size distribution

[c] Determined by colloid titration

[d] Occupied area by a charged group

[e] Critical flocculation concentration (cfc) determined in NaCl solution

into ethanol for several days, taken out of ethanol, and dried in vacuum for 3 h. They were immersed into latex dispersion whose number of particles was adjusted to 5.4×10^{11} (1/ml) for 24 h, taken out of the dispersion, and washed in water by ultrasonic irradiation of 18 W at 42 kHz oscillation frequency for 5 min to remove weakly and/or physically adsorbed particles. The morphology of the particle monolayers was observed by SEM and analyzed by determining coverage and average aggregate size (N_a) from SEM photographs [25]. The coverage was calculated using Eq. 2:

$$\text{Coverage }(\%) = \frac{N}{N_{\max}} \times 100\,, \tag{2}$$

where N and $N_{\max}$ are the number of particles per unit area and the maximum number of particles with hexagonally closed packing, respectively. N_a is defined as

$$N_a = \frac{\sum n_i N_{a,i}}{\sum n_i}\,, \tag{3}$$

where $N_{a,i}$ and n_i are the number of particles per aggregate and the number of aggregates of i particles, respectively.

Hydrophilization of polymer films with particle monolayers

CN-1.0 or CBN particle monolayers on PC films were prepared to examine the wettability of particle monolayers for water. The wettability for water was evaluated by contact angles for water and wettability test. The latter was conducted as follows: The polymer films having particle monolayers were immersed into water for few seconds, and visual observation was then made on whether the water layer on them was maintained or not. Furthermore, the resulting particle monolayers were annealed in air or water at temperatures above the T_g of particles to increase the adhesive strength. The strength of the particle monolayers against peeling was estimated using adhesive tape. Silica colloids were deposited on particle monolayers to increase the wettability of annealed particle monolayers for water. The particle monolayers formed on the PC films were immersed into Silica 300 or Silica 7 colloidal solution (1 wt%) for 24 h at 25 °C, followed by washing with water. The morphologies of the composite particle monolayers were observed by a SEM or a field emission scanning electron microscope (FE-SEM; JEOL, JSM 6330F).

Results and discussion

Synthesis and characterization of cationic polymer particles

CN particles having quaternary ammonium groups on their surfaces were prepared by emulsifier-free emulsion copolymerization at different molar ratios of C_4Br to ST in feed. The resulting CN particles having different surface charge densities were used to investigate the effect of hydrophilic–hydrophobic balance of particles on the self-organization.

The characteristics of the cationic polymer particles are listed in Table 1. Four kinds of CN particles having different diameters and surface charge densities were obtained by varying the amount of C_4Br to ST in feed. With the increase of the amount of C_4Br to ST, the particle diameter decreased and the particle surface charge density increased. Thus, the surface charge density can be controlled within a certain range by varying the molar ratio of water-soluble comonomer to water-insoluble monomer as a main component for emulsifier-free emulsion polymerization, although the particle size changes concurrently. Since cationic charges at particle surfaces are hydrophilic groups, CN particle having higher surface charge density should be more hydrophilic. The cfc was determined by increasing the concentration of NaCl as an electrolyte added to the CN latex dispersion at a given concentration and refers to the concentration that the colloids begin to flocculate. It becomes higher for the particles having higher charge

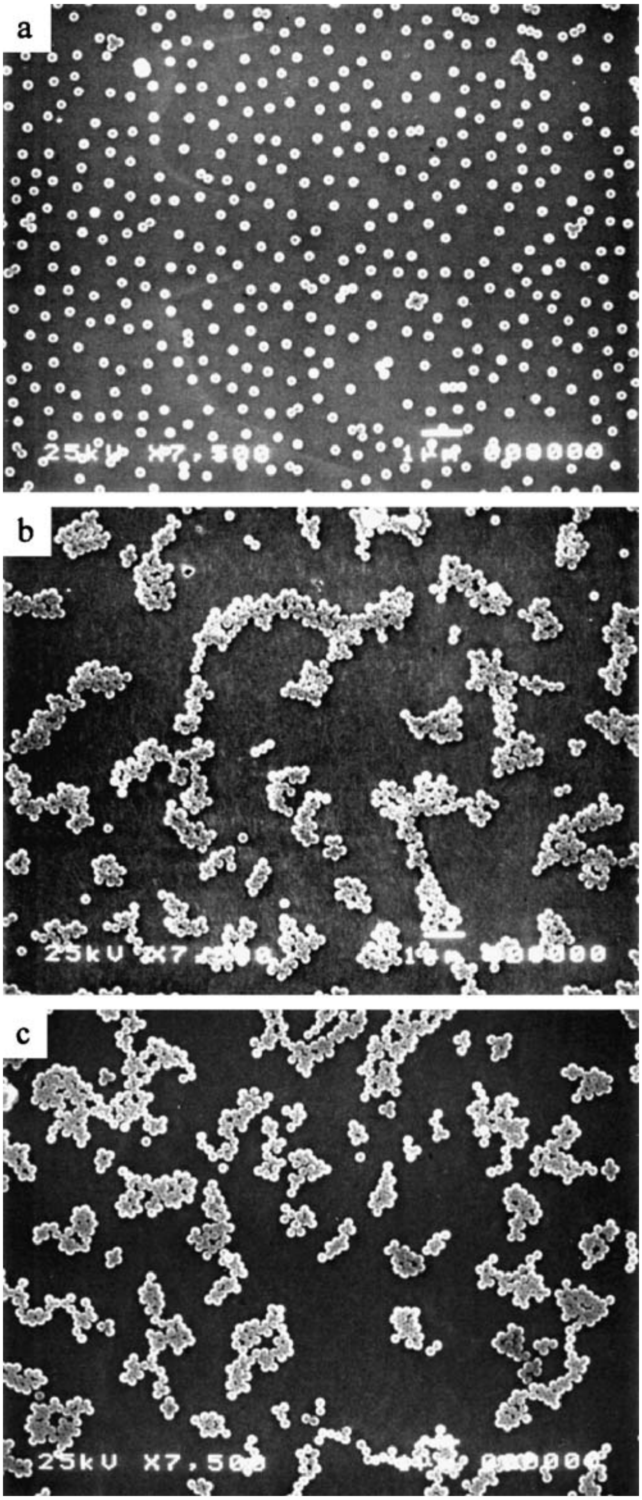

Fig. 1 **a**–**c** SEM photographs of CN-1.0 particle monolayers formed on polymer films. [Latex], number of particles=5.4×10^{11} (1/ml); temperature, 25 °C; time, 24 h. **a** PC film, **b** PE film, **c** PP film. The magnification of **a**–**c** is ×7,500

density, and hence, the hydrophobicity of particles can also be compared using the cfc values. Thus, CN-0.1 particle is the most hydrophobic and CN-2.0 particle is the most hydrophilic among these CN particles.

Self-organization of cationic polymer particles on polymer films

Yamaguchi et al. [20, 21] were the first to find that the cationic polymer particles bearing sulfonium groups on their surfaces well self-organized on hydrophobic solid substrates to form dispersed or aggregated type of particle monolayers dependent on the hydrophobicity of substrates.

In our previous paper, we reported the self-organization of cationic polymer particles bearing quaternary ammonium groups on alkylated glass plates through hydrophobic interaction and the fabrication of the close-packed type of

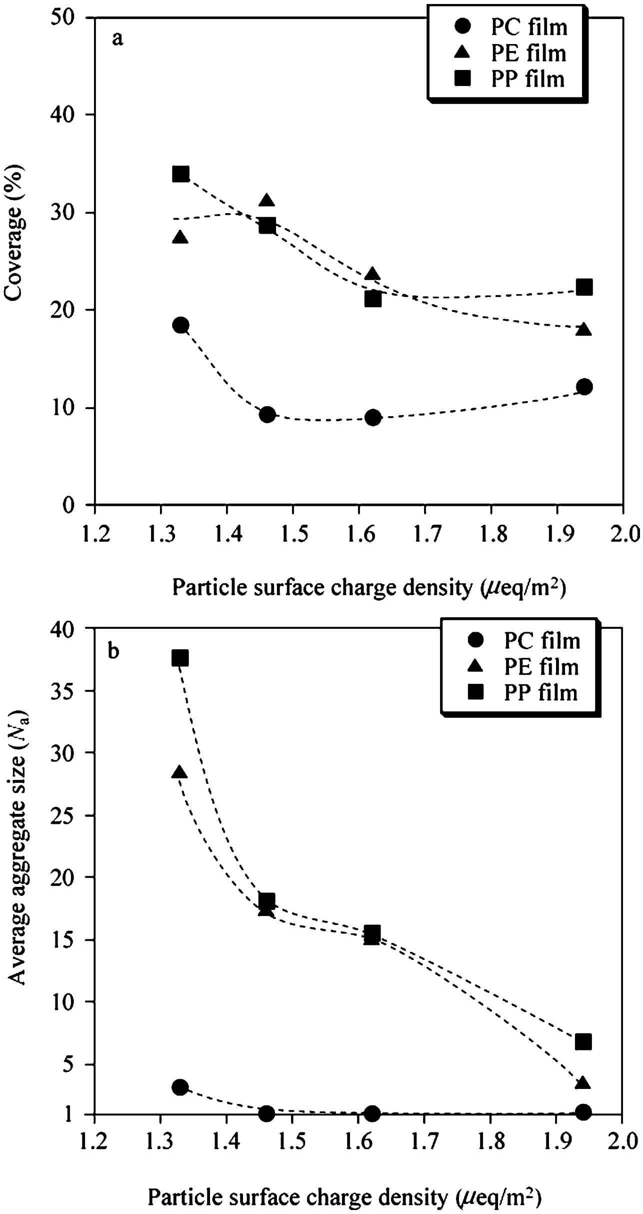

Fig. 2 **a** Coverage and **b** average aggregate size (N_a) of CN particle monolayers on polymer films against particle surface charge density. [Latex], number of particles=5.4×10^{11} (1/ml); temperature, 25 °C; time, 24 h

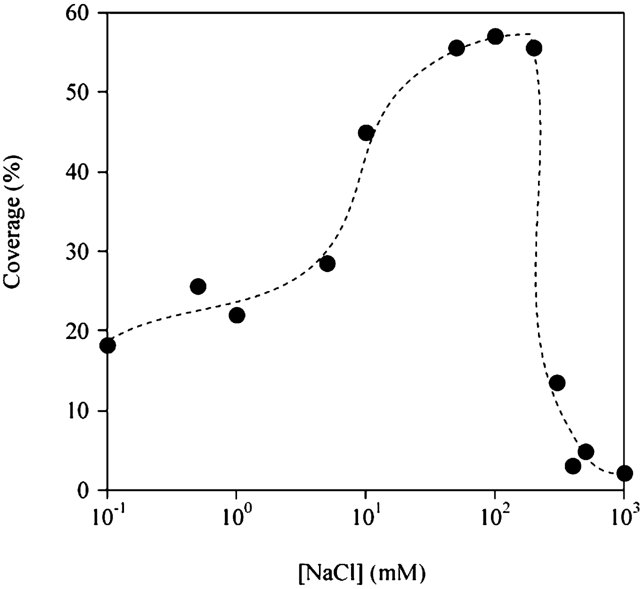

Fig. 3 Effect of electrolyte concentration on the coverage of PP films with CN-1.0 particles. [Latex], number of particles=5.4×10^{11} (1/ml); temperature, 25 °C; time, 24 h

particle monolayers with a relatively regular interval by tuning hydrophilic–hydrophobic balance of particles and temperature [22].

The average contact angles for water on the PC, PE, and PP films used in this work were 87.8°, 98.6°, and 103.4°, respectively, indicating that the surface hydrophobicity is in the decreasing order of PP film>PE film>PC film. Figure 1 shows SEM photographs of the CN-1.0 particle monolayers formed on the polymer films. CN-1.0 particles formed dispersed type of particle monolayers on less hydrophobic PC films. The self-organization on more hydrophobic PE and PP films yielded aggregated type of particle monolayers. The aggregates on PP films are slightly larger than those on the PE films. Figure 2 shows the coverage and the average aggregate size (N_a) of CN particle monolayers on the polymer films against the particle surface charge density. The average aggregate size (N_a) is related to the morphology of the particle monolayers. Larger aggregates are formed for higher N_a value, and the morphology of the particle monolayer is of aggregated type, while each particle self-organizes on the surfaces separately for N_a value close to 1, and the morphology is of dispersed type. The coverage gradually decreases with the increasing particle surface charge density, which is parallel to the hydrophilicity of particle surfaces. Furthermore, the coverages on more hydrophobic PP and PE films are higher than those on less hydrophobic PC films. These results strongly suggest that the particles self-organize on the substrate surfaces through hydrophobic interaction. N_a value also decreases with the increase of the particle surface charge density. The N_a value is higher on more hydrophobic substrates. Thus, the coverage and the morphology of the particle monolayers can be controlled by changing the particle surface charge density and the hydrophobicity of the substrates. These results are quite similar to those for the self-organization on alkylated glass plates reported previously [22].

Surface properties of charged polymer particles should be associated with the thickness of their electric double layer, which is a function of ionic strength of the media. Therefore, we investigated the effect of NaCl added as electrolyte on the particle monolayer formation. The effect of NaCl concentration on the coverage of PP films with CN-1.0 particles is shown in Fig. 3. The coverage on PP films greatly increases with the increasing NaCl concentration, exhibits a maximum approximately close to the cfc, and then decreases dramatically. When electrolytes are added to latex dispersions, the electric

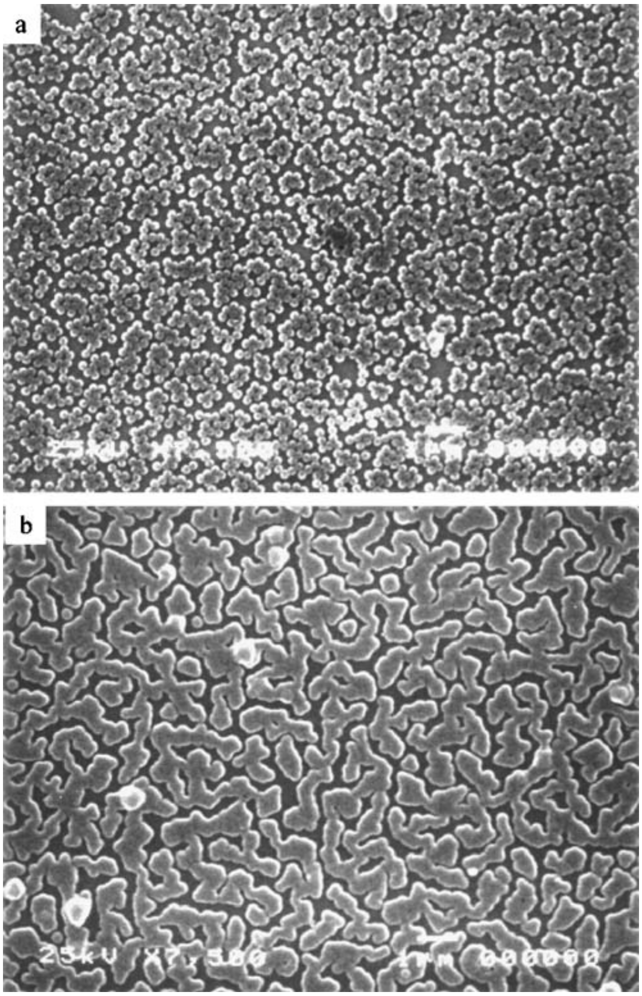

Fig. 4 **a**, **b** SEM photographs of CN-1.0 particle monolayers formed on PC films **a** before annealing and **b** after annealing at 120 °C for 1 h. Self-organization: [latex], number of particles=5.4×10^{11} (1/ml); temperature, 25 °C; time, 24 h; [NaCl], 100 mM. The magnification of **a** and **b** is ×7,500

Table 2 Surface properties of PC films with particle monolayers[a] and silica colloids[b]

Plate	Annealing temperature and time		Contact angle[c]	Wettability for water[d]	Adhesive strength[e]
	In air	In water	(degree)		
PC			87.8	×	–
CN-1.0/PC	Before annealing		76.9	○	×
	120 °C, 1 h		88.8	×	○
Silica 300/CN-1.0/PC	Before annealing		11.9	○	×
	130 °C, 30 min		88.2	○	○
	130 °C, 1 h		97.2	Δ	○
CBN/PC		Before annealing	103.1	○	×
		70 °C, 1 h	86.5	Δ	○
		60 °C, 1 h	94.9	Δ	○
		50 °C, 1 h	88.0	Δ	○
		40 °C, 1 h	88.0	○	×
		40 °C, 3 h	91.0	○	×
Silica 7/CBN/PC		Before annealing	~0	○	×
		60 °C, 1 h	~0	○	×
		70 °C, 1 h	49.1	○	○
		70 °C, 10 min	~0	○	○

[a] Self-organization: [latex], number of particles=5.4×10^{11} (1/ml); [NaCl], 100 mM; temperature, 25 °C; time, 24 h

[b] Deposition: [silica colloids], 1.0 wt%; temperature, 25 °C; time, 24 h

[c] Water, 3.0 or 2.0 µL

[d] ○, Good: Δ, dried rapidly: ×, poor

[e] ○, Adhered: ×, peeled off

double layer of particles is compressed [26]. Consequently, the electrostatic repulsion between particles on the substrates becomes weaker, resulting in the formation of more closely packed, aggregated type of particle monolayers. At NaCl concentrations above the cfc for CN-1.0 particles, particles begin to flocculate. Therefore, the preferential flocculation of the particles in aqueous phase would be responsible for the abrupt decrease of the coverage. Thus,

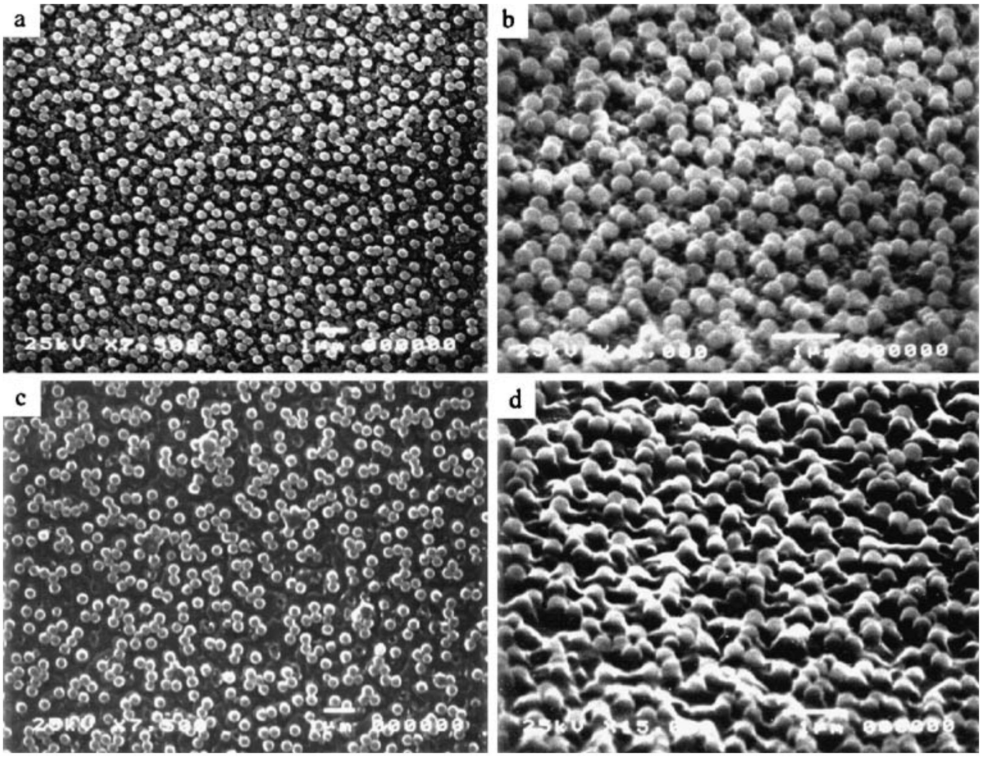

Fig. 5 a–d SEM photographs of silica colloids deposited on CN-1.0 particle monolayers. Deposition: [Silica 300], 1 wt%; temperature, 25 °C; time, 24 h. **a**, **b** Before annealing; **c**, **d** after annealing at 130 °C for 30 min; **a**, **c** top view; **b**, **d** side view. Magnification, **a** and **c** ×7,500; **b** and **d** ×15,000

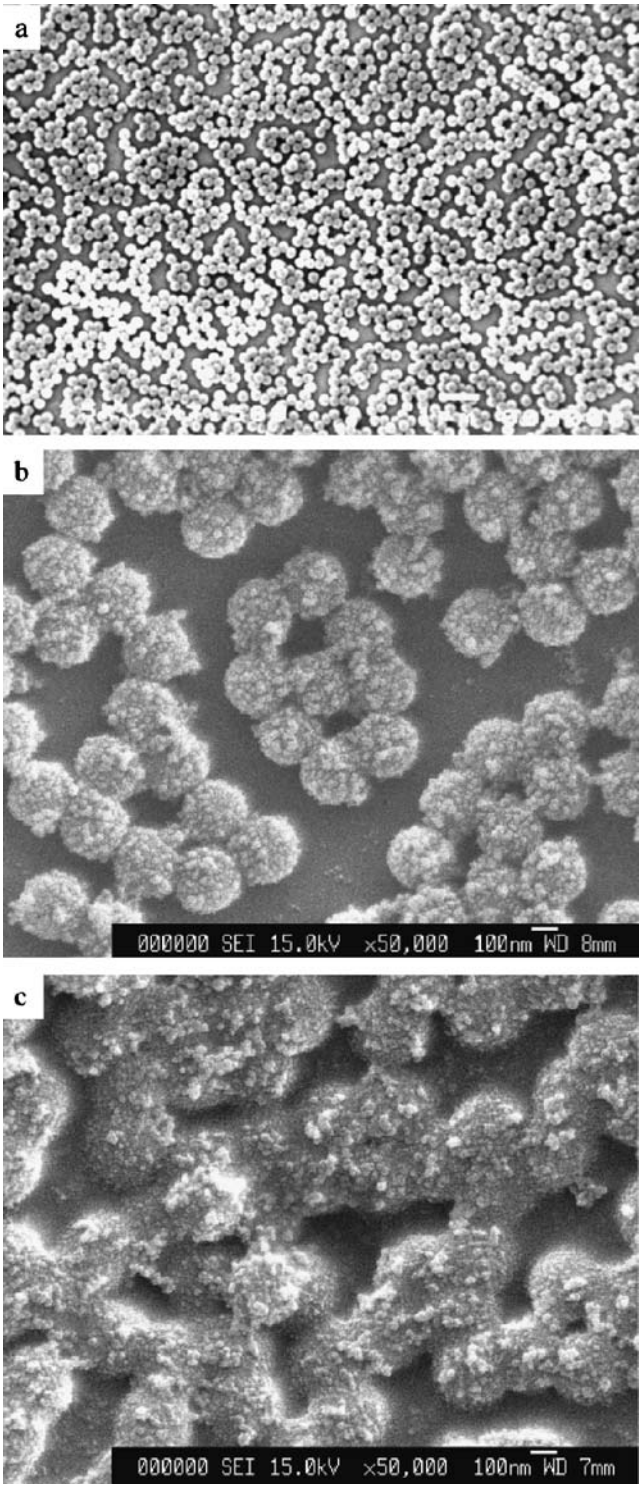

Fig. 6 a–**c** SEM and FE-SEM photographs of CBN particle monolayers formed on PC films. Self-organization: [latex], number of particles=5.4×10^{11} (1/ml); temperature, 25 °C; time, 24 h; [NaCl], 100 mM. Deposition: [Silica 7], 1.0 (wt%); temperature, 25 °C; time, 24 h. **a** CBN particle monolayers, **b** composite particle monolayers, and **c** after annealing the composite particle monolayers. Magnification, **a** ×7,500; **b** and **c** ×50,000

the hydrophobicity of particles can also be controlled by changing the electrolyte concentration (ionic strength) that leads to the variation of the coverage of particle monolayers.

Hydrophilization of polymer films with particle monolayers

The surface modification of polymer films was investigated using particle monolayers having hydrophilic cationic groups on their surfaces. Furthermore, the adhesive strength of the particle monolayers was also examined with respect to adhesion to substrates. PC films were mainly used as the substrate for surface modification. The wettability for water was evaluated by contact angles for water and visual observation. Figure 4a shows a SEM photograph of CN-1.0 particle monolayers formed on PC films in 100 mM NaCl solution, and the wettability and adhesive strength of the particle monolayers are summarized in Table 2. In general, polymer films exhibit low wettability for water and high contact angles due to low critical surface tensions [27]. As shown in Table 2, the wettability of PC films for water was improved by the formation of CN-1.0 particle monolayers. Furthermore, the contact angle also decreased, and hence, the surface of the polymer films turned hydrophilic by the cationic charges of CN particles. Thus, the fabrication of particle monolayers on polymer films can be used as hydrophilization method to increase the wettability for water. However, the adhesive strength between particle and substrate was so weak that the particle monolayers were easily peeled off. To improve both the wettability for water and the adhesive strength, the particle monolayers shown in Fig. 4a were annealed at temperatures above T_g of the particles in the air. Figure 4b shows the SEM photograph of CN-1.0 particle monolayers after annealing at 120 °C for 1 h in air. Annealed particle monolayers were strongly attached to the substrates and not peeled off, while the wettability for water was poor. This could be because the surface charges of polymer particles were buried inside the particles during annealing, owning to hydrophobic nature of air.

Next, silica colloids were deposited on particle monolayers, and then, the resulting composite particle monolayers were annealed. The CN-1.0 particle monolayers were immersed into Silica 300 colloid solution of 1 wt% for 24 h and then annealed at 130 °C in air. Figure 5 shows SEM photographs of composite particle monolayers containing Silica 300 on them before (Fig. 5a,b) and after (Fig. 5c,d) annealing. It can be seen that Silica 300 colloids are deposited separately on particle monolayers. The surface properties of Silica 300/CN-1.0 composite particle layers are shown in Table 2. After deposition of Silica 300 on particle monolayers, the contact angles for water greatly decreased. The wettability for water also became better, as expected, although the adhesive strength was still poor. The composite particle monolayers were annealed at 130 °C for

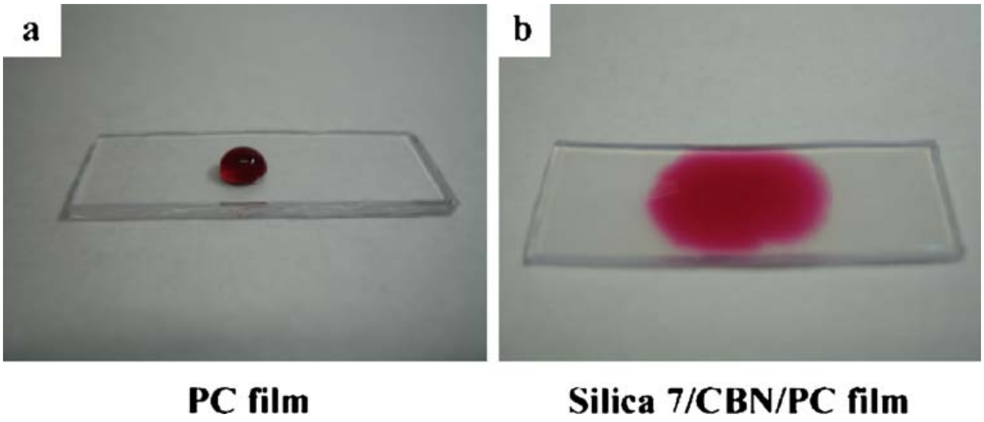

Fig. 7 **a**, **b** Digital photographs of a water droplet (5 μl) of aqueous acid red 27 solution on an unmodified PC film and **b** composite particle monolayer-modified PC film

30 min, resulting in good wettability for water and the high adhesive strength, while the contact angles for water were back to as much as that of PC films. When the composite particle monolayers were annealed at 130 °C for 1 h, the wettability for water disappeared. This could be caused by the transfer of silica colloids during particle monolayer deformation.

The annealing of particle monolayers in air resulted in the loss of the wettability for water. Therefore, CBN particles having lower T_g were self-organized on PC films and annealed in water. The characteristics of CBN particles are listed in Table 1. The T_g of CBN particles was calculated to be about 28 °C using Fox's [28] equation for the ratio of ST to BA units of 2:1 in the particles, which was determined by nuclear magnetic resonance (NMR) spectroscopy. Figure 6a shows a SEM photograph of CBN particle monolayers formed on PC films in 100 mM NaCl solution. The surface properties of CBN particle monolayers annealed in water were shown in Table 2. Although the adhesive strength was improved by annealing at 50 to 70 °C for 1 h, the water layer on the particle monolayers rapidly disappeared and the wettability for water was poor. However, the wettability of the annealed particle monolayer for water became better compared with that annealed in air. There was almost no change in the wettability for water and adhesive strength for annealing at 40 °C.

Smaller Silica 7 colloids were deposited on CBN particle monolayers and annealed in water to increase both the wettability for water and adhesive strength. Figure 7b shows a FE-SEM photograph of CBN particle monolayers containing Silica 7 colloids, and the surface properties were summarized in Table 2. Silica 7 colloids are selectively deposited on the particle monolayer and cover each CBN particle. Before annealing the Silica 7/CBN composite particle monolayers, the contact angle for water was too low to measure, indicating that Silica 7 colloids are also effective for making the particle monolayers hydrophilic. When the composite particle monolayers were annealed at 60 °C in water, the wettability for water was kept but the adhesive strength was little improved. CBN particle monolayers exhibited high adhesive strength on annealing at 60 °C in air. This difference might result from the increase in the melting point of particle monolayers caused by the smaller Silica 7 colloids. They seem to prevent from melting the composite particle monolayers. The annealing at 70 °C in water gave the films good wettability for water and high adhesive strength. The contact angles for water increased again for the particle monolayers annealed for 1 h, whereas the film annealed for 10 min was imparted with both very low contact angles and high adhesive strength. Figure 7 shows digital photographs of a droplet of aqueous acid red 27 solution on (a) unmodified PC film and (b) Silica 7/CBN/PC film. The droplet spread out on the latter film, indicating that the composite particle monolayers on polymer films works well for hydrophilization. Thus, the wettability of polymer films for water was improved by the formation of particle monolayers on them. Further improvements of both the wettability of the polymer films for water and the adhesive strength of the particle monolayers were successfully achieved by the immobilization of smaller silica colloids on the particle monolayers and subsequent annealing in water.

Conclusions

Monodisperse cationic polymer particles bearing quaternary ammonium groups effectively self-organized on polymer films through hydrophobic interaction to form particle monolayers. With the increase of particle surface charge density, the coverage and the average aggregate size of the particle monolayers decreased similarly as the self-organization on alkylated glass plates. High coverage of the aggregated type of particle monolayers was accomplished by adjusting the ionic strength of the media. The wettability of the polymer films for water was provided by fabricating particle monolayers on them. Further improvements of both good wettability of polymer films for water and high adhesive strength were achieved by the immobilization of silica colloids on the particle monolayers and subsequent annealing in water. This method will provide various applications to simple micro-surface modification of polymer films.

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