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INVITED PAPER: NANO-STRUCTURED MATERIALS (PARTICLES, FIBERS, COLLOIDS, COMPOSITES, ETC.)

Ordered arrays of electrostatically assembled SiO_2–SiO_2 composite particles by electrophoresis-induced stimulation

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Abstract

Monodispersed silica (SiO_2) nano- and microparticles can be fabricated by the sol-gel process. For device fabrication, precise formation of composite particles with a homogeneous and facile arrangement that form well-ordered, close-packed arrays is important. In this study, formation of electrostatically assembled SiO_2–SiO_2 composite particles with excellent homogeneity in arrangement was first demonstrated using sol-gel-derived monodispersed SiO_2 particles with average particles sizes of 200 nm and 16 μm. Formation of two- and three-dimensionally, close-packed arrays by electrophoresis-induced stimulation with direct-current and alternating-current electric fields was achieved for the first time using these electrostatically assembled SiO_2–SiO_2 composite particles. Detailed morphological observation by scanning electron microscopy revealed that the structure of the SiO_2–SiO_2 composite particles remained intact even after electrophoretic stimulation. The feasibility of obtaining well-ordered arrays of electrostatically assembled sol–gel-derived SiO_2–SiO_2 composite particles is important for further development of sol-gel-related technology in various applications, such as advanced composites and optical devices.

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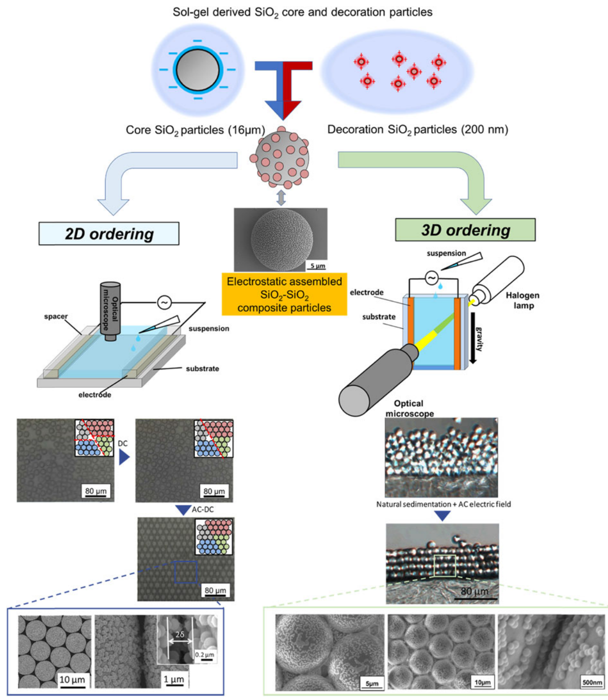
Graphical Abstract

Two- and three-dimensional ordering of electrostatically assembled SiO_2-SiO_2 composite particles using electrophoresis-induced stimulation



Keywords electrostatic nanoassembly · electrophoresis-induced stimulation · monodispersed silica particles · two-dimensional · three-dimensional · composite-particle ordering

Highlights

- Electrostatically assembled SiO_2–SiO_2 composite particles were prepared using monodispersed sol–gel-derived SiO_2 particles with two sizes.
- Ordered two- and three-dimensional SiO_2–SiO_2 composite-particle arrangements were obtained.
- Superimposition of AC and DC electric fields through electrophoresis-induced stimulation generated a two-dimensional close-packed structure.
- Simultaneous gravitational sedimentation and AC field application led to formation of three-dimensionally ordered arrays.

1 Introduction

Advanced nanomaterial fabrication by the sol-gel method has been used in various applications [1, 2]. Various methods for fabrication of silica (SiO_2) particles are available, such as spray drying [3], membrane emulsification [4], and the sol-gel method [5–8]. Use of the sol-gel method allows fabrication of high-quality monodispersed SiO_2 particles. Advancement of the sol–gel process has also enabled large-scale production of monodispersed SiO_2 particles with good size control, leading to rapid development of SiO_2-based device applications. To fully maximize this potential, arranging these monodispersed SiO_2 particles into periodic arrays or organized structures is important [9–12]. Formation of highly ordered colloidal arrays in thin or thick films has attracted the attention of researchers [13, 14].

Various methods have been used to form dense two- and three-dimensional structures using monodispersed SiO_2 colloidal suspensions, such as sedimentation [15], self-organization [16], the Langmuir–Blodgett method [13], cathodic electrodeposition [17], casting [7], self-assembly [18–20], a micromold and a groove [21], layer-by-layer assembly [22, 23], an electrostatic field [9], dip coating [24, 25], spin coating [14], and spraying of amorphous colloids [26]. Example applications that use a periodic array of SiO_2 nano- or microparticles are photonic glass/crystals [7, 14, 16, 18, 21], liquid displays [27], photonic band-gap control [11, 15, 28–30], optoelectronics [31, 32], structural color coatings [17, 26, 33, 34], superhydrophilic antireflective coating [22], superhydrophobicity [23], antifogging technology [35], reversible wettability [24], and gas sensing [19]. In a recent study reported by Xie et al. [19], the importance of the SiO_2-particle arrangement was further emphasized because they demonstrated that ordered-structure SiO_2 exhibited high selectivity in gas sensing of volatile organic compounds (methanol, ethanol, and isopropanol) compared with the disordered structure.

One method to form ordered SiO_2-particle films is electrophoretic deposition (EPD) of monodispersed SiO_2 particles. Fabrication of a SiO_2 film with a thickness of approximately 25 μm and no cracks has been reported by EPD of SiO_2 particles in the presence of poly(acrylic acid) [6]. In addition, by modifying the SiO_2-particle surface using either 3-aminopropyltriethoxysilane or vinyltriethoxysilane, formation of smooth thick films and inorganic–organic composite films with polyethylene maleate has been reported [5]. However, the stacking of the obtained SiO_2 particles was relatively random, which could hinder use in optical-related applications. For precision device applications, obtaining ordered arrays of SiO_2 particles in two and three dimensions is important [20, 29]. To form two- and three-dimensional close-packed structures using SiO_2 particles, Muto et al. [11, 12] used mechanical stimulation techniques, such as compression loading of particles sandwiched between solid bars and mechanical vibration. The aggregation process was also investigated by the discrete-element method, which revealed that gravitational sedimentation of SiO_2 colloidal particles is also important for formation of the three-dimensional close-packed structure. Various studies on the ordering of SiO_2 particles using different methods have been reported. However, only SiO_2 particles were used and not composite particles. In a previous study, we demonstrated the feasibility of composite-particle formation by electrostatic assembly to form SiO_2–SiO_2 composite particles [36]. SiO_2 decoration particles with sizes of 1 and 4 μm were homogeneously attached to the surface of 16-μm SiO_2 core particles. Because the ordering of electrostatically assembled SiO_2–SiO_2 composite particles has not been reported, it is important to investigate its feasibility and controllability in fabrication of two- or three-dimensional array films.

In the present study, using monodispersed sol–gel-derived SiO_2 particles, SiO_2–SiO_2 composite particles were first fabricated by the electrostatic assembly method. Subsequently, the ordering of the electrostatically assembled SiO_2–SiO_2 composite particles into periodic two- or three-dimensional array films by electrophoresis-induced stimulation was demonstrated. The novel findings and results of this study will be beneficial for material design using ordered composite-particle films with good scalability.

2 Experimental procedures

2.1 Formation of electrostatically assembled SiO_2–SiO_2 composite particles

Sol–gel-derived monodispersed SiO_2 particles with average particle sizes of 200 nm and 16 μm were obtained from Ube EXSYMO (Tokyo, Japan), and they were used as received. Scanning electron microscopy (SEM) images of the SiO_2 particles are shown in Fig. 1, showing their excellent monodispersitivity.

For electrostatic integrated assembly of the composite particles, the polyelectrolytes used for surface-charge modification were poly(sodium styrene sulfonate) (PSS) (weight-average molecular mass = 70,000, Sigma-Aldrich) and poly (diallyldimethylammonium chloride) (PDDA) (weight-average molecular mass = 200,000, Sigma-Aldrich). Water was used as the solvent, and the concentration of the polyelectrolytes (PSS and PDDA) was 1 wt.% with addition of 0.5 wt.% sodium chloride (Sigma-Aldrich). The concentration of the SiO_2 particles was 0.02 wt.%.

Surface-charge modification, pH adjustment, and the rinsing method were performed according to our previous report [36]. The 16-μm and 200-nm SiO_2 particles were used as the core and decoration (additive) particles, respectively. For the core SiO_2 particles, layer-by-layer polyelectrolyte adsorption in the order PDDA and PSS was performed to induce a negative surface charge. For the decoration particles, polyelectrolyte adsorption in the order PDDA, PSS, and PDDA was performed to induce a positive surface charge.

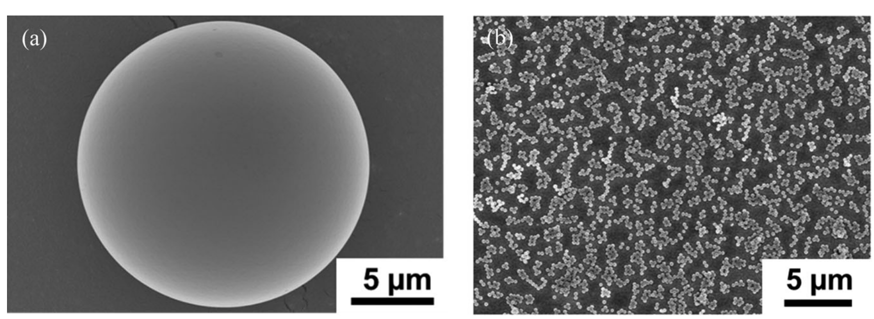


Fig. 1 SEM images of the sol–gel-derived SiO_2 particles with average diameters of **a** 16 μm and **b** 200 nm used for the electrostatic assembly process

Before mixing, the particle suspensions were dispersed by a homogenizer for 5 min. The mixture was then set aside for 3 h to allow electrostatic assembly of the SiO_2 particles [36]. The preparation processes of the two- and three-dimensionally ordered electrostatically assembled SiO_2–SiO_2 composite particles are described in Sections 2.2 and 2.3, respectively.

2.2 Formation of two-dimensionally ordered arrays by electrophoresis-induced stimulation using alternating-current and direct-current electric fields

A schematic and a photograph of the fabricated cell used for two-dimensional arrangement of electrostatically assembled SiO_2–SiO_2 composite particles by electrophoresis-induced stimulation are shown in Fig. 2. Before use, the glass slides were treated by the Radio Corporation of America (RCA) wet-cleaning procedure [37]. In the setup, parallel conductive pieces of tape attached to the RCA-cleaned slide glass were used as the electrodes. A cell or a chamber between the two electrodes was used in the electrophoretic stimulation process of the SiO_2 particles. The distance between the two electrodes was 10 mm.

The suspension consisting of SiO_2–SiO_2 composite particles in water was carefully introduced into the cell area

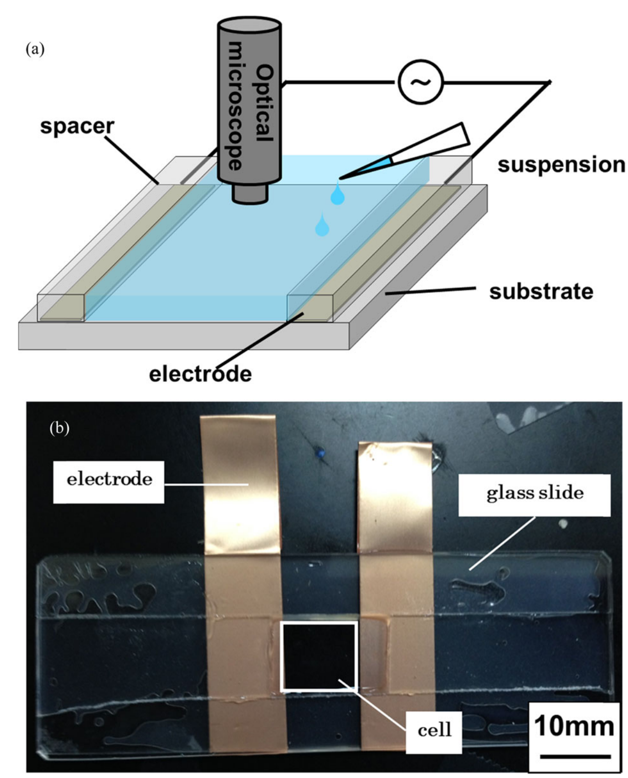


Fig. 2 **a** Schematic and **b** photograph of the fabricated cell used for two-dimensional arrangement of electrostatically assembled SiO_2–SiO_2 composite particles by electrophoresis-induced stimulation

and allowed to sediment. The concentration of the SiO_2–SiO_2 composite particles was 0.02 wt.%. Subsequently, a 5 V direct-current (DC) electric field was applied during the electrophoretic stimulation process. After the composite particles gathered at the electrode, an alternating-current (AC) electric field at 5 V with a frequency of 1 Hz was used to reorder the composite particles and remove voids. A sinusoidal AC electric field waveform was used. A programmable AC power supply (EC750S, NF Circuit Design Block) was used as the power supply.

2.3 Formation of three-dimensionally ordered arrays by concurrent gravitational sedimentation and electrophoresis-induced stimulation using an AC electric field

To form the three-dimensional periodic arrays, a different cell was fabricated and used. A schematic and a photograph of the cell are shown in Fig. 3. The cell consisted of two RCA-cleaned glass slides sandwiching two electrodes adhered to glass slides using conductive tape (thickness of approximately 70 µm). The bottom of the cell was sealed using grease, and the distance between the electrodes was 10 mm. The cell was vertically installed and filled with water.

The suspension of SiO_2–SiO_2 composite particles in water was then carefully dripped into the cell. The concentration of the SiO_2–SiO_2 composite particles was 0.02 wt.%. During gravitational sedimentation, an AC electric field was applied to induce oscillation of the composite particles. AC electric fields with voltages of 10 and 30 V were investigated, and a constant frequency of 1 Hz was used. For comparison, sedimentation of only primary SiO_2 particles and SiO_2–SiO_2 composite particles by gravitational pull was also performed.

To observe the particle-ordering structure, during the electrophoretic stimulation process, light transmitted from a halogen lamp was irradiated from one side of the cell and captured from the opposite side by an optical microscope. For the morphological observations, a laser microscope (OLS 4100, Olympus) and a field-emission scanning electron microscope (FE-SEM, Hitachi S-4800) were used. The zeta potentials of the suspensions were measured with a zeta-potential analyzer (ELSZ-1, Otsuka Electronics).

3 Results and discussion

3.1 Electrostatic assembly of SiO_2–SiO_2 composite particles

Although oxide materials exhibit the designated surface charge upon immersion in water, the charge distribution is uneven, which can lead to agglomeration and stacking faults [38, 39]. Therefore, adsorption of polyelectrolytes on the

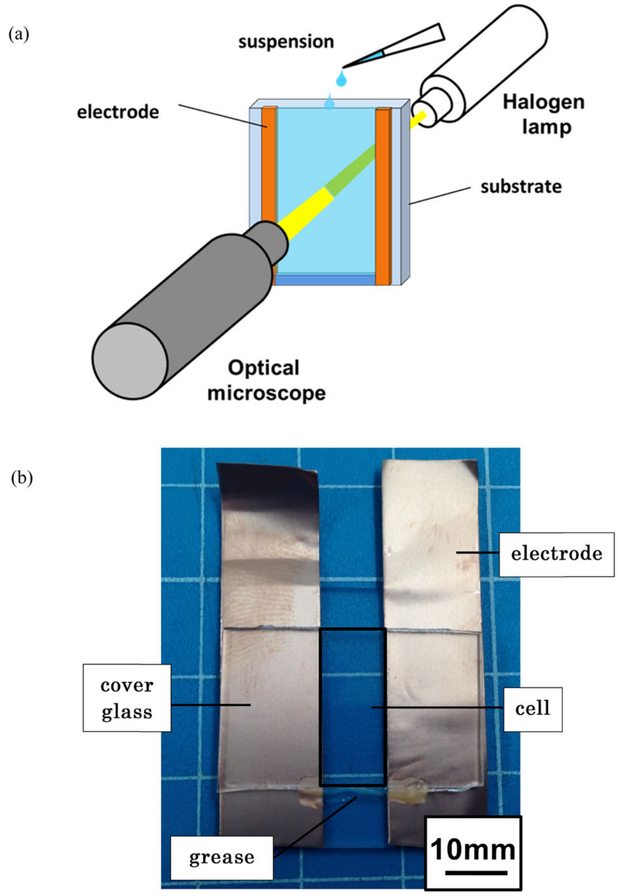


Fig. 3 **a** Schematic and **b** photograph of the fabricated cell used for three-dimensional arrangement of electrostatically assembled SiO_2–SiO_2 composite particles by electrophoresis-induced stimulation

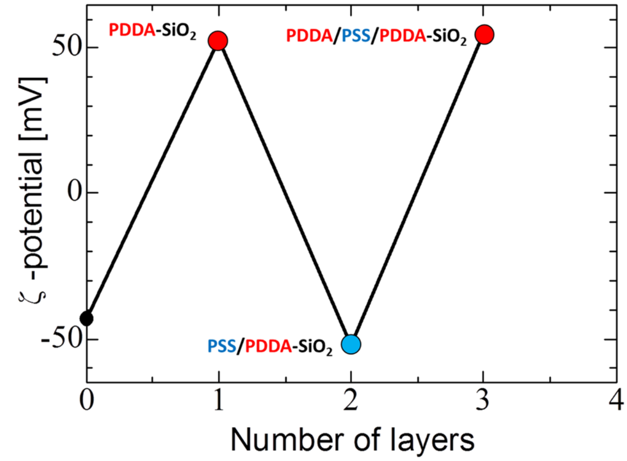


Fig. 4 Zeta potential of the SiO_2 particles obtained after surface-charge adjustment using PDDA and PSS polyelectrolytes

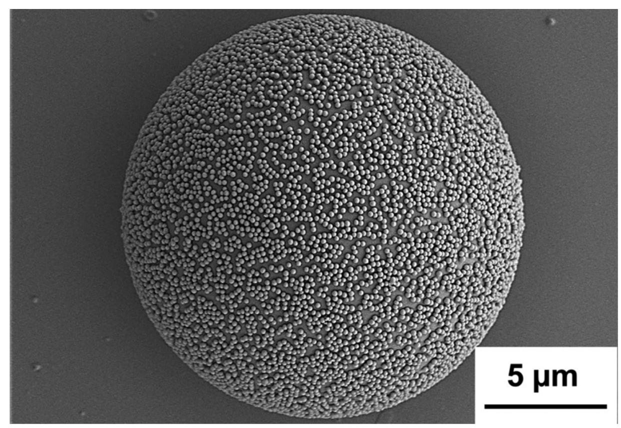


Fig. 5 SEM image of an electrostatically assembled SiO_2–SiO_2 composite particle

SiO_2 particles was performed to generate a more stable surface-charge density that could be used for electrostatic nanoassembly of materials [36, 40]. The zeta potential of the monodispersed SiO_2 particles after surface-charge modification using PSS and PDDA polyelectrolytes was measured (Fig. 4). The zeta-potential results revealed reversal of the surface charge of the SiO_2 particles from negative to positive upon adsorption of PDDA. Subsequent adsorption of PSS led to formation of a negatively charged surface. Because of the long polyelectrolyte chain, overcompensation of the charge reversal led to a slight increase in the zeta-potential strength in the inversion cycle [38, 41].

SiO_2–SiO_2 composite particles were fabricated by electrostatic assembly of negatively charged SiO_2 core particles with positively charged SiO_2 decoration particles. The electrostatically assembled SiO_2–SiO_2 composite particles were observed by SEM (Fig. 5). A very homogeneous distribution of SiO_2 decoration particles was observed on the surface of the SiO_2 core particles. The zeta potential obtained for the composite particles was approximately + 20 mV. Full coverage of SiO_2 decoration particles on the core particles was not achieved because of the steric hindrance effect generated between the core and decoration particles, as well as electrostatic repulsion between adjacent SiO_2 decoration particles [36, 42]. The steric hindrance effect is caused by the polyelectrolytes adhered to the surface of the SiO_2 particles because the long polymeric structure forms bulky chains that prevent close contact of the particles. This electrostatically assembled SiO_2–SiO_2 composite particles were then used to create two- and three-dimensionally ordered structures by electrophoresis-induced stimulation.

3.2 Formation of two-dimensionally ordered arrays by electrophoresis with AC and DC electric fields

Ordering of the electrostatically assembled SiO_2–SiO_2 composite particles was first carried out without electric-field application. A disoriented structure with large voids was observed (Fig. 6(a)). A DC electric field was then applied, which led to an improved arrangement of SiO_2 core

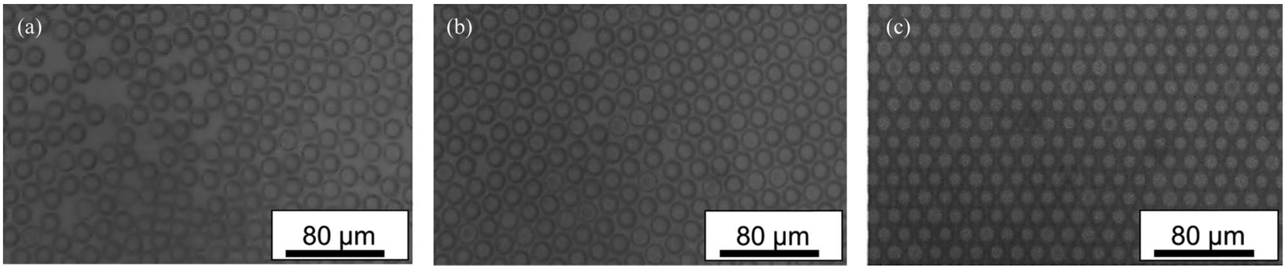


Fig. 6 Optical microscope images of the electrostatically assembled SiO_2–SiO_2 composite-particle arrangement in two dimensions **a** without electric-field application, **b** using only a DC electric field, and **c** with superimposition of AC and DC electric fields during electrophoresis-induced stimulation

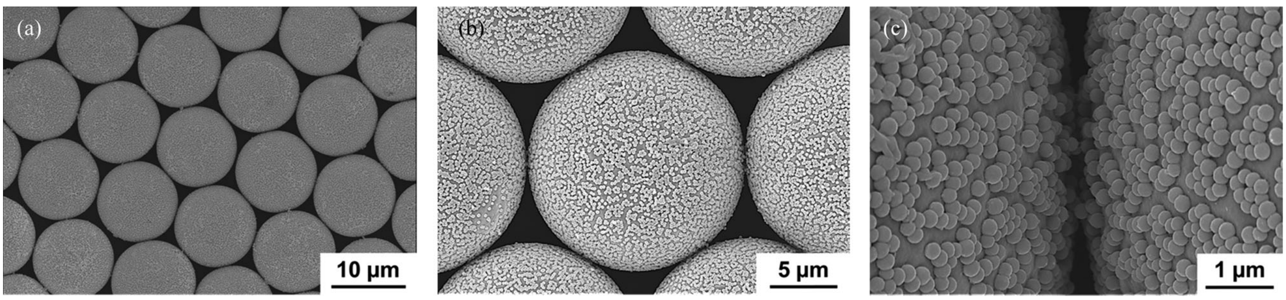


Fig. 7 SEM images of the electrostatically assembled SiO_2–SiO_2 composite particles arranged in two-dimensional ordered arrays at **a** low and **b**, **c** high magnification

particles with no large voids and a more packed structure in two dimensions (Fig. 6(b)). Even with application of a DC electric field, a close-packed arrangement was not obtained because small voids still formed. This is believed to be caused by the friction among the particles and the charge-distribution inhomogeneity on the surface of the composite particles affecting the interaction with the applied DC electric field, hindering the reordering process. Therefore, to obtain a close-packed hexagonal structure of SiO_2–SiO_2 composite particles, a sequence of AC and DC electric fields was applied during electrophoresis-induced stimulation, which resulted in formation of ordered two-dimensional arrays (Fig. 6(c)).

Detailed morphological observation of the two-dimensional close-packed SiO_2–SiO_2 composite particles was performed by SEM, and the SEM images are shown in Fig. 7. In the low-magnification image (Fig. 7(a)), compact, voidless, and well-ordered monodispersed SiO_2 core particles can be observed. Observation at higher magnification (Fig. 7(b, c)) revealed a homogeneous distribution of SiO_2 decoration particles adhered to the surface of the SiO_2 core particles. This showed that the electrostatically assembled SiO_2–SiO_2 composite particles were stable and did not detach during the electrophoretic stimulation process, even after superimposition of AC and DC electric fields. This indicates that electrostatically assembled SiO_2–SiO_2 composite particles can be ordered using electrophoresis-induced stimulation by adjusting the type of applied electric field.

A two-dimensional close-packed structure can be obtained by introducing AC and DC electric fields during the electrophoresis-induced oscillation process. The generated external stimulation can also promote rearrangement of the composite particles through cooperative interparticle or particle-cluster sliding [43, 44]. A gap of approximately 200 nm (the size of the SiO_2 decoration particles) formed at the interface between the SiO_2 core particles, as shown in Fig. 7(c). This indicates that it is possible to control the interparticle gap between the SiO_2 core particles using decoration particles with different sizes.

3.3 Formation of a three-dimensionally ordered array by gravitational sedimentation and electrophoresis with an AC electric field

To further investigate the ordered three-dimensional arrays, first, the effect of the gravitational sedimentation force on the stacked arrays using only the as-received SiO_2 core particles (without surface-charge modification) was investigated. Gravitational sedimentation is a simple yet effective technique for three-dimensional particle-array formation [11]. Sequential optical-microscope images showing the sedimentation process of the SiO_2 core particles are shown in Fig. 8(a–c). The first two layers appeared to be well-

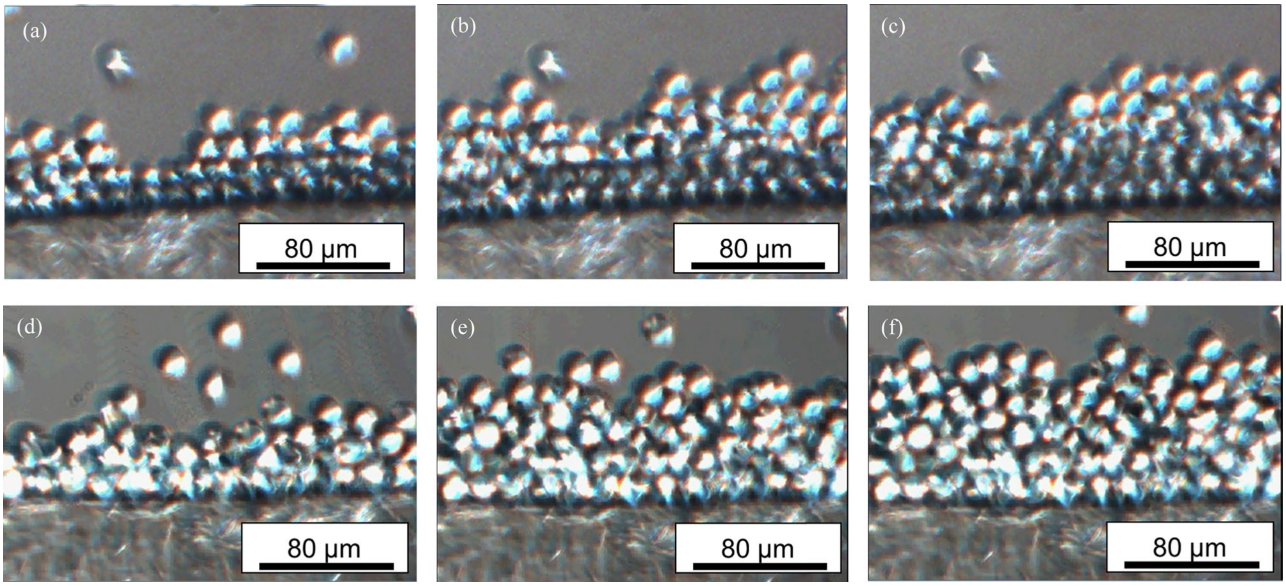


Fig. 8 Sequential optical microscope images of **a**–**c** primary SiO_2 particles and **d**–**f** electrostatically assembled SiO_2–SiO_2 composite particles by gravitational sedimentation

ordered, and the subsequent SiO_2 particles were randomly dispersed. This could be caused by the inhomogeneous repulsive force between similarly charged particles, generating aggregates and voids that gradually accumulate and disrupt the well-ordered arrangement. This phenomenon is consistent with the observation by Li et al. [30], who observed formation of agglomerates owing to the weak repulsive forces in the solution, promoting rapid Brownian coagulation that tends to form random packing states.

A similar gravitational sedimentation study was performed using the electrostatically assembled SiO_2–SiO_2 composite particles. Optical images of the electrostatically assembled SiO_2–SiO_2 composite particles are shown in Fig. 8(d–f). The particle ordering was more disoriented than that obtained using only the SiO_2 core particles. This phenomenon could be because of the rougher surface of the electrostatically assembled SiO_2–SiO_2 composite, resulting in a higher frictional barrier and an interlocking effect among the SiO_2 decoration particles. External stimulation with sufficient energy could promote reshuffling and reordering of the particles, enabling formation of close-packed three-dimensional arrays. Muto et al. [11] reported that the mechanical vibrational movement obtained using a piezoelectric actuator is efficient for reordering SiO_2 microparticles to form close-packed aggregates. From the abovementioned finding, application of an AC electric field to generate oscillating movements is believed to enable formation of two-dimensionally ordered SiO_2–SiO_2 composite-particle arrays. The oscillating vibrational movement generated using an AC electric field enables repositioning and reordering of the particles through interparticle sliding. Therefore, to achieve a close-packed three-dimensional array, an AC electric field was applied during the gravitational sedimentation process.

Optical images of the electrostatically assembled SiO_2–SiO_2 composite particles during the stacking process at different stages of electrophoresis-induced stimulation using an AC electric field with an amplitude of 10 V at a frequency of 1 Hz are shown in Fig. 9(a–c). Only the initial layers were orderly arranged, while the subsequent layers appeared to be disoriented with line defects, indicating that the supplied energy was insufficient to completely reorder the particles to form a close-packed array. Therefore, the amplitude of the AC electric field was increased to 30 V at a frequency of 1 Hz, and optical microscope images were obtained (Fig. 9(d–f)). At the higher AC electric voltage, a stronger vibrational amplitude was introduced to the SiO_2–SiO_2 composite particles, causing interparticle slip to occur among the composite particles for further rearrangement and void removal, forming an ordered close-packed array, as shown in Fig. 9(f). Although a voltage of 30 V was applied at a rather low frequency of 1 Hz, gas bubble generation was not detected, suggesting the absence of water electrolysis. To further demonstrate that there was no gas bubble generation, a supplementary video showing the formation of ordered SiO_2–SiO_2 composite particles under gravitational sedimentation and the electrophoresis-induced stimulation using AC electric field at 30 V with a frequency of 1 Hz, is included as Supplementary file 1. It has also been reported that AC electric field application during electrophoresis can be used to suppress water electrolysis and prevent coalescence of gas bubbles [45–47]. In the study of

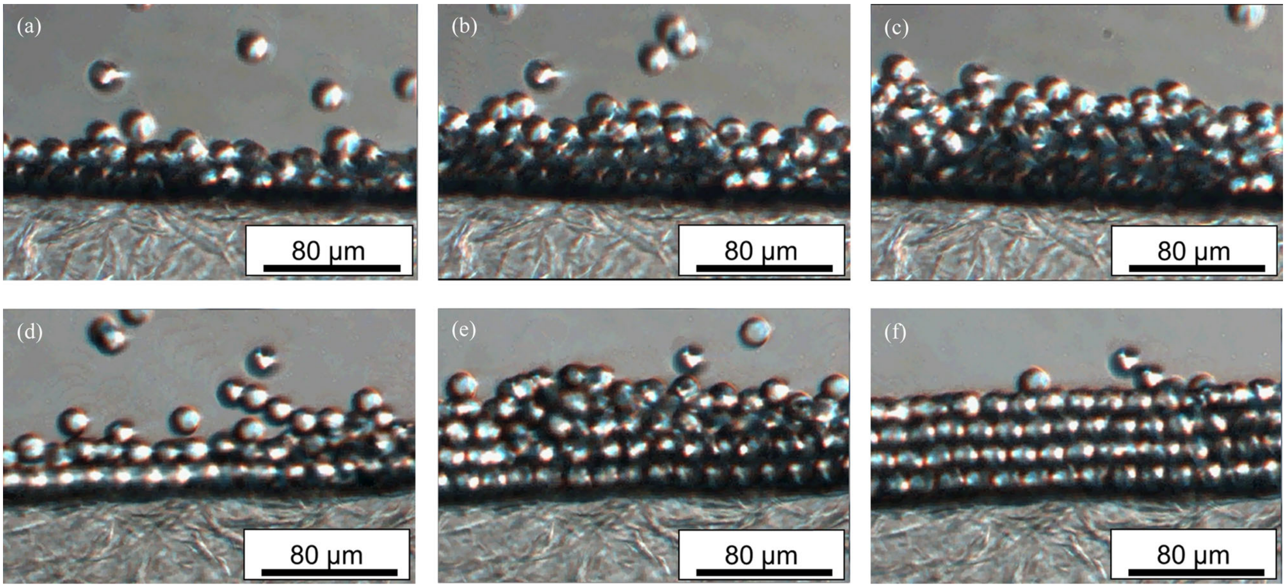


Fig. 9 Sequential optical microscope images of three-dimensionally ordered electrostatically assembled SiO_2–SiO_2 composite particles obtained using an AC electric field of **a**–**c** 10 V and **d**–**f** 30 V at a frequency of 1 Hz during electrophoresis-induced stimulation

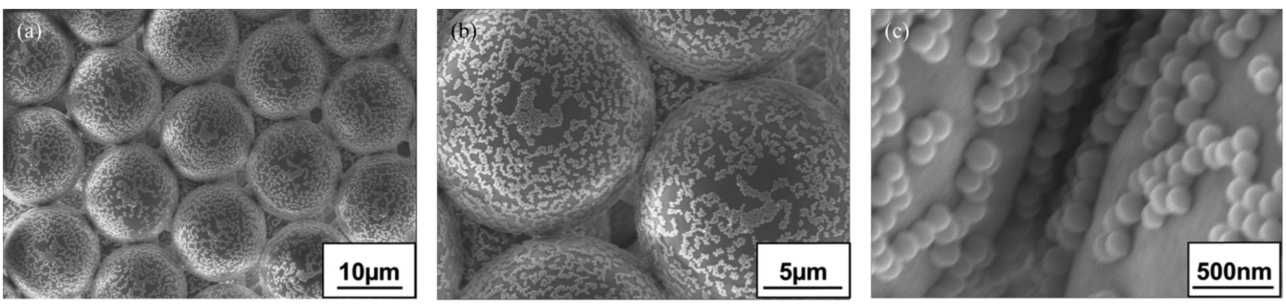


Fig. 10 SEM images of electrostatically assembled SiO_2–SiO_2 composite particles arranged in a three-dimensional close-packed structure at **a** low and **b** and **c** high magnification

Neirink et al., they demonstrated a feasible AC-EPD of aqueous alumina suspension at high voltage without water electrolysis [48]. Despite using a high voltage of 500 V under an asymmetrical AC signal at a low frequency of 1 Hz, no gas bubbles were generated, and they managed to obtain a dense alumina deposit under this condition.

The morphology of the three-dimensional close-packed SiO_2–SiO_2 composite particles was observed by SEM, and the obtained images are shown in Fig. 10. The surface morphology showed formation of a hexagonal close-packed structure consisting of SiO_2–SiO_2 composite particles (Fig. 10(a)). The SiO_2 decoration particles adsorbed on the SiO_2 core particles and did not detach. In a higher magnification image (Fig. 10(b)), the underlayer of the composite particles exhibited a similar morphology to the homogeneous distribution of SiO_2 decoration particles on the surface. In a magnified SEM image of the interparticle region (Fig. 10 (c)), a gap consisting of approximately two SiO_2 decoration particles was observed, indicating feasible interparticle gap control by changing the particle size of the decoration (additive) particles.

It is notable that during the electrophoresis-induced stimulation process with an AC electric field, bottom-up stacking of the ordered arrays was observed. This phenomenon was also reported by Li et al. [30] in their study using nanosized SiO_2 particles. Their findings suggested that formation of the close-packed patterns began at the bottom layer and propagated to the surface prior to consolidation. The driving forces for this patterned formation and growth were the coupling between the interparticle interactions and the constraints generated from the external stimulation or fields.

From these results, we have demonstrated that by applying an AC electric field with sufficient amplitude during the electrophoresis-induced stimulation process along with gravitational sedimentation, a three-dimensional close-packed arrangement of electrostatically assembled SiO_2–SiO_2 composite particles can be obtained. The findings of this study involving the formation of ordered two- and three-dimensional

SiO_2–SiO_2 composite-particle arrays will be beneficial for the development of sol–gel-related technology using electrostatically assembled composite particles for emerging applications [49, 50].

4 Conclusions

We have demonstrated formation of electrostatically assembled SiO_2–SiO_2 composite particles with a homogeneous distribution using sol–gel-derived monodispersed SiO_2 particles with average particle sizes of 200 nm and 16 µm. By applying AC and DC electric fields during electrophoresis-induced oscillation, a two-dimensional hexagonal close-packed structure of electrostatically assembled SiO_2–SiO_2 composite particles was obtained, whereas a disordered structure with voids was obtained when only a DC electric field was applied. In formation of ordered three-dimensional arrays using the electrostatically assembled SiO_2–SiO_2 composite particles, simultaneous application of an AC electric field with an amplitude of 30 V at a frequency of 1 Hz and gravitational sedimentation enabled formation of a hexagonal close-packed structure. The findings of this study will be beneficial for advancement of sol–gel-related technology using well-ordered arrays of electrostatically assembled sol–gel-derived monodispersed particles for applications such as optical devices and other emerging technologies.

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Author contributions Credit Author Statement Investigation: TA; Methodology, Validation, and Writing - First draft: WKT; Investigation and Validation: AY; Validation: GK; Validation: Atsunori Matsuda; Supervision, Conceptualization, Writing - Review & Editing, Project Administration and Funding acquisition: HM.

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Compliance with ethical standards

Conflict of interest The authors declare no competing interests.

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