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论文题目：Microfluidic-Directed Assembly of Versatile Colloidal Photonic Crystal Supraballs toward Display and Sensing

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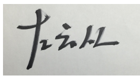
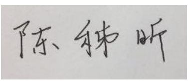
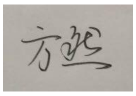
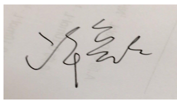
参赛队员：



指导老师：

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Microfluidic-Directed Assembly of Versatile Colloidal Photonic Crystal Supraballs toward Display and Sensing

Abstract:

Colloidal photonic crystal (CPC) materials have received considerable attention owing in part to their unique optical properties useful for various applications. The assembly of CPC supraballs with multifunction and stimuli-responsive capacity still remains challenging. Herein, we report the preparation of CPC supraballs with tunable brilliant structural color, magnetism, and stimuli-responsive behavior via microfluidic techniques. We synthesize monodispersed hydrogel microspheres poly(styrene-*co*-butyl acrylate-*co*-acrylic acid) [P(St-*co*-BA-*co*-AA)] as the building blocks of CPCs. We then construct CPC supraballs with various structural colors in the range varying from blue-violet to red by a biphase microfluidic device. The further encapsulation of Fe_3O_4 nanoparticles via a triphase microfluidic device enables the formation of Janus CPC supraballs with magneto-responsive property and structural color governed into two distinct hemispheres. Such Janus materials undergoes switching and micromanipulation responding to an external magnetic field, and hence rewritable photonic patterns were easily realized. Their switching behavior in structural color towards humidity were also demonstrated. These multifunctional Janus hydrogel CPC supraballs show potentials for display, encoding, environmentally sensing, and biomedical applications.

Keywords: Colloidal photonic crystals, structural color, Janus supraballs, stimuli-responsive behavior, display, sensing

Highlights:

1. Uniform colloidal photonic crystal (CPC) supraballs were prepared from microfluidic assembly of monodispersed hydrogel microspheres poly(styrene-co-butyl acrylate-co-acrylic acid).

2. CPC supraballs show simultaneous tunable brilliant structural color, magnetism, and stimuli-responsive behavior.

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3. CPC supraballs have wide-range tenability in structural color varying from blue-violet, yellow, green to red.

4. Rewritable photonic patterns, switching and micromanipulation can be easily realized for CPC supraballs by applying an external magnetic field.

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1. Introduction

Colloidal photonic crystals (CPCs), also known as photonic bandgap materials, are a unique kind of orderly arranged materials self-assembled by monodisperse colloidal particles. CPCs have interesting characteristics such as spatial periodic structure and manipulating the movement of photons, to present brilliant structural color.[1-3] Through changing the size of building blocks or changing the lattice constant, the structural color of CPCs can be manipulated, showing potentials for color-changing optical sensors.[4-6] Tremendous effort has been devoted to the fabrication of CPC films owing to its simplicity of manipulation.[7-9] For example, embedding CPC into elastomer or hydrogel network, the resultant materials can be stimuli-responsive as a result of shrink-swelling characteristics.[10] However, CPC films possess the instability of optical diffraction and angle-dependent optical stopbands, greatly limiting their application.

In recent years, spherical CPC supraballs (otherwise known as CPC supraparticles or CPC suprabeads) have received increasing attention.[11-13] Such materials have spherical geometries self-assembled by monodisperse micro-scale (10-100 μm) colloidal particles, which can not only maintain orderly and closely packed, but most importantly, they show angle-independent structural color. Therefore, compared with CPC films, CPC supraballs have broader potential applications in areas like displaying and sensing.[14-16] Velev's group reported the first preparation of CPC supraballs by utilizing superhydrophobic plate to form spherical colloidal droplets as a model.[17] However, this fabrication method has drawbacks including low productive rate, bad manipulation over particle size, and low monodispersion.

The development of microfluidic technology allows the availability of diverse CPC supraballs with monodisperse sizes, controllable shapes, and functionalization recently.[18] Since the introduction of the concept in 1990s, microfluidics has become one of the cutting-edge technologies in Physics, Chemistry, Biology and Chemical and biomedical engineering. Generally, the microfluidic technology utilizes either continuous flow or discontinuous flow. Continuous flow means homogeneous system with fluids continually flowing, while discontinuous flow means liquid drops are

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formed on the interface of micro-pinhole due to the flow of continuous phase. By controlling the flow rate of the fluids, microfluidic technology helps to manipulate the size and the dispersion of particles more precisely, which becomes an insightful alternative for fabricating CPC supraballs. Gu's group using microfluidic fluid for cutting fabricated well-dispersed CPC supraballs potentially useful for biological analysis.[19] Chen and his co-workers further developed triphase microfluidic techniques, enabling the formation of CPC supraballs with predictable and tunable shape and anisotropic function. [20]

Despite great achievements in this area, the exploration of CPC supraballs with multifunction and stimuli-responsive ability continues to be an intense interest. Herein, with aid of microfluidic techniques, we constructed versatile colloidal photonic crystal supraballs with tunable brilliant structural color, magnetism, and stimuli-responsive behavior, which might be useful in display devices, information encoding, environmental sensors, and biomedical applications.

2. Experimental Details

2.1. Chemicals and materials

Styrene (St), butyl acrylate (BA) and acrylic acid (AA) were purified by distillation under low pressure to remove few inhibitors. The initiator, potassium persulfate (KPS) was purified by recrystallization from water and stored in a brown bottle. N, N-Dimethylacrylamide (DMAA), N, N-methylenebisacrylamide (MBA), Trimethylolpropane triacrylate (TMPTA) and Triton X-100 were supplied by Aldrich and used as received. Methylsilicone oil was purchased from Dow Corning Corp. The photoinitiator 2-Hydroxy-4-(2-hydroxyethoxy)-2-methylpropiophenone (IRGACURE 2959) and 2-hydroxy-2-methyl-1-phenyl-1-propanone (IRGACURE 1173) were supplied by Ciba Specialty Chemicals. All other reagents were of analytical grade, purchased from commercial sources and used as received. Purified water with resistance greater than 18 MΩ cm was used in all our experiments.

2.2. Synthesis of pure polystyrene (PS) microspheres

According to the references[13, 21], typical monodispersed pure PS microspheres

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was prepared as follows: 5.5 g of St and 0.24 g of polyvinylpyrrolidone (PVP) were dispersed in 110 g of purified water in a 250 mL round flask with 200 rpm stirring under nitrogen protection. After the mixture was heated at 90 °C for 25 min, 0.04 g of KPS dissolved in 10 g of water added into the flask to initiate polymerization, and the polymerization reaction was continued for 4 h. The resulting emulsion was purified via using a nylon net with 200 meshes and were concentrated by a high-speed centrifuge routine.

2.3. Synthesis of monodispersed hydrogel microspheres

Monodispersed P(St-*co*-BA-*co*-AA) hydrogel microspheres were synthesized by emulsion copolymerization according to a modified method reported previously[22], with a universal method of copolymerization as follows: 4.5g of St, 0.5g BA, and 0.24g of PVP were dispersed in 110 g of purified water in a 250 mL round flask with 190 rpm stirring under nitrogen protection. After the mixture was heated at 90 °C for 25 min, added 0.0375g of KPS dissolved in 10g of water to initiate polymerization. The reaction was kept for 40min, and then 0.5g of AA and 0.0025 g of KPS were added, and the emulsion reaction was continued for another 3.5 h. The resulting emulsion microspheres were purified from large agglomerates using a 200 mesh of nylon net.

2,4, Fabrication of single-phase and Janus CPC supraballs

Single-phase hydrogel CPC supraballs were prepared via a facile microfluidic device. The microfluidic device was structured by inserting the capillary tube with a 32 G needle into a PDMS tube, which was fixed with ethyl a-cyanoacrylate instantaneous adhesive. Discontinuous (monodispersed P(St-*co*-BA-*co*-AA) hydrogel microspheres, i.e. CPC building block latexes) and continuous phases (methysilicone oil) were introduced into the needle and the PDMS tube, independently, with adjustable flow velocities by syringe pumps. Droplets generated at the tip of the needle into the polyethylene container, and the single-phase CPC hydrogel supraballs were obtained after water evaporation at 50℃ for 10 h.

Janus CPC supraballs were prepared with a triphase microfluidic device, which is composed of a PDMS capillary tube, and a couple of parallel 30G needles. Two

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discontinuous phases, a solution of P(St-*co*-BA-*co*-AA) hybrid latex and a dispersed mixture of TMPTA with carbon-encapsulated magnetic Fe_3O_4 suspensions were introduced into the two needles to adjust flow velocities independently by using syringe pumps. The continuous phases was selected as methysilicone oil and introduced into the PDMS capillary tube. The appropriate DMAA monomer and surfactant of Triton X-100 to guarantee photopolymerization, and adjust the shape of two-phase to form Janus structure. Typically, the 30%wt. of hybrid hydrogel latex and 0.8% wt. Fe_3O_4 of TMPTA with quantitative photoinitiator were controlled the 0.3mLh^{-1}, the flow velocities of methysilicone oil can be fixed at 20 mLh^{-1} to form liquid Janus supraballs. When the UV beam was applied towards the Janus supraballs in the PDMS capillary, it can make the DMAA and TMPTA polymerization form shapely structure. The steady Janus hydrogel CPC supraballs were obtained after water evaporation at 50℃ for about 10h.

2.5. Characterization

The dynamic light scattering (DLS) of Malvern Zetasizer 3000 was employed to measure the hydrodynamic diameters of CPC microspheres. Optical photographs of supraballs were taken using a stereoscopic microscope (SHUNYU SZM45) with a charge coupled device camera (YICHUANG, YM310). The morphologies and micro-structures of CPC supraballs were measured using a scanning electron microscope (SEM, HITACHI, S-4800). A 360 nm UV laser beam was selected as the excited light source to induce photopolymerization. Reflection spectra of CPC supraballs were recorded using an optical microscope equipped with a fiber optic spectrometer (Ocean Optics, USB4000).

3. Results and discussion

To achieve colloidal photonic crystal arrays, a long-range ordering and close-packing periodic structure is required. Thus, it is of great importance to synthesize monodispersed colloidal microspheres as the building blocks of CPC materials. We prepared series of PS colloidal microspheres by emulsion polymerization under different monomer concentrations. Figure 1 show SEM images

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and diameter distributions of the resultant PS microspheres. It can be observed from the SEM images that the prepared microspheres exhibit spherical morphology and uniform size. These PS microspheres are highly monodisperse, and hence can be close-packed into long-ordered arrays. The corresponding DLS analyses further confirm that PS microspheres obtained by the emulsion polymerization method have good monodispersity, narrow particle size distribution and less than 5% coefficient of variation, which can be used as building units of photonic crystals.

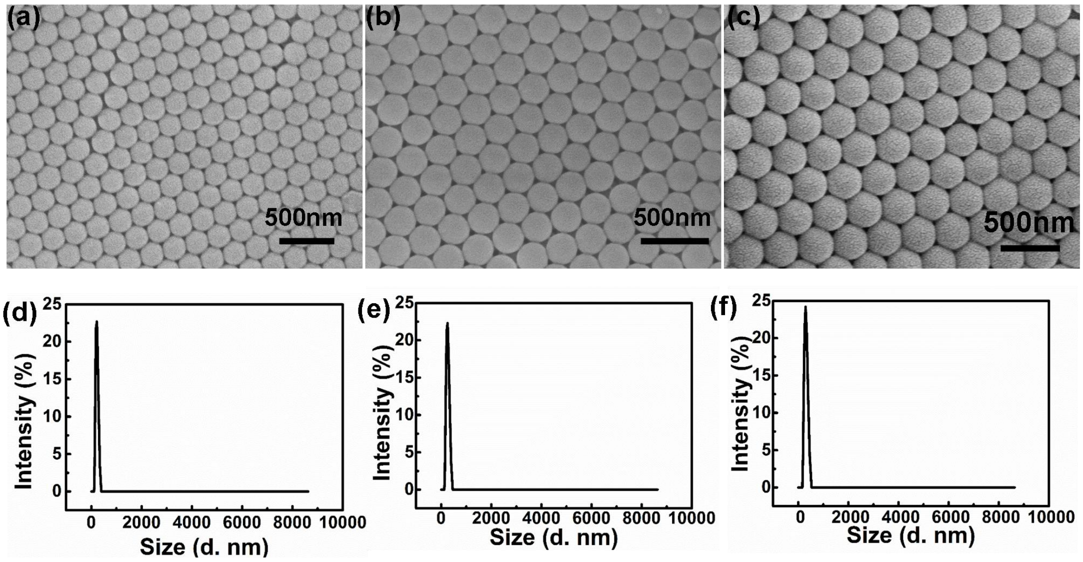

Figure 1. (a-c) Typical SEM images and (d-f) diameter distributions of PS microspheres with different average diameters: (a, d) 181 nm, (b, e) 214 nm and (c, f) 250 nm.

Hydrogels are powerful smart materials with three-dimentional network, which are widely used in sensing, tissue engineering, and biomedical applications owing to their stimuli-responsive capacity, biocompatibility, and the ability to capture metal ions or nanoparticles.[23] To confer the CPC materials with multifunction, hydrogel monomers BA and AA were introduced to grow hydrogel shells on the PS cores, affording P(St-co-BA-co-AA) latex. Then a biphase microfluidic flow-focusing device was constructed for the generation of monodisperse photonic crystal supraballs (Figure 2a). We chose methysilicone oil as continuous phase and P(St-co-BA-co-AA) latex as discontinuous phase. In a typical procedure, the latex phase was cut off by the

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continuous oil phase, resulting in uniform photonic supraballs with the assistance of surface tension between oil and water. After solvent evaporation, the photonic supraballs were self-assembled from monodispersed colloidal microspheres, ascribed to the close-packed lattice arrangement caused from minimization of the interfacial free energies. The as-prepared CPC supraballs with brilliant green structural color are shown in Figure 2b, which have uniform spherical morphology and an average diameter of 258 μm (Figure 2b-c). The results indicate we constructed an effective platform to fabricate high-quality CPC supraballs.

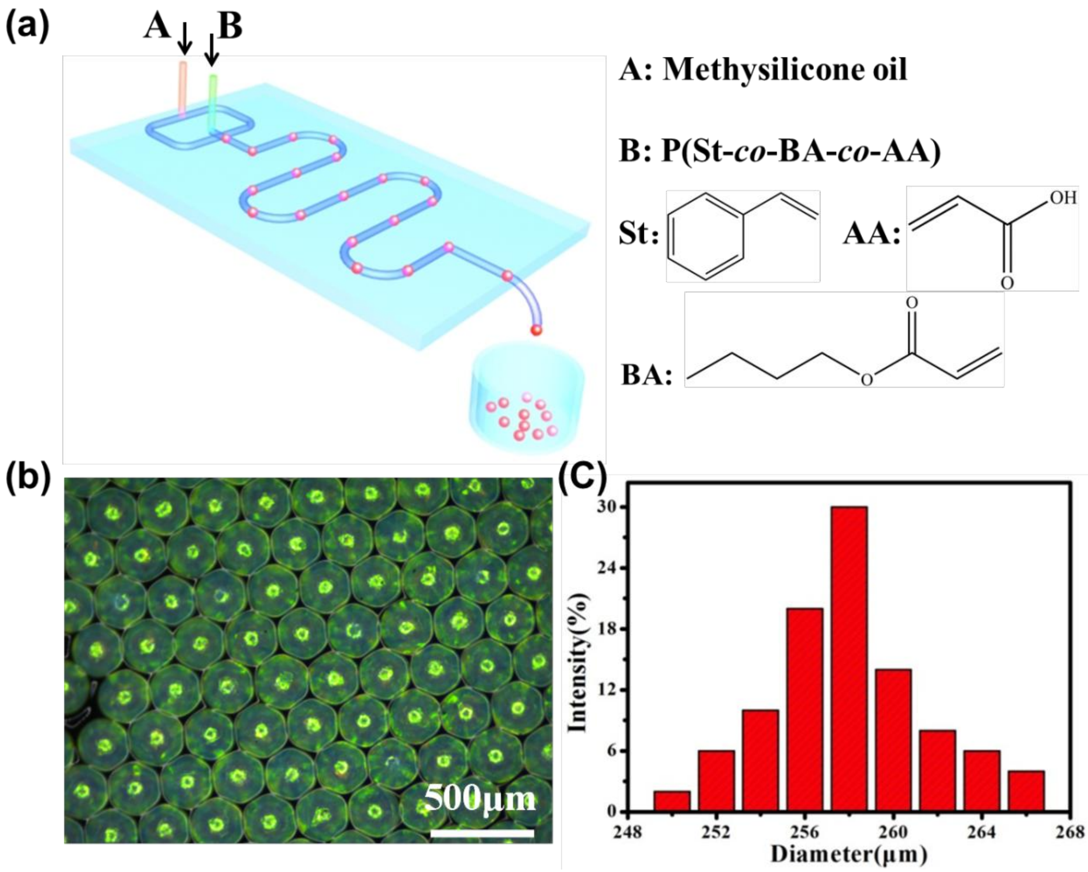

Figure 2. (a) Schematic representation of microfluidic device for preparation of monodisperse photonic supraballs. (b) Optical microscopy image and (c) the corresponding diameter distribution of the as-synthesized photonic supraballs.

As described in the Young–Dupre's equations, the velocities of oil flow and latex flow are main factors in controlling the size of the CPC supraballs. Figure 3a and 3b show the relationships between droplet diameter of the photonic supraballs and oil

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flow velocity (with a fixed latex flow velocity at 0.6 ml/h), latex flow velocity (with a fixed oil flow velocity at 16 ml/h), respectively. The drop diameter decreases with the oil flow velocity while increases with the latex flow velocity. Accordingly, the optimal conditions of the oil flow velocity and latex flow velocity were fixed at 20 mL/h and 0.1 mL/h, respectively. Moreover, by controlling the surface tension between the oil phase and the water phase, various spherical supraballs with different structural colors ranging from blue-violet, yellow to red could be facilely prepared by tuning colloidal sizes (Figure 2c-e). The as prepared photonic supraballs exhibit iridescent structural colors and present outstanding uniformity in size. Thus, by tuning the velocities of oil flow and latex flow, we are able to control the surface tension between oil phase and latex phase, and hence prepare a series of monodispersed photonic supraballs with high optical performance potentially used for anti-countering, sensing and displays.

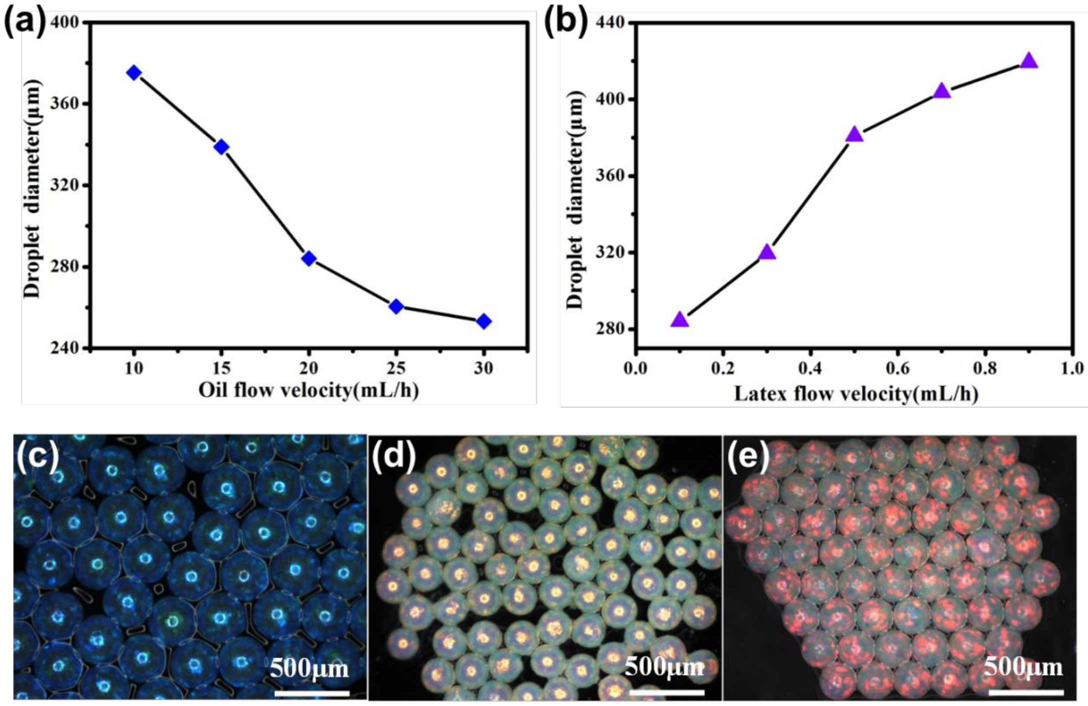

Figure 3. Relationship between droplet diameter of the CPC supraballs and (a) oil flow velocity (with a fixed latex flow velocity at 0.6 ml/h), (b) latex flow velocity (with a fixed oil flow velocity at 16 ml/h). (c-e) Optical photographs of CPC supraballs constructed by P(St-*co*-BA-*co*-AA) latex with different diameters.

We further investigated the fabrication of CPC supraballs with Janus structures by employing a triphase microfluidic device. As shown in Figure 4a, the methysilicone oil was chosen as continuous phase, P(St-*co*-BA-*co*-AA) emulsion and TMPTA/Fe_3O_4 nanoparticles were used as discontinuous phases, respectively. The converged two discontinuous phases were cut by continuous phase into biphasic droplets with CPC hemispheres and magnetic hemispheres. While applying UV light at the terminal of the microfluidic device, Janus CPC supraballs were successfully prepared. Bright blue CPC hemispheres and black hemispheres with clear boundaries can be observed in the optical image of Janus PC supraballs shown in Figure 4b. It is worth mentioning that the immiscibility among the water, TMPTA(as surfactant) and methysilicone oil makes sure the isolation of the two hemispheres in a supraballs which eliminated the diffusion of the colloidal particles in the formation progress of CPC hemispheres. The isolation of the two hemispheres was further confirmed by SEM image shown in Figure 4c and it can be seen that two sides, well-ordered colloidal particles and flat polymer, are obviously separated.

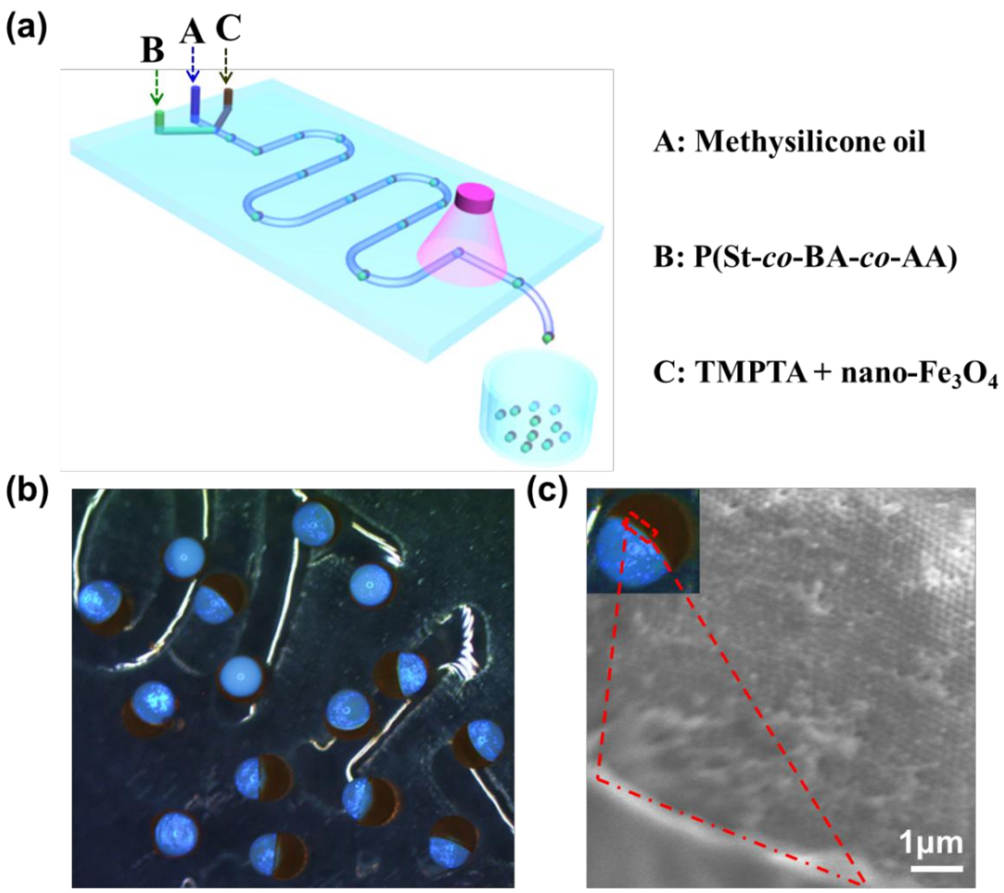

Figure 4. (a) Schematic illustration of a triphase microfluidic device for the

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fabrication of magneto-responsive Janus CPC supraballs. (b) Optical microscope image of the obtained Janus supraballs with the blue hemispheres (CPC part) and the black hemispheres (magnetic part). (c) SEM image of the equator for a Janus supraball.

Besides, we further explored the potential applications of as-obtained Janus PC supraballs. As shown in Figure 5a, a display panel with patterned "NF"(standing for the abbreviation school name "NFLS") was created using Janus PC supraballs as pixel. Initially, the pattern show ruleless color (OFF state) for the disorientation of these PC supraballs. While applying the magnetic field under the panel, all of the pixels oriented with blue hemispheres upwards (ON state) and the display panel show bright blue "NF" pattern. Therefore, such Janus materials could undergo switching in response to an external magnetic field, meaningful for bead-patterned display panel. Additionally, we endowed the supraballs with external responsive property by introducing the humidity-sensitive monomer dimethylallylamine (DMAA) into the CPC hemispheres. After UV light initiation, the colloidal particles were finely immobilized in the polymer matrix owing to the effective interactions between functionalized particle surface and matrix. Figure 5b show the changing of bead-patterned display panel by adjusting the humidity. While increasing the ambient humidity, the supraballs swelled remarkably along with the color changing from blue to while (infrared). The phenomenon can be explained by that, the absorption of water by polymer matrix from the air lead to the volume gaining of the supraballs and meanwhile the lattice constant increased without doubt, resulting in the red shift of the diffraction peak of CPC supraballs. Significantly, the reversible changing of the display panel occurred after decreasing the ambient humidity. These bead-pattern display panels utilizing functional Janus CPC supraballs as pixels exhibit great potential in the intelligent display, environment visual sensing, and information encoding areas.

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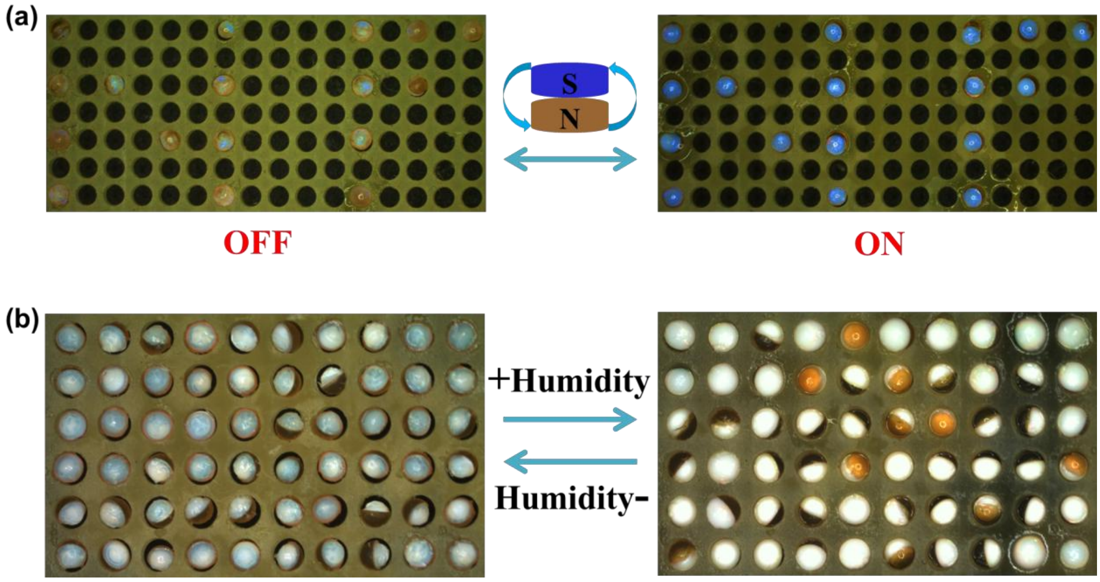

Figure 5. (a) Switching of the “NF” bead-patterned display panel while applying external magnetic field. (b) Reversible changing of the bead-patterned display panel under different humidity.

4. Conclusions

In summary, with use of monodispersed hydrogel microspheres poly(styrene-*co*-butyl acrylate-*co*-acrylic acid) [P(St-*co*-BA-*co*-AA)] as the building blocks, we successfully constructed versatile multifunctional colloidal photonic crystal (CPC) supraballs via microfluidic techniques. By controlling the surface tension between the continuous phase and the discontinuous phase in the microfluidic device, differently sized spherical CPC supraballs with various structural colors ranging from blue-violet, yellow, green to red could be facilely prepared by tuning colloidal microsphere diameters. More interestingly, we showed the availability of Janus CPC supraballs with photonic bandgap, magnetic response, and humidity-sensitive behavior via a triphase microfluidic technique. Since the building blocks of CPC supraballs are functionalized with smart hydrogel shell, the versatile ability responding to external stimuli is expectable. It is reasonable to speculate that These multifunctional Janus hydrogel CPC supraballs might find potentials in such as display, encoding, sensing, and biomedical applications.

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