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Silica particles coated with zwitterionic polymer brush: Formation of colloidal crystals and anti-biofouling properties in aqueous medium

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ABSTRACT

Silica particles (SiP) were modified with 2-bromoisobutyryl group-carrying silane coupling reagent, and a polymer brush of carboxymethylbetaine, poly[1-carboxy-*N*,*N*-dimethyl-*N*-($2'$-methacryloyloxyethyl)methanaminium inner salt] (PolyCMB), was introduced onto surface of the particles using surface-initiated atom transfer radical polymerization (ATRP). After purification by simple ultrafiltration, the layer of the PolyCMB brush-carrying silica particle (SiP-PolyCMB) in aqueous medium showed a clear iridescence at the bottom of a quartz cell upon a slow sedimentation, indicating the formation of colloidal crystals due to both electrostatic and steric repulsion between the particles. The peak of the reflection spectra gradually shifted to a smaller wavelength region due to the formation of more densely packed ordered structure of the SiP-PolyCMB. Moreover, SiP-PolyCMBs dispersed in water showed neither salt- nor freeze–thaw cycle-induced coagulation, and were resistant against the non-specific adsorption of proteins such as bovine serum albumin and egg white lysozyme, which is in contrast with the noticeable adsorption of the proteins to the bare silica particles.

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1. Introduction

Various kinds of colloidal particles have been prepared for practical uses (painting, adhesive, diagnosis, etc.) and basic researches. Among them, in these days, silica particles (SiPs) and polymer latex particles have very often been used for the construction of colloidal crystals which are induced by the electrostatic repulsion between the particles in aqueous medium [1–4]. Though the electrostatic interaction dominantly affects the colloidal stability of the particles, the physical (steric) repulsion also seems to be effective. For this purpose, polymer graft chains have often been introduced onto surfaces of colloidal particles [5–14].

Atom transfer radical polymerization (ATRP) is categorized in living radical polymerization, which can be used to prepare well-defined polymers with a wide variety of monomers, varying a topology of polymers (linear, branched, etc.) and a composition of polymeric chains (block or graft copolymers, etc.) [15–21].

Zwitterionic polymers such as polycarboxybetaine, polysulfobetaine and polyphosphobetaine are well known to be biocompatible [22]. We attributed mildness of the zwitterionic polymers to the structure of vicinal water to biocompatibilities of the polymers [23–29]. Among the zwitterionic polymers ever examined, poly[1-carboxy-*N*,*N*-dimethyl-*N*-($2'$-methacryloyloxyethyl)methanaminium inner salt] (poly(carboxymethylbetaine), PolyCMB) can most easily be prepared with less harmful chemicals: CMB (Scheme 1(a)) can be prepared by quaternization of 2-(dimethylamino)ethyl methacrylate with potassium chloroacetate in water, and subsequent electric dialysis [30,31]. We have clarified usefulness of CMB as a component of biomedical materials such as bio-sensing devices and dressing for wound healing [23–27].

In this report, we have introduced a brush of zwitterionic polymer, PolyCMB, onto a surface of SiPs with a help of ATRP, and the colloidal stability of polymer-modified SiP (SiP-PolyCMB) has been examined. Furthermore, the anti-biofouling properties of the SiP-PolyCMB have been investigated (Fig. 1).

SiPs often show rainbow-color, iridescence, due to a formation of colloidal crystals [3,4,32] induced by both the electrostatic repulsion and the sedimentation [33,34]. However, elaborate purifying processes such as treatment with ion-exchange resins are needed to obtain highly ordered colloidal crystals, with strong iridescence and high reflectivity. By the presence of polymer brushes with a narrow molecular weight distribution, steric repulsion between the brushes on the neighboring particles can also become effective to keep the distance between the colloidal particles constant, which is expected to result in an “ordered structure” of

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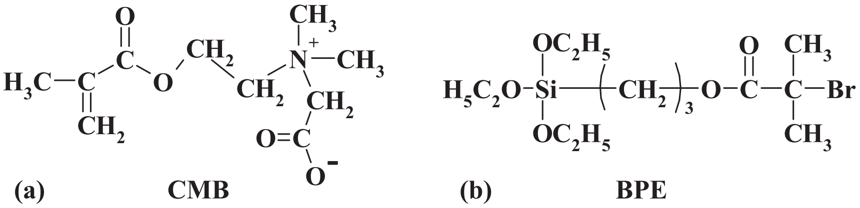


Scheme 1. Chemical structure of (a) CMB and (b) BPE.

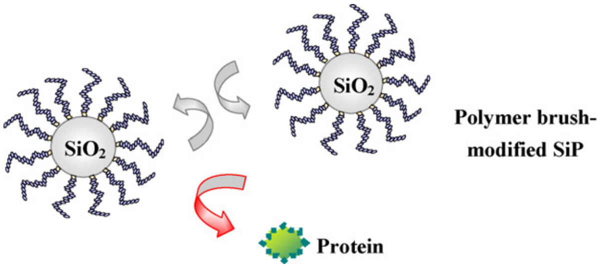


Fig. 1. Schematic of steric repulsion and anti-biofouling properties of the PolyCMB brush-modified silica particle.

colloidal particles (colloidal crystals) [7,8]. As far as we know, however, there has been no report concerning colloidal crystallization of polymer-grafted particles in aqueous medium. The introduction of well-defined zwitterionic brushes onto SiPs by surface-initiated ATRP has enabled us to observe clear reflection spectra of the dispersion of the polymer-modified SiP in aqueous medium.

2. Materials and methods

2.1. Materials

A dispersion of silica particles (Seahoster KE-P30; diameter, 280 nm according to the manufacturer) was kindly donated by Nippon Shokubai, Co., Ltd., Tokyo. A carboxymethylbetaine monomer, 1-carboxy-*N*,*N*-dimethyl-*N*-(2′-methacryloyloxyethyl)methanaminium inner salt (CMB; commercial name, GLBT, Osaka Organic Chemical Industries) was prepared as reported elsewhere [30,31]. Bovine serum albumin (BSA) and egg white lysozyme were obtained from Sigma–Aldrich. Other reagents were commercially available. A Milli-Q grade water was used for preparation of aqueous sample solutions and dispersions.

2.2. Synthesis of (3-(2-bromo-2-methyl)propionyloxy)propyltriethoxysilane (BPE, Scheme 1 (b))

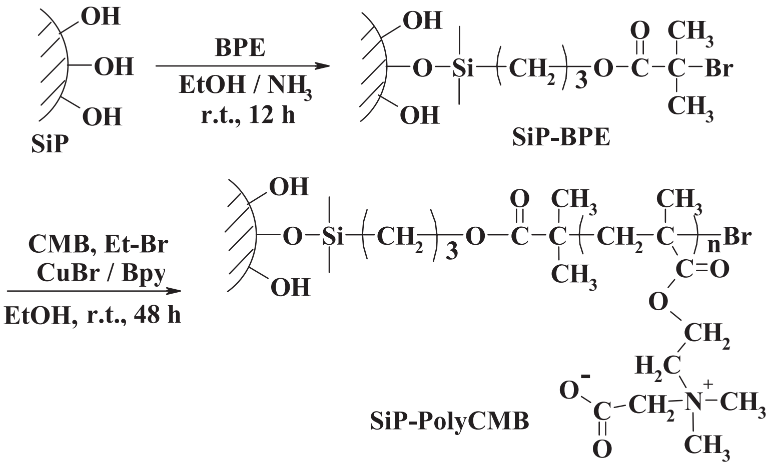
A silane ATRP initiator, BPE, was synthesized from propen-1-ol by the two-step reaction as previously described [20].

2.3. Preparation of initiator-coated silica particles via silane-coupling (Scheme 2)

At first, silica particles (400 mg), which had been mixed with degassed ethanol (EtOH) in a sample vial, were dispersed and degassed using an ultrasonic bath. The dispersion of silica particles was poured into a stirred ultrafiltration cell (Amicon; capacity, 200 mL; membrane, Amicon YM30 (molecular cut-off, 30,000)) and concentrated to 30 mL. A mixed solution of BPE (426 mg, 1.15 mmol), 20% aqueous ammonia (4.6 mL; NH_3, 49 mmol) and EtOH (15 mL) prepared in a sample vial was added to the ultrafiltration cell to give a dispersion of 1 wt% silica particles (weight ratio of BPE: 20% ammonia aq.: EtOH = 1:10:89) (total volume 50 mL). After 12 h at room temperature, the dispersion of the particles was washed with EtOH several times by ultrafiltration (membrane, YM30). The dispersion of the initiator-coated particles was ultrasonicated for 30 s and finally concentrated to 1 wt% by ultrafiltration. The dispersion was stored in a sample vial filled with Ar (SiP-BPE).

2.4. Accumulation of PolyCMB-brush on silica particles

Next, PolyCMB brush was introduced to the surface of the silica particle: the ATRP initiator-carrying silica particle, SiP-BPE, was incubated with CMB (0.5 M), ethyl 2-



Scheme 2. Construction of PolyCMB brush on a silica particle.

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