

Synthesis and properties of ellipsoidal hematite/silicone core-shell particles

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Abstract Monodispersed ellipsoidal hematite particles were synthesized and coated with silicone using a silane-coupling agent, phenyltriethoxysilane. The thickness of the silicone shell was controlled by the concentration of phenyltriethoxysilane and the presence of *n*-hexadecyltrimethylammonium chloride, which was thought to modify the surface properties of the hematite particles to be organophilic. The hematite/silicone core-shell particles were strongly hydrophobic and had a good dispersibility and stability in toluene. Hollow ellipsoidal silicone particles were obtained by the dissolution of hematite with hydrochloric acid from the core-shell particles.

Introduction

There has been a great deal of interest in coating particles of nanometer to micrometer sizes with materials of different composition for the purposes of tailoring the properties of the particles (shell) as well as emerging new materials (core-shell particles) [1–4]. As to the materials, both organic and inorganic particles have been coated by

either organic or inorganic materials ranging from molecular to micrometer level in the thickness.

Iron oxide particles including hematite possess various functions for the uses as pigments, magnetic and catalytic materials so that the surface modification of iron oxide particles has attracted attention partly for their practical applications. For example, silica coated iron oxide particles have been synthesized to prevent aggregation and stabilize the colloidal suspension [5–7]. Coating of iron oxide particles with organic species has also been reported to construct functional hybrid particles. Spiropyran and poly(*N*-isopropylacrylamide) were used to impart photo and thermo-responses to magnetite particles [8, 9]. Poly(1-vinylimidazol) was also used as metal ion trapping site on maghemite surface [10].

From the viewpoints of materials application, the particle morphology is another key issue so that the morphosyntheses of iron oxide particles have extensively been reported toward controlled particle shape and size as well as narrow particle size distribution [11–20]. As for hematite, monodispersed particles with various morphologies such as pseudocubic, spindle, peanut-type, ellipsoidal and so on, from sub-micrometer to micrometer order, have successfully been synthesized [11–17]. Taking advantage of the successes of the morphosyntheses, we use monodispersed hematite particles as the first target to be coated with silicone. Matijević and co-workers have reported the coating of spindle type hematite particles with zirconium hydrous oxide and manganese compounds and their influence on the specific surface areas and the electrophoretic mobilities [21, 22]. They have also reported the preparation of silica-coated hematite particles [23]. Coating with silicones not only makes the particle surface organophilic, but also allows the introduction of various functional groups including reactive ones onto the surface. Although the

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coating of iron oxide particles with silicones has previously been reported [24], there is still a requirements to silicone coated iron oxide particles due to their practical applicabilities, variation of the silicone compounds and so on. Materials both core (iron oxide species) and shell (silicones with different molecular structure) should be varied in addition to the controlled size and thickness of core and shell, respectively, for the optimum performances. In this study, coating of the monodispersed ellipsoidal hematite particles with silicone was investigated. We conducted the silicone coating in a suspension using silane-coupling agents, methyltriethoxysilane (MTES) and phenyltriethoxysilane (PTES), as the silicone sources. The effects of the kinds and concentrations of silane-coupling agents and the presence of surfactants on the coating reactions were examined.

Experimental

Materials

Reagent grade iron (III) chloride hexahydrate ($\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$), sodium hydroxide (NaOH), sodium nitrate (NaNO_3), anhydrous sodium sulfate (Na_2SO_4), sodium chloride (NaCl), hydrochloric acid aqueous solution (37%) (HCl), lauric acid, ammonia aqueous solution (28%), methanol, ethanol and 2-propanol were purchased from Kanto Chemical Co., Inc. (Tokyo, Japan). Methyltriethoxysilane (>98.0%) (MTES), phenyltriethoxysilane (>99.0%) (PTES) and *n*-hexadecyltrimethylammonium chloride (>98.0%) (C16TAC) were purchased from Tokyo Kasei Kogyo Co., Ltd. (Tokyo, Japan). These chemicals were used without further purification.

Synthesis of ellipsoidal hematite particles

Ellipsoidal hematite particles were synthesized according to the method reported by Sugimoto et al. [11]. An aliquot (250 ml) of 5.4 mol/l NaOH solution was added to 2.0 mol/l FeCl_3 solution (250 ml) in a polypropylene bottle for 10 min under vigorous stirring at 40 °C, followed by additional agitation for 2 min and aging at 100 °C for 6 h to obtain β -FeOOH suspension. The β -FeOOH particles were collected by centrifugation (4,000 rpm, 30 min) and subsequently washed three times with 0.5 mol/l NaNO_3 solution, and re-dispersed in 500 ml of distilled water. 20 ml of this suspension was mixed with Na_2SO_4 , NaCl and HCl, and then distilled water was added to a total volume of 40 ml. The molar ratio of FeOOH, Na_2SO_4 , NaCl and HCl was 46:3:10:10, where the amount of FeOOH was estimated from the mass of residue (Fe_2O_3) of the re-dispersed suspension after ignition. Then the

hydrothermal treatment at 140 °C for 72 h was conducted with the suspension using a Teflon lined closed vessel. The red-brown product was collected by centrifugation (4,000 rpm, 10 min), washed with distilled water, and subsequently freeze-dried. In order to remove the sulfate ion the obtained hematite particles were suspended in 1.0 mol/l ammonia aqueous solution and stirred for 1 h, then centrifuged (4,000 rpm, 10 min) and vacuum-dried.

The product was brownish-red powder. The scanning electron microscopy (SEM) image of the product (Fig. 1) demonstrated that all particles were ellipsoidal and there was no recognizable byproduct. The average particle size was 3.3 μm in major axis and 1.6 μm in minor axis, the aspect ratio was 2.1, and the standard deviation was 0.25, which were calculated from 295 particles in the SEM micrograph. Figure 2(a) shows the longitudinal particle size distribution of the observed particles. In the X-ray diffraction pattern all the diffraction peaks were ascribable to hematite (data not shown). From these results, the successful formation of monodispersed ellipsoidal hematite particles with the size of 3.3 μm in major axis and 1.6 μm in minor axis was confirmed.

Coating of the hematite particles with silicone

Reaction with PTES in the presence of C16TAC

A total of 53 mg of C16TAC, 4.41 g of deionized water, 25 ml of methanol and 4.0 ml of aqueous ammonia solution were added sequentially to 32 mg of the hematite particles, followed by stirring for 20 min. To this suspension was added PTES (the amount is shown in Table 1), followed by stirring for 17 h at room temperature. The particles were collected by centrifugation (4,000 rpm, 10 min), washed with ethanol, and subsequently vacuum-dried. For a control experiment, the same procedure was conducted without C16TAC. The samples were designated

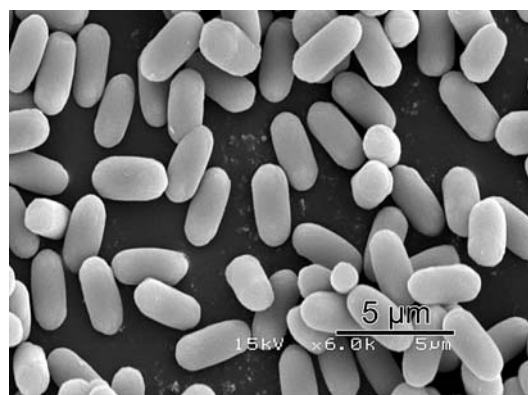


Fig. 1 SEM micrograph of the ellipsoidal hematite particles

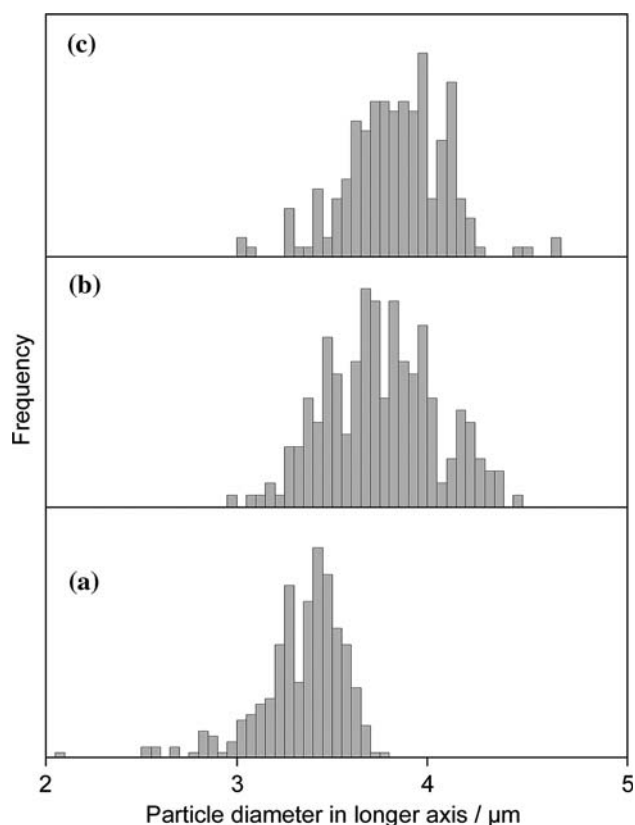


Fig. 2 Particle size distributions in major axis of (a) the ellipsoidal hematite particles (b) PTES/Hem(sonication) and (c) PTES/Hem(sonication $\times 2$)

based on the compositions and conditions and the names are summarized in Table 1.

A coating reaction under sonication was also conducted for the system of PTES/Hem(Si/Fe = 1.03). Followed by the addition of PTES and the agitation for 20 min, the suspension was sonicated for 6 h at 35 °C. After the reaction, the particles (designated as PTES/Hem(sonication)) were collected by centrifugation, washed with ethanol, and vacuum dried. The particles were subsequently

treated with PTES by the same procedure. Here, the amount of particles for the second treatment was 116% of that for the first treatment since the product of the first reaction increased ca. 16% by weight.

As a control, silicone particles were synthesized from PTES. 1.0 ml of PTES was added to the mixture of 4.41 g of deionized water, 2.0 ml of aqueous ammonia solution and 25 ml of methanol, followed by stirring for 18 h at room temperature. The particles were collected by centrifugation (4,000 rpm, 10 min), washed with ethanol, and subsequently vacuum-dried.

Reaction with PTES after the treatment with lauric acid

A total of 2.8 g of lauric acid and 80 ml of deionized water were mixed with 600 mg of the hematite particles, followed by stirring for 12 h at 60 °C. The particles were collected by centrifugation (4,000 rpm, 15 min), washed five times with deionized water, vacuum-dried, and accordingly, the lauric acid treated hematite particles were obtained.

The lauric acid treated particles were reacted with PTES under the acidic and the basic conditions. To the solution of 0.10 ml of deionized water, 2.0 ml of ethanol and 50 μ l of 0.10 mol/l hydrochloric acid were added 16 mg of the lauric acid treated hematite particles (pH of the suspension was 2.1). 0.12 ml of PTES was added to this suspension, followed by stirring for 41 h. The product was collected by centrifugation (4,000 rpm, 10 min), washed with ethanol, and subsequently vacuum-dried. For the synthesis under a basic condition, 0.3 ml of ammonia aqueous solution was added instead of hydrochloric acid to adjust to pH = 12, followed by the same procedure.

Reaction with MTES in the presence of C16TAC

MTES was allowed to react with the hematite particles in the presence of C16TAC under the same condition of

Table 1 Synthesis conditions of silicone-coated hematite particles. (unit:mmol)

Sample	Hematite	PTES/MTES	C16TAC	H ₂ O	NH ₃	Methanol
PTES/Hem(Si/Fe = 0.10)	0.20	0.041 (PTES)	0.165	245	59	620
PTES/Hem(Si/Fe = 0.21)	0.20	0.082 (PTES)	0.165	245	59	620
PTES/Hem(Si/Fe = 0.53)	0.20	0.21 (PTES)	0.165	245	59	620
PTES/Hem(Si/Fe = 0.63)	0.20	0.25 (PTES)	0.165	245	59	620
PTES/Hem(Si/Fe = 0.73)	0.20	0.29 (PTES)	0.165	245	59	620
PTES/Hem(Si/Fe = 0.83)	0.20	0.33 (PTES)	0.165	245	59	620
PTES/Hem(Si/Fe = 0.93)	0.20	0.37 (PTES)	0.165	245	59	620
PTES/Hem(Si/Fe = 1.03)	0.20	0.41 (PTES)	0.165	245	59	620
MTES/Hem(Si/Fe = 1.03)	0.20	0.41 (MTES)	0.165	245	59	620
MTES/Hem(Si/Fe = 0.25)	0.20	0.10 (MTES)	0.20	685	82	1700

PTES/Hem(Si/Fe = 1.03). The synthesis was also conducted under the lower MTES concentration condition (Si/Fe = 0.25). The products were designated as MTES/Hem(Si/Fe = 1.03) and MTES/Hem(Si/Fe = 0.25).

Properties of the hematite/silicone core-shell particles

Dispersibility

As-synthesized hematite particles and PTES/Hem(sonication) were dispersed in toluene by shaking, and then sedimentation was observed in time course. The palatability in the media was evaluated by dispersing the as-synthesized hematite particles and the core-shell particles in the same volume of toluene and deionized water in one vial.

Dissolution of the hematite

A total of 20 ml of 12 mol/l hydrochloric acid aqueous solution was added to 12.0 mg of PTES/Hem(sonication), followed by sonication for 1 min. After 15 min, the particles were collected by vacuum filtration, washed with deionized water, and subsequently vacuum dried.

Characterization

Scanning electron microscopy images were obtained with Hitachi S-2380N operated at 15–30 kV of acceleration voltage. Before the observation, samples were coated with gold by using an ion sputter. Transmission electron microscopy (TEM) images were obtained with JEOL JE-M2400EXII operated at 80 kV of acceleration voltage. Samples for the TEM observation were put on a copper micro-grid. X-Ray diffraction (XRD) patterns were obtained with Rigaku RADIIIB (Cu K α) operated at 40 kV and 20 mA in the $2\theta = 5\text{--}70^\circ$ range. Fourier transform infrared (FT-IR) spectra of the particles were measured with a Shimadzu FT-IR 8000PC in the range of 4,000–400 cm^{-1} using KBr disks. Photoluminescent (PL) measurement was carried out with a fluorescence spectrophotometer (Hitachi F4500) using a quartz cell (1 \times 1 cm) filled with the suspension.

Results and discussion

Coating of the hematite particles with silicone

Reaction with PTES in the presence of C16TAC

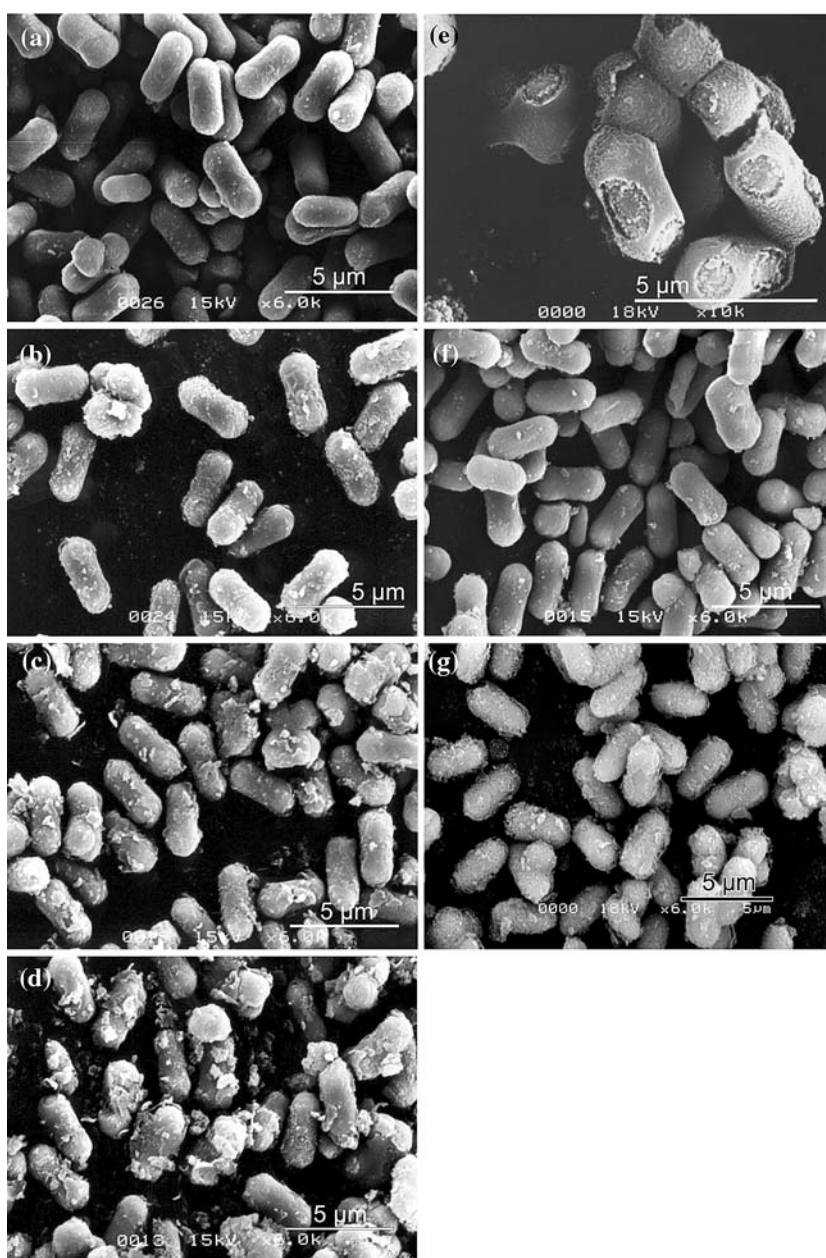
The existence of silicone on the surface of the hematite particles was demonstrated for the products prepared at the

Si/Fe ratio of ≥ 0.63 (Fig. 3(a)–(e)). No byproduct was observed in the SEM images. On the contrary, there was no recognizable change in the surface of the particles when the Si/Fe ratios were less than 0.53. The amount of the silicone deposited on the hematite particles was estimated by the weight loss after the heat treatment at 800 $^\circ\text{C}$. The mass losses were 8.2, 9.6, 9.4, 11.7, 12.8 and 16.0% for PTES/Hem(Si/Fe = 0.53), (Si/Fe = 0.63), (Si/Fe = 0.73), (Si/Fe = 0.83), (Si/Fe = 0.93) and (Si/Fe = 1.03), respectively. Figure 4 shows the relationship between the PTES concentration and the weight loss. There was a linear correlation between these values. These results suggested that the thickness of the silicone shell increased with increasing the PTES concentration, which was consistent with the SEM images. Assuming that (1) a hematite core with the size of 3.3 μm in major axis and 1.6 μm in minor axis is constructed from a cylindrical part and two half spheres, (2) there is no residual ethoxy group or silanol group in the silicone shell and all of the phenyl groups are decomposed to form silica by the heat treatment at 800 $^\circ\text{C}$, (3) the silicone shell and the hematite core are uniform and their relative densities are about 1.3 [25] and 5.26, respectively, (4) the weight losses for coated particles includes the mass loss for as-synthesized hematite particle (5.6%), the thickness of the silicone shell can be estimated from 70 nm for PTES/Hem(Si/Fe = 0.53) to 280 nm for PTES/Hem(Si/Fe = 1.03). Galembeck et al. reported the formation of poly(dimethylsiloxane) coating on iron oxide particles by heating at 250–280 $^\circ\text{C}$ [24]. In the present syntheses, the thickness of silicone shell is controllable to some extent. The mild reaction conditions if compared with the reported one [24] are another advantages of the present core-shell particles.

The hematite core and the silicone shell were distinctly identified by the TEM observation as shown in Fig. 5(a) and (b). Contrary to the assumption, the TEM images revealed that the shell was not a uniform layer but composed of small (~ 100 nm) particles of silicone. There are two possibilities of the origin of these silicone aggregates: (1) they were generated in the solution and then were adsorbed to the surface of the hematite particles, (2) they formed on the surface of the hematite particles after the adsorption of monomeric or oligomeric silicone precursors. At present, we could not determine which is the major reaction occurred in the present reactions.

The formation of silicone on the hematite surface was supported by the IR spectra of the as-synthesized hematite particles and PTES/Hem(Si/Fe = 1.03) (Fig. 6(a) and (b), respectively). For the IR spectrum of PTES/Hem(Si/Fe = 1.03), the absorption bands due to siloxane with phenyl groups were seen at 1132 cm^{-1} (Si-C $_6$ H $_5$ stretching), 1,028 cm^{-1} (Si-O stretching), 735 and 697 cm^{-1} (aromatic CH bending).

Fig. 3 SEM micrographs of PTES/Hem(X). (a) X: Si/Fe = 0.63, (b) Si/Fe = 0.73, (c) Si/Fe = 0.83, (d) Si/Fe = 0.93, (e) Si/Fe = 1.03, (f) Si/Fe = 1.03 (without C16TAC) and (g) sonication



In order to show the possible role of C16TAC on the reaction, the reaction was conducted in the absence of C16TAC. The absorption bands due to phenyl groups at 1,132 and 697 cm^{-1} were also observed for the sample prepared in the absence of C16TAC (Fig. 6(c)). The surface of the particles became less smooth after the reaction and no byproduct was observed in the SEM image (Fig. 3(f)). In addition, the heat treatment of the product at 800 °C resulted in the weight loss of 9.6%. These results suggested that the hematite particles were coated with silicone even in the absence of C16TAC. However, the amount of the coated silicone was smaller than that of the particles obtained in the presence of C16TAC (16.0% as

the mass loss). We speculate that the adsorption of C16TAC onto the surface of the hematite particles, which had negative charge under the basic condition [26], resulted in the increase in the hydrophobicity of the surface of the particles and, accordingly, PTES was adsorbed to the surface of the particles by the hydrophobic interactions between the phenyl group of PTES and the alkyl chain of C16TAC to form the silicone shell.

The SEM image of PTES/Hem(sonication) was shown in Fig. 3(g), demonstrating that the particle aggregation was suppressed if compared with that of the PTES/Hem(Si/Fe = 1.03) (Fig. 3(e)) prepared under magnetic stirring. The weight loss by the heat treatment at 800 °C was about

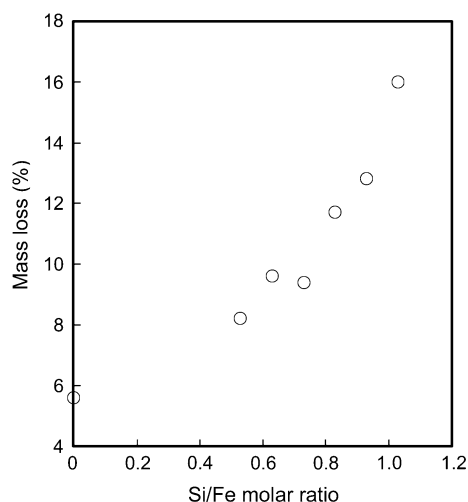


Fig. 4 Mass losses of PTES/Hem(Si/Fe = X) by the heat treatment at 800 °C

16%, which was similar to that observed for the PTES/Hem(Si/Fe = 1.03). TEM image of PTES/Hem(sonication) (Fig. 5(c), (d)) revealed that the silicone shell was formed as aggregates of small (~100 nm) particles of silicone to give a core-shell structure which was very similar to that of PTES/Hem(Si/Fe = 1.03). From the TEM image, the thickness of the shell was about 200 nm, which is in reasonable agreement with the above-mentioned speculation based on the composition.

The coating procedure under sonication was repeated for two times. The average particle size of the product in major axis increased from 3.75 μm to 3.87 μm after the second treatment (Fig. 2(b), (c)). The increase in the second

Fig. 5 TEM images of (a and b) PTES/Hem(Si/Fe = 1.03) and (c and d) PTES/Hem(sonication)

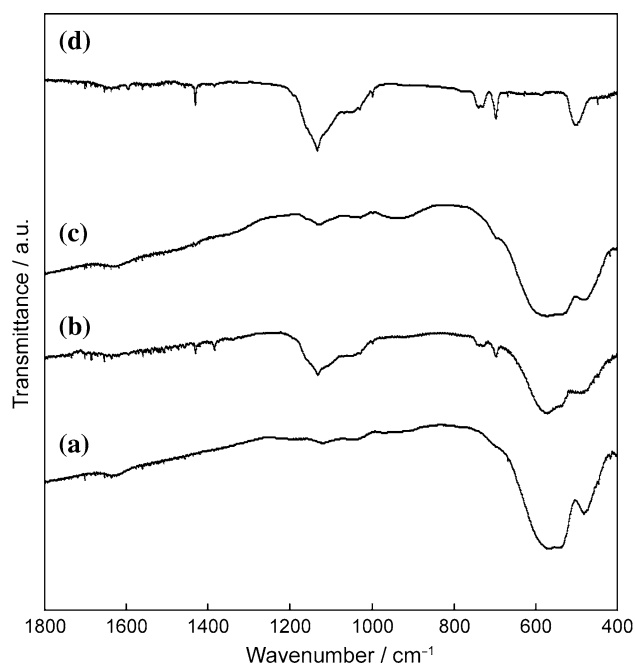
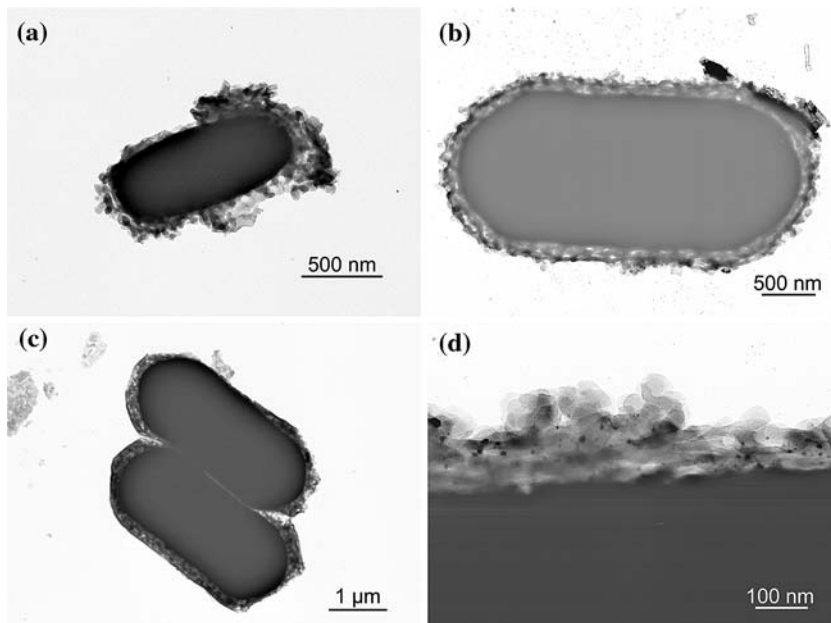


Fig. 6 IR spectra of (a) as-synthesized hematite particles (b) PTES/Hem(Si/Fe = 1.03) (c) PTES/Hem(Si/Fe = 1.03) synthesized without C16TAC and (d) the silicone shells obtained by treating PTES/Hem(sonication) with 12 mol/l HCl solution

treatment was smaller if compared with the increase in the particle size observed for the first treatment (from 3.33 μm to 3.75 μm), due to the difference in the interactions between the particle surfaces and the reactants. It is acceptable based on the above-mentioned results that the particles were coated with silicone so that the C16TAC was difficult to be adsorbed on the surface. Other reaction

parameters such as the concentration, temperature, pH of the reaction mixture may be possible variables in order to control the thickness of the shell further.

Figure 7(a) and (b) show the UV excitation and emission spectra of the PTES/Hem(sonication) and the silicone particles suspensions. An excitation and an emission peaks were observed at 268 nm and 325 nm, respectively for both the core-shell particles and the silicone particles. These results demonstrated that the quenching by the hematite core did not occur. Hamanishi et al. reported that absorption bands around 260 nm, which was close to the excitation maximum, and an excimer emission at 324–330 nm were observed in the absorption and emission spectra of phenyldisiloxanes with two, four and six phenyl groups in a molecule [27]. Since the spectra of PTES/Hem(sonication) are consistent with those of the phenyldisiloxanes, the distance between the adjacent phenyl groups in the silicone shell is very short to be able to form excited dimer upon irradiation.

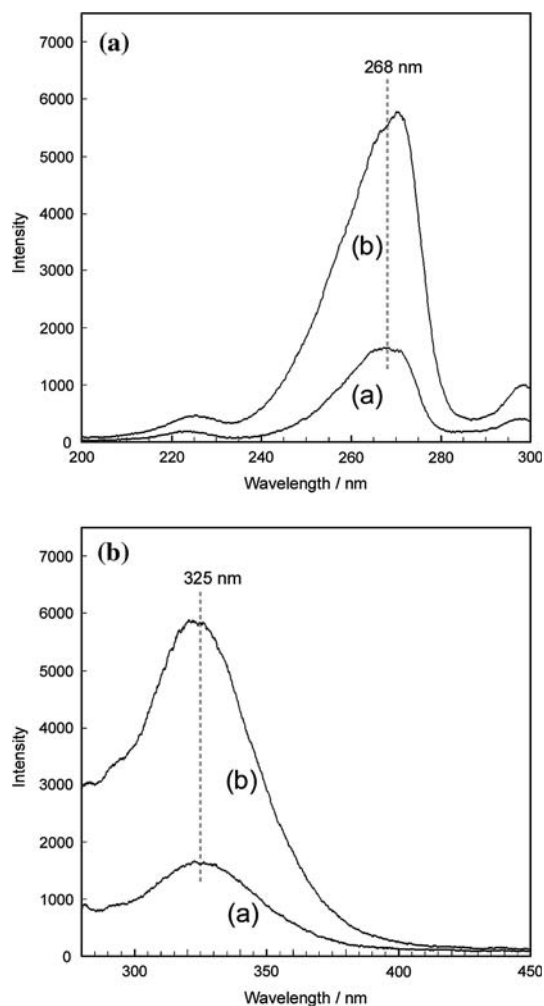


Fig. 7 PL spectra of (a) PTES/Hem(sonication) and (b) the pure silicone particles. (A: excitation, B: luminescence)

Reaction with PTES after the treatment with lauric acid

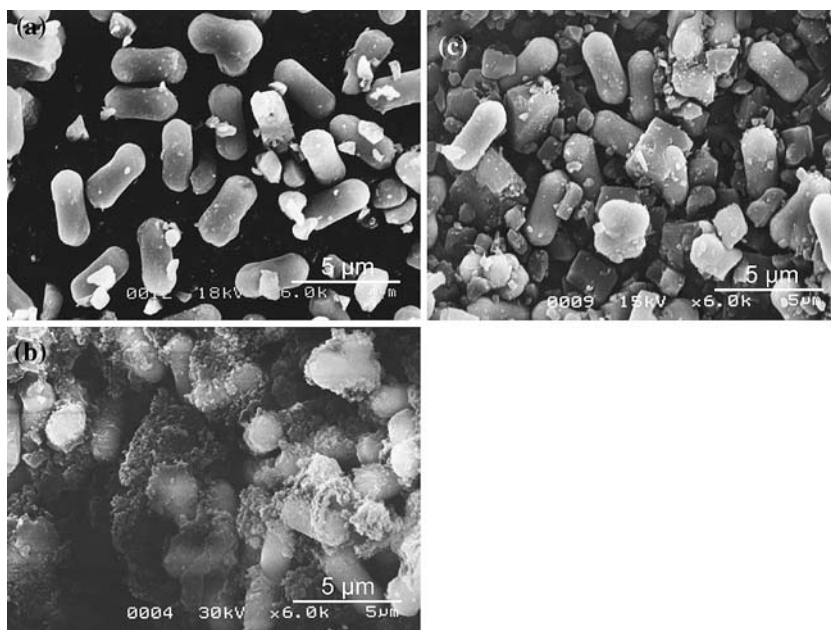
Lauric acid was used in place of C16TAC in order to support the reaction mechanism discussed above. The reaction mixture became acidic (pH = 2.1) and the color of the particles changed from brownish-red to orange after the reaction. The morphology of the particles changed as shown in Fig. 8(a) probably due to the dissolution. The formation of silicone shell on the particle surface was not shown by the SEM observation. The weight of the powder decreased by about 25% after the reaction. Even when the reaction was conducted under a basic condition (pH = 12), core-shell structure did not form as shown in the SEM micrograph (Fig. 8(b)). The hematite particles seemed to be embedded in the polycondensation product of PTES. The surface charge of hematite particles is dependent on pH so that cationic species such as C16TAC is appropriate to modify the surface under the basic condition where PTES polycondensates.

Reaction with MTES in the presence of C16TAC

The SEM micrograph of MTES/Hem(Si/Fe = 1.03) (Fig. 8(c)) revealed that the core-shell structure was not obtained and byproducts which thought to be polycondensation products of MTES were generated. For MTES/Hem(Si/Fe = 0.25), any byproduct was not observed in the SEM image, however, the silicone was not observed on the surface of hematite particles. There was no recognizable exothermic peak due to the decomposition of the methyl groups in the DTA curve (data not shown).

From these results, it was concluded that the silicone shell was not formed on the hematite surface when MTES was used as the silicone source instead of PTES. This difference in the obtained products between MTES and PTES may arise from the difference in the solubility of them to methanol, and/or the hydrophobic interaction with the alkyl groups of C16TAC. The solubility of MTES to methanol is higher than that of PTES and the hydrophobic interactions with the alkyl groups of C16TAC of MTES are weaker than those of PTES, because the methyl groups of MTES are less hydrophobic compared to the phenyl groups. As a consequence, MTES was not adsorbed to the surface of the hematite particles modified by C16TAC. To synthesize hematite/silicone core-shell particles by using silicon alkoxides, hydrophobicity of the organic groups of the silicon alkoxide and the hydrophobic interactions between the surface of the hematite particles and the reaction media are important factors. The effects of the silicone coating will be discussed in the following section using the product obtained by the reaction with PTES.

Fig. 8 SEM micrographs of (a) the lauric acid treated hematite particles after the reaction with PTES at pH = 2.1, (b) the lauric acid treated hematite particles after the reaction with PTES at pH = 12 and (c) MTES/Hem(Si/Fe = 1.03)



Properties of the hematite/silicone core-shell particles

Dispersibility

The dispersibility of the as-synthesized hematite particles in toluene was very low, so that the particles sedimented just after stopping the shaking. On the other hand, it took ten minutes for the core-shell particles to sediment completely, suggesting the improvement of the dispersibility in toluene. The improvement of the dispersibility may be attributed to the affinity of the silicone shell of the particles with toluene.

When PTES/Hem(sonication) and the as-synthesized hematite particles were added to a mixture of water and toluene (phase separated), PTES/Hem(sonication) dispersed only in the toluene layer, the uncoated hematite particles, on the other hand, distributed both within the toluene and water layers, which suggested the core-shell particles had strong hydrophobicity due to the silicone.

Dissolution of the hematite core

The particles floated on the solution just after the PTES/Hem(sonication) was added to hydrochloric acid aqueous solution. Immediately the particles dispersed in the solution. After the sonication for 1 min, brownish-red color of the particles changed to white, while the solution color changed from colorless to yellow during standing for 15 min at ambient temperature. In the SEM image of the solid product (Fig. 9), hollow particles with the shape very similar to the original hematite particles were seen. Figure 6(d) shows the IR spectrum of the product. All

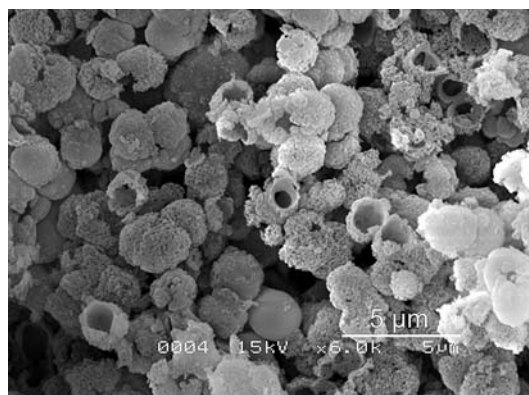


Fig. 9 SEM micrograph of the silicone shells obtained by treating PTES/Hem(sonication) with 12 mol/l HCl solution

absorption bands in the spectrum were ascribable to the silicone with phenyl groups, except for the unknown absorption band at 500 cm^{-1} , and the absorption bands due to hematite were not detected. Thus, it was shown that the acid treatment of the core-shell particles resulted in the dissolution of the hematite and the generation of the hollow particles comprised of silicone.

Monodispersed ellipsoidal hematite particles were successfully coated with silicone by the reaction with PTES under basic conditions in the presence of C16TAC, where the thickness of the silicone shell was controlled by changing the concentration of PTES. C16TAC was thought to make the surface of the hematite particles hydrophobic, which caused the interactions between the alkyl groups of C16TAC and the phenyl groups of PTES more effective. Sonication during the coating procedure led well-dispersed

particles in the reaction mixture. The silicone coated hematite particles obtained in the present study may be useful as industrial materials such as a pigment doped or mixed with various matrices, and its controlled morphology is potentially valuable for optical materials. Furthermore, the present reaction procedure is also useful to coat other oxide particles because of the ease of operation and mild reaction conditions.

Conclusions

Monodispersed ellipsoidal hematite particles with the size of 3.3 μm in major axis and 1.6 μm in minor axis were successfully coated with silicone by using a silane-coupling agent, phenyltriethoxysilane. In the presence of *n*-hexadecyltrimethylammonium chloride, the thickness of the silicone shell was controlled by changing the concentration of phenyltriethoxysilane. On the other hand, silicone shell did not form on the hematite particles when methyltriethoxysilane was used as the silicone source under the experimental conditions employed in the present study. The hematite/silicone core-shell particles had strong hydrophobicity due to the silicone shell, and had good dispersibility and stability in toluene. Hollow silicone shells were obtained by treating the hematite/silicone core-shell particles with hydrochloric acid.

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