

Influence of hollow silica-alumina composite spheres prepared using various amount of L(+)-arginine on their activity for hydrolytic dehydrogenation of ammonia borane

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ABSTRACT

In this work, we investigate influence of amount of L(+)-arginine on morphology of hollow silica-alumina composite spheres and their activity for hydrolytic dehydrogenation of ammonia borane. Hollow silica-alumina composite spheres were prepared by polystyrene templates method. In this method, silica-alumina composite shell were coated on polystyrene particles via a sol-gel reaction using L(+)-arginine as promoter, and the polystyrene template particles were removed by calcination. From the result of transmission electron microscopy, shell thickness of the hollow spheres prepared amount of L(+)-arginine of 0.0581, 0.1163, 0.2325, and 0.4650 g were 10, 20, 26, and 30 nm, respectively. From the result of nitrogen sorption, average pore size of the hollow spheres prepared amount of L(+)-arginine of 0.0581, 0.1163, 0.2325, and 0.4650 g was 6.2, 4.2, 3.4, and 2.9 nm, respectively. These results indicate that pore size and shell thickness changed into adjusting amount of L(+)-arginine. Activity of the hollow spheres prepared using various amount of L(+)-arginine for hydrolytic dehydrogenation of ammonia borane were compared. The result indicates that amount of hydrogen evolution of all the hollow spheres was almost the same level. On the other hands, hydrogen evolution rate increase with decrease of amount of L(+)-arginine. Copyright © 2016 VBRI Press.

Keywords: Ammonia borane; hollow silica-alumina composite spheres; amount of L(+)-arginine; average pore size; shell thickness.

Introduction

Hydrogen is a clean energy carrier. It is considered a very promising fuel, and can be an alternative to fossil fuels [1, 2]. Ammonia borane (NH₃BH₃) has attracted attention because of its high hydrogen content (19.6 %wt), low molecular weight (30.9 g·mol⁻¹), and high stability [3-10]. Additionally, NH₃BH₃ can evolve hydrogen via hydrolysis reaction in the presence of suitable acids and catalysts under mild conditions (Eq. 1). [10-24].



It has been reported that solid acids such as H-type zeolites (H-BEA and H-MOR) [11], hollow silica-alumina composite spheres [12-17], and hollow silica-zirconia composite spheres [18] show high activity for hydrolytic dehydrogenation of NH₃BH₃. However, there are few reports on the influence of morphology such as material size and pore size of solid acids on the activity for hydrolysis of NH₃BH₃.

To investigate this effect, we have focused on hollow spheres because of their intrinsic features such as high

specific surface area, low density, adsorption capacity, and easily controllable size. The hollow spheres are typically prepared hard template methods using polystyrene (PS) particles [25-30]. PS particles are attractive templates because they are monodisperse particles and easily controllable size. In this method, shells were coated onto the PS template particles and then the PS templates were removed by calcination.

Previously, we prepared hollow silica-alumina composite spheres using L(+)-arginine as a promoter [14-17]. It has also reported that morphology of hollow silica spheres was influenced by amount of aqueous ammonia solution as a promoter [31, 32]. In this work, we investigate influence of various amount of L(+)-arginine on morphology of the hollow silica-alumina composite spheres and their activity for hydrolytic dehydrogenation of NH₃BH₃.

Experimental

Preparation of hollow composite spheres

The hollow silica-alumina composite spheres were fabricated by the PS template method as described

previously. The monodisperse PS particles were prepared by emulsifier-free emulsion polymerization as follows: 9.0 mL of styrene (Kanto Chem. Co., > 99.0 %), 1.5 g of polyvinylpyrrolidone K30 (Fluka, Mw \approx 40000), 0.26 g of cationic initiator 2,2'-azobis-(isobutyramidine) dihydrochloride (Kanto Chem. Co., > 95.0 %), and 100 mL of ion-exchanged water were charged into a 250-mL three-neck flask equipped with a mechanical stirrer, a thermometer with a temperature controller, a nitrogen (N_2) inlet, a Graham condenser, and a heating oil bath. The reaction solution was deoxygenated by bubbling N_2 gas through the solution at room temperature for 1 h. The reaction was then, stirred at a rate of 250 rpm, and the reaction was heated to 343 K for 24 h. The obtained PS suspension was centrifuged at 6000 rpm for 5 min and washed three times with ethanol (Kanto Chem. Co., > 99.5 %), and the content of the PS suspension could be tailored through the addition of ethanol. 0.0057 g of aluminum isopropoxide (Aldrich, > 98.0 %), 0.0581-0.4650 g of L(+)-arginine (Wako, > 98.0 %), 40 mL of ethanol, and 0.1551 mL of tetraethoxysilane (TEOS, Kanto Chem. Co., > 99.9 %) were added to 15 g of the PS suspension. The sol-gel reaction was carried out at 323 K for 17 h, and the as-prepared composites could be obtained. After drying in a desiccator overnight, the hollow spheres were obtained by calcination in air at 873 K for 0 h at a heating rate of 0.5 K min^{-1} . The white powders were obtained in this fashion.

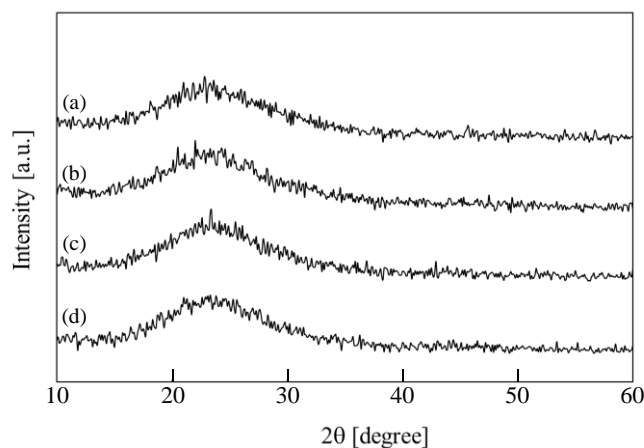


Fig. 1. Powder XRD pattern of hollow silica-alumina composite spheres prepared using amount of L(+)-arginine of (a) 0.0581, (b) 0.1163, (c) 0.2325, and (d) 0.4650 g.

Characterizations

The morphology of the hollow spheres was observed by Transmission electron microscopy (TEM) using a Hitachi FE2000 operating at an acceleration voltage of 200 kV. The specific surface area, average pore size, and pore volume of the hollow spheres were measured by N_2 sorption at 77 K with an ASAP 2010 Automatic Physical Adsorption Instrument. Powder X-ray diffraction (XRD) patterns were recorded on a Rigaku MultiFlex X-ray diffractometer using $Cu \text{ K}\alpha$ radiation (0.15406 nm) operating at 30 kV and 16 mA. Solid-state ^{27}Al magic angle spinning (MAS) nuclear magnetic resonance (NMR) measurements were performed by a JEOL ECA800

(18.8 Tesla) spectrometer equipped with a home-made 3.2 mm CPMAS probe set to 20 kHz spinning.

Activity for hydrolytic dehydrogenation of NH_3BH_3

A weight of 0.8 g of the hollow spheres was placed in a two-necked round-bottomed flask in air at room temperature. One neck was connected to a gas burette, and the other was connected to an addition funnel. The reaction was started by stirring the mixture of the hollow spheres, and aqueous NH_3BH_3 (Aldrich, 90 %) solution (0.14 wt. %, 3.5 mL) was added from the addition funnel. The evolution of gas was monitored using the gas burette.

Results and discussion

The crystalline structure of the hollow spheres prepared using various amount of L(+)-arginine was investigated by powder X-ray diffraction measurements. **Fig. 1** shows the XRD patterns of the hollow spheres prepared using various amount of L(+)-arginine. The XRD pattern of all the hollow spheres shows the characteristic peaks of silica in an amorphous phases between $2\theta = 20^\circ$ and 40° , indicating that the hollow spheres consist of amorphous silica phases. The morphology of hollow silica-alumina composite spheres were observed using TEM measurement. **Fig. 2** shows TEM images of hollow silica-alumina composite spheres prepared using various amount of L(+)-arginine. From the results, shell thickness of the hollow spheres prepared using amount of L(+)-arginine of 0.0581, 0.1163, 0.2325, and 0.4650 g were 10, 20, 26, and 30 nm, respectively. The result indicates that shell thickness of the hollow spheres increase with increase of amount of L(+)-arginine.

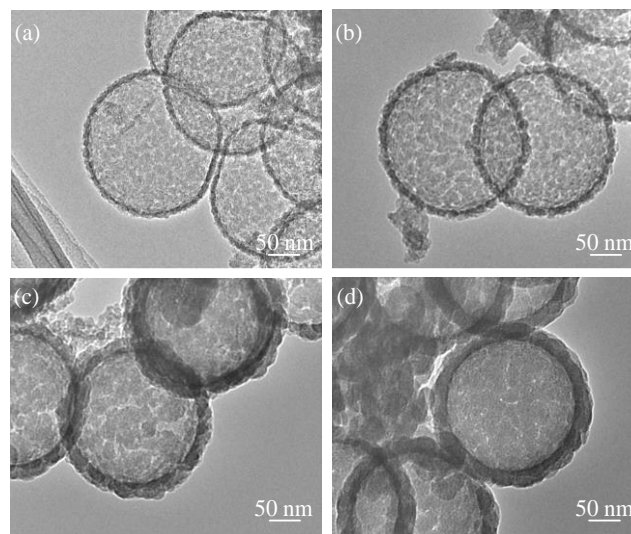


Fig. 2. TEM images of hollow silica-alumina composite spheres prepared using amount of L(+)-arginine of (a) 0.0581, (b) 0.1163, (c) 0.2325, and (d) 0.4650 g.

Table 1 shows physiochemical properties of the hollow spheres prepared using various amount of L(+)-arginine. Specific surface area, average pore size, and pore volume were measured by N_2 sorption using the Brunauer-Emmett-Teller (BET) and Barrett-Joyner-Halenda (BJH) methods. From the result, specific surface area increase with increase

of amount of L(+)-arginine. On the other hands, average pore size (4V/A by BET) and single point total pore volume (at $P/P_0=0.97$) decrease with increase of amount of L(+)-arginine.

Table 1. Physicochemical properties of hollow silica-alumina composite spheres prepared using various amount of L(+)-arginine.

L(+)-arginine [g]	Specific surface area [m^2g^{-1}]	Average pore size [nm]	Pore volume [cm^3g^{-1}]
0.0581	469	6.3	0.74
0.1163	520	4.4	0.57
0.2325	576	3.5	0.46
0.4650	590	2.8	0.40

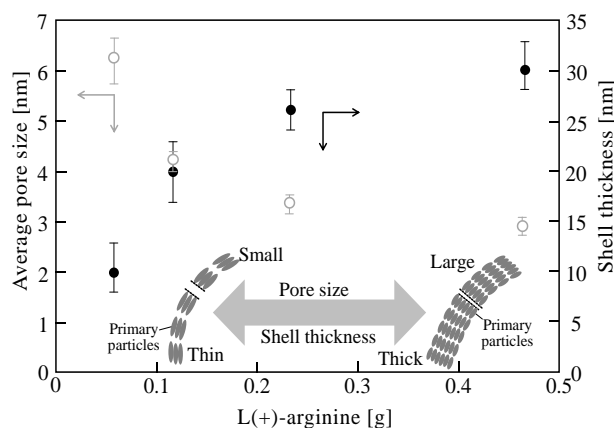


Fig. 3. Average pore size and shell thickness of the hollow spheres versus various amount of L(+)-arginine.

Fig. 3 shows average pore size and shell thickness of the hollow spheres versus various amount of L(+)-arginine. From the result, average pore size and shell thickness of the hollow spheres changed into adjusting amount of L(+)-arginine. The silica-alumina composite shell was composed of primary particles. It has been reported that the size of primary particles decreases with increase of amount of promoter [33]. Based on the result, the size of primary particles probably decreases with increase of amount of L(+)-arginine because a number of silica-alumina nucleus increase with increase of amount of L(+)-arginine.

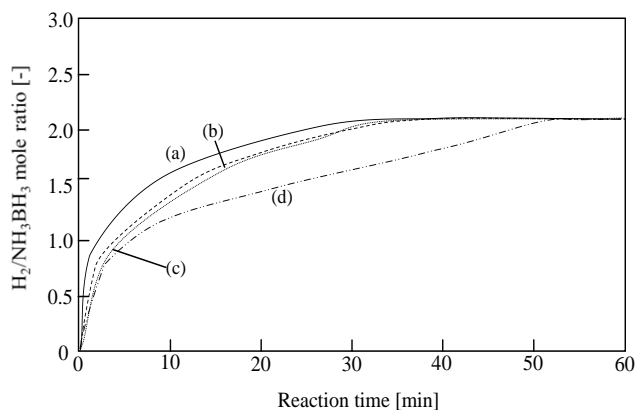


Fig. 4. $\text{H}_2/\text{NH}_3\text{BH}_3$ molar ratios of hydrogen evolved from aqueous NH_3BH_3 solution (0.14 wt.%, 3.5 mL) in the presence of hollow silica-alumina composite spheres prepared using amount of L(+)-arginine of (a) 0.0581, (b) 0.1163, (c) 0.2325, and (d) 0.4650 g.

Activities of the hollow spheres prepared using amount of L(+)-arginine of 0.0581, 0.1163, 0.2325, and 0.4650 g for hydrolytic dehydrogenation of NH_3BH_3 were compared. **Fig. 4** shows time course of hydrogen generation in the presence of the hollow spheres prepared using various amount of L(+)-arginine. From the result, hydrogen evolution of 8 mL was occurred at 29, 33, 35, and 54 min in the presence of the hollow spheres prepared using amount of L(+)-arginine of 0.0581, 0.1163, 0.2325, and 0.4650 g, respectively. The molar ratios of the hydrolytically generated hydrogen to the initial NH_3BH_3 in the presence of all the hollow spheres were 2.1 (theoretical value: 3.0). On the other hand, hydrogen evolution rate calculated with the data produced up to 50 % of the amount of hydrogen at reaction completion. The hydrogen evolution rate of the hollow spheres prepared using amount of L(+)-arginine of 0.0581, 0.1163, 0.2325, and 0.4650 g was 1.3, 1.0, 0.8, and 0.7 mL min^{-1} , respectively.

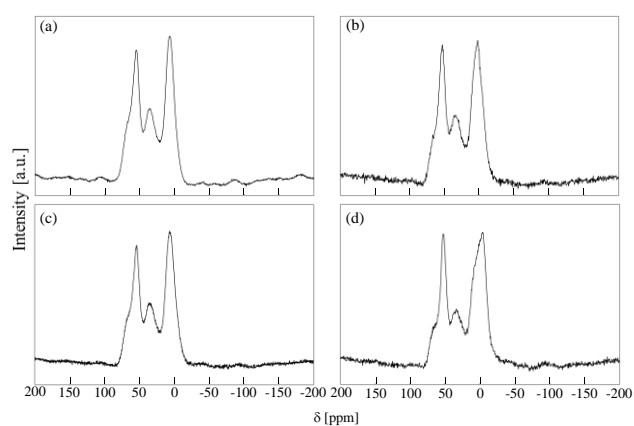


Fig. 5. Solid-state ^{27}Al MAS NMR spectra of hollow silica-alumina composite spheres prepared using amount of L(+)-arginine of (a) 0.0581, (b) 0.1163, (c) 0.2325, and (d) 0.4650 g.

It has been reported that amount of hydrogen evolution depends on amount of Brønsted acid sites [12, 15, 16]. Amount of acid sites of the hollow spheres prepared using various amount of L(+)-arginine was measured by neutralization titration with n-butyl amine using methyl red as the indicator. From the result, amount of acid sites of all the hollow spheres were approximately 0.10 mmol g^{-1} . The coordination number of the hollow silica-alumina composite spheres prepared using various amount of L(+)-arginine were examined using solid-state ^{27}Al MAS NMR measurement. From the result, all the hollow spheres have 4-, 5-, and 6- coordinated aluminum species, as evidenced by three spectra with separated ^{27}Al signals at about 52, 30, and 0 ppm as shown in **Fig. 5**. Additionally, peak area ratios of 4-, 5-, and 6- coordinated aluminum species ($I_4:I_5:I_6$) of all the hollow spheres were 9:4:7, respectively. It has been reported that 4-coordinated aluminum was associated with Brønsted acid sites [34-36]. These results indicate that the amount of Brønsted acid sites in all the hollow spheres were almost the same level.

On the other hand, the hydrogen evolution rate increase with decrease of the pore size and/or shell thickness. It has been reported that the shell thickness under about 30 nm of the hollow silica-alumina composite spheres has little effect on the hydrogen evolution rate for hydrolytic

dehydrogenation of NH_3BH_3 [16]. Fig. 6 shows the hydrogen evolution rate from aqueous NH_3BH_3 solution versus average pore size of the hollow spheres prepared using various amount of L(+)-arginine. From the result, the hydrogen evolution rate increase with increase of average pore size of the hollow spheres. The result suggests that the hydrogen evolution rate depends on the pore size of the hollow spheres.

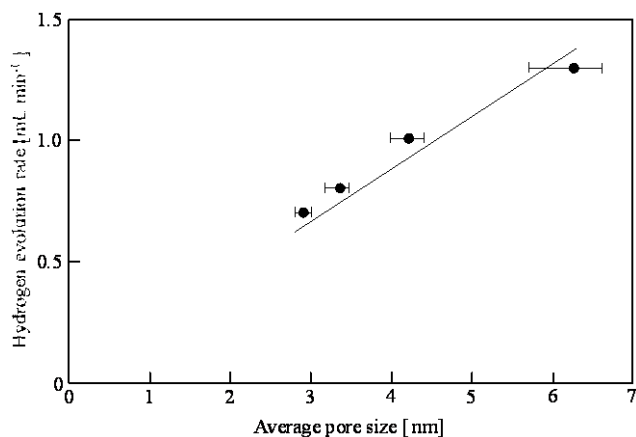


Fig. 6. Hydrogen evolution rate from aqueous NH_3BH_3 solution (0.14 wt.%, 3.5 mL) versus average pore size of hollow silica-alumina composite spheres prepared using various amount of L(+)-arginine.

Conclusion

We investigated effect of various amount of L(+)-arginine on the morphology of hollow silica-alumina composite spheres and their activity for hydrolytic dehydrogenation of NH_3BH_3 . From the result of TEM images, shell thickness of the hollow spheres increase with increase of amount of L(+)-arginine. From the result of N_2 sorption measurement, average pore size of the hollow spheres decrease with increase of amount of L(+)-arginine. These results indicate that the shell thickness and average pore size of the hollow spheres changed into adjusting amount of L(+)-arginine because the size of primary particles decreases with increase of the amount of L(+)-arginine. Activities of the hollow spheres prepared using various amount of L(+)-arginine for hydrolytic dehydrogenation of NH_3BH_3 were compared. From the results, the amount of hydrogen evolution of all the hollow spheres was the same level. From the result of neutralization titration and solid-state ^{27}Al MAS NMR spectra, the amount of Brønsted acid sites for hydrolytic dehydrogenation of NH_3BH_3 was almost the same level. On the other hand, the hydrogen evolution rate increases with increase of average pore size of the hollow spheres. The result indicates that the hydrogen evolution rate depends on the pore size of the hollow spheres.

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Author contributions

Conceived the plan: NT, TU; Performed the experiments: NT, HI; Data analysis: NT, HI, SO; Wrote the paper: NT (xx, xy, yz are the initials of authors).

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