The synthesis of urchin-like $\gamma$-Al$_2$O$_3$ hierarchical microspheres from Al foils and its rapid adsorption ability towards Congo red

Kaizhong Feng, Jie An, Weian Ren, Xiaoling Lei, Gaoying Li, Daoqing Rong and Xiaogang Wen

School of Materials Science and Engineering, Sichuan University, Chengdu, 610065, People’s Republic of China

E-mail: wenxg@scu.edu.cn

Keywords: $\gamma$-Al$_2$O$_3$, NH$_4$Al(OH)$_2$CO$_3$, hierarchical nanostructures, adsorption, congo red

Abstract

In this paper, we present a convenient hydrothermal method to synthesize hierarchical microspheres consisting of ammonium aluminum carbonate hydroxide (AACH) nanowires with a diameter of about 400 nm and length $\sim$50 $\mu$m on aluminum foils. After calcination at 900 °C, hierarchical $\gamma$-Al$_2$O$_3$ microspheres with mesoporous structures were obtained successfully, which possess a high specific surface area of 124 m$^2$ g$^{-1}$ and high porosity of 0.71 cm$^3$ g$^{-1}$. The possible formation mechanism was discussed. The as-prepared mesoporous $\gamma$-Al$_2$O$_3$ hierarchical nanostructures exhibited improved adsorption performance towards Congo red in aqueous solution, 90% CR could be removed rapidly in 20 min, and its saturation adsorption capacity in 80 mg l$^{-1}$ Congo red can reach to 180 mg g$^{-1}$, it suggests that the hierarchical $\gamma$-Al$_2$O$_3$ microspheres with unique microstructure have potential in wastewater treatment.

1. Introduction

Due to its low density, high hardness and strength, excellent thermal and chemical stability, and good optical properties [1–4], alumina has been used as catalyst, catalyst support [5, 6], adsorbent [4, 7] and ceramic [8, 9] etc widely. Recently, one dimensional (1D) and two dimensional (2D) nanostructure arrays have attracted extensive interests because of their better charge transportation ability, higher surface area and enhanced mechanical properties. Especially, the hierarchical nanostructures constructed with 1D or 2D nanounits demonstrated obvious advantages in adsorption, because it provided high specific surface area, high surface-to-volume ratio, high pore volume, enough mechanical strength and easy recovery, which combined the characteristics of both the nanosized block units and the overall microsized aggregations [4]. The 1D & 2D alumina, including the corresponding hierarchical alumina nanostructures, were mainly synthesized by thermal evaporation at high temperature [2, 10], electrochemical deposition [11] or chemical etching [12, 13], and the thermal decomposition from boehmite (AlOOH) [14] or ammonium aluminum carbonate hydroxide (NH$_4$Al(OH)$_2$CO$_3$, AACH) [15, 16]. As a low-temperature phase, $\gamma$-Al$_2$O$_3$ nanostructures were mainly synthesized by the last two strategies. For the 1D $\gamma$-Al$_2$O$_3$ synthesis via thermal decomposition from Al-contained precursors, mostly, various kinds of soluble Al salts were used as Al source. Due to the rapid growth in the solution with relative high Al ion concentration, it is difficult to tune the growth direction, morphology and microstructure of final nanomaterials. Whereas the growth rate of nanostructures can be controlled well when it grow from metal because of the relative low atom or ion diffusion rate in metal source, and low solubility in solution, hence, nanomaterials with well-controlled morphology could be formed on metal substrate. For example, Cu$_2$S nanowire or CuO nanorod arrays on Cu substrate [17, 18], Fe$_3$O$_4$ nanowire or nanobelt array on Fe substrate [19, 20], ZnO nanowire arrays on Zn substrate [21], TiO$_2$ nanowire array on Ti substrate [22] etc have been successfully synthesized by this strategy. Up to now, to our best knowledge, there is no report about the synthesis of Al-based nanowire arrays from metal Al source via a surfactant-free solution phase growth process.

In this paper, we report a novel approach to fabricate ammonium aluminum carbonate hydroxide nanowire arrays (AACH) on Al foils via a simple hydrothermal process. And after calcination of AACH, urchin-like
\( \gamma - \text{Al}_2\text{O}_3 \) and \( \alpha - \text{Al}_2\text{O}_3 \) hierarchical microspheres could be obtained respectively by controlling the annealing temperature. And the synthesized \( \gamma - \text{Al}_2\text{O}_3 \) hierarchical microspheres exhibit good adsorption ability towards Congo red dye. 90\% CR could be removed in 20 min, the saturated adsorption amount can reach to 180 mg g\(^{-1}\).

2. Experimental

All the reagents used were analytical grade and used as received without further purification.

The AACH nanowire arrays were synthesized via a hydrothermal process on the surface of aluminum foils. In a typical synthesis, firstly, high purity Al foils \((10 \times 10 \text{ mm})\) with 1 mm thickness was degreased in acetone for 15 min and then rinsed in distilled water. After that, it was placed into 1 M NaOH solution for 20 min to eliminate the oxide layer and subsequently rinsed with distilled water for four times. The as-cleaned aluminum foils were fully immersed into 20 ml 4 M urea solution in a 25 ml Teflon-lined autoclave. The autoclave was heated \((5 \text{ °C min}^{-1})\) to 180 °C and kept at this temperature for 24 h. Then the autoclave was cooled to room temperature naturally, and the Al foils covered with a compact white layer were obtained. The samples were washed several times with distilled water and then dried at 80 °C for 8 h in an oven. To obtain \( \gamma - \text{Al}_2\text{O}_3 \) and \( \alpha - \text{Al}_2\text{O}_3 \) nanowires, the prepared AACH nanowire were scraped carefully from the Al foils, and then were calcined at 900 °C and 1200 °C for 3 h in air, respectively. Finally, the obtained white powders were collected for further characterization and measurement.

The morphology of the samples before and after annealing was observed using scanning electron microscopy (SEM, JEOL JSM-6490LV) at an accelerating voltage of 15 kV, and their crystal phases were identified by X-ray diffraction (XRD; Philips X’Pert PRO) with Cu K\( \alpha \) radiation. More microstructure and crystal structure analysis of the samples before and after calcination was characterized by transmission electron microscopy (TEM, JEM 2100 F) equipped with a selected area electron diffraction (SAED). Thermogravimetric and differential thermal analyses (TG-DTA) of samples were conducted on a thermogravimetric analyzer (STA449F3 NETZSCH). The Brunauer–Emmett–Teller (BET) surface of the sample was calculated from nitrogen adsorption data collected on a Micromeritics ASAP 2020 gas adsorption apparatus (USA). The BET surface area was determined by BET model using the adsorption data in the relative pressure \((P/P_0)\) range of 0.05–0.3. The pore size distribution was calculated by the Barrett–Joyner–Halender (BJH) method using adsorption isotherm. The nitrogen adsorption volume at the relative pressure \((P/P_0)\) of 0.992 was used to determine the pore volume. Fourier transform infrared spectrometer (FT-IR, Nicolet 6700) was employed to detect the composition of AACH precursor and \( \gamma - \text{Al}_2\text{O}_3 \) nanowires.

Adsorption measurements of Congo red (CR, \( C_{22}H_{22}N_6O_6S_2Na_2 \)) on \( \gamma - \text{Al}_2\text{O}_3 \) were performed at room temperature. In a typical process, 20 mg of absorbent was added into 50 ml of CR solutions \((80 \text{ mg l}^{-1}, \text{pH ca.7})\), after a continuous stirring at a rate of 150 rpm for different time, 5 ml of solutions was taken out and centrifuged for 5 min at 7000 rpm, and then the upper transparent solutions were collected and measured using UV–vis spectrophotomer (UV–vis). The characteristic absorption of CR around 500 nm is chosen to monitor the adsorption process.

The percentage removal of CR (CR\(_{\text{eq}}, \%\)) was determined by the following formula:

\[
\text{CR}_{\text{eq}} = \frac{(C_0 - C_e)}{C_0} \times 100
\]

3. Results and discussion

The crystal phases of obtained samples were characterized by XRD. Figure 1 shows the XRD patterns of samples before and after calcination. It can be seen from figure 1(a), the as-prepared sample on Al foil (before calcination) demonstrates strong and sharp peaks, which indicate the high crystallinity of the sample, and all the diffraction peaks can be indexed well to orthorhombic ammonium aluminum carbonate hydroxide \((\text{NH}_4\text{Al(OH)}_2\text{CO}_3)\), the calculated lattice constants \((a = 6.639 \text{ Å}, b = 11.929 \text{ Å} \text{ and } c = 5.717 \text{ Å})\) were close to the reference values \((a = 6.618 \text{ Å}, b = 11.944 \text{ Å} \text{ and } c = 5.724 \text{ Å}, \text{JCPDS No. 42-0250})\). After calcination at 900 °C for 3 h (figure 1(b)), the diffraction signal become much weaker, which implies the tremendous crystal structure transformation during the calcination process, the diffraction peaks at 31°, 42°, 63° can be attributed
planes of $\gamma$-Al$_2$O$_3$ (JCPDS No. 10-0425) and no other impurity peaks were detected. The calculated lattice constants ($a = b = c = 7.900$ Å) are very consistent with the reference values ($a = b = c = 7.900$ Å). And the broaden diffraction peaks of $\gamma$-Al$_2$O$_3$ reveal its nanosized nature, the crystallite size of $\gamma$-Al$_2$O$_3$ nanostructures was estimated to be 12.6 nm based on Scherer’s formula ($D_{hk\ell} = \frac{0.89\lambda}{B_{hk\ell} \cos \theta}$). As shown in figure 1(c), while the sample was calcined at higher temperature of 1200 °C, it will be transformed to $\alpha$-Al$_2$O$_3$ (JCPDS Card No.10-0173) and the crystallization become better than $\gamma$-Al$_2$O$_3$. All the diffraction peaks can be indexed to corundum Al$_2$O$_3$ and no impurity phases are observed.

It is well known that different alumina phases including amorphous Al$_2$O$_3$, $\gamma$-Al$_2$O$_3$, $\theta$-Al$_2$O$_3$ and $\alpha$-Al$_2$O$_3$ can be obtained during the thermal decomposition of AACH by release of H$_2$O, NH$_3$, and CO$_2$ based on the following equation [23, 24]:

$$2\text{NH}_4\text{Al(OH)}_2\text{CO}_3 \rightarrow \text{Al}_2\text{O}_3 + 2\text{CO}_2 + 2\text{NH}_3 + 3\text{H}_2\text{O}$$

(3)

In order to investigate the thermal decomposition processes of the AACH nanowires, thermogravimetric and differential scanning calorimetry (TG-DSC) analysis was employed. The temperature increased from 42.7 °C to 1500 °C at a rate of 10 °C min$^{-1}$ in a flowing nitrogen environment. The TG-DSC curves of the AACH are shown in figure 2. Based on TG curve, it is clear that the AACH has two major weight loss. The first weight loss below 123 °C can be attributed to the desorption of physically adsorbed water. The second weight loss between the temperature range of 123 °C–300 °C is associated with the decomposition of AACH, releasing CO$_2$, NH$_3$, and H$_2$O and forming AlOOH. As the temperature continues to increase, the TG curve keeps descending.

Figure 1. XRD patterns of the samples: (a) as-synthesized from solution, (b) after annealing at 900 °C for 3 h, and (c) 1200 °C for 3 h.

Figure 2. TG-DSC plots of the as-synthesized AACH nanowires.
between temperature of 300 °C–500 °C, which is corresponding to the weight loss from AIOOH to Al₂O₃ [23, 24]. When the temperature is above 500 °C, there is no further weight loss is observed. The total weight loss is about 55.82%, which is lower than the theoretical value of 63.30%, and it can be ascribed to the quasi-aluminum hydroxycarbonates which are contained in the as-synthesized sample [16]. For DSC curves, there is a strong endothermic peak at 283 °C, which corresponds to the release of CO₂, NH₃, and H₂O with increasing temperature. Three exothermic peaks at 800.6 °C, 984.5 °C and 1161.2 °C appear, which can be ascribed to the phase transition process of the alumina from amorphous Al₂O₃ to γ-Al₂O₃, γ-Al₂O₃ to θ-Al₂O₃ and θ-Al₂O₃ to α-Al₂O₃, respectively [24]. Based on the TG-DSC analysis of AACH, urchin-like γ-Al₂O₃ and α-Al₂O₃ microspheres can be obtained after calcined at 900 °C and 1200 °C for 3 h, respectively.

Figure 3(a) shows the SEM image of AACH nanowires arrays as-grown on the Al foil in 4 M urea solution. These nanowires are about 400 nm in average diameters, and uniform along the whole length, the dense nanowires seem to originated from some small spots and grow radially, and finally, a lot of urchin-like AACH nanowire arrays are formed on the surface of Al foil. Figure 3(b) is the TEM image of several AACH nanowires. It indicates that the nanowires are straight and rigid, and the diameter range from 200 to 500 nm. After annealing at 900 °C for 3 h, the nanowires become more flexible, but the wire-like morphology were maintained well (figure 3(c)), the average diameter of the nanowire decrease to 350 nm, indicating the crystal shrinkage of nanowire induced by the release of CO₂, NH₃, and H₂O. Figure 3(d) shows the magnified TEM image of a single γ-Al₂O₃ nanowire, it demonstrates a porous nature, and the corresponding SAED pattern (Inset of figure 3(d)) demonstrates several concentric circles, indicating the polycrystalline characteristics of γ-Al₂O₃ nanowire. Two clear diffraction rings are responding to (440) and (400) planes of γ-Al₂O₃ respectively. These results are agreement with previous XRD analysis, and further confirm that the AACH nanowires have been completely converted to γ-Al₂O₃ nanowires.

In order to give further insight into the porous structure and the pore size distribution of γ-Al₂O₃, the BET measurement was performed. As shown in figure 4(a), the N₂ adsorption–desorption isotherm of urchin-like γ-Al₂O₃ hierarchical nanostructures presents as a type IV isotherm with a H3 hysteresis according to the International Union of Pure and Applied Chemistry (IUPAC) classification [25], suggesting the existence of abundant slit-like pores in the hierarchical microspheres. This also can be confirmed by SEM and TEM image of γ-Al₂O₃ in figures 3(c) and (d). The calculated BET surface area of γ-Al₂O₃ nanowires is about 124 m² g⁻¹. Figure 4(b) presents the pore size distribution of γ-Al₂O₃ nanowires. It is obvious that γ-Al₂O₃ exhibits a narrow pore size distribution. There are two kinds of pores in the urchin-like hierarchical nanostructures, the smaller pores centered at about 3.1 nm should come from the interior of nanowires, which formed by crystal transformation from AACH to γ-Al₂O₃, companying the release of gaseous CO₂, NH₃, and H₂O. The larger pores centered at about 8.9 nm can be ascribed to the slit-like pores among the nanowires [4, 25]. The pore

Figure 3. SEM and TEM images AACH nanowires (a) and (b) and γ-Al₂O₃ (c) and (d). Inset of D: corresponding SAED pattern of a single γ-Al₂O₃ nanowire.
The volume of γ-Al2O3 nanowires is calculated to be about 0.71 cm^3 g⁻¹. The large surface area and mesoporous structures suggest that γ-Al2O3 may be a potential candidate for adsorbent and catalyst.

For the preparation of AACH hierarchical nanostructures, urea, which is used as a source of carbonate and pH adjusting agent, has played a crucial role. Figure 5 shows the SEM images of AACH nanostructures on the Al foils grown with different urea concentration. When the concentration of urea solution was 2 mol l⁻¹, no AACH wires is observed on aluminum foils, instead, a thin nanoplates layer grow from the Al substrate directly (figures 5(a) and (b)). However, once the concentration of urea solution was increased to 3.33 M, a small amount of white powders appear on aluminum foils, and urchin-like hierarchical nanostructures consisting of smooth 1D nanowires can be observed clearly from the SEM image (figures 5(c) and (d)), based on the diameter of whole urchin-like hierarchical nanostructures, the measured length of AACH nanowires (equal to the radius of urchin-like hierarchical microspheres) can reach to 40 μm, some disperse nanowire and nanowire bundles, lying down on the substrate, can also be observed, which imply that a lot of nanowires prefer to radially grow from a same nucleus, and it also indicate that the nanowires does not grow from the Al substrate directly, the urchin-like AACH hierarchical nanostructures should be formed in solution and deposited on the Al substrate finally. When the concentration of urea was increased to 4 M, the whole aluminum foil was covered with thick white powders. As depicted in figures 5(e) and (f), abundance of urchin-like hierarchical microspheres consisting of nanowires with ~400 nm in diameter deposit on the substrate, and the nanowire length is about 50 μm. Comparing with the samples obtained from 3.33 M and 4 M urea solutions, the samples prepared from 5 M urea solution are relatively higher yield, but no obvious morphology changes of the urchin-like microspheres were observed (figures 5(e) and (f)).

Based on the previous analysis, the formation process of AACH nanowires on aluminum foil can be suggested as the following reactions [16, 26]:

\[
\begin{align*}
\text{CO(NH}_2\text{)}_2 + 3\text{H}_2\text{O} & \rightarrow 2\text{NH}_4^+ + \text{HCO}_3^- + \text{OH}^- \\
2\text{Al} + 2\text{OH}^- + 6\text{H}_2\text{O} & \rightarrow 2\text{Al(OH)}_3^- + 3\text{H}_2 \\
\text{Al(OH)}_3^- + \text{NH}_4^+ + \text{HCO}_3^- & \rightarrow \text{NH}_4\text{Al(OH)}_2\text{CO}_3 + \text{H}_2\text{O} + \text{OH}^- 
\end{align*}
\]

At first, the urea will produce sufficient NH₄⁺, HCO₃⁻ and OH⁻ at the hydrothermal conditions (reaction 4), and then Al(OH)₃ chain will be formed near the surface of the Al foil in the basic solution (reaction 5). In the presence of NH₄⁺, OH⁻ and HCO₃⁻, NH₄Al(OH)₂CO₃ nanoparticles, which can be used as the crystal nucleus, could be produced. Schematic 1 shows the formation process of novel urchin-like γ-Al₂O₃ hierarchical microcrystals. At first, 1D NH₄Al(OH)₂CO₃ nanowires is easy to form in the presence of low concentration urea [27–29], due to NH₄⁺ and OH⁻ ions can prefer to adsorb on some of the crystal planes of AACH nanocrystallites, it promote the nucleus grow along a preferential direction to form AACH nanowires. And increasing urea concentration will produce more OH⁻ in the solution, which results in the decrease of particle size, so the rod-like surface of AACH start separating from each other [27], various separated nanowires originated from a same nucleus grow radially and form the urchin-like hierarchical microspheres. Finally, after annealing at 900 °C, urchin-like γ-Al₂O₃ hierarchical microspheres can be obtained from AACH by releasing H₂O, NH₃ and CO₂ (reaction 3).
Figure 6 shows the Fourier transform infrared (FT-IR) spectra of samples before and after annealing. The as-synthesized AACH nanowires without annealing (curve a) display strong adsorption for $\text{OH}^-$, $\text{NH}_4^+$, $\text{CO}_3^{2-}$ and Al-O etc. groups. The peaks at 3445 and 981 cm$^{-1}$ are associated with stretching and bending of hydroxyl group respectively, the absorption bands between 3174 and 2850 cm$^{-1}$ are due to symmetric
stretching of NH$_4^+$ group, and the absorption peaks at 1832, 1723 and 1391 cm$^{-1}$ are due to asymmetric stretching of NH$_4^+$ group [27, 30–32]. The absorption bands at 1538, 1452 and 1106 cm$^{-1}$ can be ascribed to CO$_3^{2−}$ group [27, 30–32]. And the bands between 856 and 483 cm$^{-1}$ are associated with the lattice vibrational modes of Al–O bond [27, 32]. After calcination at 900 °C for 3 h, as can be seen from curve (b), the sample’s FT-IR spectrum change obviously, most O–H, N–H, –CO$_3$ become much weaker or even disappear, whereas, the peaks in the region 500–1000 cm$^{-1}$, which may be assigned to Al–O, become much stronger. The broad band at 3446 cm$^{-1}$ and the weak peak at 1643 cm$^{-1}$ may be caused by the O–H of the adsorbed water in the sample, while the peaks at 1400 and 1113 cm$^{-1}$ may be come from the adsorbed non-structural carbonate group [32]. The IR spectra before and after calcination further confirm that the AACH precursor has completely transformed into γ-Al$_2$O$_3$ after annealing at 900 °C for 3 h.

Figure 7 shows the CR adsorption performance of urchin-like γ-Al$_2$O$_3$ hierarchical microspheres. As can be seen from figure 7(a), the adsorption of γ-Al$_2$O$_3$ towards CR is very fast, 87% Congo red was removed by the γ-Al$_2$O$_3$ within 10 min, and the removal rate can reach to 90% after 20 min. It confirms that the γ-Al$_2$O$_3$ hierarchical microspheres consisting of nanowires possess rapid adsorption ability towards CR. Figure 7(b) compares the adsorption rate of synthesized urchin-like γ-Al$_2$O$_3$ microspheres, α-Al$_2$O$_3$ microspheres and commercial Al$_2$O$_3$ powder. It is clear that the adsorption of CR onto γ-Al$_2$O$_3$ microspheres is faster than that onto the α-Al$_2$O$_3$ microspheres and commercial Al$_2$O$_3$ powder, the saturation adsorption capacity of urchin-like γ-Al$_2$O$_3$ hierarchical microspheres in 80 mg l$^{-1}$ Congo red (adsorbent dosage: 400 mg l$^{-1}$) can reach to...
180 mg g$^{-1}$, which is much higher than that of $\alpha$-Al$_2$O$_3$ microspheres (140 mg g$^{-1}$) and commercial Al$_2$O$_3$ (120 mg g$^{-1}$) powder. It is well known that Congo red is easy to be absorbed onto the metal oxide surface due to coordination effect between metal ions and amine groups at the end of Congo red molecules and the adsorption capacity of a material is determined by its specific surface area, porous size, surface chemical structure, and active sites etc [4]. Therefore, the good adsorption performance should be mainly ascribed to its unique urchin-like hierarchical nanostructures, which provide effective slit-like pores for fast adsorption and accommodation to CR molecular, as well as its suitable crystalline structure and good dispersion.

Figure 8 shows the adsorption recyclability of the urchin-like $\gamma$-Al$_2$O$_3$ hierarchical microspheres towards CR. It indicates that the as-prepared $\gamma$-Al$_2$O$_3$ microspheres have good adsorption recyclability, after 5 adsorption cycles, the Congo red removal efficiency can maintain at more than 90% with no significant decrease.

4. Conclusions

In summary, AACH nanowire arrays have been fabricated under hydrothermal condition using metal aluminum as aluminum source. After simple calcination process, novel mesoporous microspheres consisting of $\gamma$-Al$_2$O$_3$ nanowires could be obtained easily. The urchin-like $\gamma$-Al$_2$O$_3$ hierarchical microspheres with high specific surface area (124 m$^2$ g$^{-1}$) and unique slit-like pore structure exhibits much improved adsorption performance towards CR dye. 90% dye could be removed in 20 min, and the saturation adsorption capacity in 80 mg l$^{-1}$ Congo red (adsorbent dosage: 400 mg l$^{-1}$) can reach to 180 mg g$^{-1}$. the results suggest a novel strategy for the facile synthesis of hierarchical Al$_2$O$_3$ nanostructures, which could be a promising candidate adsorbent for wastewater treatment. Besides, it is also expected to have good potential in applications on catalyst and catalyst supporting etc.

Acknowledgments

The authors thank the financial supporting of the National Natural Science Foundation of China (No. 51072124), the Program for New Century Excellent Talents in University (No. NCET100605) and the Sichuan Science and Technology Program (No. 2018GZ0463).

ORCID iDs

Xiaogang Wen  https://orcid.org/0000-0002-4653-1793

References

[1] Zhu Z F, Sun H J, Liu H and Yang D 2010 PEG-directed hydrothermal synthesis of alumina nanorods with mesoporous structure via AACH nanorod precursors J. Mater. Sci. 45 46–50

[2] Peng X S, Zhang L D, Meng G W, Wang X F, Wang C Z and Wu G S 2002 Photoluminescence and infrared properties of $\gamma$-Al$_2$O$_3$ nanowires and nanobelts J. Phys. Chem. B 106 11163–7
[1] Green S, Badán J A, Gilles M, Cortes A, Riveros G, Ramírez D, Gómez H, Quaglia E, Dalchíe E A and Marotti R E 2007 Optical properties of nanoporous Al2O3 obtained by aluminium anodization Phys. Stat. Sol. (C) 4 618–21
[2] Cai W Q, Yu J G and Jaroniec M 2010 Template-free synthesis of hierarchical spindles-like γ-Al2O3 materials and their adsorption affinity towards organic and inorganic pollutants in water J. Mater. Chem. 20 4587–94
[3] Shimizu K, Satsuma A and Hattori T 2000 Catalytic performance of Ag-Al2O3 catalyst for the selective catalytic reduction of NO by higher hydrocarbons Appl. Catal. B: Environ. 25 239–47
[4] Han Y F, Zhong Z Y, Ramesh K, Chen F X and Chen L W 2007 Effects of different types of γ-Al2O3 on the activity of gold nanoparticles for CO oxidation at low-temperatures J. Phys. Chem. C 111 3163–70
[5] Knaup J M, Köhler C, Frauenheim T, Blumenau A T, Amkreutz M, Schüpfel P, Schneider B and Hennemann O D 2006 Computational studies on polymer adhesion at the surface of γ-Al2O3, I. The adsorption of adhesive component molecules from the gas phase J. Phys. Chem. B 110 20460–8
[6] Ganesh I, Olherro S M, Torres P M C and Ferreira J M F 2009 Gelcasting of magnesium aluminate spinel powder J. Am. Ceram. Soc. 92 350–7
[7] Waka Y, Nakagawa N, Wakamoto T, Ohtsubo H, Shimizu K and Kohtoku Y 1998 High-temperature strength and thermal stability of a unidirectionally solidified Al2O3/YAG eutectic composite J. Mater. Sci. 33 1217–25
[8] Zhao Q, Xu X, Zhang H, Chen Y, Xu J and Yu D 2004 Catalyst-free growth of single-crystalline alumina nanowire arrays Appl. Phys. A 79 1721–4
[9] Pang Y T, Meng G W, Zhang L D, Shan W J, Zhang C, Gao X Y, Zhao A W and Mao Y Q 2003 Electrochemical synthesis of ordered alumina nanowire arrays J. Solid State Electrochem. 7 544–7
[10] Yuan Z H, Huang H and Fan S S 2002 Regular alumina nanopillar arrays Adv. Mater. 14 303–6
[11] Sun X X, Liang J, Zhao J F, Ma Q and Xu B S 2010 Preparation of alumina nanowires, nanorods, and nanowalls by chemical etching Appl. Phys. A 98 263–7
[12] Chen X Y, Zhang Z J, Li X L and Lee S W 2008 Controlled hydrothermal synthesis of colloidal boehmite (AlOOH) nanorods and nanoflakes and their conversion into γ-Al2O3 nanocrystals Solid State Commun. 145 368–73
[13] He X M, Li G J, Liu H, Li J Q and Zhu Z F 2012 Thermal behavior of alumina microfibers prepared by surfactant assisted microwave hydrothermal J. Am. Ceram. Soc. 95 3638–42
[14] Li G C, Liu Y Q, Guan L L, Hu X F and Liu G G 2012 Mesoporous γ-Al2O3 fabricated by thermal decomposition of nanorods ammonium aluminium carbonate hydroxide Mater. Res. Bull. 47 1073–9
[15] Wang S H and Yang S H 2001 Growth of crystalline Cu3S nanowire arrays on copper surface: effect of copper surface structure, reagent gas composition, and reaction temperature Chem. Mater. 13 4794–9
[16] Anandan S, Wen X G and Yang S H 2005 Room temperature growth of CuO nanorod arrays on copper and their application as a cathode in dye sensitized solar cells Mater. Chem. Phys. 93 35–40
[17] Yang Z H, Wang K, Shao Z M, Tian Y, Chen G D, Wang X Z, Dou Y and Zhang W X 2017 In situ preparation of Fe3O4 hierarchical arrays on stainless steel substrate for high efficient catalysis J. Solid State Chem. 246 278–83
[18] Dlugosch T, Chinnari A, Muralidharan P, Schirmer A, Biskupek J and Stehrle S 2017 Thermal oxidation synthesis of crystalline iron oxide nanowires on low-cost steel substrates for solar water splitting Semicond. Sci. Technol. 32 084001
[19] Mei Z W, Liu Y, Wang H, Gao S J, Wen X G, Gu L, Qiu Y F and Yang S H 2010 Facile and controllable growth of ZnO 1D nanostucture arrays on Zn substrate by hydrothermal process J. Nanosci. Nanotechnol. 10 3123–30
[20] Wei Z S, Liu Y, Wang H, Mei Z W, Ye J W, Wen X G, Gu L and Xie Y T 2012 A gas-solid reaction growth of dense TiO2 nanowire arrays on Ti foils at ambient atmosphere J. Nanosci. Nanotechnol. 12 516–23
[21] Mirzaajani R, Alizadeh M, Rahimipour M R and Sarem M 2019 Seed-assisted hydrothermally synthesized AACH as the alumina precursors Mater. Chem. Phys. 221 188–96
[22] Morinaga K, Torikai T, Nakagawa K and Fujino S 2000 Fabrication of fine α-alumina powders by thermal decomposition of ammonium aluminium carbonate hydroxide (AACH) Acta Mater. 48 4735–41
[23] Sing K S W, Everett D H, Haul R A W, Moscou L, Pierotti R A, Rouquerol J and Stiemieniewska T 1985 Reporting phisorption data for gas/solid systems Pure Appl. Chem. 57 603–19
[24] Lin Y X, Cai W F, He H, Wang X B and Wang G Z 2011 Three-dimensional hierarchically structured PAN@γ-Al2O3 fiber films based on a fiber templated hydrothermal route and their recyclable strong Cr(VI)-removal performance RSC Adv. 2 17695
[25] Abdullah M, Mehmood M and Ahmad M 2012 Single step hydrothermal synthesis of 3D urchin like structures of AACH and aluminium oxide with thin nano-spikes Ceram. Int. 38 3741–5
[26] Lebedeva I I, Starostin A S, Valsiţer I V and Valsiţer V A 2018 Hydrothermal synthesis of urchin-like alumina for fire-extinguishing powders J. Mater. Sci. 53 3915–26
[27] Wang F, Zhu J F and Liu H 2018 Urea controlled hydrothermal synthesis of ammonium aluminium carbonate hydroxide rods AIP Adv. 8 035103
[28] Stoicea G and Pérez-Ramírez J 2007 Reforming Dawsonite by memory effect of AACH-derived aluminas Chem. Mater. 19 4783–90
[29] Lafficher R, Ding M, Salvatori F, Boualleg M, Colson D and Paud F 2017 Ammonium aluminium carbonate hydroxide NH4Al(OH)2CO3 as an alternative route for alumina preparation: comparison with the classical boehmite precursor Powder Technol. 320 565–73
[30] Zhu Z F, Liu H, Sun H J and Yang D 2009 PEG-directed hydrothermal synthesis of multilayered alumina microfibers with mesoporous structures Micropor. Mesopor. Mater. 123 39–44