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Controlled mesoporous carbon as support of AuCl₃ catalyst for acetylene

hydroclorination

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Abstract: Mesoporous carbon materials with controllable pore sizes within the range of 5.6–40.2 nm were successfully synthesized using colloidal silica as hard templates and boric acid as the pore expanding agent. The catalytic properties of the 0.5% AuCl₃ loaded mesoporous carbon catalysts towards acetylene hydrochlorination were tested in a fixed-bed reactor. Under reaction conditions of 180 °C, C_2H_2 hourly space velocity = 720 h⁻¹ and the feed HCl/ C_2H_2 volume ratio = 1.15, it was found that larger mesoporous carbon supports could accelerate reaction rate, resulting in higher acetylene conversion. Supported AuCl₃ catalyst on mesoporous carbon with pore size about 40.2 nm displayed excellent catalytic activity (acetylene conversion above 83%). The dependence of regular catalytic performance on pore size is important for acetylene hydrochlorination because it provides fast molecular diffusion, thus suppressing coke formation.

Keywords: mesoporous carbon, controlled pore size, acetylene hydrochlorination, coke formation

1. Introduction

Acetylene hydrochlorination is the main method for synthesizing vinyl chloride monomer (VCM) in domestic industrial production of polyvinyl chloride from calcium carbide.¹ Currently, acetylene hydrochlorination is primarily performed with a mercuric chloride catalyst supported on activated carbon (HgCl₂/AC). However, the active component of toxic HgCl₂ causes serious environmental problem and causes damage to human health. Therefore, replacing poisonous HgCl₂ with an environment-friendly catalyst for acetylene hydrochlorination has received increasing attention.²

Hutchings et al.^{3–6} found a positive relationship between the catalytic activity of metal chlorides and the standard electrode potential of cations. Accordingly, metal catalysts, such as Au,⁷ Bi,^{8,9} and Pt,¹⁰ have been intensely investigated to obtain prominent reactivity toward acetylene hydrochlorination. AuCl₃ demonstrates optimal catalytic activity, and its initial catalytic activity is higher than that of the HgCl₂ catalyst. However, the stability of AuCl₃ remains inadequate for industrial application. The deactivation mechanism of Au-based catalysts in the acetylene hydrochlorination reaction has been investigated.^{11,12} The deposition of carbonaceous materials on the catalyst dominates the deactivation process at temperatures lower than 100 °C, whereas the reduction of Au³⁺ to Au⁰ is the deactivation pathway at temperatures higher than 120 °C. Improving the stability of gold catalyst by introducing other metals to inhibit the reduction of Au³⁺ to Au³⁺ catalyst improves the stability of the Au³⁺

catalyst and prolongs its lifetime.¹³ Additionally, an Au–Co catalyst can be used for 50 h without obvious deactivation to obtain an acetylene conversion of 91.6% under a gas hourly space velocity (GHSV, C_2H_2) of 360 h⁻¹ at 150 °C.¹⁴ Moreover, TiO₂–AuCl₃/AC catalyst¹⁵ shows outstanding stability in acetylene hydrochlorination; adding TiO₂ species partially inhibits the reduction of Au³⁺ to Au⁰.

The coke formation of carbonaceous materials leads to catalyst deactivation; hence, the physicochemical properties of supports have an important role in acetylene hydrochlorination.^{16,17} SiO₂ and TiO₂ with surface areas lower than 200 m^2/g have been used as supports of Au catalyst for acetylene hydrochlorination; a negligible acetylene conversion was obtained compared with that of the carbon supports.^{18,19} However, activated carbons contain micropores (< 2 nm) only and have no interconnected porous network; moreover, activated carbons have no sufficient open-pore windows for reactant molecular diffusion and rapid reaction product transport. Therefore, mesoporous carbons with regularly interconnected mesopores (2-50 nm) may function as an excellent catalyst support for acetylene hydrochlorination; the mesoporous channels and a noticeable volume of mesopores can provide a large space for dispersion of active sites, and thus, may favor the transportation of substances.²⁰⁻²² However, the effect of different mesoporous carbons on Au-based catalysts for acetylene hydrochlorination remains unknown; determining such effects is fundamental to develop highly efficient and environment-friendly non-mercuric catalysts for industrial VCM production.

To investigate the relationship between the pore sizes of carbon supports and the

catalytic properties of AuCl₃/C catalysts, we synthesized mesoporous carbon materials with controllable pore sizes within the range of 5.6–40.2 nm. The synthesized carbon material was used to support AuCl₃ catalyst. The catalytic activity of the obtained AuCl₃/C catalysts was evaluated under the same conditions. A reasonable catalytic activity was explained through coke deposition.

2. Experimental

2.1. Materials

Sucrose (99.0%, J&K Chemical), boric acid (H₃BO₃, 99.5%, J&K Chemical), silicon (IV) oxide (4 nm, 15% in H₂O colloidal dispersion, Alfa Aesar), Ludox HS-40 (12 nm, Sigma Aldrich), silicon (IV) oxide (20 nm, 15% in H₂O colloidal dispersion, Alfa Aesar), C₂H₂ (gas, 98%), and HCl (gas, \geq 99%) were used in the present study.

2.2. Catalyst preparation

The synthesis of C-*x* carbon with tunable pore sizes was performed using the previous method reported by Ji Man Kim et al, ²³ where *x* represents the different average pore sizes of carbon. In the synthesizing process, sucrose was used as the carbon precursor, and colloidal silica with different particle sizes was used as hard template and boric acid was used as pore expanding agent to achieve the various pore sizes of carbon material. To synthesize C-5 materials, we added 13.72 g of 4 nm colloidal silica solution to sucrose (6.25 g), sulfuric acid (0.71 g), H₂O (50 mL), and boric acid solutions (5.65 g). The mixtures were dried at 100 °C for 6 h and at 160 °C for another 6 h in an oven. The brown powder was carbonized at 900 °C with N₂ flow and heating rate of 5 °C/min for 3 h. The carbonized powder was then etched with

hydrofluoric acid solution to remove silica and boron species. The C-20/30/40 materials were synthesized by the same method using different silica with larger colloid particle sizes as the template and increasing the amount of boric acid.

AuCl₃/C-*x* catalysts were prepared through an incipient wetness impregnation technique using aqua regia as solvent, as described in the literature.²⁴ A solution of HAuCl₄ · 4 H₂O (Strem, 31.98 mg) in aqua regia (3.2 mL, 0.5wt. % Au) was added drop-wise to the C-*x* materials (3.0 g), which were then stirred at room temperature for 1 h. AuCl₃/C-*x* catalysts were soaked for 24 h and dried at 140 °C for 14 h.

2.3. Catalytic reaction

Catalyst performance in acetylene hydrochlorination was evaluated using a fixed-bed microreactor (i.d. of 10 mm). Before initiating the reaction, the reactor was purged with nitrogen to remove water and air in the system. HCl gas was passed through the pipeline at a flow rate of 20 mL/min to activate the catalyst (0.8 g). Subsequently, the microreactor was heated at 180 °C. Afterward, acetylene and hydrogen chloride were fed through the microreactor, thus producing a GHSV (C_2H_2) of 720 h⁻¹. The exit gas mixture was passed through an absorption bottle containing clean water and then injected into a Shimadzu GC-2014C chromatograph for analysis. 2.4. Characterization

Physical characterization of the sample was performed using a transmission electron microscope (TEM, JEOL, JEM 2010 operating at 200 kV) forits morphological features. Brunauer–Emmett–Teller (BET) surface area data were collected using a Micromeritics ASAP 2020 instrument by obtaining nitrogen

adsorption isotherms at 77 K. The micropore surface area was estimated using the t-plot method. The pore size distribution was analyzed with the adsorption branch using the Barrett-Joyner-Halenda (BJH) algorithm. The total pore volume was calculated from the amount adsorbed at a relative pressure of 0.99. Thermo gravimetric analysis (TGA) was conducted with TGA/DTA system (SDT Q600, America) at room temperature to 900 °C with air flow of 10 mL/min.

3. Results and discussion

N₂ adsorption-desorption isotherms and the corresponding pore size distributions determined from the adsorption branches of C-x materials are shown in Fig. 1. All porous carbon samples exhibited type IV nitrogen adsorption isotherm with a broad intermediate hysteresis loop between H_1 and H_2 ; this isotherm is typically attributed to the adsorption in mesoporous pores. A capillary condensation step occurred at a relative pressure that ranged from 0.62 to 0.99, which is consistent with the results of previous studies.^{25,26} With the increase in the diameter of colloidal silica particles and the boron content in the carbon precursor, the step gradually shifted to higher relative pressures. This result implies that pore size increased, which is also confirmed by the narrow pore size distribution curves shown in Fig. 1b. The structural properties of C-xmaterials are summarized in Table 1. The representative structures of mesoporous carbon with 5.8, 19.6, 29.2, and 40.5 nm pore diameter were also demonstrated. The results indicated that the pore size of carbon support could be successfully controlled using boric acid as a pore-expanding agent and colloidal silica as templates. The TEM images shown in Fig. 2 represent the pore morphologies of C-x after AuCl₃ catalyst

loading. Structural integrity, namely, roughly spherical and closely packed structure,							
was conserved after catalyst loading, further indicating that pore size increased.							
However, distinguishing the homogeneous dispersions of small, spherical, and							
uniform dark spots, which correspond to Au nanoparticles, in TEM images is difficult							
because of the active components in the state of Au^{3+} in the supported catalysts.							

Table 1 Physical properties of C-x materials									
Samples	$Dp (nm)^{(a)}$	$S_{\rm BET} ({\rm m^2/g})^{(b)}$	$V_{\rm tot} (\rm cm^3/g)^{(c)}$	$V_{\rm micro} ({\rm cm}^3/{\rm g})^{\rm (d)}$	$V_{\rm meso}$ ×100 (%)				
C-5	5.6	949.3	1.08	0.32	70.37				
C-20	19.6	866.5	1.08	0.26	75.93				
C-30	29.2	763.1	1.21	0.21	82.64				
C-40	40.5	658.8	1.30	0.15	88.46				

(a) Mesopore diameters calculated from the N₂ adsorption branches using BJH method. (b) Surface areas calculated using BET method. (c) Total pore volumes estimated from the N₂ sorption isotherms at $p/p_0 = 0.99$. (d) Micropore volume with diameters less than 2 nm calculated at $p/p_0 = 0.16$ using the Horvath–Kawazoe formula.



Fig. 1 (a) The corresponding pore size distribution curves of the obtained C-*x* materials, and (b) N₂ adsorption–desorption isotherms. Pore size distribution curves were calculated from adsorption branches using BJH method.



Fig. 2 TEM and HRTEM images of fresh catalyst: (a) AuCl₃/C-5, (b) AuCl₃/C-20, (c) AuCl₃/C-30, and (d) AuCl₃/C-40.

To investigate the effect of pore size on the catalytic performance of AuCl₃ catalyst for acetylene hydrochlorination, we prepared four mesoporous carbons with different pore sizes (5.9, 19.6, 29.2, and 40.5 nm) to support 0.5% AuCl₃. Catalytic activity was evaluated under the same conditions, and the results are presented in Fig. 3. In general, acetylene conversion systematically increases with increasing pore sizes. Initial acetylene conversion increased from 10.2% to 36.6% as the pore sizes of the carriers increased from 5.9 nm to 40.5 nm. Furthermore, the catalysts exhibited a sequential increase in their catalytic performances during acetylene hydrochlorination. For the AuCl₃/C-5 catalyst, the initial acetylene conversion is relatively low and after 8 h reaction it becomes 46.7%. For AuCl₃/C-20 and AuCl₃/C-30 catalysts, the final acetylene conversion increases to 59.0% and 69.8%, respectively. AuCl₃/C-40 catalyst shows the best catalytic performance for acetylene hydroclorination in our experiments (maximum acetylene conversion of 84.7%). Fig. 4 shows the effect of the carriers' pore sizes on acetylene conversion. The final acetylene conversion of AuCl₃/C-*x* catalysts significantly increased from 46.7% to 84.7% as pore sizes increased. Thus, using larger mesoporous carbon to support 0.5% AuCl₃ would be favorable in catalysis because it could provide high pore accessibility and facilitate molecular diffusion. With time-on-stream, pore expansion can accelerate the initial activity; the consequential fast reaction rate can result in high acetylene conversion.



Fig. 3 Acetylene conversion of 0.5% AuCl₃/C-*x* catalysts in acetylene hydrochlorination. Reaction conditions: temperature (T) = 180 °C, C_2H_2 gas hourly space velocity (GHSV) = 720 h⁻¹, feed volume ratio V_{HCl}/V_{C2H2} = 1.15.



Fig. 4 Effect of the pore sizes of 0.5% AuCl₃/C-x catalysts on acetylene conversion.

These findings correspond to the catalytic behavior of AuCl₃/C-*x* catalysts and confirm that pore entrance size is relatively significant. However, different pore sizes have fundamentally different catalytic properties. As HCl and C_2H_2 molecules flow into the catalyst pore through diffusion, the collision of the pore wall and other molecules in the active sites produces a reaction. In particular, HCl and C_2H_2 are catalyzed by AuCl₃ to produce C_2H_3Cl . The deep transport of the reactant gas into the AuCl₃ active sites and the inefficient transport of organic products, namely, C_2H_3Cl , from the reaction zones within a very short distance could accelerate coke formation.





Fig. 5 TGA curves of different 0.5% AuCl₃/C-*x* catalysts before and after acetylene

hydrochlorination.



Fig. 6 The coke contents of different 0.5% AuCl₃/C-x catalysts after acetylene hydrochlorination.

Fig. 5 shows TGA analysis of different 0.5% AuCl₃/C-x catalysts before and after acetylene hydrochlorination, being used to evaluate the degree of coke deposition on the surface of catalysts. Both fresh and spent AuCl₃/C-x catalysts had a visible mass loss before 100 °C, indicating that there is miner water adsorption on the surface of catalyst. Whereas there have very little weight loss in the range of 100–250 °C, when the temperature continues to rise from 250 to 458 °C, the AuCl₃/C-x catalysts exhibit a slow weight loss, mainly due to the burning of carbon deposition and carbon carrier

begin to be oxidized. When the temperature exceeds 458 °C, the AuCl₃/C-x catalysts rapidly decrease in weight indicating that carbon is burned off. Fig. 6 presents the coke deposition content on porous catalysts; the content was based on previous reported calculation method.¹⁴ Therefore, the amount of coke deposition can be calculated by the difference of the mess loss between the fresh and spent AuCl₃/C-*x* catalysts in the temperature range of coke burning (250-458 °C). It is obvious that the order of carbon deposition is AuCl₃/C-5 (4.8%) > AuCl₃/C-20 (3.3%) > AuCl₃/C-30 (1.7%) > AuCl₃/C-40 (0.5%). Moreover, the interconnected pore system in larger mesoporous carbon can favor efficient acetylene conversion better compared with that in the porous carbons with smaller pore size. Hence, larger and open mesoporous networks can facilitate the rapid diffusion of organic products from the reaction zones, resulting in suppressed coke formation and improved catalytic activity.

Samples	$D_{\rm p}({\rm nm})$	$S_{\rm BET}({\rm m^2/g})$	$V_{\rm tot}({\rm cm}^3/{\rm g})$	$V_{\rm micro} ({\rm cm}^3/{\rm g})$	V _{meso} ×100 (%)
AuCl ₃ /C-5 (fresh)	5.8	949.3	1.10	0.34	69.09
AuCl ₃ /C-20 (fresh)	19.6	763.1	1.09	0.25	77.06
AuCl ₃ /C-40 (fresh)	40.5	658.8	1.30	0.19	85.38
AuCl ₃ /C-5 (spent)	6.1	727.4	0.97	0.17	82.47
AuCl ₃ /C-20 (spent)	21.8	670.0	1.04	0.12	88.46
AuCl ₃ /C-40 (spent)	44.2	546.9	1.21	0.09	93.56

Table 2 Textural properties of catalysts



Fig. 7 (a) The corresponding pore size distribution curves of fresh and spent AuCl₃/C-x catalysts,

and (b) N₂ adsorption-desorption isotherms.

Porosity should be considered to verify coke formation. The BET results listed in Table 2 provide an indirect evidence for the coke formation on AuCl₃/C-x catalysts. The specific surface area and total pore volume of $AuCl_3/C-x$ catalysts were moderately reduced after acetylene hydrochlorination. Interestingly, the mesoporosity of the spent AuCl₃/C-x catalysts significantly increased compared with that of the fresh catalysts. For AuCl₃/C-5, AuCl₃/C-20 and AuCl₃/C-40 catalysts, the increasing degree of mesoporosity are 13.38%, 11.40% and 8.18% after 8 h reaction, respectively. It is indicated that there is significant difference among the AuCl₃/C-x catalysts for variation of mesoprosity. The increasing of mesoporosity further proved that micropores, which are the key components, were filled or blocked, and the coke formation increased with decreasing pore sizes of mesoporous carbon support. Therefore, the mesopore in AuCl₃/C-x catalysts is an important fraction that functions as an efficient catalyst support to produce better molecule diffusion. Micropores can be identified as voids where some pore channels may become inaccessible, thereby

expanding coke formation. These findings are in good agreement with the N_2 sorption curves and the pore size distribution curves shown in Fig. 7. All N_2 adsorption isotherms are maintained as type IV isotherms with hysteresis loops for mesoporous adsorption; moreover, well-defined steps appear in the adsorption–desorption curves without significant shift in relative pressure. Remarkably, Fig. 7b shows that the BJH pore size distribution curves of different AuCl₃/C-*x* catalysts narrow down and sharpen after acetylene hydrochlorination. Thus, the systematic increase in mesoporosity is mainly due to the fraction of the micropores blocked in mesostructural supports; hence, the total volume for the coke formation in the carbon framework decreases, resulting in more dense packing mesopores. This finding is consistent with the catalytic behavior aforementioned, which exhibited a relatively constant acetylene conversion after a period of activation that originated from the active component. AuCl₃ in the mesoporous networks exhibited maximum catalytic performance even when the micropores were plugged.

4. Conclusions

In summary, C-*x* mesoporous carbon materials with systematic controllable pore sizes within the range of 5.6–40.2 nm were successfully prepared using boric acid as pore-expanding agent and different colloidal silica as templates. Loading 0.5% AuCl₃ in C-*x* mesoporous carbon materials were assessed for acetylene hydrochlorination. Moreover, the effect of pore sizes on catalytic activity was investigated. Pore size variation significantly affects the initial activity. Larger mesoporous carbon supports could accelerate the reaction rate, resulting in higher acetylene conversion. In addition,

the analysis on coke deposition in catalyst supports revealed that catalytic reaction mainly relies on mesopores even if the micropores are blocked to some extent. The dependence of regular catalytic performance on pore sizes will be beneficial for acetylene hydrochlorination, which requires fast molecular diffusion to suppress coke formation.

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