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Electrooxidation of Formaldehyde on Silver/Ordered Mesoporous Carbon Composite Electrode in Alkaline Solutions

Ling-Bin Kong, Ru-Tao Wang, Xiao-Wei Wang, Zhen-Sheng Yang, Yong-Chun Luo, and Long Kang

Abstract—**Metal nanocatalysts, as the anodic materials, have become increasingly important in fuel cells, due to their unique physical and chemical properties. Here, the porous silica was employed as a hard template to prepare the ordered mesoporous carbon (CMK-3). The as-prepared CMK-3 has a large surface area of 877 m^2/g and the hierarchical porous structure with the pore size distribution centered at 3.7 nm. The CMK-3 supported silver nanocatalysts have been prepared through the wet chemical reduction by using the reduction of formaldehyde. The electrochemical properties of the Ag/CMK-3 nanocatalysts for formaldehyde oxidation are studied by cyclic voltammerry (CV) and chronoamperometric curves (i-t) in alkaline aqueous solutions. The results show that the peak current density (CV) of the Ag/CMK-3 electrode is 112 mA/cm^2, above 2 times higher than that of Ag/XC-72 at the same Ag loading (14.15 μg/cm^2). Furthermore, i-t curves demonstrate that the Ag/CMK-3 nanocatalysts are efficient and stable electrocatalysts for anodic oxidation of formaldehyde in alkaline solutions. Therefore, Ag/CMK-3 nanocatalysts with the improved electrocatalytic activity suggest their application potential towards fuel cells and sensors.**

Index Terms—**Nan catalysts; Ag nanoparticles; ordered mesoporous carbon; formaldehyde electro oxidation.**

I. INTRODUCTION

It's controversial that the formaldehyde is a promising fuel in fuel cells because of the possible relationship between formaldehyde exposure and cancer [1]. Formaldehyde is also one of the initial products of methanol oxidation which has its fundamental and technological importance in industrial catalytic processes [2]. The electrooxidation mechanism of formaldehyde has been widely investigated on Pt [3], Pd [4], and Rh [5] electrodes which are mostly in connection with fuel cells and sensors. Whereas, these anodic materials are the noble metal with their scarcity, and can't lower the commercial cost. New metal nanocatalysts with high electrochemical catalytic properties and low commercial cost can be explored by rational designing of their compositions and structures. As been well known, silver is more abundant in nature and less expensive than platinum and palladium, as a result, cost associated issues is one of the main driving force behind the increasing interest in silver as the anodic materials in fuel cells. Moreover, the real and most important attraction for Ag-based electro catalysts is originated by the fact that they can be highly active in the electro oxidation of formaldehyde in alkaline environment, where other non-noble metals are not sufficiently active and stable for electrochemical applications. Recently, Gang and Lu [6] reported that the Ag nimrods had the satisfying electro oxidation property for formaldehyde in alkaline solution.

In general, the catalytic properties of metal are dependent upon the size, composition, structure, as well as the support materials [7]-[9]. Especially, the support materials [10]-[12], such as MCM-n, SBA-n, CMK-n, can disperse the metal clusters homogeneously and stably owing to their desirable hosting structure. The ordered Mesoporous carbon (CMK-3) is the promising material for its numerous applications because of its large specific surface areas, high chemical inertness, good conductivity, and mechanical stability [13]. It is an ideal host for synthesis of nanocatalysts. Ryoo and co-worker investigated oxygen reduction using Pt-loaded mesoporous carbonized SBA-15, and observed a significant increase in reduction current, compared to Pt loaded in conventional carbon (such as carbon black, charcoal and activated carbon fibers), using a rotating electrode and Nations as the binding incomer [14]. Therefore, the higher electro oxidation properties for formaldehyde of nana-sized Ag, combined with the desirable hosting structure of CMK-3 mentioned above, strongly suggest that the Ag/CMK-3 nanocatalysts have the higher activity for formaldehyde electro oxidation as the anodic materials.

In this paper, chemically synthesized Ag/CMK-3 nanocatalysts are prepared by reducing silver nitrate with the formaldehyde in aqueous CMK-3 solution. The structure and nature of the resulting Ag/CMK-3 nanocatalysts are characterized by transmission microscopy (TEM) and X-ray diffraction (XRD). The catalytic properties of the Ag/CMK-3 nanocatalysts for formaldehyde electro oxidation have been investigated in alkaline environment. The results demonstrate that the as-prepared Ag/CMK-3 nanocatalysts exhibit excellent electro catalytic activity and long-term stability for formaldehyde electro oxidation, implying its potential to substitute for noble metal catalysts in fuel cells and sensors.

II. EXPERIMENTAL

A. *Synthesis of CMK-3*

All reagents were of analytical grade. The CMK-3 was synthesized, following the method described by Ryoo and

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co-workers [15]. 1 g of SBA-15 was added to a solution obtained by dissolving 1 g of sucrose and 0.147 g of H_2SO4 in 5 g of H_2O. The SBA-15 was synthesized, following the method reported by Zhao et al [16]. The mixture was placed in a drying oven for 6 h at 373 K, and subsequently the oven temperature was increased to 433 K and maintained for 6 h. The silica sample, containing partially polymerized and carbonized sucrose at the present step, was treated again at 373 and 433 K using the same drying oven after the addition of 0.8 g of sucrose, 0.09 g of H_2SO_4 and 5 g of H_2O. The carbonization was completed by pyrolysis with heating to typically 1173 K under vacuum. The carbon-silica composite obtained after paralysis was washed with 5 wt% hydrofluoric acid at room temperature, to remove the silica template. The template-free carbon product thus obtained was filtered, washed with ethanol, and dried at 373 K.

B. Synthesis of Ag/CMK-3 nanocomposite

The CMK-3 supported Ag catalysts were synthesized via a wet chemical reduction method [17]. Briefly, 50 mg of pristine CMK-3 and 19.7 mg $AgNO_3$ were added into 10 ml distilled water, and then were ultra-sonicated about 30 min to disperse them uniformly. Then 10 ml formaldehyde as the reduction agent solved in 10 ml water was slowly drop wised into the suspension solution. The pH value of the mixture was adjusted to 11 by adding the aqueous 1 M NaOH solution under vigorous stirring. Finally, the mixture was refluxed at 85 °C for 2 h in a heating mantle, after it was agitated vigorous under room temperature for 4 h. The black solid slurries were filtrated, washed and dried at 90 °C for more than 10 h in a vacuum oven, and then, the Ag/CMK-3 nanocatalysts were obtained. For comparison, Ag nanocatalyst supported on Vulcan XC-72R carbon black (Cabot, USA, BET: 213 cm^2/g), denoted as Ag/XC-72, was also prepared under the same preparation conditions.

C. Preparation of electrodes and electrochemical measurements

Electrochemical measurements were performed using an electrochemical working station (CHI660C, Shanghai Chinua, and China) and a standard three-electrode cell at room temperature (about 22 °C). A commercial saturated calomel electrode (SCE) and a platinum foil were used as reference electrode and counter electrode respectively. The glassy carbon electrode (GC, 3 mm in diameter, geometric area: 0.07 cm^2) was polished mechanically to be a mirror by successively using the alumina powder of size 30 nm, followed by cleaning in an ultrasonic bath and dring in an oven at 313 K. 1 mg of catalysts and then were dispersed ultrasonically in 1mL of an ethanol solution of 5 wt% Nafion to obtain a homogeneous black suspension. Then, 5 μl of the suspensions were dropped onto the GC electrode surface using a micropipette. The coating was then dried at room temperature in air. To measure the catalytic activity of Ag/CMK-3 nanocatalysts towards electro oxidation of formaldehyde oxidation, cyclic voltammeters (CV) and current-time (i-t) curves methods were employed and performed at a sweep rate of 50 and 100 mV/s.

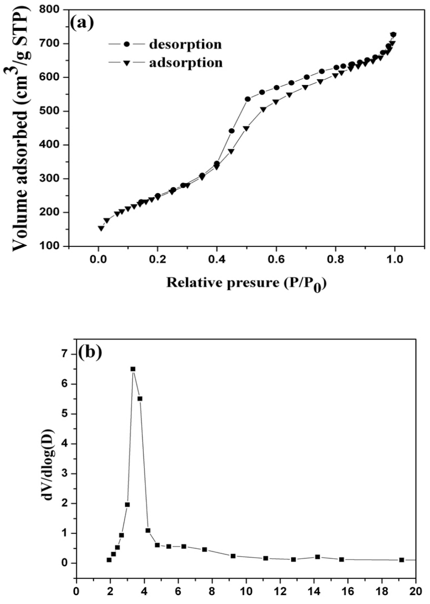
D. Sample characterization

The samples were characterized by using a Rigaku D/Max-2400 X-ray diffract meter (XRD) with a Cu Kα radiation resource to determine the crystalline structure. Transmission electron microscope (TEM) images were obtained with a JEOL JEM-2010 microscope (Japan) operated at 200 KV and equipped with an energy-dispersive spectrometer (EDS). Nitrogen adsorption-desorption data was obtained by using an AASP 2010M (Micromeritics, US). The surface area of CMK-3 was calculated using the Brinier Emmett Teller (BET) equation. Pore size distributions of CMK-3 were calculated by the Barrett-Joyner-Helena (BJH) method using the desorption branch of the isotherm.

III. RESULTS AND DISCUSSION

Fig.1 shows the N_2 adsorption-desorption isotherm of the synthesized CMK-3 materials. In terms of shape, the isotherm for CMK-3 can be classified as type Ⅳ according to the IUPAC. A distinct hysteresis loop can be observed in the range of ca. 0.4-1.0 P/P_0, indicating the presence of mesopores. The as-prepared CMK-3 have a large surface area of 877 m^2/g and the hierarchical porous structure with the pore size distribution centered at 3.7 nm, as measured by the Brunauer-Emmett-Teller (BET) measurements. The large surface area of CMK-3 indicates that the CMK-3 is an ideal host for synthesis of nanocatalysts.

Fig. 2 shows the XRD patterns of the two samples of Ag/CMK-3 and Ag/XC-72 nanocatalysts. The diffraction



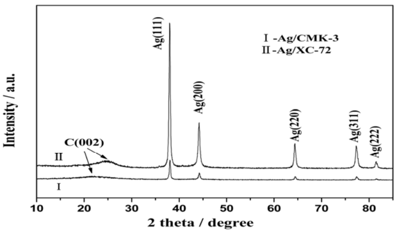


Fig. 2. XRD pattern of the Ag/CMK-3 and Ag/XC-72 samples nanocatalysts.

Peaks of Ag nanoparticles corresponding to different planes can be observed from the pattern obviously. The peaks can be assigned to the Ag face-centered cubic (fcc) phase, namely the planes (111), (200), (220), (311), (222), indicating the successful fabrication of Ag nanopaticles onto the CMK-3 and XC-72. No diffraction peaks could be attributed to silver oxides in the XRD. The X-ray diffraction peak at 22° and 24.1° can be attributed to the hexagonal graphite structure (002) [18].

The typical morphology of Ag/CMK-3 and Ag/XC-72 nanocatalysts was characterized by transmission electron microscopy (TEM). The ordered 2-D mesoporous texture of the Ag/CMK-3 can be seen in Fig. 3(a) and (b). In our case Ag nanoparticles with a broad size distribution ranging from 80 to 200 nm are homogeneous dispersed on the surface of CMK-3, and some large aggregates of metal Ag in shape could also be found on the surface of CMK-3, due to the Ag nanoparticles with the characteristic of easy aggregation are not generally more stable than Au, Pt nanoparticles [19]. The chemical nature of these Ag/CMK-3 nanocomposites can be detected by ED's measurements (Fig. 3(c)). A typical EDS spectrum for Ag nanoparticle modified CMK-3 supported by peaks, arising from the Ag nanoparticles, CMK-3 and copper foil substrate. In addition, ED pattern for the Ag/CMK-3 nanocatalysts show several bright concentric rings, polycrystalline metal Ag. The morphology of Ag/XC-72 was shown in fig. 3(d).

A better and direct understanding of the catalytic properties of the resulting Ag catalysts are sought by carrying out electrochemical measurements in 1 M KOH aqueous solution in the absence and presence of 1 M formaldehyde. The typical behavior for Ag electrode in alkaline medium and a broader discussion of the oxidation-reduction results were discussed elsewhere [20, 21]. In the anodic potential (fig. 4(a)), current peaks are related to the electro dissolution of Ag to $[Ag(OH)_2]^-$ through adsorption of OH^- and the electroformation of multilayer of Ag_2O and AgO. On the other hand, the more negative cathodic peaks could be attributed to the processes involved in the electro-reduction of Ag_2O and AgO to Ag. From the CV measured in 1 M KOH, it also shows the electrochemical active surfaces of Ag/CMK-3 nanocomposites [6], and Ag/CMK-3 samples exhibit higher oxidation-reduction peak than the Ag/XC-72 samples, indicating that the Ag/CMK-3 nanocatalysts have the higher electrode active surface.

The electrooxidation of formaldehyde on metal, such as Pt, Cu, Rh et al., has been widely reported [1]-[5]. In aqueous solutiongs, formaldehyde exists mainly in its hydrated form, methylene glycol ($H_2C(OH)_2$), and the following electrochemical oxidation maybe occur on Ag surface:

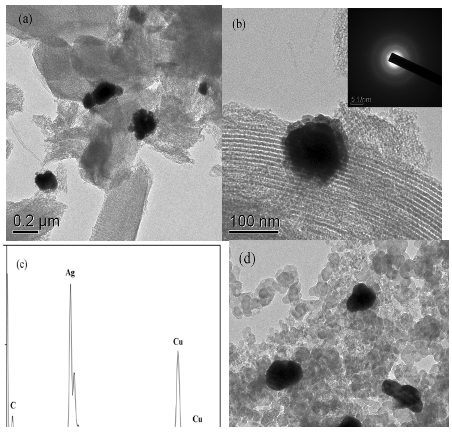


Fig. 3. (a), (b) TEM images of Ag/CMK-3 nanocatalysts; Top-right inset shows the corresponding ED pattern. (c) EDS spectrum of Ag/CMK-3. (d) TEM image of Ag/XC-72.

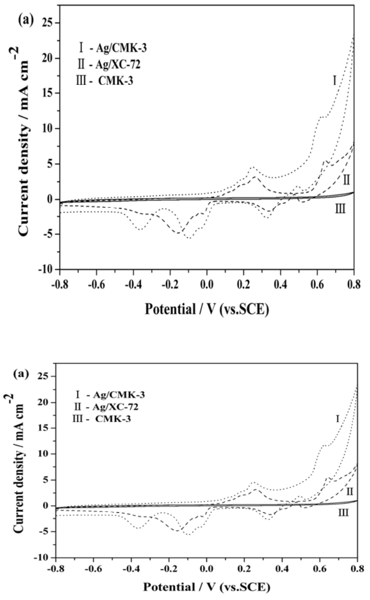


Fig. 4. CV at glassy carbon electrode modified by Ag nanocatalysts (a) in 1 M KOH, (b) in 1 M KOH + 1 M formaldehyde. Scan rate: 100 mV/s.

$$HCHO + H_2O \rightarrow H_2C(OH)_2 \quad (1)$$

$$H_2C(OH)_2 \rightarrow CO_{ads} + H_2O + 2H^+ + 2e^- \quad (2)$$

$$H_2C(OH)_2 \rightarrow H2COO_{ads} + 2H^+ + 2e^- \quad (3)$$

$$CO_{ads} + H_2O \rightarrow CO_2 + 2H^+ + 2e^- \quad (4)$$

$$H_2COO_{ads} \rightarrow CO_2 + 2H^+ + 2e^- \quad (5)$$

Here, CO_{ads} and H_2COO_{ads} mean the adsorbed species on metal electrode surface. The reaction procedure presented above can show the formaldehyde oxidation behavior at Ag electrode is the four-electron oxidation process and yields CO_2 as the final produce on the basis of parallel pathway involving two adsorbed intermediates (CO_{ads} and H_2COO_{ads}). Fig. 4(b) shows the typical cyclic voltammograms of Ag in 1 M formaldehyde + 1 M KOH solution. It can be observed that

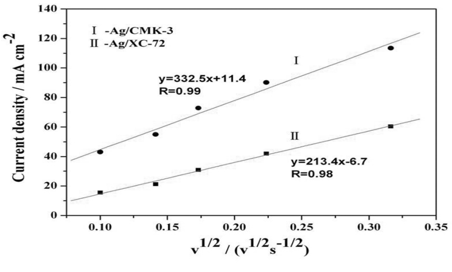


Fig. 5. Dependence of peak current densities on the square root of scan rates of Ag/CMK-3 and Ag/XC-72 nanocatalysts measured in 1 M KOH + 1 M formaldehyde solution.

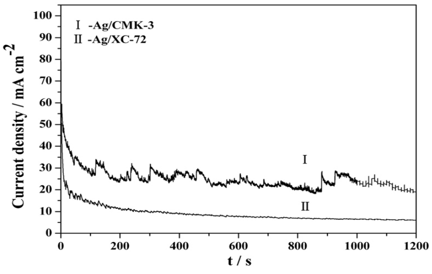


Fig. 6. Current-time curves of formaldehyde electro-oxidation on Ag/CMK-3 and Ag/XC-72 nanocatalysts in 1 M KOH + 1 M formaldehyde solution

the CMK-3 modified GC electrode appear to be inactive for anodic oxidation of formaldehyde. In the typical curve of Ag catalysts, the main oxidation peak potentials are located at 0.2 V and 0.4 V (vs. SCE), corresponding peak current density is 60.9 and 112 mA/cm^2, respectively. It clearly shows the electrocatalytic activity of Ag/CMK-3 nanocatalysts is higher than that of Ag/XC-72 nanocatalysts and also demonstrates the Ag/CMK-3 nanocatalysts have the higher electrochemical active surfaces. As stated above, it should be mentioned that the large Ag nanoparticles, even exceeding 100 nm, also show the high electrocatalytic activity for formaldehyde electrooxidation. The enhanced electrocatalytical properties can be attributed to the CMK-3 supports with the large surface areas and the highly ordered mesoporous texture which are in favor of dispersing the Ag nanoparticles homogeneous and providing Ag electrode with high electrocatalytic activity. Moreover, the loadings of Ag on the XC-72 or CMK-3 coated GC electrode are about 1 μg, and the corresponding maximum current densities of Ag were calculated about 4.3 A/mg and 7.91 A/mg, respectively. To best of our knowledge, these results are obviously higher than the reports of Ag nanorods for formaldehyde electrooxidation [6].

Compared with the relation between the peaks current obtained from CVs scan and the square root of scan rates, which is shown in Fig. 5, a linear relationship is observed, suggesting that the electrocatalytic oxidation of formaldehyde on Ag/CMK-3 and and Ag/XC-72 nanocatalysts is the diffusion-controlled process [22].

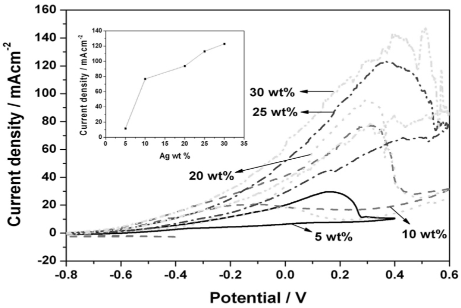


Fig. 7. CV at glassy carbon electrode modified by Ag/CMK-3 composites with different Ag loading in 1 M KOH + 1 M formaldehyde. Scan rate: 50 mV/s. Inset shows dependence of peak current densities on Ag/CMK-3 composites with different Ag loading

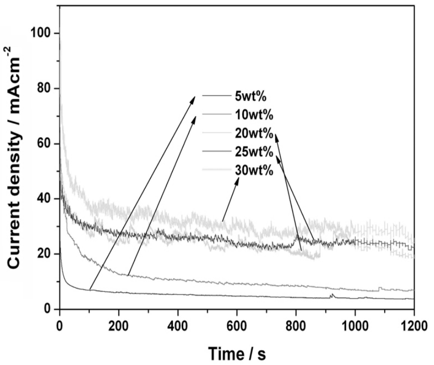


Fig. 8. Current-time curves of formaldehyde electro-oxidation on Ag/CMK-3 composites with different Ag loading in 1 M KOH + 1 M formaldehyde solution.

It can be further discussed the electrocatalytic stability and activity of the catalysts under continuous operating conditions, then long-term chronoamperometric (current-time) experiments are investigated in 1 M formaldehyde and 1 M KOH aqueous solution. Fig. 6 shows the current-time (i-t) curves of formaldehyde oxidation which are applied at 0.2 and 0.4 V (vs. SCE), corresponding to the anodic current peak (CV) for Ag/XC-72 and Ag/CMK-3 electrodes, respectively. It can be observed that the limiting current densities on Ag/CMK-3 and Ag/XC-72 after 1200 s are 18.8 and 6 mA/cm^2, respectively, indicating a significantly enhanced catalytic activity and stability to formaldehyde electrooxidation of Ag/CMK-3 nanocatalysts. This result is consistent with that of CV. As the weight percentage of Ag in Ag/CMK-3 composite increases, the electrocatalytic activity for formaldehyde oxidation of Ag/CMK-3 electrode also increases, this can be observed in cyclic voltammograms test (Fig. 7) and long-term chronoamperometric (current-time) experiments (Fig. 8). On the basis of these results, as mentioned above, it strongly suggested that the Ag/CMK-3 nanocatalysts have the higher catalytic activity for the electrooxidation of formaldehyde.

IV. CONCLUSION

In summary, Ag/CMK-3 nanocatalysts have been successfully synthesized through the facile wet chemical reduction. The electrocatalytic activity of Ag/CMK-3 nanocatalysts has been tested for formaldehyde electrooxidation in alkaline media. The results of CV and i-t curves demonstrate that Ag/CMK-3 nanocatalysts have the significantly enhanced activity and high stability towards the electrooxidation of formaldehyde. This finding represents a significant step toward the implementation of individual Ag/CMK-3 nanocatalysts as anodic materials in fuel cells and sensors.

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