Research Article

Enhanced Supercapacitance of Hydrous Ruthenium Oxide/Mesocarbon Microbeads Composites toward Electrochemical Capacitors

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A facile hydrothermal strategy was proposed to synthesize RuO$_2$$ \cdot $nH$_2$O/mesocarbon microbeads (MCMBs) composites. Further physical characterizations revealed that RuO$_2$$ \cdot $nH$_2$O nanoparticles (NPs) were well dispersed upon the surfaces of the MCMB pretreated in 6 M KOH solution. Electrochemical data indicated that the RuO$_2$$ \cdot $nH$_2$O/MCMB composites owned higher electrochemical utilization of RuO$_2$ species, better power property, and better electrochemical stability, compared with the single RuO$_2$ phase. The good dispersion of RuO$_2$$ \cdot $nH$_2$O NPs and enhanced electronic conductivity made the H$^+$ ions and electrons easily contact the RuO$_2$$ \cdot $nH$_2$O phase for efficient energy storage at high rates.

1. Introduction

Electrochemical capacitors (ECs) are a kind of charge-storage devices possessing higher power density, more excellent reversibility, longer life cycle than batteries and much higher energy density compared to conventional capacitors [1]. Owing to these extraordinary properties, ECs have attracted increased interests during the past years with projected applications in the hybrid vehicle systems and memory backup systems. The electrochemical performance of ECs depends upon the electroactive materials greatly. Among these electroactive materials, hydrous ruthenium oxide (RuO$_2$$ \cdot $nH$_2$O) has been recognized as the state-of-the-art electrode material, due to its facile transport pathways for both protons and electrons, high specific capacitance (SC), highly reversible redox reactions, and so forth, [2–8]. However, a very high cost and toxic nature greatly preclude its commercial application. For this reason, the approaches to reduce its amount and further enhance its electrochemical utilization are essential to make the RuO$_2$-based devices more cost-effective.

It is well established that the good dispersion of RuO$_2$$ \cdot $nH$_2$O nanoparticles (NPs) upon the surface of the carbon matrices is much favorable for enhancing their electrochemical utilization [4–8]. And the RuO$_2$$ \cdot $nH$_2$O/carbon-based composites delivered a wide SC range from 150 to 1580 F g$^{-1}$ [5, 8], which was greatly depending upon the loading and thickness of the RuO$_2$$ \cdot $nH$_2$O existing in the carbon materials. Commonly, the used carbon materials include carbon nanotubes [4], activated carbon [5], carbon black [6], and carbon nanofibres [7]. Since mesocarbon microbeads (MCMB) were first separated by Honda and Yamada from the mesophase pitches [9], this typical carbon material has been used in many applications, such as high-density carbon material [10, 11], filler for high-performance liquid chromatography [12], active carbon with super high surface [13], anodes of lithium ion battery [14–18], alkaline zinc-air cells [19], and supports of NiO NPs for ECs [20].

To the best of our knowledge, the investigation of the MCMB as a support to disperse RuO$_2$$ \cdot $nH$_2$O NPs for ECs has not been reported as yet. In this work, the RuO$_2$$ \cdot $nH$_2$O NPs were well dispersed upon the surfaces of the MCMB via mild hydrothermal method. Such unique RuO$_2$$ \cdot $nH$_2$O/MCMB composites can not only enhance the dispersion of RuO$_2$$ \cdot $nH$_2$O NPs but remit their serious aggregation...
electrochemical workstation. Electrochemical impedance spectroscopy (EIS) was performed with a frequency response analyzer (Solatron 1255B) interfaced with potential galvano-stat (Solatron 1287) controlled by a personal computer. Chronopotentiometry (CP) curves of the electrodes were evaluated with an Arbin BT2042 battery workstation system in the certain potential ranges.

3. Results and Discussion

3.1. Characterization of the RuO\textsubscript{2} · nH\textsubscript{2}O/MCMB Composites. XRD patterns of the as-synthesized RuO\textsubscript{2} · nH\textsubscript{2}O/MCMB composites with different loadings are presented in Figure 1 as indicated. As shown in Figure 1(a), the peak intensity of the MCMB at 2\(\theta\) = 26.5\(^\circ\) dramatically diminishes after loading RuO\textsubscript{2} · nH\textsubscript{2}O NPs. Moreover, with the increase of RuO\textsubscript{2} · nH\textsubscript{2}O NPs loadings, the peak intensity of the MCMB at 2\(\theta\) = 26.5\(^\circ\) decreases more and more. Notably, the peak intensities of the MCMB from 40\(^\circ\) to 60\(^\circ\) also decrease greatly after loading RuO\textsubscript{2} · nH\textsubscript{2}O NPs and keep the same decreasing trend as the peak at 2\(\theta\) = 26.5\(^\circ\), as depicted in Figure 1(b). All these data support that the MCMB has been successfully coated with RuO\textsubscript{2} · nH\textsubscript{2}O NPs after hydrothermal treatment. However, the obvious diffraction peaks of the RuO\textsubscript{2} · nH\textsubscript{2}O phase cannot be found in Figures 1(a) and 1(b). Thus, the typical XRD pattern of RuO\textsubscript{2} · nH\textsubscript{2}O/MCMB composite with 100 wt.% RuO\textsubscript{2} · nH\textsubscript{2}O NPs, that is, the pure RuO\textsubscript{2} · nH\textsubscript{2}O phase, is further shown in Figure 1(b). In sharp contrast, the broad diffraction peaks with very low intensity are presented for the RuO\textsubscript{2} · nH\textsubscript{2}O phase. And these typical diffraction peaks should result from the relative poor crystalline quality and/or nanometer-scale size of the as-prepared hydrous RuO\textsubscript{2} NPs. To more clearly identify the existence of RuO\textsubscript{2} phase in the composites, the enlarged XRD patterns of the pure RuO\textsubscript{2} · nH\textsubscript{2}O and RuO\textsubscript{2} · nH\textsubscript{2}O/MCMB composite with 12.5 wt.% RuO\textsubscript{2} · nH\textsubscript{2}O NPs were shown in Figure 1(c), respectively. Evidently, four obvious broad diffraction peaks contributed by the rutile RuO\textsubscript{2} phase (ICPDS card no. 43-1027) can be found both in the two samples, which indicates the real existence RuO\textsubscript{2} · nH\textsubscript{2}O in the RuO\textsubscript{2} · nH\textsubscript{2}O/MCMB composites.

The FESEM images of the RuO\textsubscript{2} · nH\textsubscript{2}O/MCMB composite with the 24.8 wt.% RuO\textsubscript{2} · nH\textsubscript{2}O NPs are shown in Figure 2. Evidently, the RuO\textsubscript{2} · nH\textsubscript{2}O/MCMB composite exhibits uniform spherical grains with the size of 1~2 \(\mu\)m, as shown in Figure 2(a). From the image with the higher magnification (Figure 2(b)), the nanosized RuO\textsubscript{2} · nH\textsubscript{2}O NPs are evidently dispersed well onto the surfaces of the MCMB, the reasons for which are mainly related to the unique surface characteristics of the MCMB after the alkaline hydrothermal pretreatment. As reported before, substantial amounts of OH\textsuperscript{−} would exist upon the surfaces of the MCMB after such pretreatment [20]. The OH\textsuperscript{−} existing upon the surfaces of the MCMB not only improves the surface hydrophilic property of the MCMB but also acts as anchors for the subsequent deposition of RuO\textsubscript{2} · nH\textsubscript{2}O NPs on their surfaces [20]. Specifically, during stirring the suspension of the MCMB in the solution with Ru\textsuperscript{3+} ions, some precipitation would form onto the surfaces of the MCMB. Thus,
Figure 1: XRD patterns of RuO$_2$·$n$H$_2$O/MCMB composites with different RuO$_2$·$n$H$_2$O loadings as indicated.

Figure 2: SEM images with different magnifications for the RuO$_2$·$n$H$_2$O/MCMB composite with 24.8 wt.% RuO$_2$·$n$H$_2$O NPs.
with the following hydrothermal treatment at 180°C, initial precipitation can act as nucleation centers, which results in more and more RuO$_2$·nH$_2$O NPs coating onto the surfaces of the MCMB after hydrothermal treatment. Therefore, the alkaline hydrothermal treatment of MCMB plays a great role in the formation of RuO$_2$·nH$_2$O/MCMB composites with good dispersion of RuO$_2$·nH$_2$O NPs.

3.2. Electrochemical Profiles of the RuO$_2$·nH$_2$O/MCMB Composites. Cyclic voltammetry was used to determine the electrochemical properties of the RuO$_2$·nH$_2$O/MCMB composites. Figure 3(a) shows the CV plots of the RuO$_2$·nH$_2$O/MCMB composite with 24.8 wt.% RuO$_2$·nH$_2$O NPs in 0.5 M H$_2$SO$_4$ aqueous solution. Obviously, the CV curves of the composite all display a good rectangular shape with respect to the zero-current line and a repaid current response on voltage reversal at each end potential at various scanning rates as indicated. Also, the E-I response of the composite on the positive sweep is mirror-image symmetric to their corresponding counterpart on the negative sweep within the electrochemical window from 0.0 to 1.0 V (versus SCE), revealing a good supercapacitive behavior. An important parameter, columnic efficiency ($\eta$) of the composite electrode, can be evaluated from (2) based on the CP plots depicted in Figure 4(a):

$$\eta = \frac{t_d}{t_c} \times 100\%,$$

where $t_d$ and $t_c$ are the time for galvanostatic discharging and charging, respectively. The columnic efficiencies at different current densities ranged from 0.5 to 5 A g$^{-1}$ all keep above 99.4%, revealing its good supercapacitive behavior.

Furthermore, the SCs of the composite electrode were calculated from the CP curves (Figure 4(a)) based on (3) and the typical data are depicted in Figure 4(b):

$$SC_{composite} = \frac{I}{AV},$$

$$SC_{Ru} = \frac{SC_{composite}}{w},$$

where SC$_{composite}$, SC$_{Ru}$, $I$, $t$, $AV$, and $w$ are the SC (F g$^{-1}$) of the composite electrode, the SC (F g$^{-1}$) contributed by the single RuO$_2$·nH$_2$O species, the charge/discharge current density (A g$^{-1}$), the time (s) elapsed for the discharge cycles, the potential interval (V) of the discharge, and the percentage of RuO$_2$·nH$_2$O existing in the composites, respectively.

The SCs contributed by the RuO$_2$·nH$_2$O species as a function of current densities are shown in Figure 4(b). Impressively, a SC$_{Ru}$ of 1084 F g$^{-1}$ can be delivered at a current density of 0.5 A g$^{-1}$ by the composite electrode with electrochemical reversibility, and power property of any electrode material. Thus, typical CP curves of the RuO$_2$·nH$_2$O/MCMB composite (24.8 wt.% RuO$_2$·nH$_2$O NPs) at various current densities are shown in Figure 4(a). The E-t responses present a symmetric triangular shape, and the potential is linearly dependent on the charge-discharge time, which exhibits its good supercapacitive behavior. An important parameter, columnic efficiency ($\eta$) of the composite electrode, can be evaluated from (2) based on the CP plots depicted in Figure 4(a):

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24.8 wt.% RuO$_2$·nH$_2$O NPs. Furthermore, the electrode not
only exhibit high SCs but also maintain them well at much
higher current densities. Specifically, the electrode preserves
more than 74% of its SC delivered at 0.5 A/g as the current
density increases to 5 A g$^{-1}$, that is, even 812 F g$^{-1}$ at 5 A g$^{-1}$. For other composites with different RuO$_2$·nH$_2$O loadings,
the SC$_\text{composite}$ and SC$_\text{Ru}$ were also estimated and collected
in Figure 4(c). As reported before [3], the SC$_\text{Ru}$ of the bare
RuO$_2$·nH$_2$O, that is, the composite with 100 wt.% RuO$_2$·nH$_2$O loading, is just ca. 477 F g$^{-1}$, which is much less than the
SC$_\text{Ru}$ of the composite electrode with 24.8 wt.% RuO$_2$·nH$_2$O NPs. It indicates that the electrochemical utilization
of the RuO$_2$·nH$_2$O species is enhanced greatly when RuO$_2$·nH$_2$O NPs are well dispersed upon the surface of the MCMB.
It can be further verified by other composites with different
RuO$_2$·nH$_2$O loadings. As seen from Figure 4(c), the RuO$_2$·nH$_2$O/MCMB composites with 12.5 wt.% RuO$_2$·nH$_2$O NPs
can deliver a large SC$_\text{Ru}$ of 1115 F g$^{-1}$. In addition, a SC$_\text{Ru}$ of
987 F g$^{-1}$ still can be obtained even in the case of 52.1 wt.% RuO$_2$·nH$_2$O NPs. It is worthy of noting that the SC$_\text{composite}$ of
the composite electrode with 52.1 wt.% RuO$_2$·nH$_2$O NPs is
even larger than that of the bare RuO$_2$·nH$_2$O NPs. Therefore,
the existence of MCMB in the composites enhances the dis-
persion of RuO$_2$·nH$_2$O NPs, which avoids the serious aggre-
gation of RuO$_2$·nH$_2$O NPs themselves and makes a great
portion of ruthenium oxide NPs with large exposed surface
contacted easily by the H$^+$ ions and electrons to participate in
the electrochemical reaction for much efficient energy stor-
age at high rates. Thus, a higher electrochemical utilization
can be obtained for the composite electrodes.

To further determine the power performance of the elec-
trodes, high-rate dischargeability (HRD) of the electrode was

![Figure 4: CP plots (a) and SC$_\text{Ru}$ as a function of current densities (b) of the RuO$_2$·nH$_2$O/MCMB composite with 24.8 wt.% RuO$_2$·nH$_2$O; specific capacitances (c) and HRD curves (d) of the RuO$_2$·nH$_2$O/MCMB composites with 12.5 wt.%, 52.1 wt.% and 100 wt.% RuO$_2$·nH$_2$O NPs, respectively.](image-url)
investigated in a current density range from 0.5 to 5 A g\(^{-1}\). Here, the HRD is defined as the ratio of SC\(_{Ru}\) at a certain current density to that at 0.5 A g\(^{-1}\) and calculated according to

\[
HRD(\%) = \frac{SC\_{Ru,d}}{SC\_{Ru,0.5}} \times 100, \quad (4)
\]

where SC\(_{Ru,d}\) and SC\(_{Ru,0.5}\) are the discharge SC\(_{Ru}\) at a certain current density and 0.5 A g\(^{-1}\), respectively. Figure 4(d) presents the HRD properties of the composite electrodes.

For further understanding the electrochemical performances, the long-term cycle ability of the electrodes was also evaluated by repeating the charge/discharge test at a current density of 5 A g\(^{-1}\) for 1000 cycles. The SC as a function of the cycle number is presented in Figure 5(b). After 1000 continuous cycle tests, the SC degradation of the RuO\(_2\) \(\cdot n\)H\(_2\)O/MCMB composite with 24.8 wt.% RuO\(_2\) \(\cdot n\)H\(_2\)O is ca. 8%, much less than ca. 14% for the pure RuO\(_2\) \(\cdot n\)H\(_2\)O NPs, demonstrating that the RuO\(_2\) \(\cdot n\)H\(_2\)O/MCMB composite can maintain better electrochemical stability than the single RuO\(_2\) \(\cdot n\)H\(_2\)O NPs phase.

4. Conclusions

In conclusion, an efficient hydrothermal strategy was proposed here to disperse RuO\(_2\) \(\cdot n\)H\(_2\)O NPs upon the surfaces of mesocarbon microbeads. Electrochemical capacitance of the RuO\(_2\) \(\cdot n\)H\(_2\)O/MCMB composites were systematically investigated in 0.5 M H\(_2\)SO\(_4\) aqueous solution. Electrochemical data indicated that the RuO\(_2\) \(\cdot n\)H\(_2\)O/MCMB composites own higher electrochemical utilization, better power property, and better electrochemical stability than the pure RuO\(_2\) \(\cdot n\)H\(_2\)O NPs. The good dispersion of RuO\(_2\) \(\cdot n\)H\(_2\)O NPs and the enhanced electronic conductivity make the RuO\(_2\) \(\cdot n\)H\(_2\)O NPs with large exposed surface contacted easily by H\(^+\) ions and electrons to participate in more efficient Faradaic reactions for energy storage at high rates.
Acknowledgments

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