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A novel rapid microwave synthesis of MoS_2 nanosheets for supercapacitor electrode

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Abstract. We report a novel rapid microwave synthesis of two-dimensional MoS_2. Morphological and structural characterizations were performed by XRD and SEM, which showed that the obtained MoS_2 was a sheet-like structure with good crystallization. The electrochemical performance illustrated that the MoS_2 electrode prepared with 0.0300 mol Na_2MoO_4 has a higher specific capacitance is 83.4 F g^{-1} at 0.5 A g^{-1}, and favorable electrochemical durability up to 91.0 %, after 4000 cycles.

1. Introduction

Recently, supercapacitors (SCs) have fascinated more attention, than batteries and conventional capacitors because of its advanced performance in specific energy and power density [1]. Thus, SCs is considered a prospective energy storage device and is suitable for situations requiring lasting cycle life and big power density [2]. Nevertheless, for actual applications and meet modern energy storage requirements, innovative SCs of higher capacitance ratios must be developed without immolating power transportation and cycle life.

The nature of the electrode material determines the capacitive performance of the SCs. Consequently, it is very important for the selection of appropriate materials as SCs electrodes [3]. Over the past decade, two-dimensional (D) transition metal dichalcogenides (TMDCs), typically $MoSe_2$, MoS_2, WSe_2, and WS_2 have been considered as potential electrode materials to SCs by their good conductivity, relatively high power and capacity [1]. Especially, MoS_2 is a typical TMDCs, which consists of S-Mo-S assemble and held together by Van der Waals forces [4]. Theses diversity are due to its structure, similar to graphene with better SCs performance [5]. Many studies have focused on the synthesis of 2D MoS_2, consist of mechanical exfoliation [6], physical vapor deposition [7], chemical exfoliation [8], and hydrothermal synthesis [9]. But these methods are time-consuming and energy-intensive. Therefore, developing an easy, rapid and cut-price technique is required to synthesize MoS_2 with graphene-like structure.

In the article, we have reported the synthetic of different concentrations of two-dimensional MoS_2 nanosheets by microwave method. And the influence of different concentrations of reactants on the topography and properties of the obtained MoS_2 materials were investigated. The results illustrated that the MoS_2 nanosheets had the highest specific capacitance when using 0.0300 mol Na_2MoO_4. In addition, with the concentration of reactants increases, MoS_2 nano-sheet gradually becomes nanosphere.

2. Experiment

2.1. Preparation

Firstly, a certain amount of $Na_2MoO_4 \bullet 2H_2O$ (0.0075/0.0150/0.0300 mol) was added to a beaker holding 100 ml H_2O and stirred for 0.5 h. Then $CS(NH_2)_2$ (0.03/0.06/0.12 mol) was the addition of the solution and mixing for 23.5 h. After that, the resulted solution was transferred to microwave muffle furnace at 450 °C, heating power with 30 W, holding temperature 450 °C, holding power with 30 W, holding time 10 min. At last, the products were collected and washed with C_2H_6O and H_2O.

2.2 Material Characterizations

Prepared products were analyzed by X-ray diffractometer (XRD), and scanning electron microscope (SEM). XRD study conducted on a PANalytical X'Pert PRO. SEM study was performed.

2.3 Electrochemical Characterizations

Electrochemical properties were measured by Autolab potentiostat (PGSTAT302N) in 1M KCl aqueous solution using a three-electrode system. Platinum (Pt) and 1M $Ag/AgCl/Cl^-$ were used as the counter and reference electrodes, separately. The working electrode was MoS_2, and the acetylene black and PVDF are a mixture of 80:15:5 by a mass ratio in some N-methyl pyrrolidine-2-one (NMP). Cyclic voltammetry (CV) curves were tested at 0-1 V for MoS_2 at different scan rates. Galvanostatic charge/discharge (GCD) process was tested at different current densities at 0-1V. Cycle stability was analyzed using CV measurement 4000 cycles at 50 mV s^{-1}.

3. Results and discussion

The XRD pattern of MoS_2 prepared with the different amount of Na_2MoO_4 in Figure 1. From the Joint committee on Powder Diffraction (JCPDS card No. 37-1492, Molybdenite-2*H*, a=b=3.1612 Å, c=12.2985 Å, Space group: P63/mmc), the three main characteristic peaks at 14.38°, 39.54°, and 49.79° corresponding to (002), (103), and (105) planes, indicates that the MoS_2 thin film has Hexagonal phase. No characteristic peaks from impurity have been detected, indicating the pure MoS_2 was prepared. Furthermore, the diffraction peak of MoS_2 prepared with 0.0075 mol Na_2MoO_4 can be seen from the pattern was sharp and intense, suggesting that its crystallinity was the best.

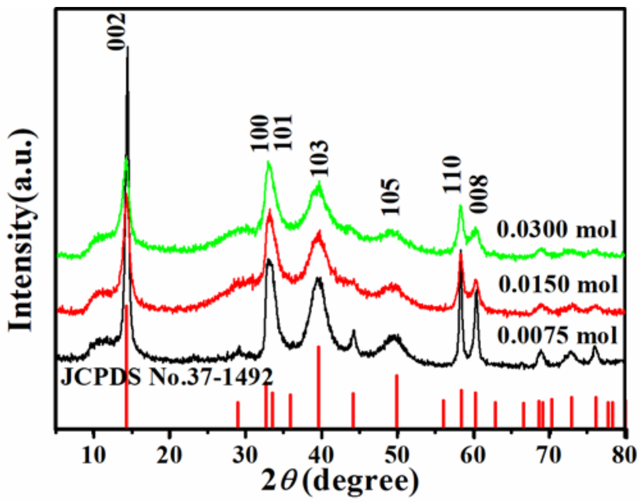

Figure 1. XRD pattern of MoS_2 prepared with the different amount of Na_2MoO_4

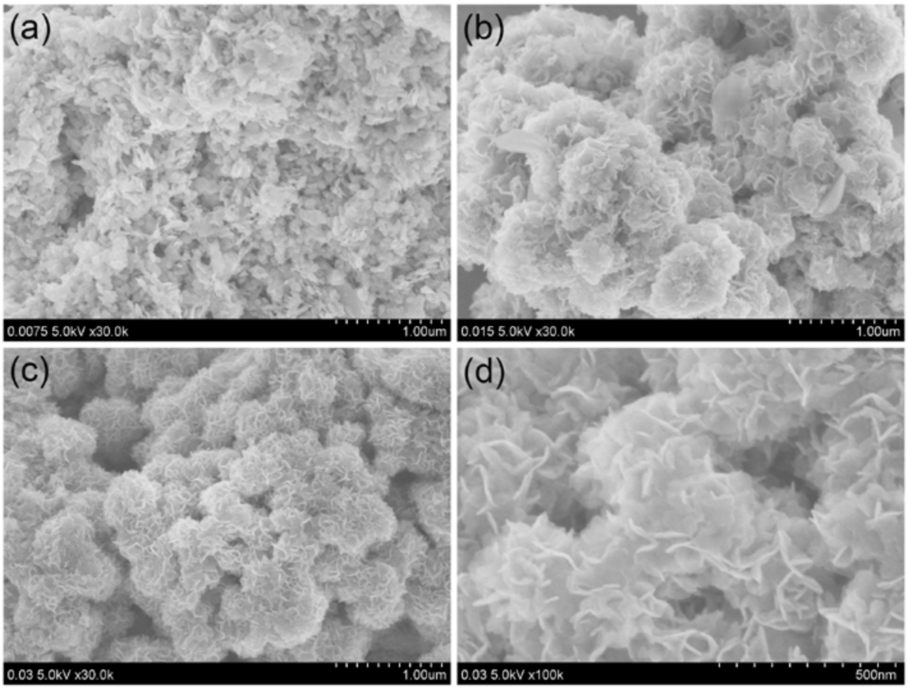

Figure 2. SEM of MoS_2 prepared with different amount of Na_2MoO_4. (a) 0.0075 mol, (b) 0.0150 mol, (c) 0.0300 mol. (d) Enlarged SEM of MoS_2 prepared with 0.0300 mol Na_2MoO_4.

Figure 2 demonstrates SEM of MoS_2 prepared with the different amount of Na_2MoO_4. Figure 2a is a low-magnification SEM image of MoS_2 prepared with 0.0075 mol Na_2MoO_4, and it can be seen that MoS_2 is a uniform sheet-like morphology. Interestingly, the MoS_2 prepared with 0.0150 mol Na_2MoO_4 of figure 2b shows the form of dense nanoblocks that are tightly connected t. The SEM image of MoS_2 prepared with 0.0300 mol Na_2MoO_4 is shown in Figure 2c, which exhibited the morphology of the nanospheres with spaces. The uniform distribution of nanospheres with space can facilitate electrolyte to access. An enlarged SEM image of MoS_2 prepared with 0.0300 mol Na_2MoO_4 is shown in figure 2d, as can be seen, these nanospheres are assembled from nanosheets with uniform morphology.

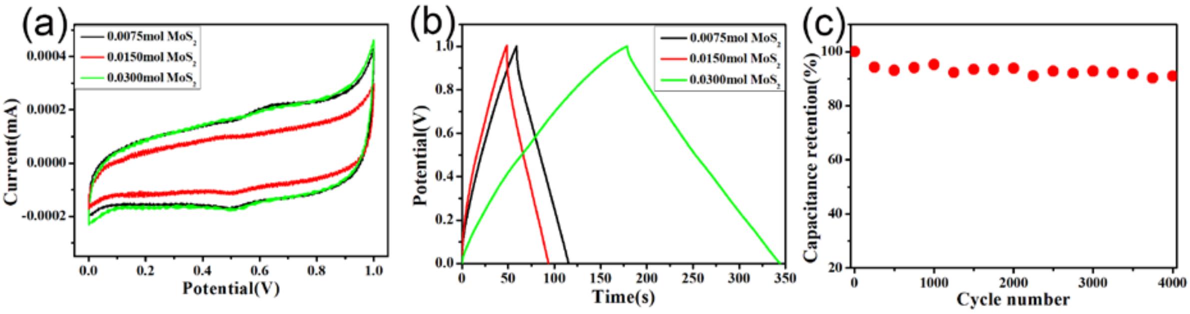

Figure 3. (a) CV curve of MoS_2 prepared with the different amount of Na_2MoO_4 at 1 mV s^{-1}, (b) GCD curve at 0.5 A g^{-1}, (c) cyclic stability curve of MoS_2 prepared with 0.0300 mol Na_2MoO_4.

The CV and GCD of MoS_2 prepared with the different amount of Na_2MoO_4 was tested at 0-1V as shown in Figures 3. Figure 3a indicates the CV curves of MoS_2 with different amount of Na_2MoO_4 at 1 mV s^{-1}. The Approximate-rectangle and symmetric CV curves were detected, demonstrating that the faraday redox reaction was electrochemically invertible in the three samples. Compared with 0.0075 mol and 0.0150 mol, the CV curve of MoS_2 prepared with 0.0300 mol Na_2MoO_4 exhibited a larger area, which is due to the formation of MoS_2 nanospheres with uniform structure, promoting electrolyte to access. Figure 3b shows the GCD curves for different MoS_2 electrodes at 0.5 A g^{-1}. It shows a symmetrical natural voltage distribution, which is consistent with CV results, pointing to an excellent invertible redox reaction in the whole potential area of MoS_2. The specific capacitance of MoS_2 electrodes prepared with different the amount of Na_2MoO_4 was calculated from the GCD curve by the formula; $C_{sp} = (I\ \Delta t)/(m\ \Delta V)$, where '$C_{sp}$' is the specific capacitance, 'I (A)'is the discharge current, 'Δt(s)' is the discharge time, 'm (g)' is the active mass of the material, and 'ΔV' is the potential windows. The specific capacitance of MoS_2 with different amount of Na_2MoO_4 at 0.5 A g^{-1} is given in table 1. The specific capacitance of MoS_2 prepared with 0.0075, 0.0150, and 0.0300 mol Na_2MoO_4 were calculated to be 29.1, 23.5 and 83.4 F g^{-1}, respectively. Figure 3c illustrates the long-term cycle stability of the MoS_2 electrode prepared with 0.0300 mol Na_2MoO_4, which was tested through CV tests repeating 4000 cycles at 50 mV s^{-1}. It could be noted that its specific capacitance retention rate exhibits outstanding stability, and the increase in the number of cycles has only a slight fluctuation. The capacitance retention study showed that the MoS_2 prepared with 0.0300 mol Na_2MoO_4 retained 91.0% after 4000 cycles.

Table 1. The specific capacitance of MoS_2 electrodes prepared with different the amount of Na_2MoO_4 at 0.5 A g^{-1}.

Amounts of Na_2MoO_4 (mol)	0.0075	0.0150	0.0300
Specific capacitance (F g^{-1})	29.1	23.5	83.4

4. Conclusion

In summary, we have reported a rapid and efficient synthesize of 2D MoS_2 nanosheets through the microwave method. Morphological and structural characterizations were performed by XRD and SEM showed that the prepared MoS_2 was a sheet-like structure with good crystallization. The electrochemical performance illustrated that the MoS_2 electrode prepared with 0.0300 mol Na_2MoO_4 exhibited a higher specific capacitance of 83.4 F g^{-1} at 0.5 A g^{-1}, and favorable cycle life, which is up to 91.0 % after 4000 cycles.

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