

## Defect-rich molybdenum disulfide as electrode for enhanced capacitive deionization from water

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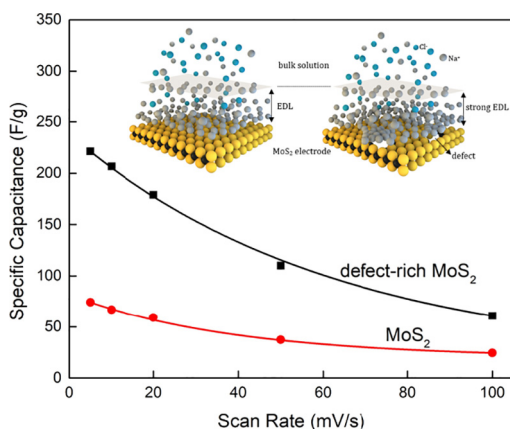
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### GRAPHICAL ABSTRACT



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### ABSTRACT

Molybdenum disulfide (MoS<sub>2</sub>) could be a promising electrode candidate for capacitive deionization (CDI) process benefited from the high specific area, excellent electronic conductivity, sound electrochemical stability, and good regenerability. In this work, a CDI study was performed on the desalination performance of water with defect-rich MoS<sub>2</sub> sheets as the electrode, in which the defects were fabricated through thermal treatment. The MoS<sub>2</sub> with abundant defects on the surface exhibited a much higher desalination capacity than that without any surface modification. The enhancement could be attributed to the abundant negative charges, high specific capacitance and low inner resistance of the defect-rich MoS<sub>2</sub>, which promoted the electrostatic attraction and electrosorption of Na<sup>+</sup> to the electrode surfaces, as well as the formation of a thick electric double layer in the CDI process. The results suggested that defects can be incorporated on the surface of MoS<sub>2</sub> for being a superb CDI electrode.

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## 1. Introduction

Capacitive deionization (CDI) is a promising technology for desalination because of its competence at efficiency, cost, and environmental issues [1–3]. It is an electrosorption process that operates by adsorbing ions through electric double layer (EDL) formed at the interface of electrodes and a feed water stream [4,5]. When the electrodes are being charged, the sodium and chloride ions are firstly being attracted to approach the electrodes, and then adsorbed electrostatically in the EDL of the electrode materials. Once the electrodes are discharged, the adsorbed ions are released back to the effluent water stream. Based on the principle of EDL, the CDI efficiency is largely dependent on the specific surface area and electronic conductivity of the electrode materials [6]. Carbon materials, such as activated carbon [7,8], carbon aerogel [9], are conventional materials for the preparation of electrodes for CDI application due to their high surface and good electronic conductivity, however they offer a long diffusion path for the access of sodium ions to the EDL of electrode because of the small pore size. To bring a fast access of sodium ions into the electrode, 2D materials might be more suitable for being the CDI electrode material profited from their ultra-high surface-to-volume ratio.

Molybdenum disulfide ( $\text{MoS}_2$ ) is a typical layered transition-metal dichalcogenite, which consists of two hexagonal sheets of sulfur atoms and one intermediate hexagonal sheet of molybdenum atoms. The 2D  $\text{MoS}_2$  possesses huge specific surface area, providing an ideal platform for the physical and chemical reaction. Recent studies indicated that  $\text{MoS}_2$  exhibited outstanding performances and superior ability for various dyes and heavy metals removal due to the strong  $\pi$ - $\pi$  interaction and S-heavy metal complexation [10–12]. However,  $\text{MoS}_2$  has not drawn widespread attention as a desalination material until it was revealed that  $\text{MoS}_2$  might be a promising candidate for desalination. Molecular dynamics simulations demonstrated that nanoporous 2D molybdenum disulfide would be an efficient filter membrane in sea water desalination because it could prevent salt ions and allow transport of water through nanopores, exhibiting high water transparency and a strong salt filtering capability [13,14]. However the above researches were based on theoretic calculation, the translation of applying  $\text{MoS}_2$  to a real water desalination was in 2017, where pure  $\text{MoS}_2$  or  $\text{MoS}_2$ /carbon nanotube composite was used as electrodes [15,16]. Surprisingly, the experimental results indeed verified the excellent desalination capacity and good cycling stability of  $\text{MoS}_2$ , which was even better than that of graphene [17,18]. In the investigation of desalination performance, Wang stated that the unique two-dimensional thin sheet structure enabled  $\text{MoS}_2$  a sound CDI electrode [15,16]. Capitalizing on the two-dimensional layered structure of  $\text{MoS}_2$ , Presser further proposed that faradaic ion intercalation might be the main contributor for the efficient removal of  $\text{Na}^+$  and  $\text{Cl}^-$  from a feed water [15,16]. These studies revealed the important role of two-dimensional structure in the desalination performance of  $\text{MoS}_2$ , however no profound study has been performed on the surface property of  $\text{MoS}_2$  in its CDI behavior.

Thus in this study, an attempt was made to reveal the role and mechanism of surface properties on  $\text{MoS}_2$  in the CDI process through comparing the desalination performance of  $\text{MoS}_2$  and defect-rich  $\text{MoS}_2$ . The  $\text{MoS}_2$  sample was first subjected to thermal treatment for its property modification and then characterized by high resolution transmission electron microscope (HRTEM) and atomic force microscope (AFM), while the desalination performance was investigated through the adsorption isotherm and kinetics experimental. The regenerability and stability of  $\text{MoS}_2$  electrode were also studied through the electrochemical and CDI cycle experiments. The object was to obtain a clear understanding in the role and influence mechanism of defects in the desalination process with  $\text{MoS}_2$  as electrodes, as well as to enhance the desalination performance of  $\text{MoS}_2$  and give a guidance for the preparation of  $\text{MoS}_2$  as electrode.

## 2. Experimental

### 2.1. Materials and reagents

Hexaammonium heptamolybdate tetrahydrate ( $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24}\cdot 4\text{H}_2\text{O}$ ), thiourea ( $\text{CN}_2\text{H}_4\text{S}$ ), sodium chloride ( $\text{NaCl}$ ), silver nitrate ( $\text{AgNO}_3$ ), potassium chromate ( $\text{K}_2\text{CrO}_4$ ) and sodium hydroxide ( $\text{NaOH}$ ) used in this study were purchased from the Sinopharm Chemical Reagent Co., Ltd. (China). Sulfuric acid ( $\text{H}_2\text{SO}_4$ ) was supplied by Xinyang chemical reagent (China). All the reagents were of analytical grade. Titanium plate was purchased from Yongsheng Company, China, respectively. Milli-Q water (Millipore, Bedford, MA) with a resistivity of 18.2  $\text{M}\Omega\cdot\text{cm}$  was used in the experiments.

### 2.2. Methods

#### 2.2.1. Preparation of molybdenum disulfide nanosheets

Flower-like molybdenum disulfide was prepared according to the synthesis in previous work [19]. Firstly, 2.48 g of  $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24}\cdot 4\text{H}_2\text{O}$  and 4.56 g of  $\text{CN}_2\text{H}_4\text{S}$  were dissolved in 72 mL deionized water, followed by magnetic stirring the mixture to form a homogeneous solution. After 30 min, the solution was transferred into a 100 mL Teflon-lined stainless steel autoclave, and then thermally treated at 220 °C for 6 h. After cooling down, the synthetic precipitate was collected by filtration, and washed with deionized water for 6 times to remove the residual chemical reagents. So far, the flower-like molybdenum disulfide was synthesized. The as-prepared molybdenum disulfide was subsequently ultra-sonicated by a Cole-Parmer at 300 W for 10 min to be exfoliated and obtain the  $\text{MoS}_2$  nanosheets. Finally, the suspension was freeze-dried for 24 h. For the preparation of defect-rich  $\text{MoS}_2$ , the  $\text{MoS}_2$  nanosheets were subjected to thermal treatment at 300 °C in a Neytech Vulcan 3–550 muffle furnace for 2 h, followed by washing the as-prepared sample with deionized water for the removal of oxides. The thermally treated sample was named as T- $\text{MoS}_2$  in this work.

#### 2.2.2. Fabrication of electrodes

The electrode material was a mixture of active material ( $\text{MoS}_2$ , T- $\text{MoS}_2$  or activated carbon (AC)), conductive carbon black, and poly vinylidene fluoride (PVDF) with a mass ratio of 8:1:1. The mixture with a total mass of 37.5 mg, among which the active material accounted for 30 mg, was ground in an agate mortar for 20 min to form a homogeneous slurry before spreading on a titanium plate. Afterwards, the electrode materials were coated on one side of the titanium plate to form a 40  $\mu\text{m}$  thickness and 2 cm  $\times$  6 cm rectangle area. Then, the electrode was heated at 60 °C in vacuum for 12 h to remove the residual organic solvent.

#### 2.2.3. Electrosorption experiments

The desalination experiments were performed in a self-made CDI system, which was schematically represented in Fig. 1. Two pairs of titanium plate coated with AC,  $\text{MoS}_2$  or T- $\text{MoS}_2$  composite were placed in the poly(methyl methacrylate) cell as electrodes. The distance between the positive and negative electrode was 1 mm and the potential applied to the electrode was 0.8 V. During the desalination, 100 mL of  $\text{NaCl}$  solution with a desired concentration was continuously recycled in the system at a flow rate of 25 rpm by a peristaltic pump. 1 mL of the solution was taken from the outlet at different time intervals for the determination of  $\text{Na}^+$  concentration. The concentration of  $\text{Cl}^-$  at initial and final time was determined by Mohr method. The electrosorption capacity of  $\text{Na}^+$  on cathode and  $\text{Cl}^-$  on anode was calculated according to Eq. (1) to evaluate the performance of the electrode in the desalination process.

$$q = \frac{(C_0 - C) \times V}{m} \quad (1)$$

where  $C_0$  and  $C$  are the  $\text{Na}^+$  and  $\text{Cl}^-$  concentrations at the initial time

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