Supporting Information

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Composition of $\text{MoO}_2$ Nanoparticles with RGO Sheets as Improved Lithium Ion Battery Anode

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Experimental Section:

All required chemicals were procured from Merck and proceeded without additional purification.

Preparation of graphene oxide:

Modified Hummer’s method was implemented to synthesize graphene oxide (GO).

Conventionally, 0.5 g of NaNO$_3$ was added to 0.5 g of graphite powder and dissolved in conc. H$_2$SO$_4$ (25 mL). The mixture was stirred for 30 min to achieve a homogeneous solution. Gradually, 3.5 g of KMnO$_4$ was added to the above reaction mixture and transferred to an oil bath (temp= 40 °C) and stirred for 2 h. Further, double distilled (DD) water (25 mL) was added to the above solution and stirred for 30 min at 70 °C. Eventually, 30 % H$_2$O$_2$ (5 mL) in 50 mL of DD water was added. The retrieved GO suspension was washed with conc. HCl and DD water and dried at 80 °C.

Preparation of MoO$_2$–rGO nanocomposite:

MoO$_2$–rGO was synthesized in 2 steps. First step involved the synthesis of MoO$_2$ NPs. MoO$_2$ NPs was synthesized by annealing method in presence of Ar environment. In an atypical method, 0.247 g of ammonium heptamolybdate [(NH$_4$)$_6$Mo$_7$O$_{24}$.4H$_2$O] and 0.026 g malic acid [C$_4$H$_6$O$_5$] were dissolved in 5 mL of DD water (1:1 molar ratio). Homogeneous solution was achieved by stirring the above mixture for 10 min. The solution was transferred to 30 mL alumina boat and was positioned in a tubular furnace. Inert atmosphere was created by passing Ar gas for 30 min. The temperature of the furnace was raised from room temperature to 500 °C at a heating rate of 6 °C min$^{-1}$. Inert atmosphere was maintained throughout the reaction by continuous flow of Ar gas. After 30 min, the furnace was allowed to cool down to room temperature naturally. The synthesized product (black powder) was confirmed as MoO$_2$ by XRD characterization.

The reaction of formation of MoO$_2$ NPs can be written as:

$$6 \text{(NH}_4\text{)}_6\text{Mo}_7\text{O}_{24}\.4\text{H}_2\text{O} + 7 \text{C}_4\text{H}_6\text{O}_5 + \text{H}_2\text{O} = 42 \text{MoO}_2 + 28 \text{CO}_2 + 36 \text{NH}_3 + 64 \text{H}_2\text{O}$$  \(1\)

In the second step, GO (100 mg) was dispersed in 20 mL of DD water and sonicated for 30 min. Ascorbic acid (C$_6$H$_8$O$_6$, 50 mg) was added as a surfactant and reducing agent, and further sonicated for 15 min. 500 mg of synthesized MoO$_2$ NPs was added to the above suspension and sonicated for additional 30 min. The black suspension was transferred to 23 mL Teflon container and hydrothermal treatment was carried out for 6 h at 180 °C. The end product MoO$_2$–rGO was washed several times with DD water, followed by methanol and dried overnight at 60 °C. Scheme.1 shows the schematic representation of synthesis of MoO$_2$–rGO nanocomposite.
Scheme 1: Schematic representation of synthesis of MoO$_2$-rGO nanocomposite

References: