



# Hierarchical structured graphene/metal oxide/porous carbon composites as anode materials for lithium-ion batteries



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## ABSTRACT

As a novel anode material for lithium-ion batteries, CeO<sub>2</sub> displays imperceptible volumetric and morphological changes during the lithium insertion and extraction processes, and thereby exhibits good cycling stability. However, the low theoretical capacity and poor electronic conductivity of CeO<sub>2</sub> hinder its practical application. In contrast, Co<sub>3</sub>O<sub>4</sub> possesses high theoretical capacity, but undergoes huge volume change during cycling. To overcome these issues, CeO<sub>2</sub> and Co<sub>3</sub>O<sub>4</sub> nanoparticles are formed inside the pores of CMK-3 and display various electrochemical behaviors due to the different morphological structures of CeO<sub>2</sub> and Co<sub>3</sub>O<sub>4</sub> within CMK-3. Moreover, the graphene/metal oxide/CMK-3 composites with a hierarchical structure are then prepared and exhibit better electrochemical performances than metal oxides with or without CMK-3. This novel synthesis strategy is hopefully employed in the electrode materials design for Li-ion batteries or other energy conversion and storage devices.

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## 1. Introduction

The demand for the energy storage and conversion devices is gradually increasing owing to the rapidly incremental global energy consumption. As one of the most important technologies for energy conversion and storage, lithium-ion batteries (LIBs) are widely used in portable electronic devices (e.g., cell phones) due to their outstanding advantages such as high energy density, long cycle life, no memory effect and environmental friendliness [1]. The selection of electrode materials is of great importance since the electrochemical performance of LIBs relies heavily on the electrode properties. Carbon-based materials (e.g., graphite) are usually used as anode materials for LIBs because of their low cost, high abundance and outstanding kinetics [2,3]. However, graphite suffers great danger from the formation of lithium dendrite during the overcharge process owing to its low Li-intercalation potential (0.2 V vs. Li/Li<sup>+</sup>) [4]. Transition metal oxides are another one of the promising anode materials for LIBs and usually possess high theoretical capacities based on a redox mechanism. For instance, the theoretical capacity of Co<sub>3</sub>O<sub>4</sub> is ~890 mA h g<sup>-1</sup>, much higher than that of commercial graphite anode (372 mA h g<sup>-1</sup>) [5–7]. But the severe volume change and poor electronic conductivity of

Co<sub>3</sub>O<sub>4</sub> during the charge and discharge processes may reduce its cycle and rate performances [8,9]. Recently, CeO<sub>2</sub> is reported as a novel LIB anode material and displays opposite electrochemical properties such as low theoretical capacity but imperceptible volumetric and morphological changes because of a fully reversible phase transformation between fluorite CeO<sub>2</sub> and cubic Ce<sub>2</sub>O<sub>3</sub> during the electrochemical process [10–12]. In addition, the intrinsic low electronic conductivity of CeO<sub>2</sub> is also an impediment to its practical use for LIBs.

Carbon materials are usually used to support metal oxides in order to improve their electrochemical performances due to its high electronic conductivity. Moreover, porous carbon such as CMK-3 can further supply the improved electronic conducting network, buffers their volume variation during cycling, and hosts the nanosized metal oxide from aggregation and disintegration through its ordered mesopores [13–15]. Therefore, metal oxides-loaded porous carbons (e.g., CMK-3) ought to show better cycling stability and rate capability in principle. However, in our previous study and other relevant literatures, it has been found that the content of metal oxides in CMK-3-supported materials highly influences the electrochemical performances [16–18]. Namely, too high loading of metal oxides may destroy the mesostructure of CMK-3, and some large particles inevitably form outside the pores of CMK-3, leading to a capacity fading and poor cycle performance, whereas too low loading of metal oxides may reduce the theoretical capacities of the CMK-3-supported composites. Thus

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it is still a challenge to well integrate metal oxides into porous carbon and avoid the formation of large particles outside the mesopores in order to pursue high-performance electrode materials for supercapacitors or LIBs.

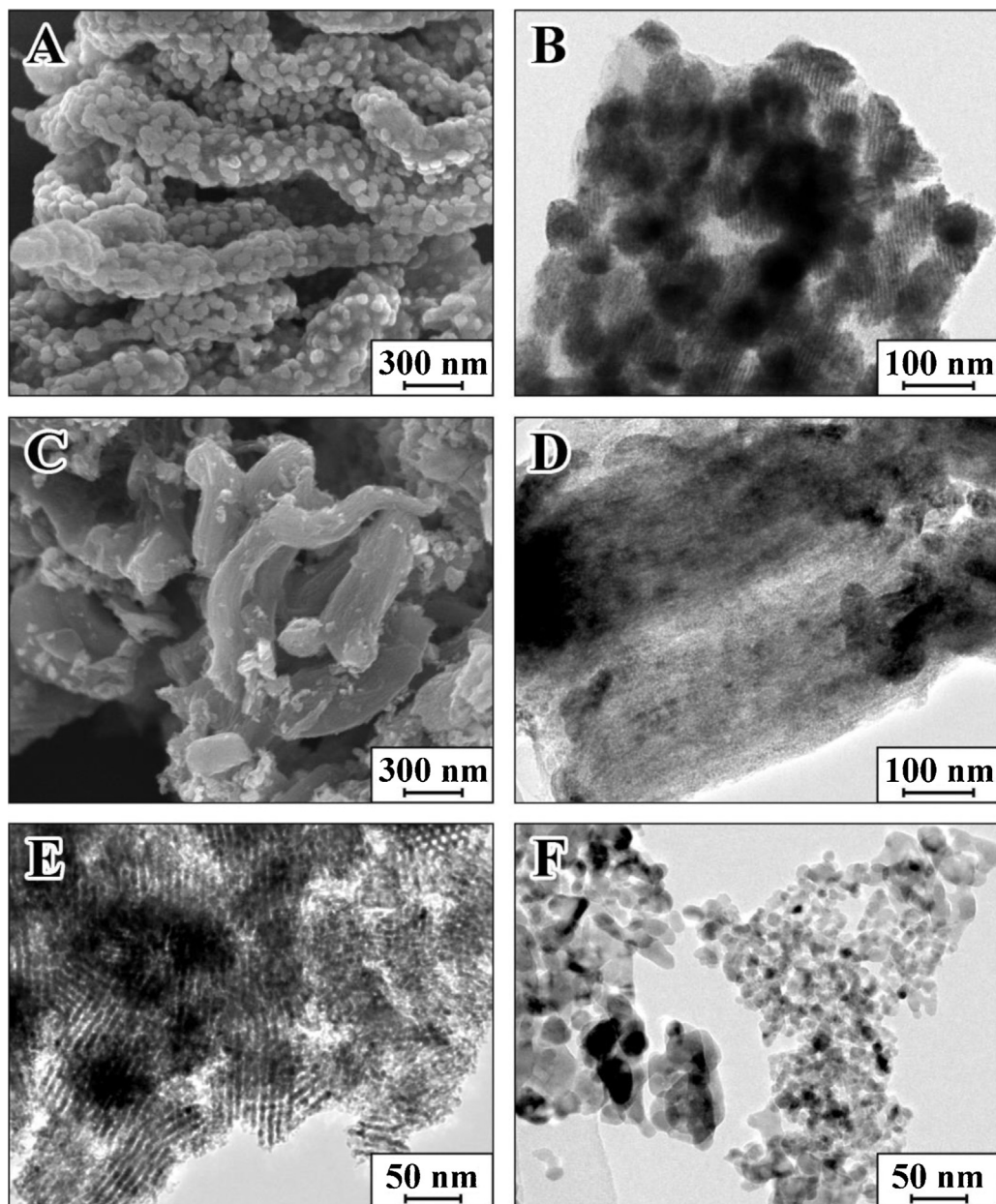
Graphene, as a novel two-dimensional carbon nanosheet, has attracted many attentions due to its excellent properties such as large specific surface area, great mechanical strength, high electrical and thermal conductivity [19]. Accordingly, various graphene-based metal oxides are fabricated as a new class of advanced electrode materials with enhanced cycle and rate performances [20–23]. In the present work,  $\text{CeO}_2$  and  $\text{Co}_3\text{O}_4$  have been selected as functional guest materials to be loaded into CMK-3. One of the reasons of choosing them is the significantly different melting points of their precursors, which is a critical factor in the crystallization of oxides inside the template pores. Their structure, size and morphology have been revealed by electron microscopic studies. The apparent difference in the constructions of CMK-3-

$\text{CeO}_2$  and CMK-3- $\text{Co}_3\text{O}_4$  (referred to as mC- $\text{CeO}_2$  and mC- $\text{Co}_3\text{O}_4$ ) influences their electrochemical performances. Furthermore, these CMK-3-supported metal oxides were further encapsulated by graphene nanosheets through the electrostatic interaction and hydrogen bonding. The hierarchical structure of graphene/metal oxide/porous carbon composites (referred to as G-mC-MOs, e.g., G-mC- $\text{CeO}_2$  and G-mC- $\text{Co}_3\text{O}_4$ ) may highly improve the electrochemical properties of the composite electrodes.

## 2. Experimental

### 2.1. Preparation of CMK-3- $\text{CeO}_2$ and CMK-3- $\text{Co}_3\text{O}_4$

CMK-3 was synthesized using sucrose as the precursor and SBA-15 as the template (see the Supporting information) [24]. CMK-3-supported metal oxides were synthesized by thermal decomposition of metal nitrates inside the pores of CMK-3. In a



**Fig. 1.** SEM and TEM images of CMK-3/oxide composites, (A, B) mC- $\text{CeO}_2$  and (C, D) mC- $\text{Co}_3\text{O}_4$ . TEM images of the specimens after removing the CMK-3 template, (E) m- $\text{CeO}_2$  and (F) n- $\text{Co}_3\text{O}_4$ .

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