



## Short communication

## Antimony nanoparticles anchored in three-dimensional carbon network as promising sodium-ion battery anode

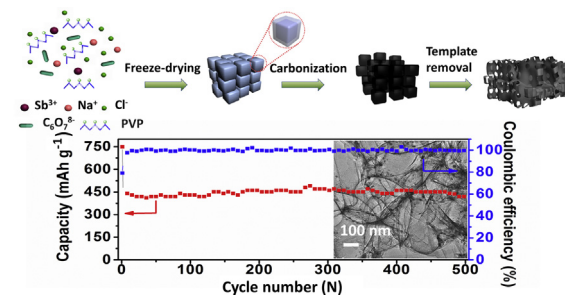
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## HIGHLIGHTS

- SbNPs@3D-C was achieved by template-assisted freeze-drying and carbonization.
- SbNPs@3D-C showed nano-sized Sb particles anchored in 3D carbon network structure.
- Long-life performance (500 cycles) and superior rate ability were demonstrated.
- NaCl template-assisted strategy combined with freeze-drying can be widely extended.

## GRAPHICAL ABSTRACT



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## ABSTRACT

A novel composite with antimony (Sb) nanoparticles anchored in three-dimensional carbon network (denoted as SbNPs@3D-C) is successfully synthesized via a NaCl template-assisted self-assembly strategy, followed by freeze-drying and one-step *in-situ* carbonization. The three-dimensional interconnected macroporous carbon framework can not only stabilize the architecture and buffer the volume expansion for Sb nanoparticles, but also provide high electrical conductivity for the whole electrode. Consequently, as a sodium-ion battery anode, the SbNPs@3D-C delivers a high reversible capacity ( $456 \text{ mAh g}^{-1}$  at  $100 \text{ mA g}^{-1}$ ), stable cycling performance (94.3% capacity retention after 500 cycles at  $100 \text{ mA g}^{-1}$ ) as well as superior rate capability ( $270 \text{ mAh g}^{-1}$  at  $2000 \text{ mA g}^{-1}$ ). When compared with commercial Sb particles, the SbNPs@3D-C exhibits dramatically enhanced electrochemical performance. Free from expensive template sources and complex manipulation, this work might shed some light on the synthesis of low-cost and high-performance materials for the next “beyond lithium” battery generation.

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## 1. Introduction

Widespread implementation of advanced energy-storage systems requires high efficiency, low cost, and sustainability. Rechargeable lithium-ion batteries (LIBs) are one of the most successful energy storage devices, which have rapidly penetrated into everyday life

[1–4]. However, the utilization of LIBs for electrical grid is a controversial plan due to the limited lithium resource. Sodium, which locates below lithium in the periodic table, shares similar chemical properties in many aspects. In addition, it is abundant and low-cost. Thus, sodium is an ideal alternative to lithium in energy storage systems [5–8]. Over the past decades, many layered oxides [9], oxides [10], and polyanionic compounds [11] have been intensively investigated as cathode materials for sodium-ion batteries (SIBs). With regard to anode materials, the pristine graphite has a very low capacity when used as a SIBs anode because of insufficient interlayer spacing [12,13]. Various carbonaceous materials, including soft [14]

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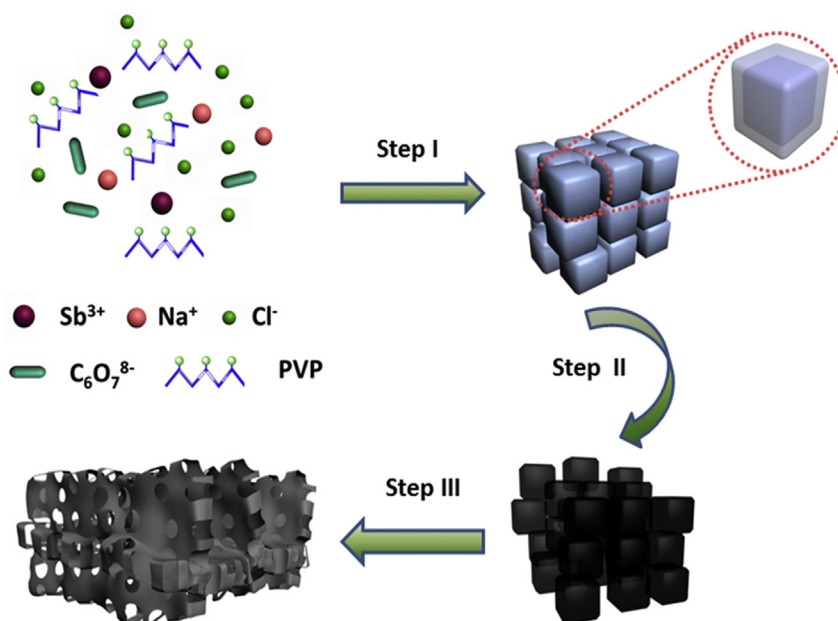


Fig. 1. Schematic illustration of the synthesis procedure. Step I: freeze drying; step II: carbonization; step III: template removal.

and hard carbon [15], hollow carbon nanospheres [16], hollow carbon nanowires [17], carbon nanosheets [18], reduced graphene oxides [19,20] have been investigated. Nevertheless most of these carbonaceous materials possess low specific capacities less than 300 mAh g<sup>-1</sup>, for instance, the hard carbon C1600 [15] delivers a reversible capacity of 290 mAh g<sup>-1</sup> at a specific current of 20 mA g<sup>-1</sup>; the hollow carbon nanospheres [16] exhibit a reversible capacity of 200 mAh g<sup>-1</sup> at a specific current of 50 mA g<sup>-1</sup>, which cannot meet the ever-increasing demand for high-capacity energy storage devices. Moreover, most intercalation type anodes display high working potentials, resulting in low energy density [21]. Therefore, to improve the overall performance of SIBs, the pursuit of new anode materials with high capacity and long life remains a great challenge.

Recently, antimony has been investigated as a promising anode due to its high specific capacity of 660 mAh g<sup>-1</sup> when fully sodiated to Na<sub>3</sub>Sb:  $3\text{Na}^+ + \text{Sb} + 3\text{e}^- \leftrightarrow \text{Na}_3\text{Sb}$  [22]. Nevertheless, the large volume change during sodium ion insertion/extraction causes active material fracture and loss of electrical contact, resulting in poor electrochemical performance. Previous reports on Sb anode for SIBs mainly include mechanically milled Sb/C composites [23], reduced graphene oxide/Sb particles [24], electrospun Sb/C nanowires [25,26], Sb/N-rich carbon nanosheets [27], hollow Sb particles [28], monodisperse Sb nanoparticles [29], mechanically milled Sb/MWCNT composites [30], one-dimensional Sb@C [31] etc. For example, Cao et al. [26] reported the synthesis of Sb/C nanofibers with a high reversible capacity of 631 mAh g<sup>-1</sup> and an impressive capacity retention of 90% for 400 cycles. Hu et al. [32] reported the assembly of full-cell with Sb/rGO anode and Na<sub>3</sub>V<sub>2</sub>(PO<sub>4</sub>)<sub>3</sub>/rGO cathode. However, most of the above-mentioned strategies are complicated and time-consuming. Besides, the electrochemical performances of the resulted anode materials are not very satisfactory. In this regard, a direct synthesis route for Sb-based anodes with high performance is urgently desired.

Currently, three-dimensional conductive nanostructures have become a rapidly growing field of energy storage research because of their large surface area, continuous interconnected network, and excellent interface [33,34]. On the other hand, reducing the particle size from bulk to nanoscale can effectively enhance electrochemical reaction kinetics [35]. By combining nanosized active materials with three-dimensional network, some encouraging

advances have been achieved in lithium-ion batteries [36] and supercapacitors [37]. However, conventional template-assisted methods for macroporous three-dimensional construction are complex. First, the templates should be concisely modified to endow themselves with compatibility between core and shell. Then, most of the template-removal processes are often energy-consuming or danger-risking. Herein we develop a novel NaCl template-assisted self-assembly strategy followed by freeze-drying and carbonization to construct SbNPs@3D-C composites. The synthesized SbNPs@3D-C exhibits long cycling stability and improved rate performance. We believe this work might shed some light on the synthesis of high-performance hybrid electrode materials for future low-cost, large-scale energy storage system.

## 2. Results and discussion

Fig. 1 schematically illustrates the synthesis procedure. In the first step (step I), SbCl<sub>3</sub>, citric acid, polyvinyl pyrrolidone (PVP), and NaCl were dissolved in water, frozen by liquid nitrogen, and subjected to freeze-drying. Herein, SbCl<sub>3</sub> was used as the Sb precursor. Citric acid, a weak organic acid with three carboxyl groups, coordinated with and inhibited the hydrolysis of Sb<sup>3+</sup>. PVP, an amphiphilic and water-soluble polymer [38], acted as the capping agent to confine the size and morphology of NaCl particles. It is well known that NaCl has a face-centered cubic (FCC) crystal structure [39]. During freeze-drying, the NaCl grew into cubes, which further self-assembled into three-dimensional structure and acted as the template for the carbon network. Meanwhile, the *in-situ* formed SbCl<sub>3</sub>/citric acid/PVP composite coated evenly on the surface of the NaCl template. In step II, the as-obtained sponge-like sample after freeze-drying was ground and carbonized. Upon heating in H<sub>2</sub>/Ar, the PVP and citric acid were pyrolyzed, forming a three-dimensional interconnected carbon network. Meanwhile, the Sb<sup>3+</sup> was reduced to Sb nanoparticles firmly anchored in the three-dimensional carbon network. Finally (step III), the NaCl template was thoroughly removed by deionized water and pure SbNPs@3D-C was obtained.

X-ray diffraction (XRD) is used to study the crystalline structure of the obtained samples. As depicted in Fig. 2a, the diffraction peaks can be indexed to crystalline Sb (space group:  $R\bar{3}m$ ,  $a = b = 0.4307$  nm,  $c = 1.1273$  nm, JCPDS: 01-071-1173), attesting

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