Electronic Supplementary Information

Synthesis of NASICON-type structured NaTi₂(PO₄)₃-graphene nanocomposites as anodes for aqueous rechargeable Na-ion batteries

Gang Pang,^{a,b} Ping Nie,^a Changzhou Yuan,^b Bing Ding,^a Jiajia Zhu ^a and Xiaogang Zhang^{*a}

^aCollege of Material Science & Engineering and Key Laboratory for Intelligent Nano Materials and Devices of Ministry of Education, Nanjing University of Aeronautics and Astronautics, Nanjing, 210016, P. R. China. E-mail: azhangxg@163.com; Fax: +86 025 52112626; Tel: +86 025 52112918
^bAnhui Key Laboratory of Metal Materials and Processing, School of Material

Science & Engineering, Anhui University of Technology, Maanshan, 243002, P. R. China

Synthesis of Graphite oxide (GO)

Graphite oxide (GO) was prepared by a modified Hummers method (ref.30 in main text). The graphite powder (12 g) was added slowly into an 90 °C solution of concentrated H_2SO_4 (50 mL), $K_2S_2O_8$ (10 g), and P_2O_5 (10 g). While maintaining agitation at 80°C for 4.5 h, the resultant dark blue mixture was allowed to cool to room temperature. The mixture was then carefully diluted with distilled water, filtered until the rinse water pH became neutral. The product was dried in air at 80 °C for 12 h. This preoxidized graphite was then subjected to oxidation by Hummers' method.

The oxidized graphite powder (6 g) was put into concentrated H_2SO_4 (230 mL) in an ice-bath and stirred for 30 min. KMnO₄ (30 g) was added gradually to the suspension with stirring and cooling. The rate of addition was controlled carefully to prevent the temperature of the suspension from exceeding 20 °C as a safety measure. Then, the mixture was slowly heated to 35 °C and stirred for 2 h. Subsequently, distilled water (460 ml) was dropwise added under vigorous stirring, causing violent effervescence and a quick rise in temperature to near 100 °C. The slurry was stirred at the temperature for another 30 min. Afterwards, 30 % H₂O₂ solution (50 ml) and distilled water (800 ml) were added sequentially to reduce insoluble manganese species and stirred for 12 h. The resulting bright yellow suspension was filtrated and washed using 2000 ml dilute HCl (1 mol/L) solution. After dispersing the collected precipitate into 2000 ml distilled water, the solution was centrifuged and washed twice with distilled water. Subsequently, the resulting product was dried under vacuum at 80 °C for two days. The GO was obtained and ground into powder (100 mesh).



Fig. S1 TG curve of the $NaTi_2(PO_4)_3/GNS$ in air atmosphere.



Fig. S2 AFM image and the thickness (a, b), FESEM image (c) and XRD pattern (d) of GO.



Fig. S3 XRD pattern (a) and FESEM image (b) of TiO_2/GNS .



Fig. S4 XRD pattern (a) and FESEM image (b) of $Ti_5P_4O_{20}/GNS$.



Fig. S5 XRD pattern (a) and FESEM image (b) of $NaTi_2(PO_4)_3/GNS$.