Electronic Supplementary Material (ESI) for Nanoscale

Supporting Information

One-pot synthesis of Fe_2O_3 yolk-shell particles with two, three, and four shells for application as anode material in lithium-ion batteries

Mun Yeong Son, Young Jun Hong, Jung-Kul Lee and Yun Chan Kang*

Department of Chemical Engineering, Konkuk University, 1 Hwayang-dong, Gwangjin-gu, Seoul 143-701, Korea

This file includes:

- Experimental section
- Schematic diagram of the ultrasonic spray pyrolysis process.
- SEM images of the Fe_2O_3 yolk-shell particles prepared by spray pyrolysis at various temperatures; (a) 600°C, (b) 800°C, (c) 1000°C.
- XRD patterns of the yolk-shell and densely structured Fe_2O_3 particles prepared at various temperatures.
- Enlarged TEM images of the Fe_2O_3 yolk-shell particles prepared by spray pyrolysis from the spray solution with sucrose at 800°C.
- Thermogravimetric curve of the Fe_2O_3 yolk-shell particles prepared by spray pyrolysis at 1000°C.

- Charge and discharge curves of the Fe_2O_3 yolk-shell particles prepared at 1000°C cycled at 300 mA g^{-1} in the voltage ranges of 0.005-3.0V.
- Coulombic efficiencies of the Fe_2O_3 particles prepared by spray pyrolysis from the spray solutions with and without sucrose. (Cycled at 300 mA g^{-1} in the voltage ranges of 0.005-3.0V)
- Cycling and Coulombic efficienciency of the Fe_2O_3 yolk-shell particles prepared at 1000°C at a high current density of 1000 mA g^{-1}.

Experimental section

Preparation of Fe_2O_3 particles: The multishelled Fe_2O_3 yolk-shell-structured particles were directly prepared using spray pyrolysis from an aqueous spray solution containing Fe nitrate and sucrose. The sucrose was used as the carbon source to form the yolk-shell-structured particles. The spray pyrolysis system consisted of a droplet generator, a quartz reactor, and a powder collector. The length and diameter of the quartz reactor were 1000 and 55 mm, respectively. A 1.7-MHz ultrasonic spray generator with six vibrators was used to generate a large quantity of droplets, which were carried into the high-temperature tubular reactor by air as a carrier gas at a flow rate of 5 L min^{-1}. The reactor temperature was increased from 600 to 1000°C. The concentrations of the Fe nitrate and sucrose were 0.25 and 0.7 M, respectively.

Characterization: The crystal structures of the Fe_2O_3 yolk-shell-structured particles were investigated using X-ray diffractometry (XRD, Rigaku DMAX-33). The morphologies of the Fe_2O_3 yolk-shell-structured particles were characterized using scanning electron microscopy (SEM, JEOL JSM-6060) and high-resolution transmission electron microscopy (HR-TEM, FEI Tecnai^{TM} 300K). The Brunauer-Emmett-Teller (BET) surface area and Barrett-Joyner-Halenda (BJH) pore diameter of the particles were measured using N_2 gas as the adsorbate.

Electrochemical measurements: The capacities and cycle properties of the electrodes produced using the Fe_2O_3 yolk-shell-structured particles were measured using 2032-type coin cells. The electrodes were made of 70 wt% active material, 20 wt% carbon black (Super-P) as conductive material, and 10 wt% sodium carboxymethyl cellulose (CMC) as a binder with a few drops of distilled water. Lithium metal and a microporous polypropylene film were used as the counter electrode and separator, respectively. $LiPF_6$ (1

M) in a mixture of ethylene carbonate (EC) and dimethyl carbonate (DMC) in a 1:1 volume ratio with 2 wt% vinylene carbonate (VC) was used as the electrolyte. The entire cell was assembled under an argon atmosphere in a glove box. The charge/discharge characteristics of the samples were measured at various current densities in the range 5 mV–3 V.

Schematic diagram of the ultrasonic spray pyrolysis process

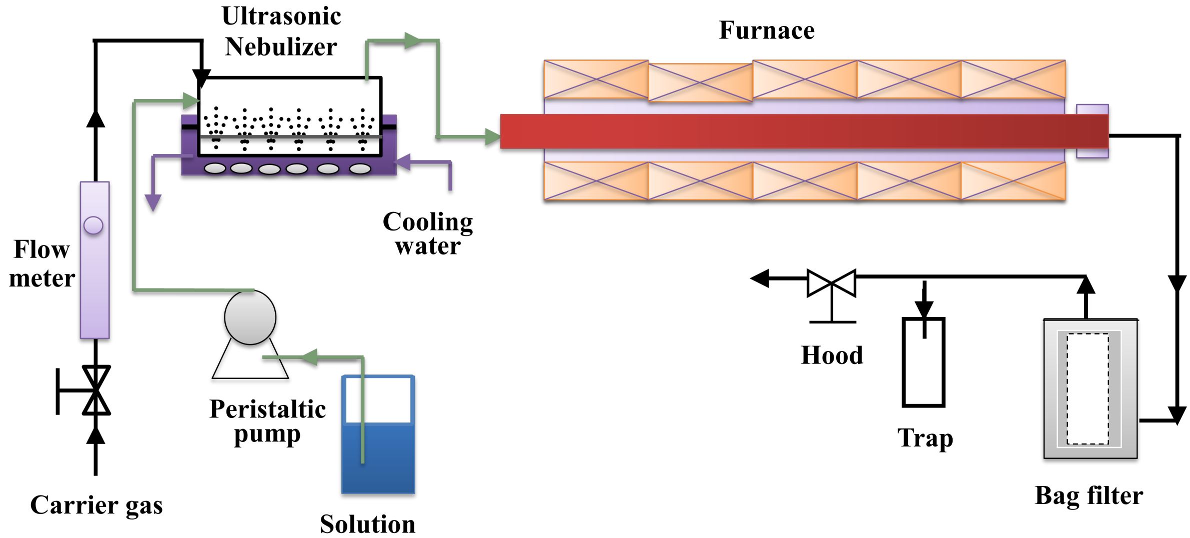

Fig. S1 Schematic diagram of the ultrasonic spray pyrolysis process.

Morphologies of the Fe_2O_3 yolk-shell prepared at various temperatures

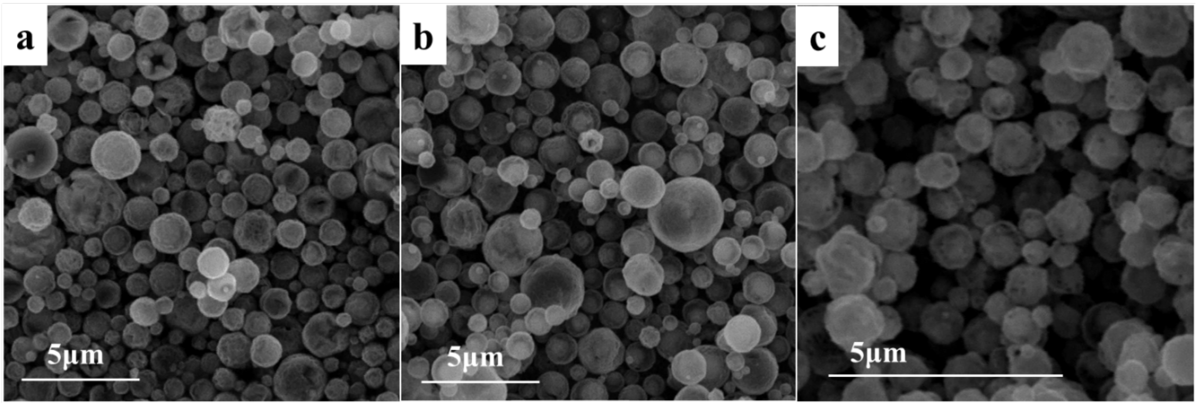

Fig. S2 SEM images of the Fe_2O_3 yolk-shell particles prepared by spray pyrolysis at various temperatures; (a) 600^{o}C, (b) 800^{o}C, (c) 1000^{o}C.

XRD patterns of the Fe_2O_3 particles

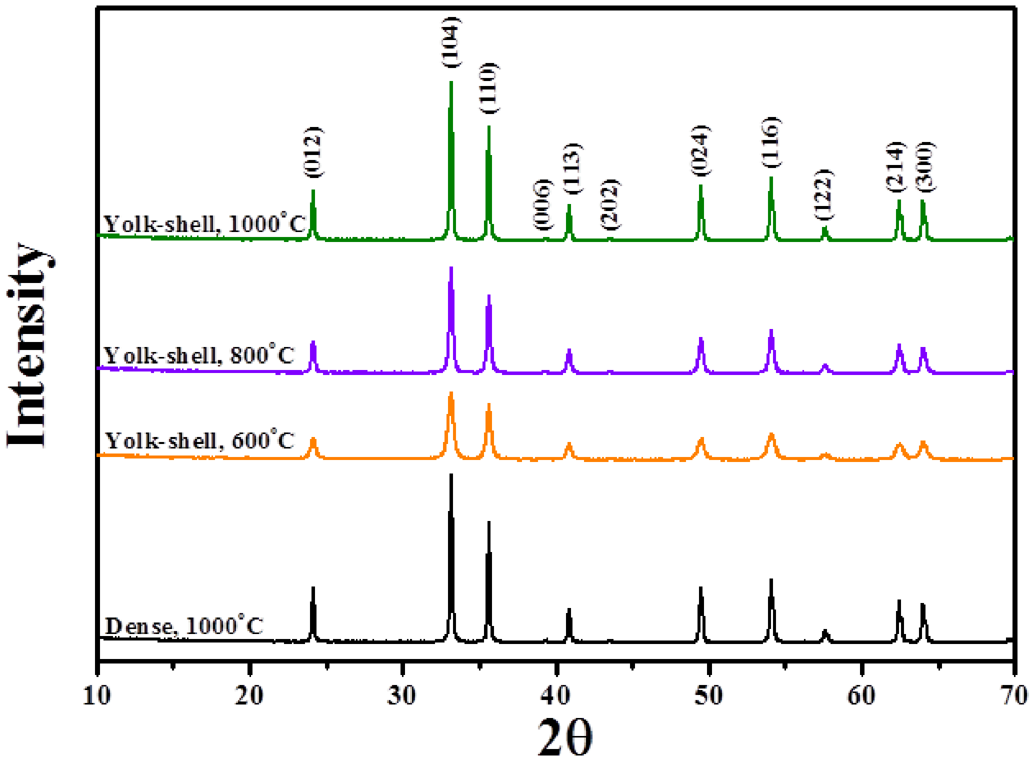

Fig. S3 XRD patterns of the yolk-shell and densely structured Fe_2O_3 particles prepared at various temperatures.

Enlarged TEM images of the Fe_2O_3 yolk-shell particles

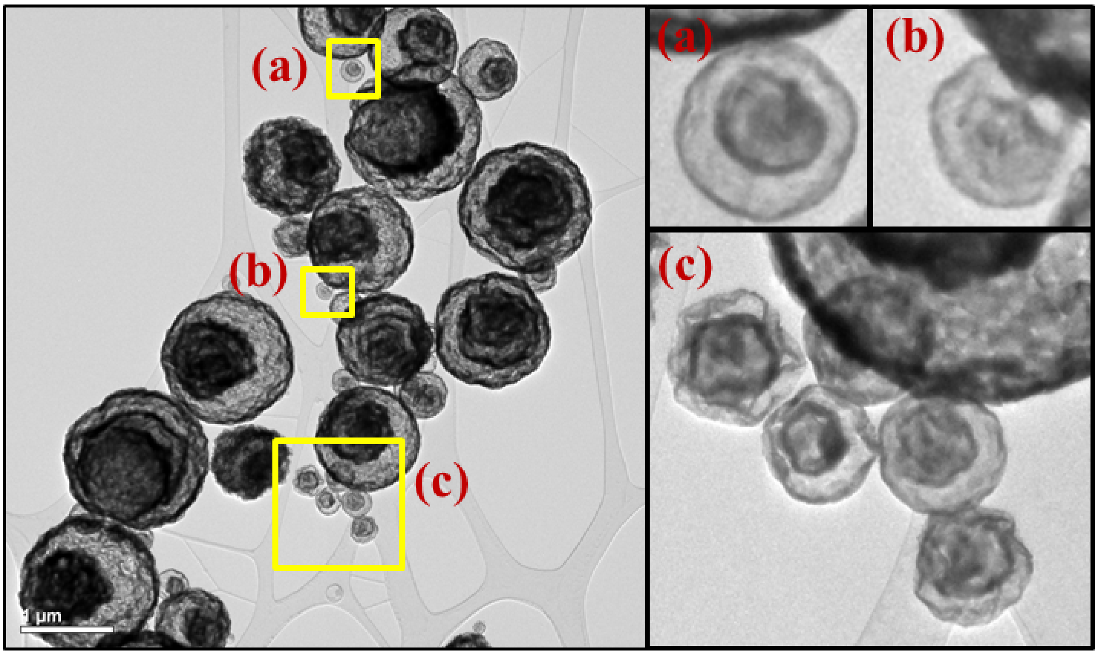

Fig. S4 Enlarged TEM images of the Fe_2O_3 yolk-shell particles prepared by spray pyrolysis from the spray solution with sucrose at 800^{o}C.

TG curve of the Fe_2O_3 yolk-shell particles

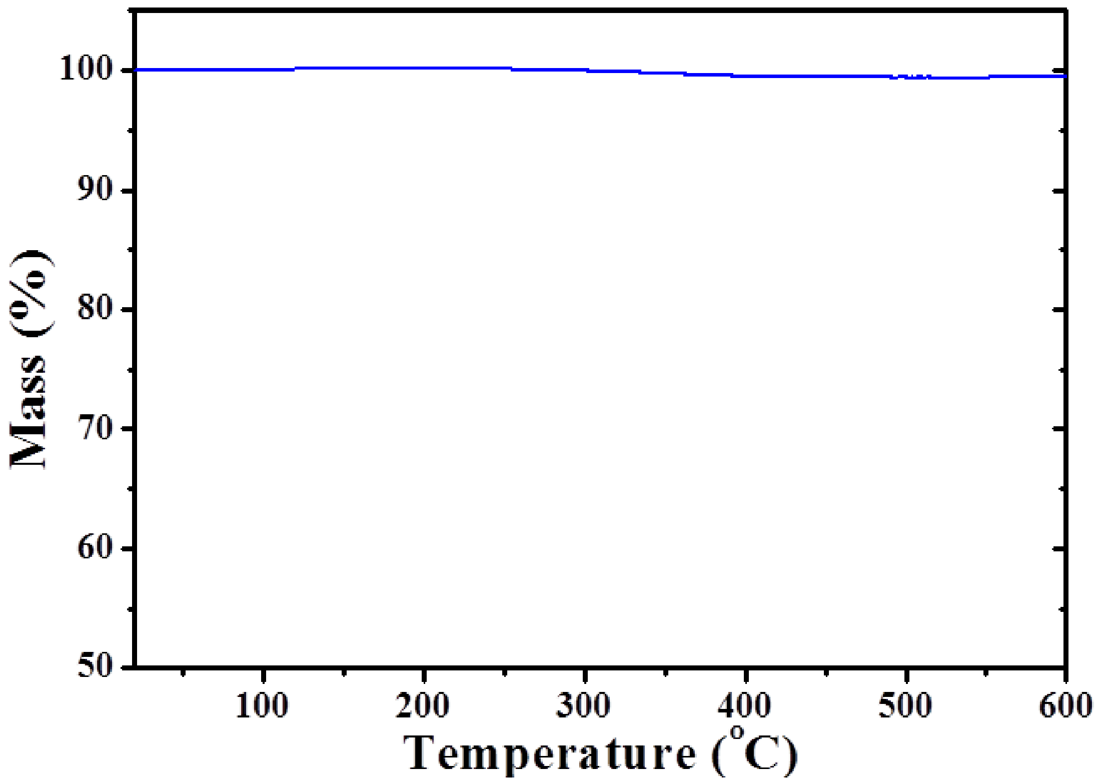

Fig. S5 Thermogravimetric curve of the Fe_2O_3 yolk-shell particles prepared by spray pyrolysis at 1000°C.

Cycle profiles of the Fe_2O_3 yolk-shell particles prepared at 1000°C

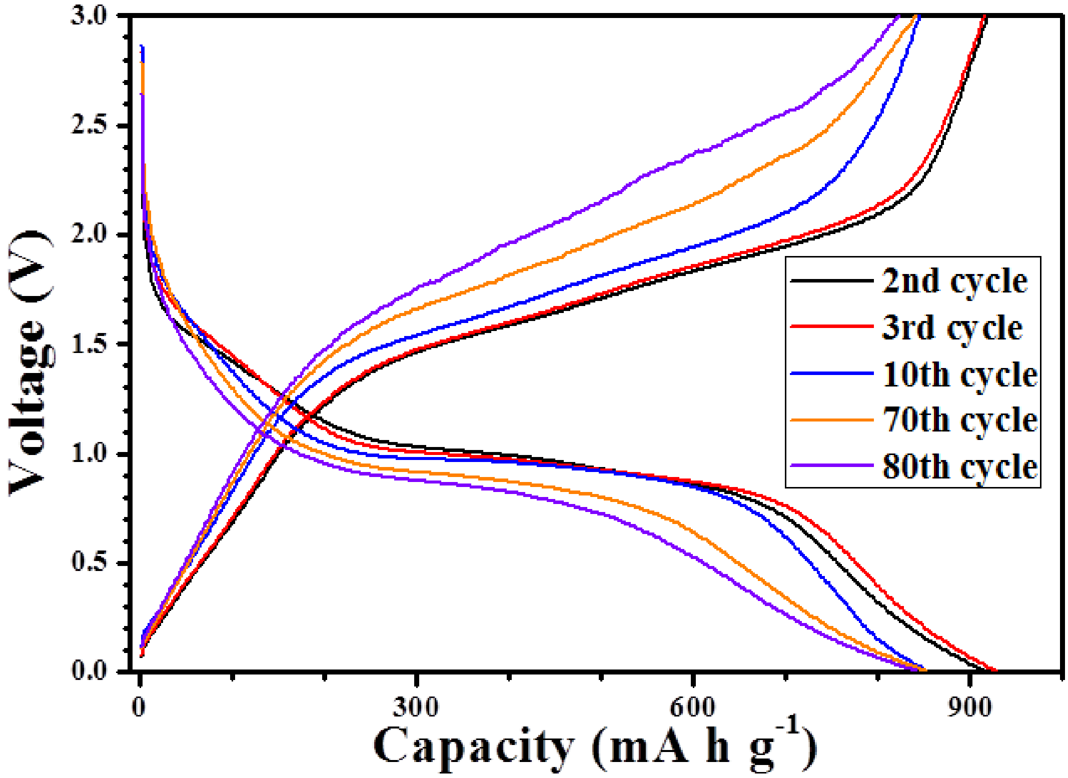

Fig. S6 Charge and discharge curves of the Fe_2O_3 yolk-shell particles prepared at 1000°C cycled at 300 mA g^{-1} in the voltage ranges of 0.005-3.0V.

Coulombic efficiencies of the Fe_2O_3 particles

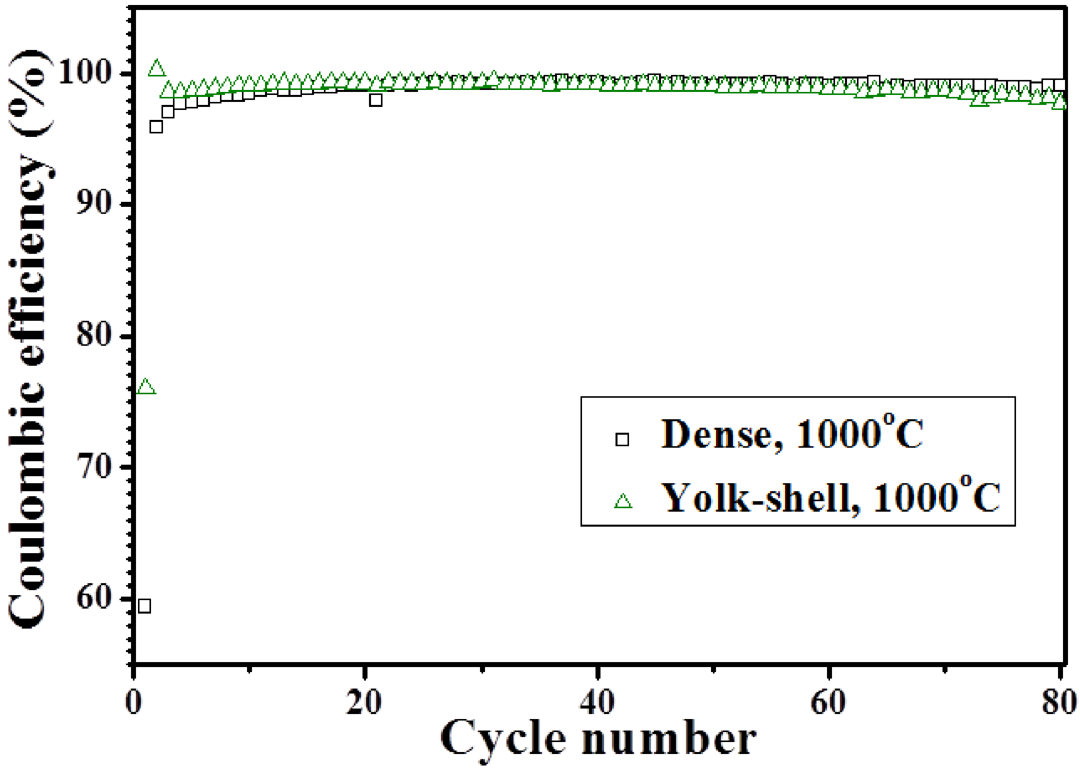

Fig. S7 Coulombic efficiencies of the Fe_2O_3 particles prepared by spray pyrolysis from the spray solutions with and without sucrose. (Cycled at 300 mA g^{-1} in the voltage ranges of 0.005-3.0V)

Cycling and Coulombic efficienciency of the Fe_2O_3 yolk-shell particles prepared at 1000°C

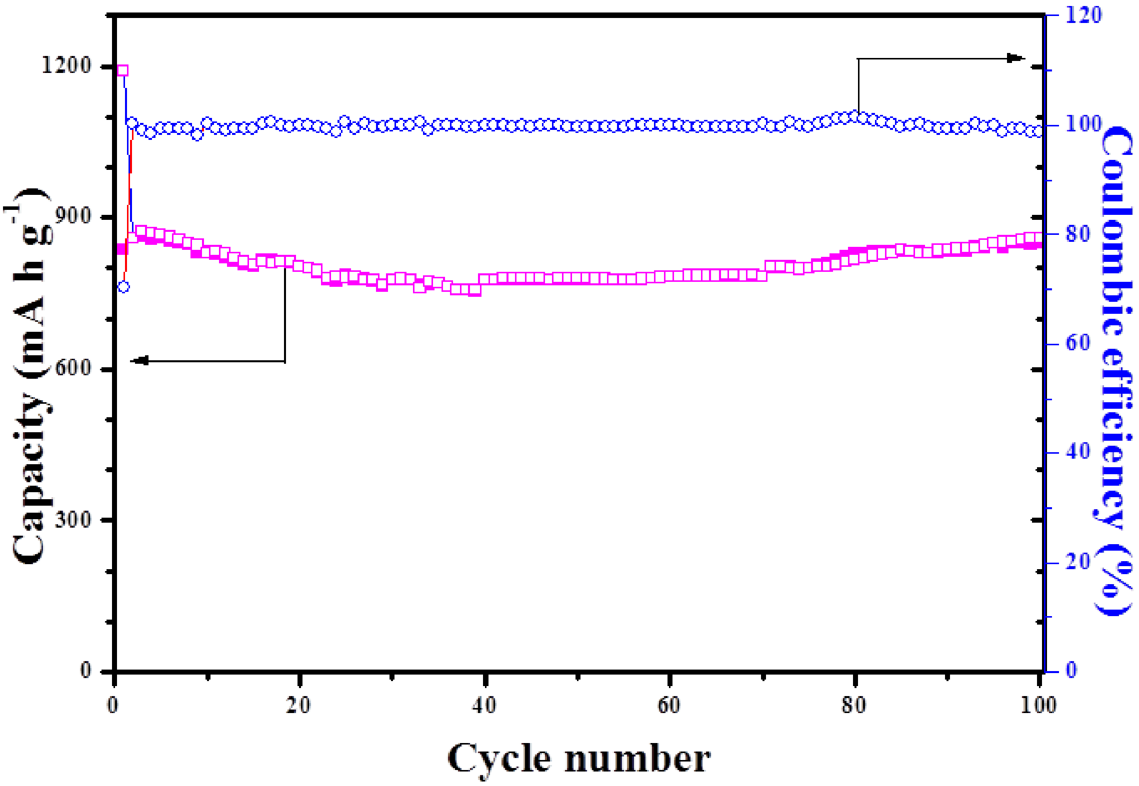

Fig. S8 Cycling and Coulombic efficienciency of the Fe_2O_3 yolk-shell particles prepared at 1000°C at a high current density of 1000 mA g^{-1}.