Electronic Supplementary Material (ESI) for Nanoscale.

SUPPLEMENTARY INFORMATION

Amorphous iron oxide-selenite composite microspheres with yolk-shell structure as highly efficient anode material for lithium-ion batteries

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Experimetal Section

1. Materials characterization

The morphologies of the prepared microspheres were investigated using scanning electron microscopy (SEM, VEGA3) and transmission electron microscopy (TEM, JEM-2100F). The crystal structures and chemical properties of the prepared microspheres were analyzed using X-ray diffraction spectroscopy (XRD, X'pert PRO with Cu K□ radiation, □□□ 1.5418 Å) at the Korea Basic Science Institute, Daegu, and X-ray photoelectron spectroscopy (XPS, Thermo Scientific™, K-Alpha™). Ex-situ XPS analyses at the first fully discharged and charged states after argon-ion gun etching were also performed with the above measuring equipment. The properties and the amount of pitch derived carbon was characterized via thermogravimetric analysis (TGA, Pyris 1 Thermogravimetric Analyzer, PerkinElmer) in the range of 25–700 ^{o}C at 10 ^{o}C min^{-1} in an air-based atmosphere, respectively. The surface area and porosities of samples were analyzed using the Brunauer–Emmett–Teller (BET) method with high-purity N_2.

2. Electrochemical measurements

The electrochemical properties of the the prepared microspheres were analyzed using a 2032-type coin cell. The anode was prepared by mixing the active material, carbon black, and sodium carboxymethyl cellulose in a weight ratio of 7:2:1. Lithium metal and microporous polypropylene films were used as counter electrode and separator, respectively. The electrolyte was 1.0 M $LiPF_6$ dissolved in a mixture of fluoroethylene carbonate−dimethyl carbonate (FEC/DMC; 1:1 v/v). The discharge and charge characteristics of the samples were investigated by cycling in the potential range of 0.001−3.0 V at various current densities. Cyclic voltammograms (CVs) were measured at a scan rate of 0.1 mV s^{-1}. Electrochemical impedance spectroscopy (EIS) was performed on the electrode over a frequency range of 0.01–100 kHz. In-situ EIS analysis was performed at preselected potentials during the discharge and charge processes at a current density of 0.1 A g^{-1}.

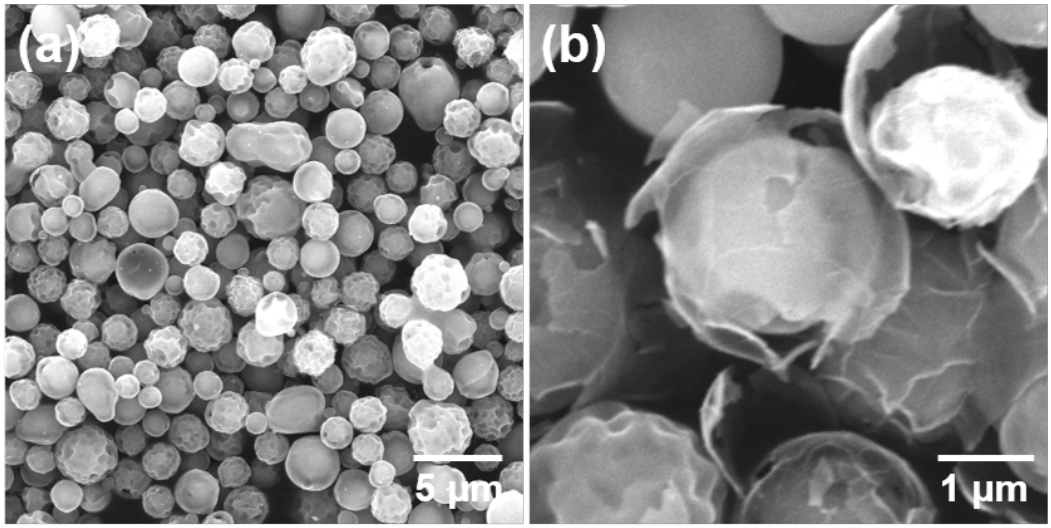


Fig. S1. Morphologies of yolk-shell structured c-Fe_2O_3 microspheres.

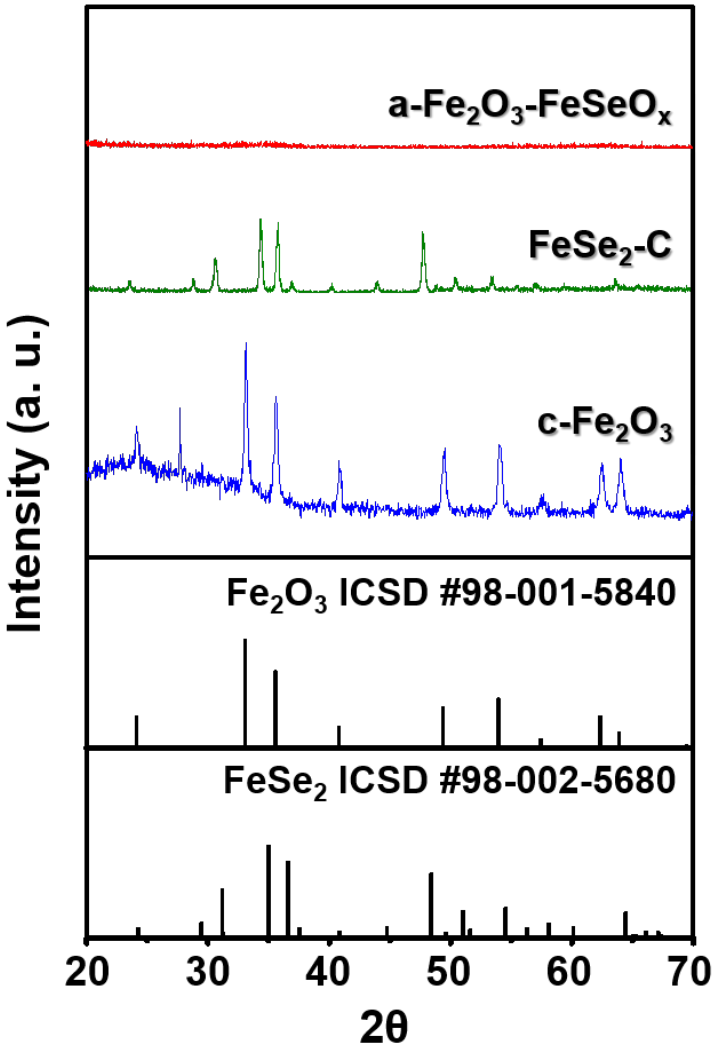


Fig. S2. XRD patterns of c-Fe_2O_3, $FeSe_2$-C, and a-Fe_2O_3-$FeSeO_x$ microspheres.

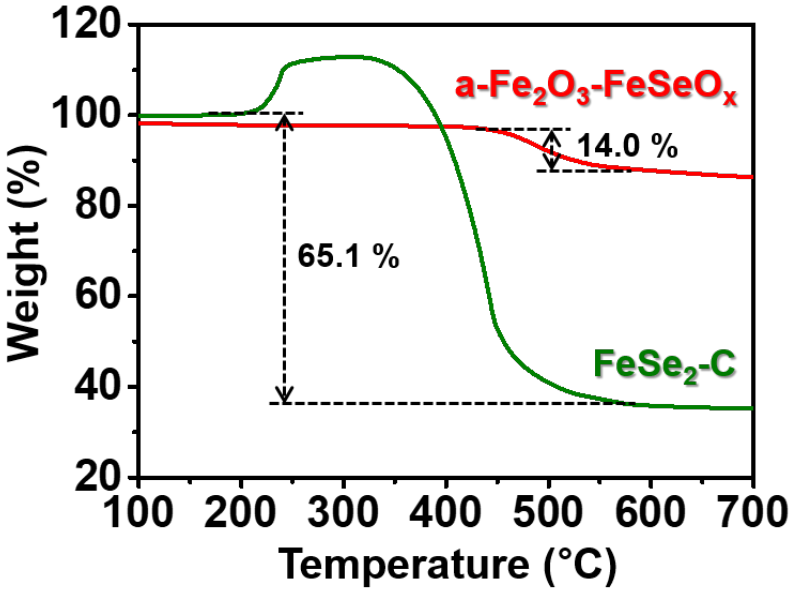


Fig. S3. TGA curves of $FeSe_2$-C and a-Fe_2O_3-$FeSeO_x$ microspheres.

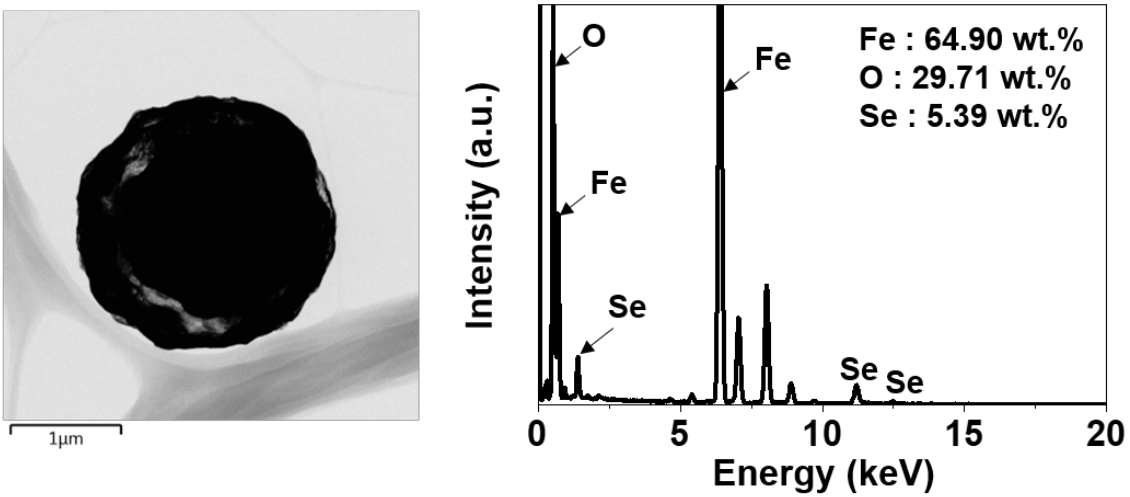


Fig. S4. EDX spectrum of a-Fe_2O_3-$FeSeO_x$ microspheres.

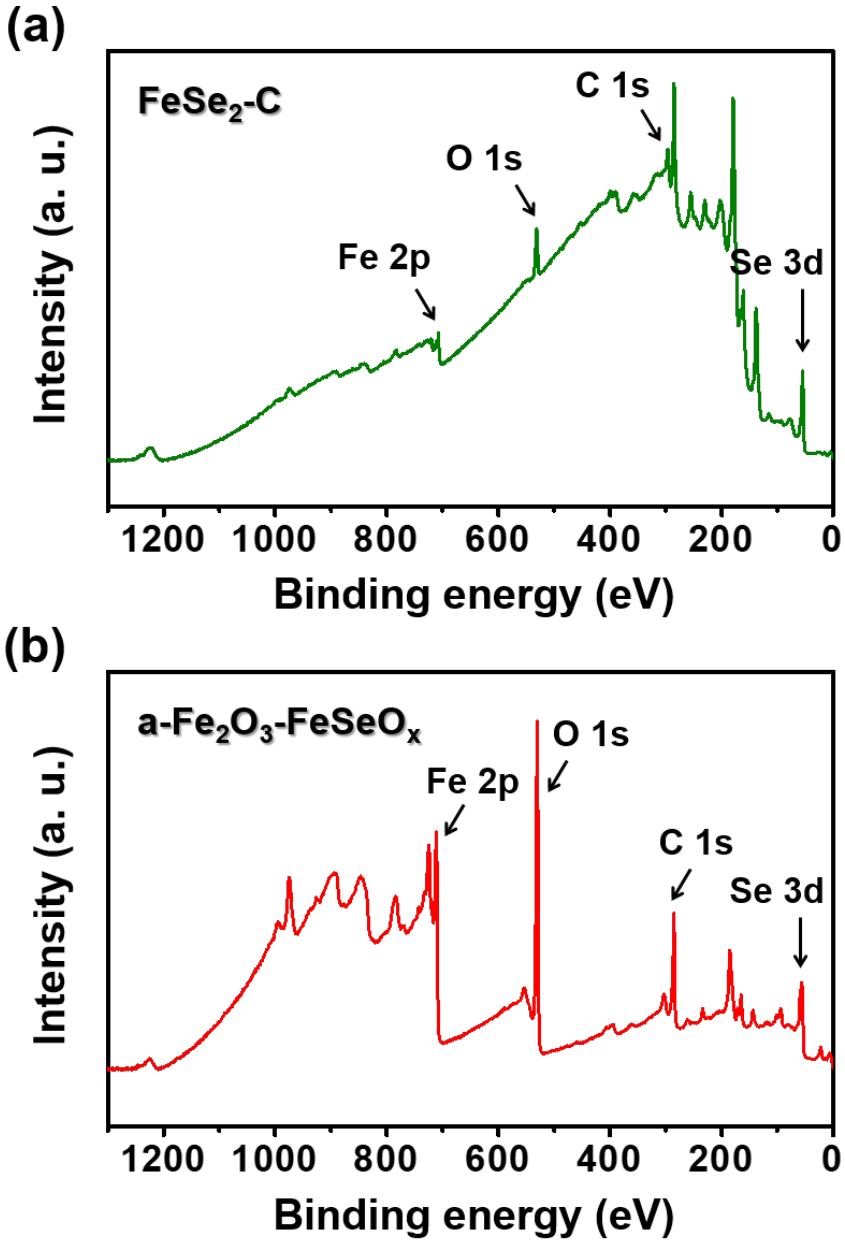


Fig. S5. XPS survey scans of $FeSe_2$-C and a-Fe_2O_3-$FeSeO_x$ microspheres.

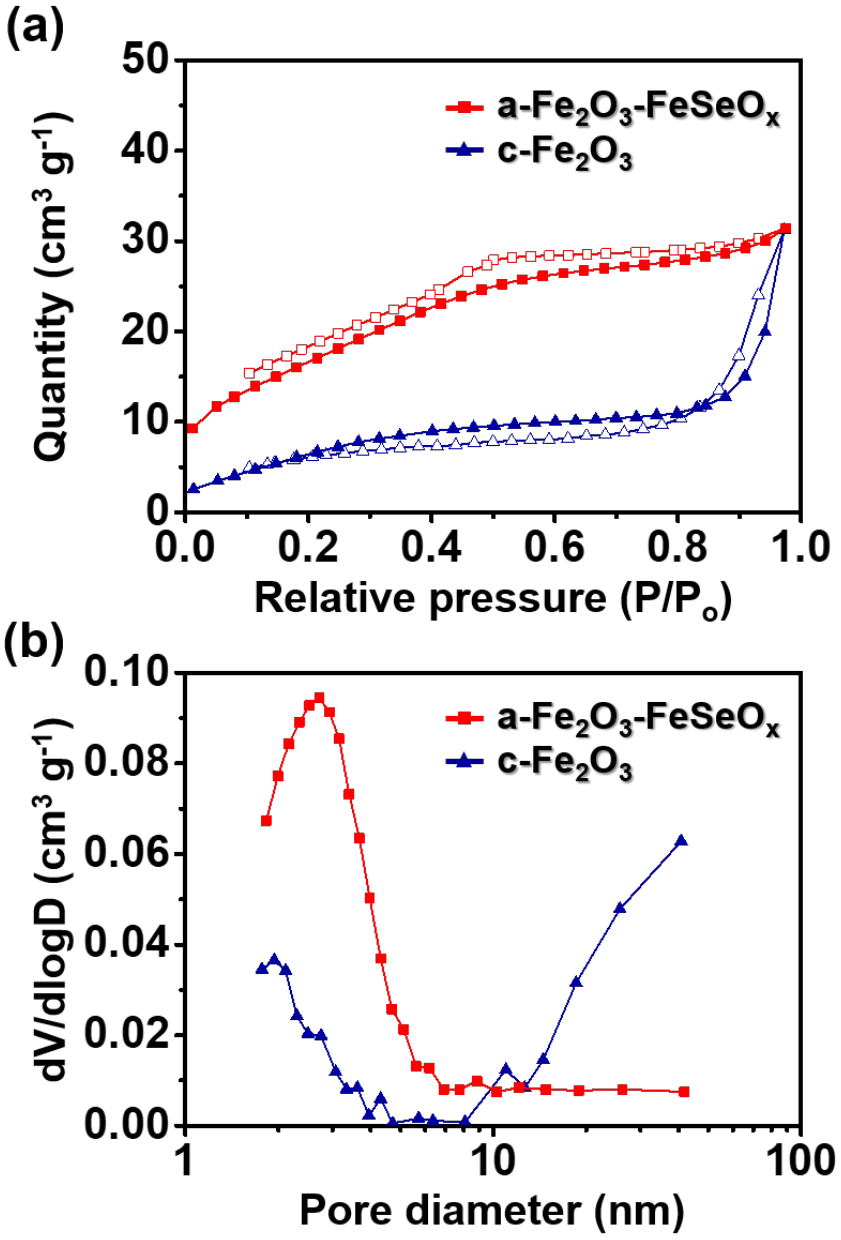
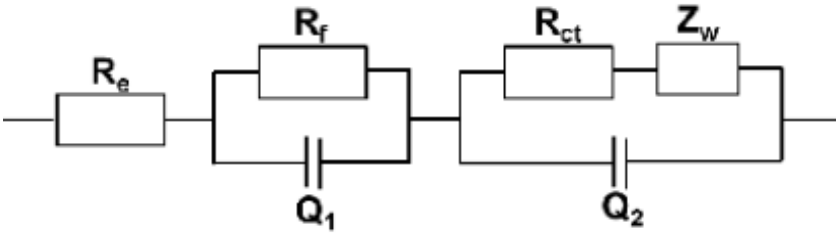


Fig. S6. (a) N_2 gas adsorption-desorption isotherm curves and (b) BJH pore size distributions of c-Fe_2O_3 and a-Fe_2O_3-$FeSeO_x$ microspheres.



R_e : the electrolyte resistance, corresponding to the intercept of high frequency semicircle at Z_{re} axis

R_f : the SEI layer resistance corresponding to the high-frequency semicircle

Q_1 : the dielectric relaxation capacitance corresponding to the high-frequency semicircle

R_{ct}: the denote the charger transfer resistance related to the middle-frequency semicircle

Q_2 : the associated double-layer capacitance related to the middle-frequency semicircle

Z_w : the Li-ion diffusion resistance

Fig. S7. Equivalent circuit model used for AC impedance fitting.

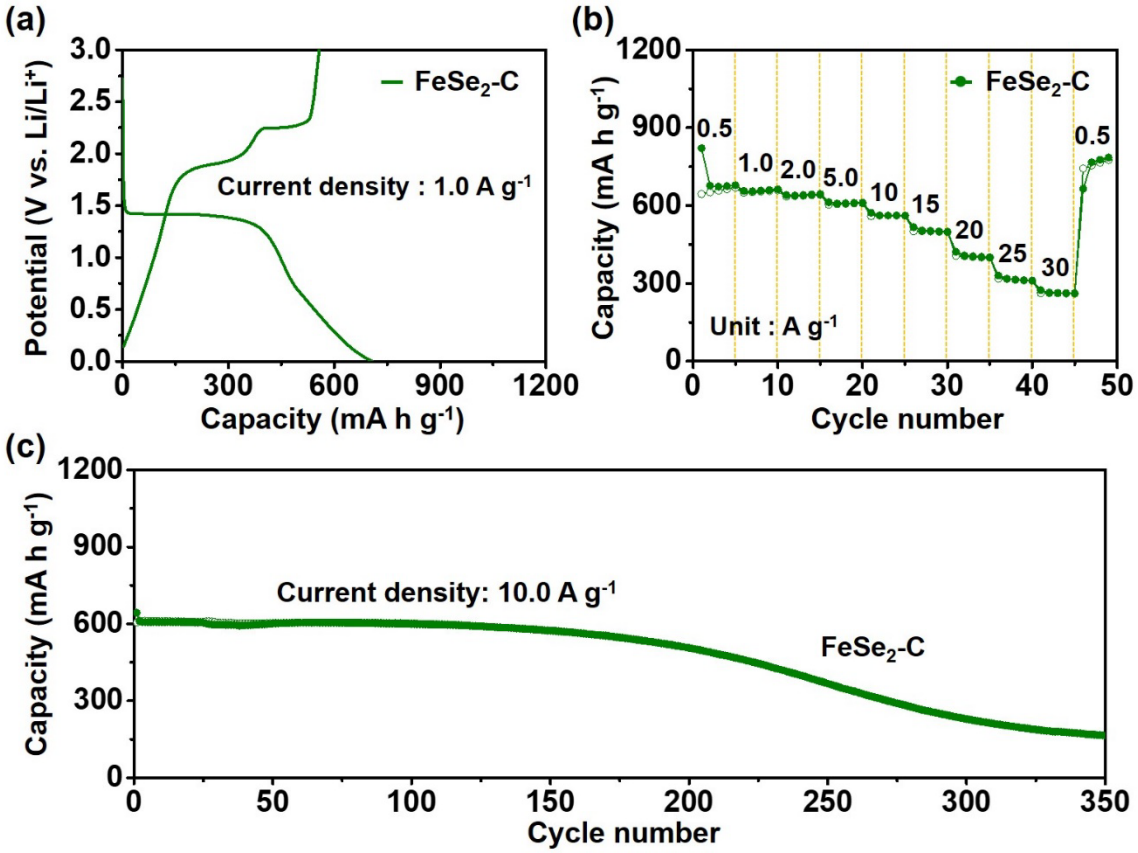


Fig. S8. Electrochemical properties of $FeSe_2$-C electrodes: (a) first galvanostatic discharge-charge profile, (b) rate performance, (c) cycling performance at a current density of 10 A g^{-1}.

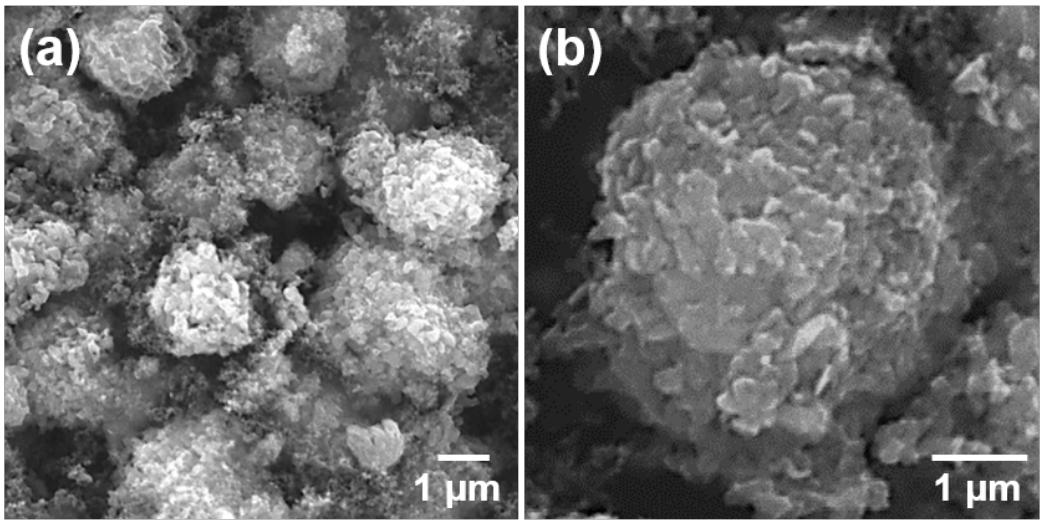


Fig. S9. SEM images of a-Fe_2O_3-$FeSeO_x$ microspheres obtained after 100 cycles.

Materials	Fe [wt%]	O [wt%]	Se [wt%]
a-Fe_2O_3-$FeSeO_x$	**65.40**	**29.12**	**5.48**

Table S1. Compostions of the a-Fe_2O_3-$FeSeO_x$ microspheres determined from ICP-OES analysis.