

Supplementary Information for

Facile large scale preparation and electromagnetic properties of silica-nickel-carbon composite shelly hollow microspheres

Zhenguo An,* Jingjie Zhang*

Technical Institute of Physics and Chemistry, Chinese Academy of Sciences, Beijing
100190 China.

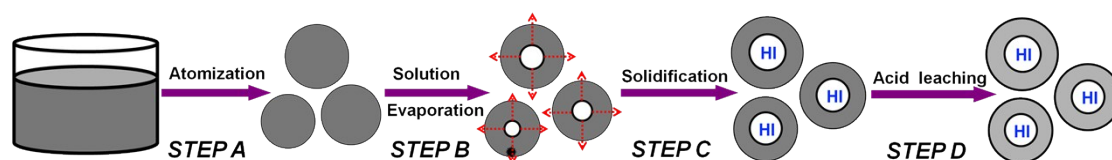


Fig. S1 Schematic illustration of the formation process of the single shell silica hollow microspheres (SHMs), HI stands for the hollow interior of the microspheres.

The stepwise fabrication process of the CSHMs is schematically shown in Figure S1. In the preparation of single shell silica hollow microspheres (SHMs), precursor microspheres were firstly prepared by spray drying of water glass (steps A to C in Figure S1). Liquid droplets with different diameters were formed through atomization by a centrifugal atomizer and then went into the drying chamber immediately. These droplets tend to form a spherical shape by surface tension (to minimize the surface energy). ~~When the droplets~~ went into the drying chamber with relatively high

* To whom correspondence should be addressed. Technical Institute of Physics and Chemistry, Chinese Academy of Sciences, Beijing 100190 China. Tel/fax: +86 10 82543690.

E-mail address: zgan@mail.ipc.ac.cn (ZG An); jjzhang@mail.ipc.ac.cn (JJ Zhang)

temperature, a dry shell was formed at first on the surface of the droplet by fast evaporation of water. As the evaporation process went on, the liquid water glass in the inner part of the droplets transferred to the preformed dry shell and transpired, leaving the solute to deposit on the dry shell.¹⁻² This process continues till all the water in the droplets is evaporated and precursor microspheres (composed of Na_2SiO_3 and SiO_2 , see TABLE S1) with a hollow interior (HI) and certain shell thickness are obtained (see Fig. 2 (A)). It should be noted that a few of the droplets may adhere with each other randomly and failed to form bigger spherical droplets before surface solidification, leading to the existence of some irregular particles, as were marked by black arrows in Fig. 2 (A) and (B). In the acid leaching process, the Na_2SiO_3 component in the precursor microspheres reacted with HCl to form SHMs with the hollow spherical shape preserved according to the following reaction:

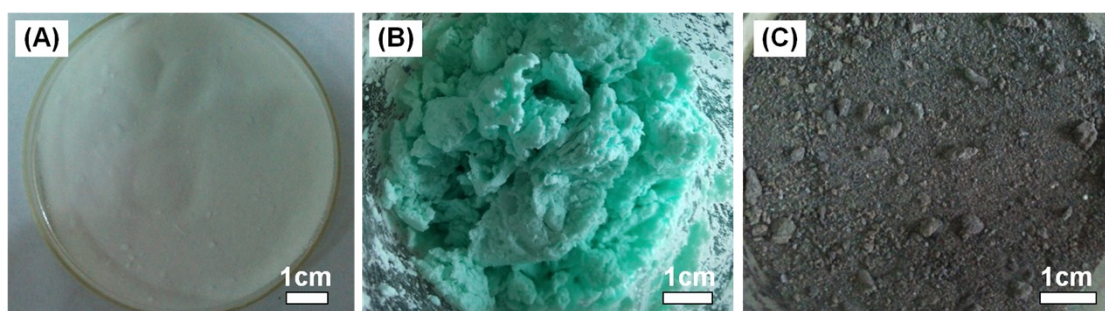
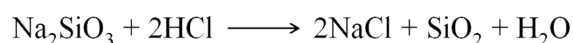


Fig. S2 Photographs of the products at different reaction stages: (a) SHMs, (b) the blue-green cream-like mixture (precursor mixture) before calcination reaction, (c) the as-obtained silica-nickel-carbon CSHMs (S2).

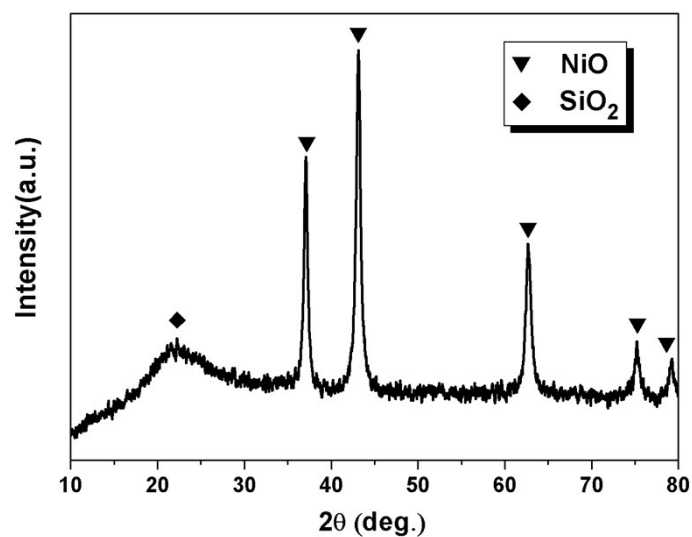


Fig. S3 XRD patterns of the silica-nickel oxide CSHMs obtained by heating treatment of the precursor mixture in air atmosphere.

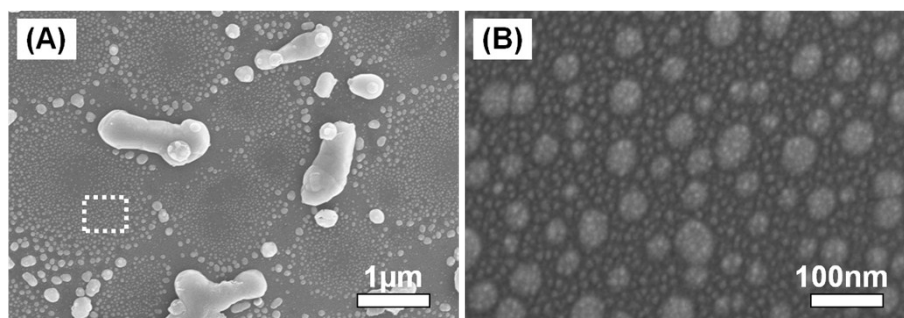


Fig. S4 SEM images of the silica-nickel CSHMs obtained by reduction of the silica-nickel oxide CSHMs (S2) by hydrogen.

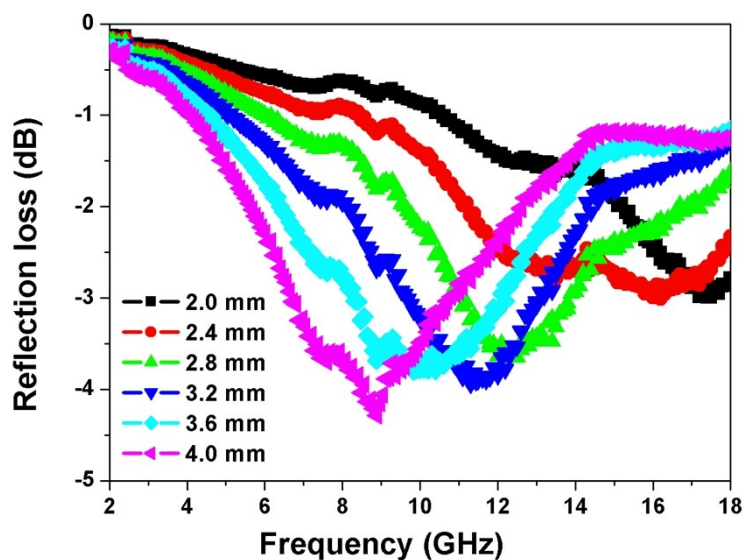


Fig. S5 Microwave reflection losses of the as-obtained SHMs at different absorber layer thicknesses.

TABLE S1. Mole percentages of various components in different samples.

Samples	Components	SiO ₂ / mol%	Na ₂ O / mol%	Ni / mol%	C / mol%	Impurities / mol%
Precursor microspheres		77.5	21.6	—	—	0.9
Silica SHMs		99.6	0.04	—	—	0.36
CSHMs (S1)		50.7	—	48.9	—	0.4
CSHMs (S2)		14.3	—	14.1	71.3	0.3

References

1. P. Walzel, *Chem. Eng. Technol.*, 2011, **34**, 1039.
2. A. B. D. Nandiyanto, K. Okuyama, *Adv. Powder Technol.*, 2011, **22**, 1.