Supplementary information

Eco-friendly, catalyst-free synthesis of highly pure carbon spheres using vegetable oils as renewable source and their application as a template for ZnO and MgO hollow spheres

Sandesh Y. Sawant[a,b], S. Senthilkumar[a] , Rajesh S. Somani[a]*, Moo Hwan Cho[b]* and Hari C. Bajaj[a]*

[a]Discipline of Inorganic Materials and Catalysis,

Central Salt & Marine Chemicals Research Institute,

Council of Scientific & Industrial Research (CSIR),

G. B. Marg, Bhavnagar 364002, Gujarat, India.

[b]School of Chemical Engineering, Yeungnam University,

Gyeongsan-si, Gyeongbuk 712-749, South Korea.

* Corresponding author: Tel: +91 278 2471793; Fax: +91 278 2567562; E-mail: rssomani@csmcri.org (Rajesh S. Somani); hcbajaj@csmcri.org (Hari C. Bajaj); Tel: +82-53-810-2517; Fax: +82-53-810-4631; E-mail: mhcho@ynu.ac.kr (Moo Hwan Cho)

The present address of Sandesh Y. Sawant is School of Chemical Engineering, Yeungnam University, Gyeongsan-si, Gyeongbuk 712-749, South Korea

1. Characterization of vegetable oils

1.1 Estimation of acid value and % free fatty acids

2.0 g of oil was titrated against 0.1 N ethanolic KOH in 20 ml of ethanol: diethyl ether (1:1) mixture as solvent using phenolphthalein as indicator. Percentage free fatty acids (FFAs) were calculated using oleic acid as a factor.

1.2 Saponification value

2.0 g of oil was heated at 60 °C for 30 minutes in 25 ml of 0.5 N ethanolic KOH and titrated against 0.5 N hydrochloric acid using phenolphthalein indicator.

1.3 Fatty acid compositions

Fatty acid composition of vegetable oil was determined by Gas chromatography equipped with mass spectroscopy (GC-MS) (GCMS-QP 2010, Shimadzu) using helium as carrier gas with flame ionization detector and RTX-5 capillary column. 1 g of oil was converted to methyl ester using 0.1 g of NaOH in 15 ml of methanol at 50 °C for 5 h; and extracted with n-hexane before being injected into the GC.

Table S1

Chemical characteristics of the vegetable oils used for synthesis of the carbon spheres

Parameter	Jatropha oil	Groundnut oil	Coconut oil	Mustard oil	Cotton seed oil	Cotton seed oil (used)
Free fatty acid (mg/g)	6.27	0.63	0.34	0.51	0.08	0.33
Acid value (mg KOH/g)	12.54	1.27	0.68	1.02	0.16	0.66
Saponification value (mg KOH/g)	160	185	216	165	195	182

Table S2

Fatty acid compositions of the vegetable oils under study determined by GC-MS analysis

Fatty acid (x:y)	Jatropha oil	Groundnut oil	Coconut oil	Mustard oil	Cotton seed oil	Cotton seed oil (used)
Palmitic (16:0)	17.84	11.09	10.40	---	28.82	28.13
Stearic (18:0)	8.63	5.87	3.44	2.33	3.91	3.66
Arachidic (20:0)	0.42	2.09	---	---	0.44	0.47
Oleic (18:1)	32.19	73.84	7.88	16.92	18.35	16.99
Linoleic (18:2)	40.21	---	1.61	14.93	46.32	48.76
Gondoic (20:0)	---	1.38	---	4.94	---	---
Behenic (22:0)	---	3.75	---	2.45	0.22	0.28
Lignoceric (24:0)	---	1.98	---	---	---	0.14
Caproic (6:0)	---	---	0.45	---	---	---
Caprylic (8:0)	---	---	8.98	---	---	---
Capric (10:0)	---	---	5.38	---	---	---
Lauric (12:0)	---	---	41.95	---	---	---
Myristic (14:0)	---	---	19.91	2.52	1.11	0.98
Erucic (22:1)	---	---	---	53.86	---	---
Palmitoleic (16:1)	0.71	---	---	---	0.83	0.59
Nervonic (24:1)	---	---	---	2.05	---	---

x: total number of carbon atoms: y: number of unsaturations

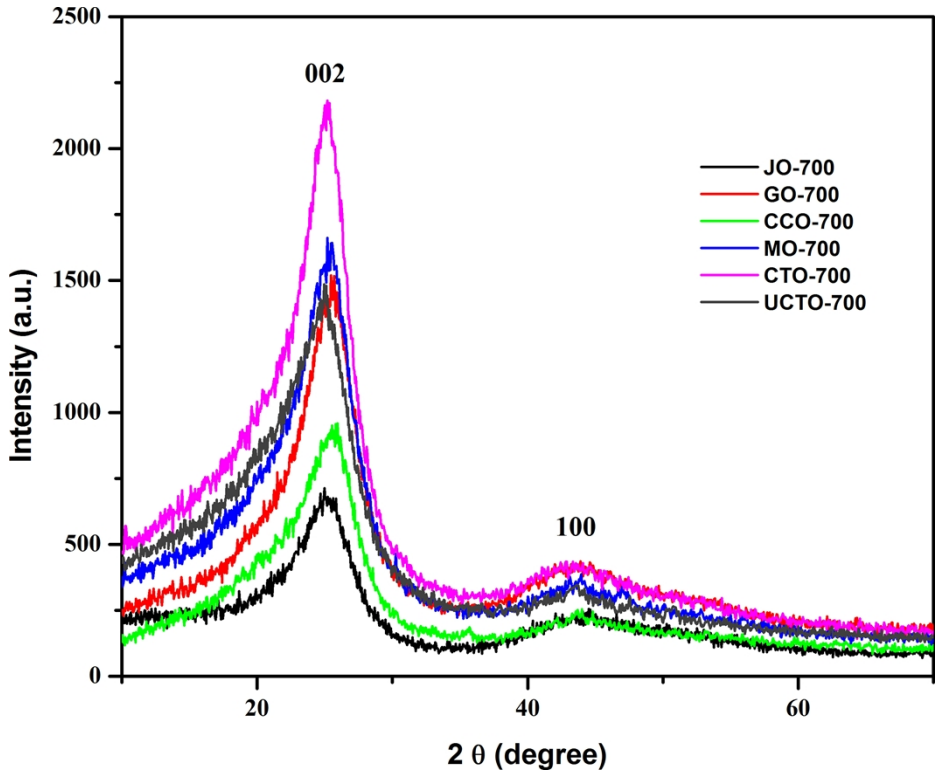

Fig. S1 X-ray diffraction pattern of the carbon spheres prepared using different vegetable oils at 700 °C for zero minute under autogenic pressure.

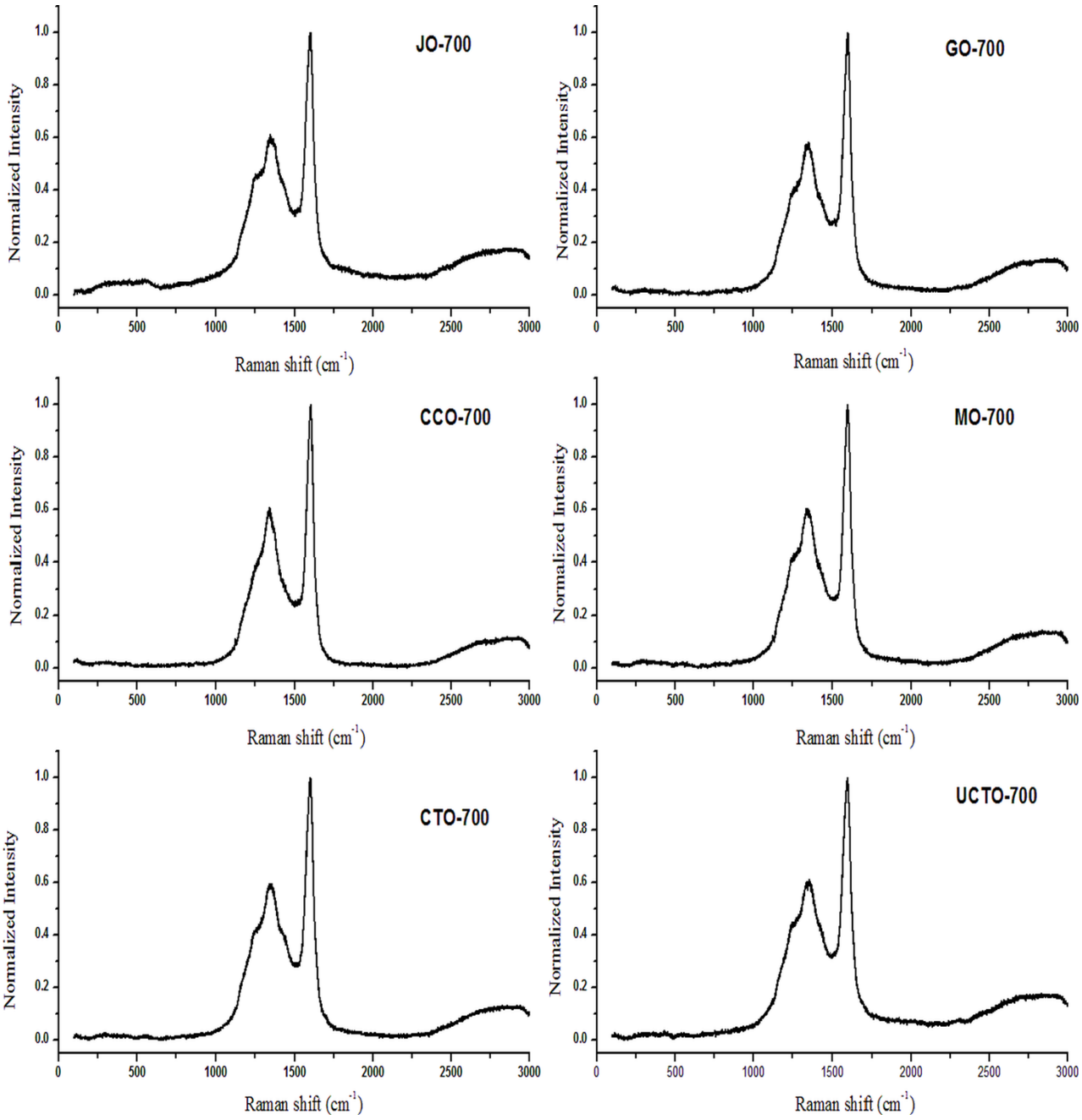

Fig. S2 FT-Raman spectrum of the carbon spheres prepared using different vegetable oils under optimized conditions.

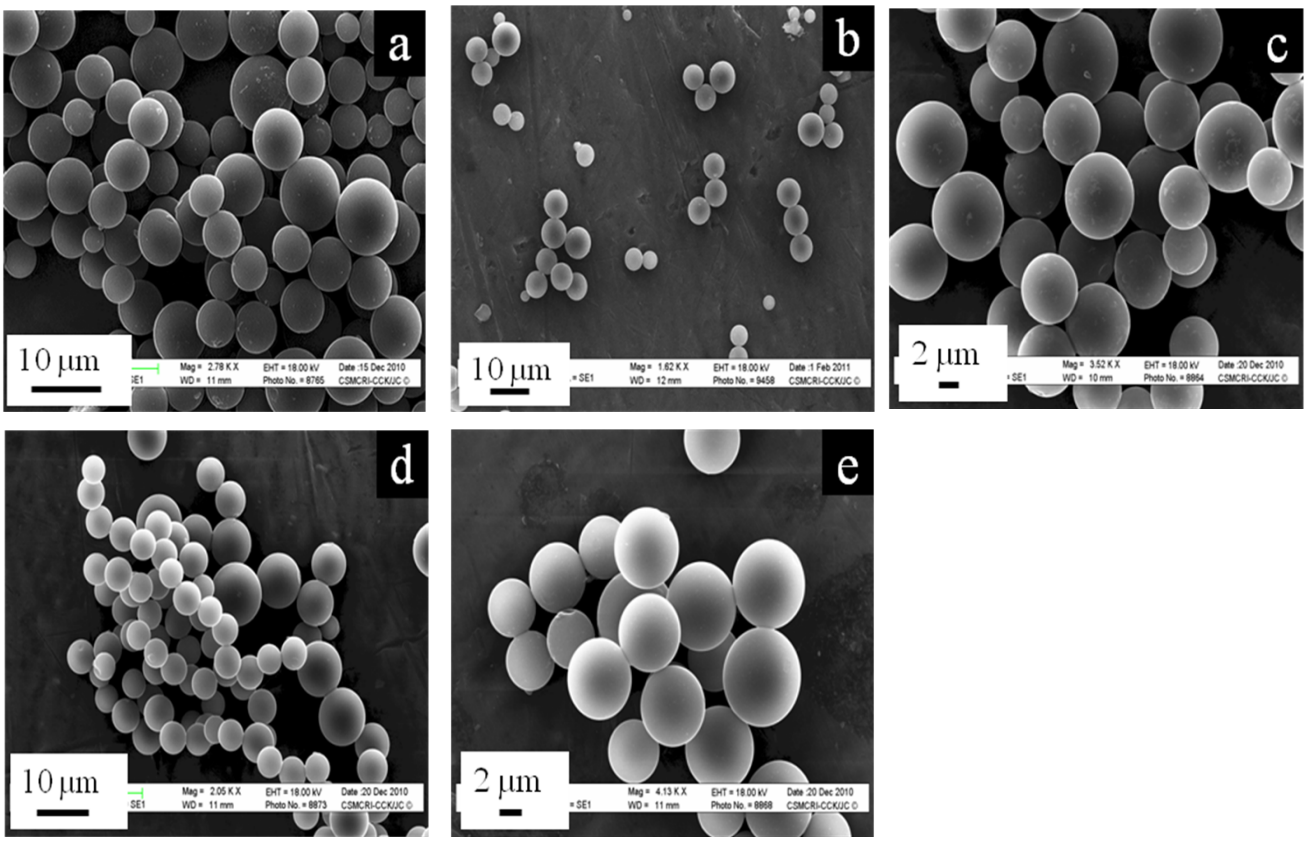

Fig. S3 SEM images of (a) GO-700, (b) CCO-700, (c) MO-700, (d) CTO-700 and (e) UCTO-700.

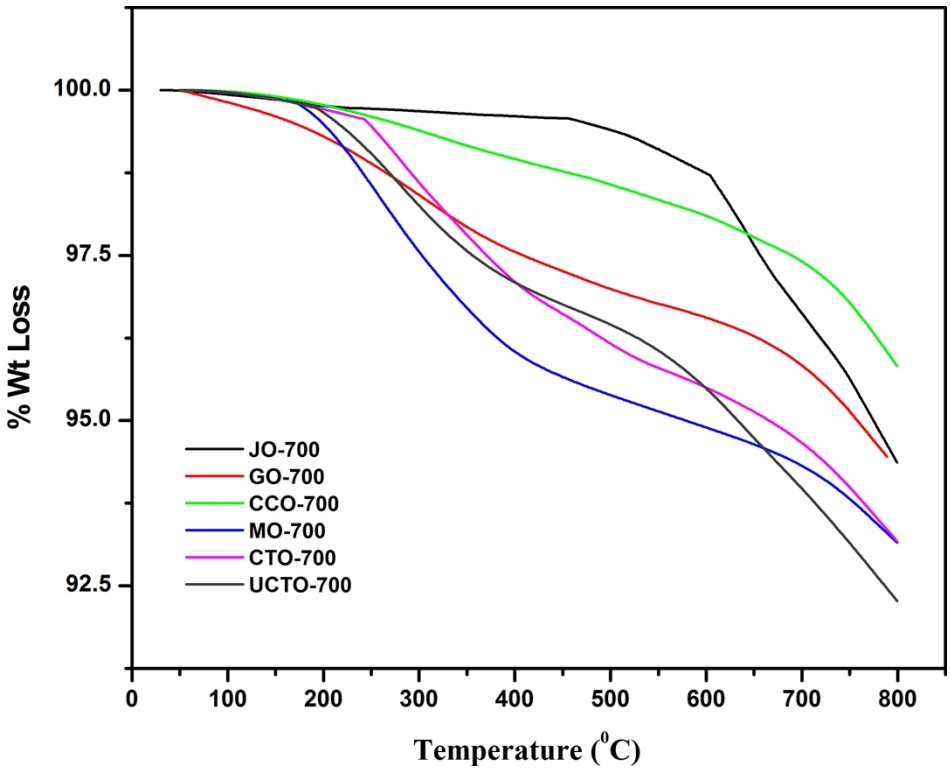

Fig. S4 TGA plot of the carbon spheres prepared using different vegetable oils.

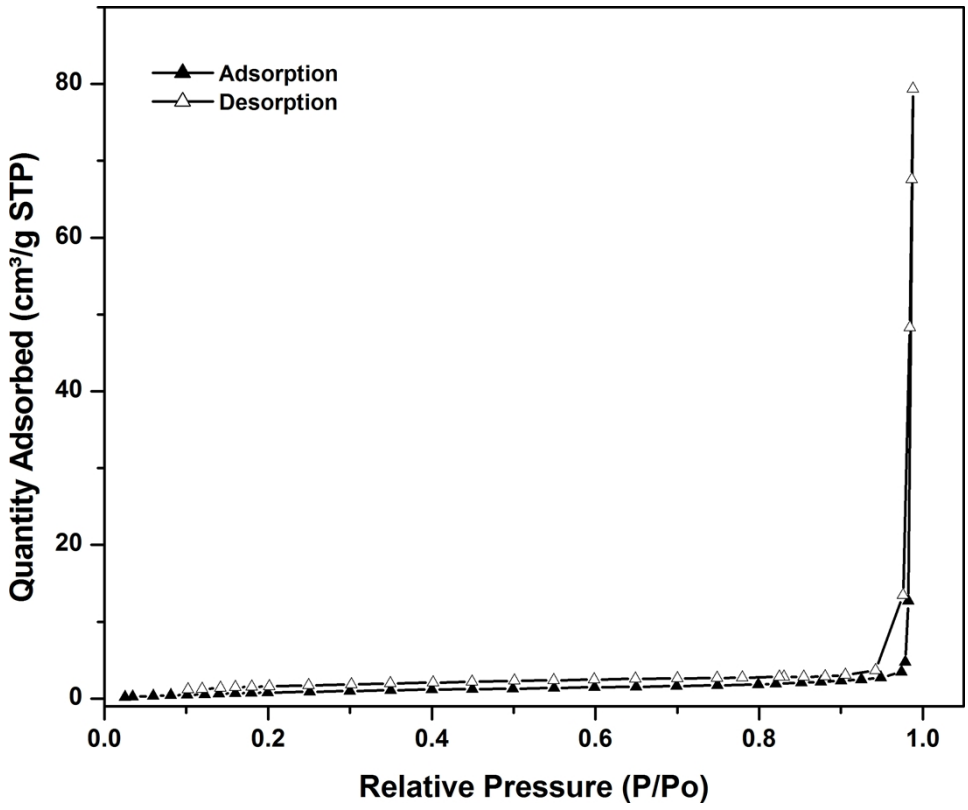

Fig. S5 N_2 Sorption isotherm of the carbon spheres prepared from jatropha oil as carbon source (JO-700) at 77 K.

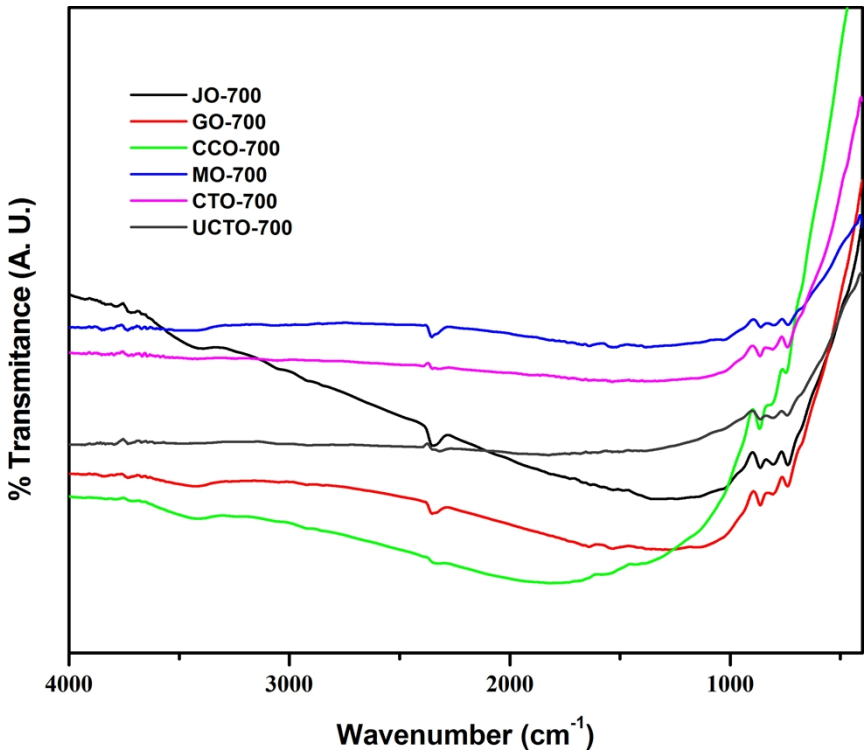

Fig. S6 FT-IR spectra of the carbon spheres prepared using different vegetable oils.

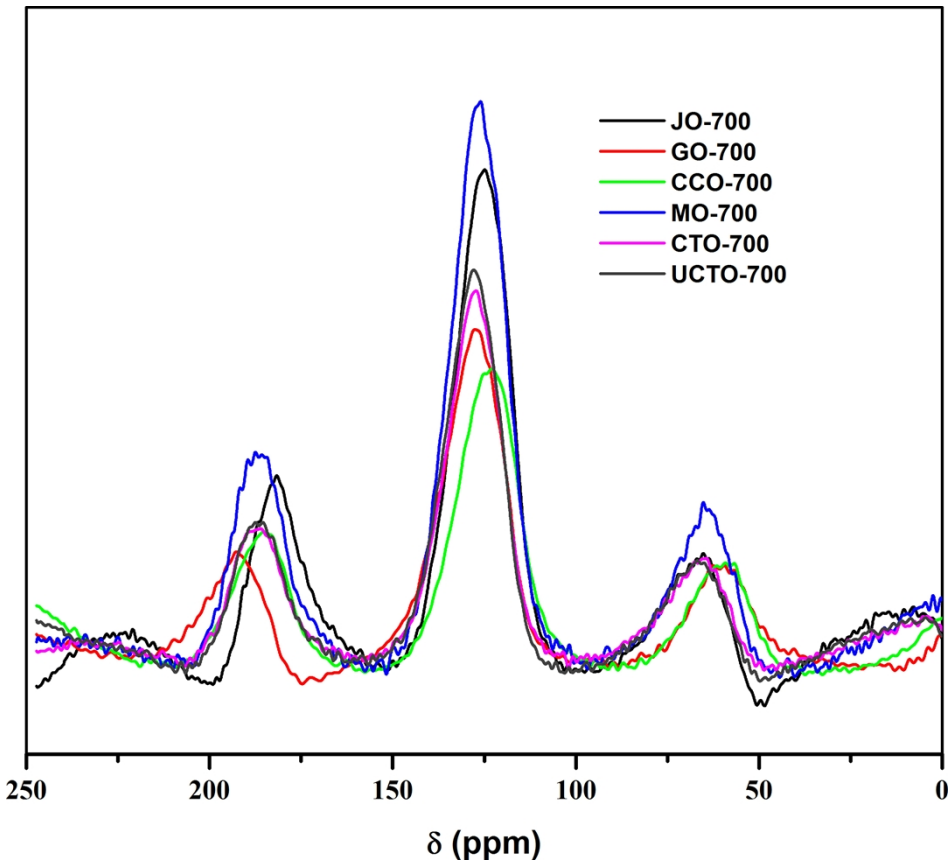

Fig. S7 ^{13}C CP-MAS NMR spectra of the carbon spheres prepared using different vegetable oils at 700 °C.

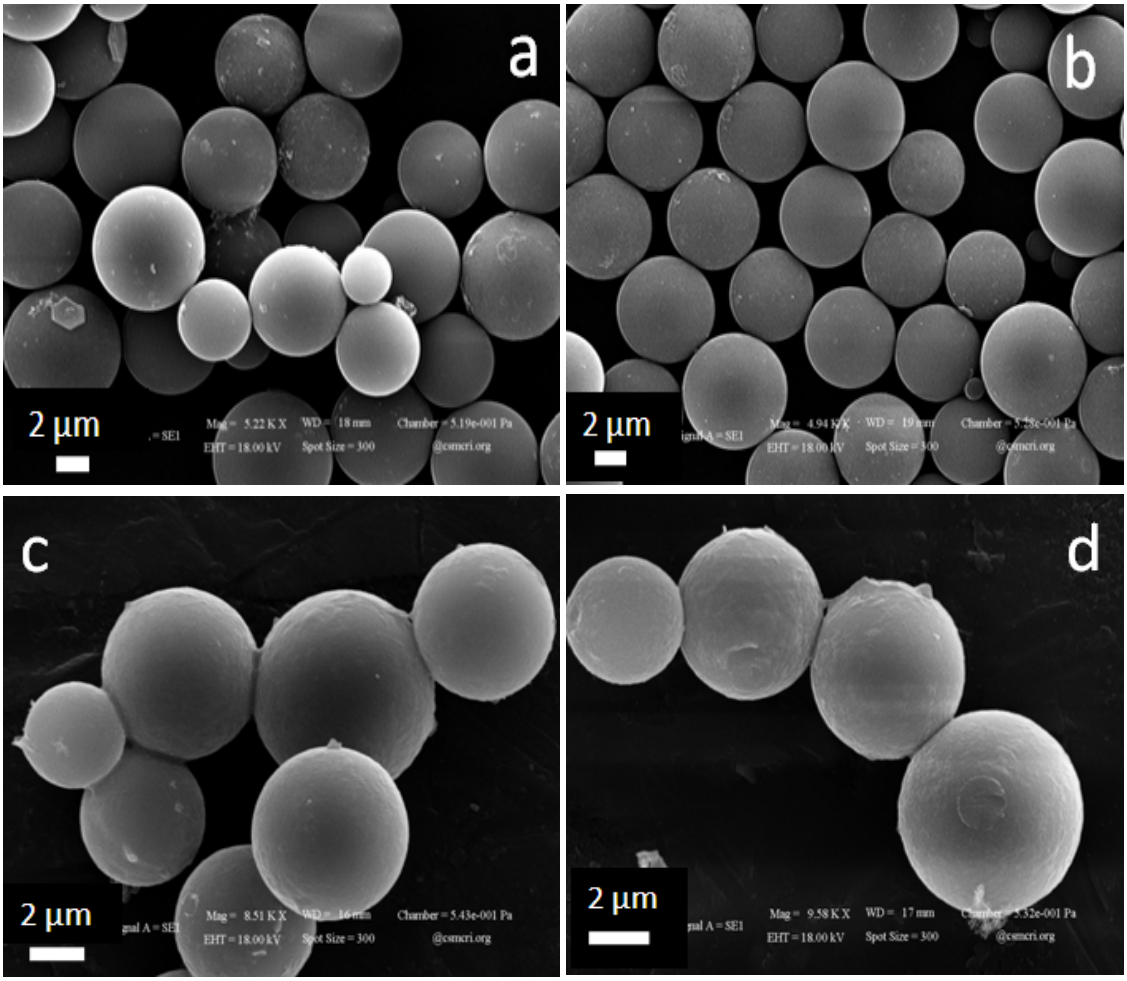

Fig. S8 SEM images of carbon spheres uniformly coated with zinc hydroxide and magnesium hydroxide prepared by (a) 0.05g $ZnCl_2$ (b) 0.1g $ZnCl_2$, (c) 0.05g $MgCl_2.6H_2O$ and (d) 0.1g $MgCl_2.6H_2O$.

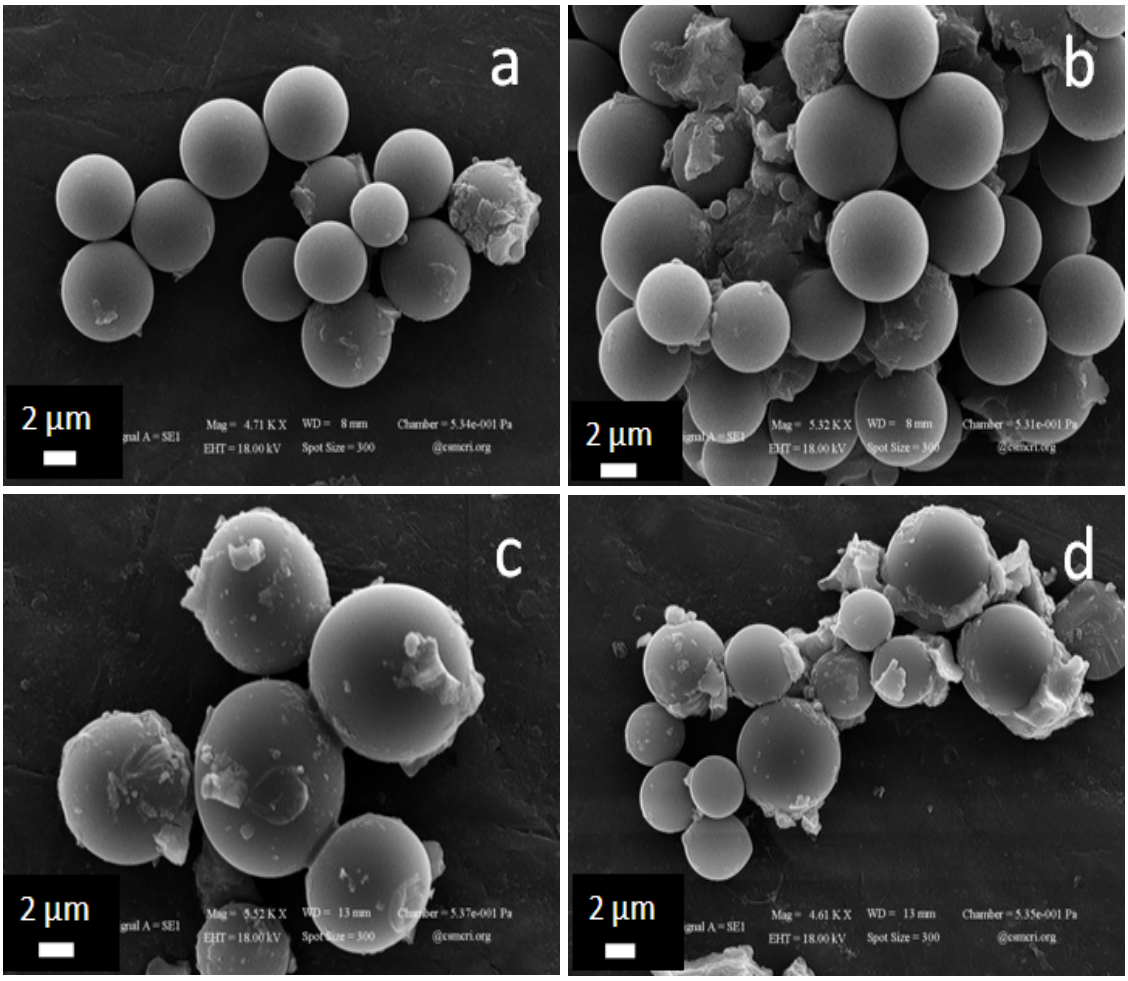

Fig. S9 SEM images of carbon spheres coated with zinc hydroxide and magnesium hydroxide prepared by (a) 0.15g $ZnCl_2$ (b) 0.2g $ZnCl_2$, (c) 0.15g $MgCl_2.6H_2O$ and (d) 0.2g $MgCl_2.6H_2O$.

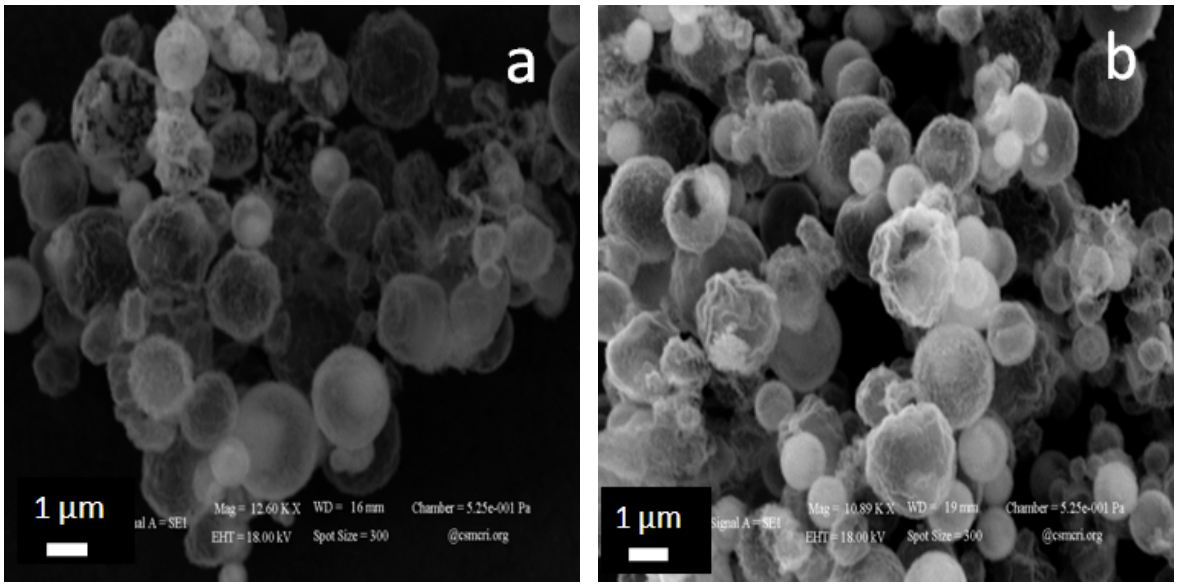

Fig. S10 SEM images of (a) ZnO and (b) MgO hollow sphere obtained by calcinations of carbon metal hydroxide core shell structure (0.1g of metal salt concentration) at 600 °C for 2 hr.

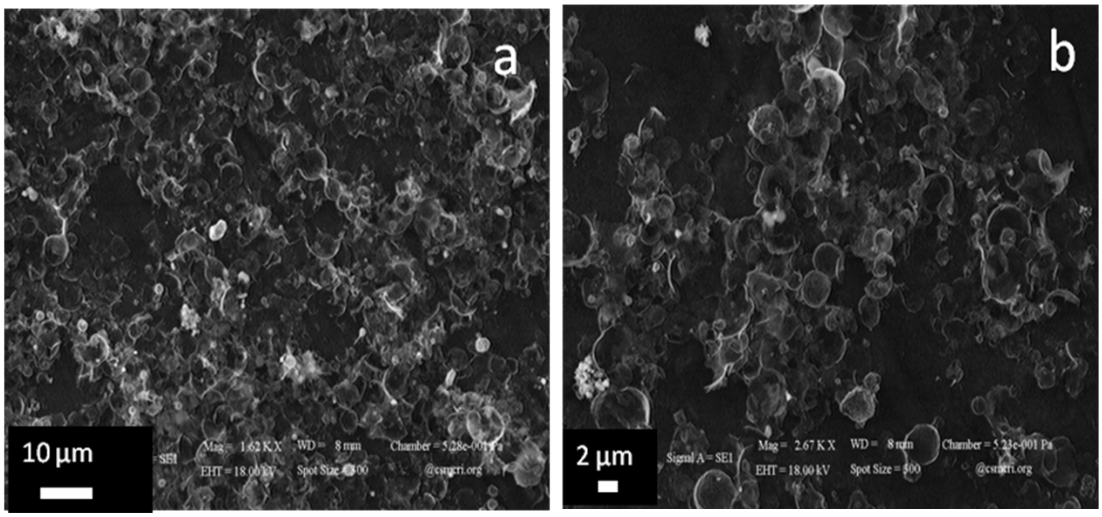

Fig. S11 SEM images of (a) ZnO and (b) MgO break hollow spheres obtained after calcinations of carbon metal hydroxide core shell structure (0.1g of metal salt concentration) at 700 °C for 2 hr.

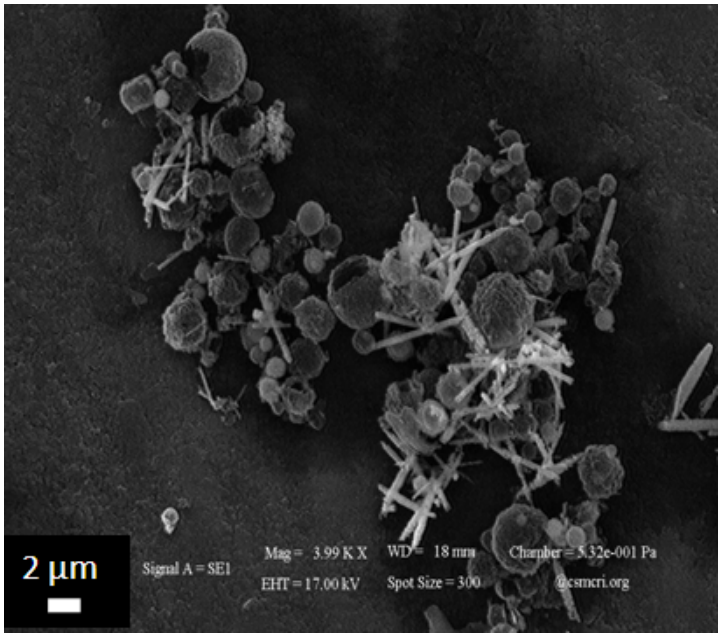

Fig. S12 SEM images of ZnO solid rods and hollow spheres produced using ammonium hydroxide as precipitating agent.