ISSN(PRINT) : 2320 -1967
ISSN(ONLINE) : 2320 -1975

GLOBAL SCIENTIFIC INC.

ChemXpress

ORIGINAL ARTICLE

CHEMXPRESS 9(2), 119-123, (2016)

# Glucose hydrothermal preparation of monodispersed carbon spheres with various particle sizes

**Xinwei Chen, Zhibo Liu, Zhuangzhuang Shen, Yingwen Duan***

**Key Laboratory of Special Function Materials and Structure Design, Ministry of Education, Lanzhou University, Lanzhou 730000, (CHINA)**

***Received : 13th February, 2015 ; Revised : 20th June, 2015 ; Accepted : 01st July, 2015***

**Abstract :** Using glucose as starting material, monodispersed carbon nano-spheres with a regular and perfect shape and narrow particle size distribution are synthesized via simple hydrothermal method. Characterizated by XRD, SEM, TEM and FTIR. Experimental results reveal that the as-prepared solid carbon sphere has smooth surface, the particle size and dispersibility can be well tuned by changing experimental condition of solution concentration, reaction temperature and time. Adding PEG [poly(ethylene glycol)] into glucose solution can increase the particle sizes and inhibite the particle aggregation. The as-prepared carbon sphere reserves a large number of active groups of glucose on its surface, which made it even contributes to the further application. **© Global Scientific Inc.**

**Keywords :** Glucose; Carbon sphere; Hydrothermal method; Particle size.

## INTRODUCTION

Spherical carbon materials have achieved continuous research attentions owing to their unique properties, such as chemical inertness, perfect conductivity of heat and electricity, self-sintering character and high density[1]. Which are always used as high strength high density materials[2], electrode material of lithium battery[3], the padding of liquid chromatography[4], the activated carbon with high specific surface area[5], catalysis support materials[6], and super capacitors[7]. Usually, carbon spheres are classified as three types[8]: (1) fullerenes family and carbon onion (with enclosed graphite layer structure and diameter 2~20 nm), like C60 and C70; (2) not fully graphitized carbon nano-spheres (diameter 50 nm~1 um); (3) carbon micro-spheres (over 1 um diameter)[2]. Besides, they are also classified as hollow[9], solid[10], porous[11], core-shell structure and colloidal carbon spheres[12] according to their morphology. Many methods have been used to fabricate carbon spheres. For example template method[13], hollow and porous carbon spheres can be prepared by using spherical inorganic (like silica beads) and porous silicate as template. Synthesis process includes modifying silica beads, polymer monomer or organic molecules acting on silicone template, carbonizing in inert atmosphere and at last removing template with acids and bases. Chemical vaporous deposition for preparing carbon spheres usually includes two types according to catalyst used or not[14,15]. The former made carbon vapors and decomposes on the catalyst surface, the later use the thermolysis of carbon-containing or-

ORIGINAL ARTICLE

ganics in the inert atmosphere. Actually, the most popular method for preparing carbon spheres is hydrothermal method[16,17], i.e., using solvent as medium in reactor, taking advantage of high temperature and pressure of autoclave to make full and better reaction, here used solvent usually is glucose, sucrose and starch etc[16-18].

Using hydrothermal technique and starting materials of glucose solution, we fabricate carbon spheres, whose particle size is quite different with reported literatures although under a similar experimental conditions[16,17]. It seems that morphology and particle sizes of the synthesized product are not only controlled via experimental condition of reaction temperature and time, the used raw materials and solution concentration, but also influenced via other factors of volume of the reaction container, atmospheric pressure, humidity of the experimental environment, and even source of the used raw materials. Here the related experimental results will be presented

## EXPERIMENTAL

A glucose solution (0.5-1.0 M, 20 mL) was transferred into a stainless-steel autoclave of 30 mL capacity, and was heated in reaction furnace under temperature of 180 °C for 6-16 h. Then, the solution was cooled down to ambient temperature. The black precipitate was collected by centrifugation, washed several times with deionized water and ethanol, and dried at 80 °C in an oven for 8 h. The used glucose was analytical reagents, purchased from the Recovery of Tianjin Science and Technology Development Co., Ltd., without additional purification before use.

The morphology of the sample was investigated by a Hitachi S-4800 field-emission scanning electron microscope (SEM) and a FEI Tecnai G2 F30 transmission electron microscopy (TEM). The phase structure of the sample was determined by powder X-ray diffraction (Rigaku D/MAX-2400) equipped with Cu Kα radiation (λ=1.5406 Å). Fourier transform infrared (FT-IR) spectrum was recorded on a Nexus 670 FT-IR spectrometer with the standard KBr pellet method.

## RESULTS AND DISCUSSION

### SEM and TEM characterization

In this experiment, the range of temperature for the synthesis reaction is very narrow. i.e., no carbon spheres are obtained at low temperatures of T <180 °C, and produced particles fuse at T ≥ 200 °C, temperature between 180 and 190 °C is suitable for carbon spheres formation. Here we choice 180 °C as reaction temperature. Figure 1 shows the morphology and particle size distribution of the carbon nano-spheres produced under the different concentrations of glucose

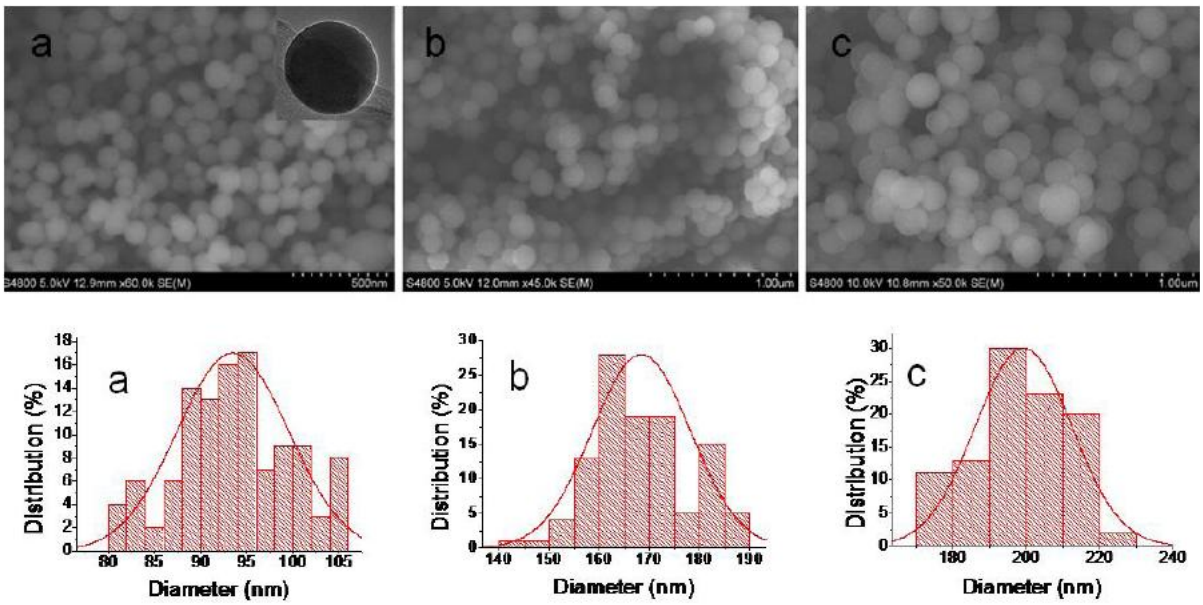


**Figure 1 : SEM and histograms of the size distribution of the carbon nano-spheres produced at different concentration of glucose solution at temperature of 180 °C for 6 h. (a) 0.5 M, (b) 0.75 M, (c) 1.0 M, inset is the corresponding TEM image of a single carbon nano-spheres**

solution. It is seen that produced carbon solid spheres consist of a large quantity of the monodispersed carbon nano-spheres, with good morphologies (regular spherical shape and smooth outer surface) and narrow particle size distributions. A higher concentration of glucose leads to an increase in the diameter of the carbon nano-spheres and a change in size distribution width, while without any alteration in the morphologies of the carbon nano-pheres. The average particle size is about 90 nm at 0.5 M, 160 nm at 0.75 M and 200 nm at 1.0 M of glucose.

Figure 2 is SEM images and the histograms of the size distribution of the as-prepared carbon nano-spheres under the different reaction time at 0.5 M of glucose. It is seen that the particle size increases as reaction time, and the average particle size is about 120 nm at 8 h, 170 nm at 12 h and 200 nm at 14 h, respectively. A longer reaction time, of 16 h or above, the as-prepared carbon nano-spheres aggregate together, as shown in Figure 2d. In order to reduce the particle aggregation, PEG-200 is added into glucose solution. The experimental results reveal that addition of PEG-200 can inhibite the particle aggregation, meanwhile, it also increases the particle size of the as-prepared carbon spheres. As shown in Figure 3. The average particle size of the as-prepared carbon spheres is about 170 nm at adding 0.004 g of PEG-200 into 0.5 M glucose solution, and about 200 nm at doubling PEG-200 under reaction time of 6 h. As adding 0.016 g of PEG-200 into glucose solution of 0.5 M, the average particle size of the as-prepared carbon spheres increases to 500 nm at reaction time of 6 h, and reaches to 1.3 um at the reaction time of 8 h. PEG-200 is a non-ionic polymeric surfactant that can be used as size-controlling agent to decrease particle size. In our experiment, addition of PEG into reaction solution lead to the carbon spheres size increases probably due to the amount of added PEG is little.

**XRD and FTIR characterization**

The structure expression of glucose is $CH_2OH$-$(CHOH)_4$-CHO, with C, H and O three elements and hydroxyl and aldehyde groups. Aldol reaction occurs under the hydrothermal environment. Figure 4a is the typical XRD pattern of the as-prepared CSs. No diffraction peaks of carbon are observed, the broad and weak diffraction peak means the fabricated product is amorphous. The FTIR spectrum is employed to identify the functional groups on the surface of the products. The bands at $\upsilon$ = 3423.9 and 1704.5 $cm^{-1}$, attribute to vibration of alcohol – OH, C=O and conjugated alkenes groups. At 1513.1 and 1300.5 $cm^{-1}$ probably support aromatic rings vibration, as shown in Figure 4b. These functional groups on the surface of the carbon nano-spheres indicate that the as-prepared carbon nano-spheres reserve a large number of active groups from glucose, meanwhile, further aromatized of glucose molecule from dehydration synthesis leads to conjugated double bonds and carbon-carbon bonds, which made products carbonized into spheres.

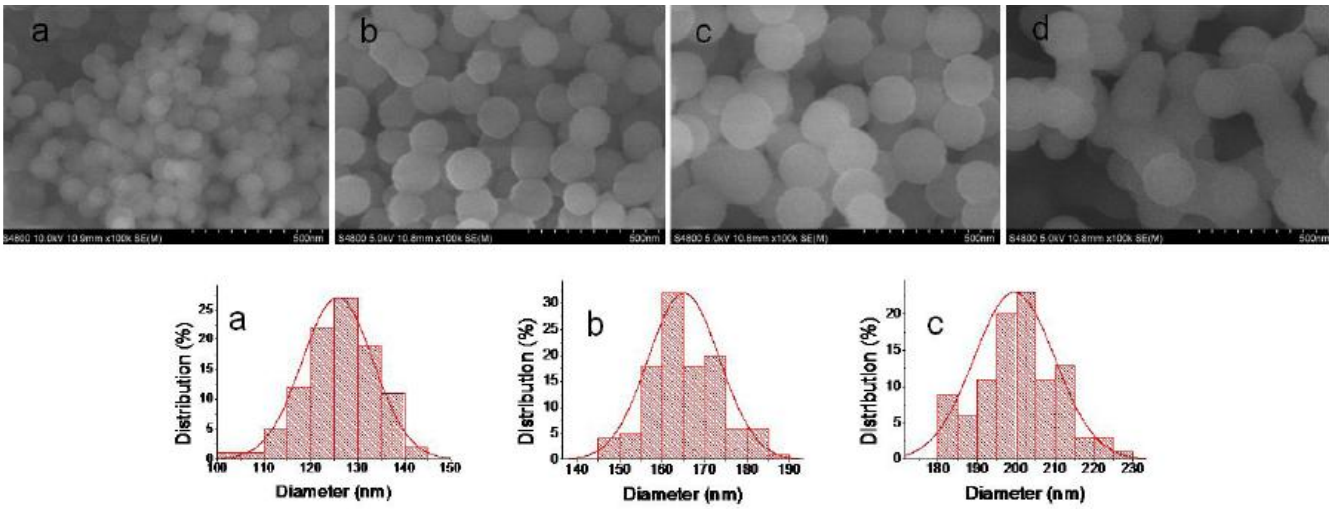


**Figure 2 : SEM images and histograms of the size distribution of the carbon nano-spheres produced at 0.5 M of glucose and temperature of 180 °C under different reaction time. (a) 8 h, (b) 12 h, (c) 14 h, (d) 16 h**

ORIGINAL ARTICLE

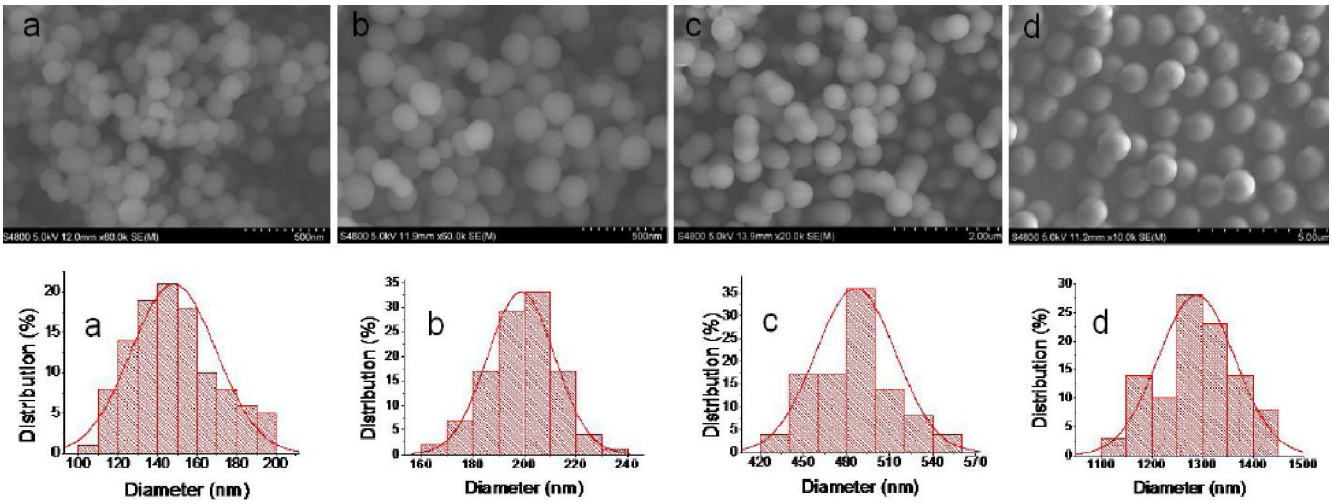


**Figure 3 : SEM images and histograms of the size distribution of the carbon spheres produced through adding different dosage of PEG-200 into glucose solution of 0.5 M (180 °C). (a) 0.004 g, 6 h; (b) 0.008 g, 6 h; (c) 0.016 g, 6 h; (d) 0.016 g, 8 h**

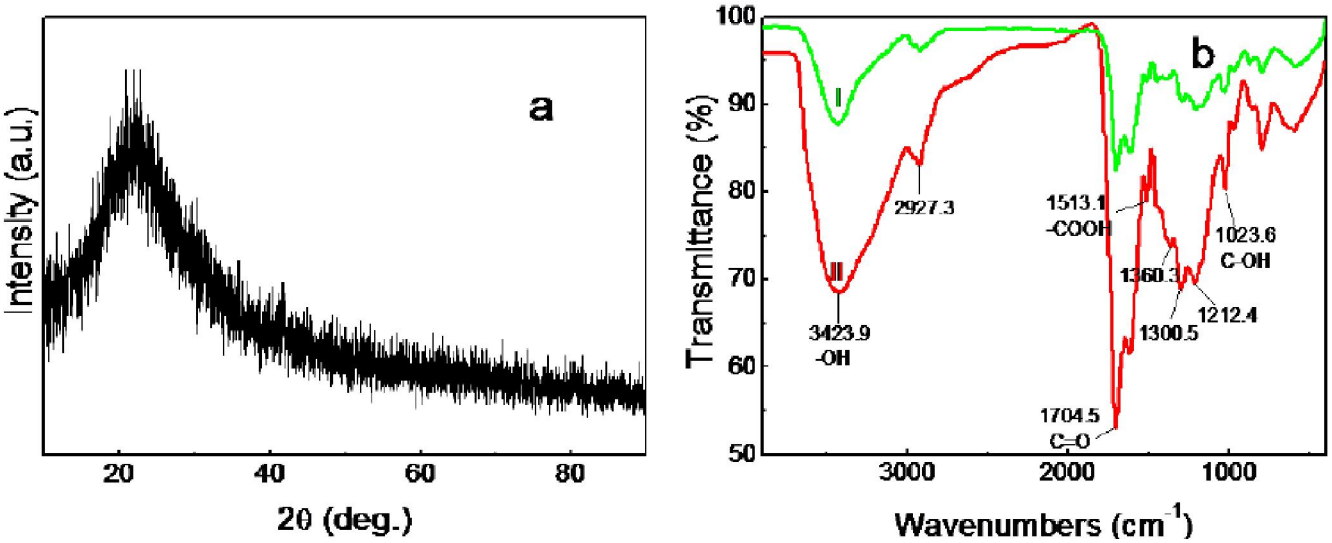


**Figure 4 : (a) The typical XRD pattern of the as-prepared carbon nano-spheres, (b) FTIR spectra of the as-prepared carbon nano-spheres, (I) no PEG, (II) adding PEG-200**

## CONCLUSIONS

Using glucose as starting materials, monodispersed solid carbon spheres with various particle sizes are prepared by hydrothermal method through controlling the experiment condition and adding PEG-200. The as-prepared solid carbon sphere has smooth surface, it reserves a large amount of functional groups on its surface, maybe which is beneficial for its further application.

## ACKNOWLEDGEMENTS

This work was supported by the National Science Foundation for Fostering Talents in Basic Research of the National Natural Science Foundation of China (Nos. 041105 and 041106).

## REFERENCES

**[1]** H.Honda; Mesophase putch and meso-carbon microbeads, Mol.Cryst.Liq.Cryst., **94**, 97–108 **(1983)**.

**[2]** Y.G.Wang, Y.Korai, I.Mochida; Carbon disc of high density and strength prepared from synthetic pitch-derived mesocarbon microbeads, Carbon, **37(7)**, 1049–1057 **(1999)**.

**[3]** M.X.Liu, X.M.Ma, L.H.Gan, Z.J.Xu, D.Z.Zhu,

L.W.Chen; A facile synthesis of a novel mesoporous Ge@C sphere anode with stable and high capacity for lithium ion batteries, J.Mater.Chem. A, **2**, 17107–17114 **(2014)**.

**[4]** A.A.Deshmukh, S.D.Mhlanga, N.J.Coville; Carbon spheres, Mat.Sci.Eng.R, **70**, 1–28 **(2010)**.

**[5]** Y.G.Lu, L.H.Ling, L.Liu, B.J.Zhang; Preparation of high surface area active carbon microbeads from meso-carbon microbeads, Coal Conversion, **22**, 66–70 **(1999)**.

**[6]** J.B.Joo, Y.J.Kim, W.Kim, P.Kim, J.Yi; Simple synthesis of graphitic porous carbon by hydrothermal method for use as a catalyst support in methanol electro-oxidation, Catal.Commun., **10**, 267–271 **(2008)**.

**[7]** Y.Zhao, Y.N.Meng, P.Jiang; Carbon@$MnO_2$ core-shell nanospheres for flexible high-performance supercapacitor electrode materials, J.Power Sources, **259**, 219–226 **(2014)**.

**[8]** Ph.Serp, R.Feurer, Ph.Kalck, Y.Kihn, J.L.Faria, J.L.Figueiredo; A chemical vapour deposition process for the production of carbon nanospheres, Carbon, **39(4)**, 621–626 **(2001)**.

**[9]** G.Hu, D.Ma, M.J.Cheng, L.Liu, X.H.Bao; Direct synthesis of uniform hollow carbon spheres by a self-assembly template approach, Chem. Commun., 1948–1949 **(2002)**.

**[10]** Q.Wang, H.Li, L.Q.Chen, X.J.Huang; Monodispersed hard carbon spherules with uniform nanopores, Carbon, **39**, 2211–2214 **(2001)**.

**[11]** S.B.Yoon, K.Sohn, J.Y.Kim, C.H.Shin, J.S.Yu, T.Hyeon; Fabrication of carbon capsules with hollow macroporous core/mesoporous shell structures, Adv.Mater., **14**, 19–21 **(2002)**.

**[12]** X.M.Sun, Y.D.Li; Colloidal carbon spheres and their core/shell structures with noble-metal nanoparticles, Angew.Chem.Int.Ed., **43**, 597–601 **(2004)**.

**[13]** J.Jang, B.Lim; Selective fabrication of carbon nanocapsules and mesocellular foams by surface-modified colloidal silica templating, Adv.Mater., **14**, 1390–1393 **(2002)**.

**[14]** Z.L.Wang, Z.C.Kang; Pairing of pentagonal and heptagonal carbon rings in the growth of nanosize carbon spheres synthesized by a mixed-valent oxide-catalytic carbonization process, J.Phys.Chem., **100**, 17725–17731 **(1996)**.

**[15]** Y.Z.Jin, C.Gao, W.K.Hsu, Y.Q.Zhu, A.Huczko, M.Bystrzejewski, M.Roe, C.Y.Lee, S.Acquah, H.Kroto, D.R.M. Walton; Large-scale synthesis and characterization of carbon spheres prepared by direct pyrolysis of hydrocarbons, Carbon, **43**, 1944–1953 **(2005)**.

**[16]** Y.Z.Mi, W.B.Hu, Y.M.Dan, Y.L.Liu; Synthesis of carbon micro-spheres by a glucose hydrothermal method, Mater.Lett., **62**, 1194–1196 **(2008)**.

**[17]** M.Li, W.Li, S.X.Liu; Hydrothermal synthesis, characterization, and KOH activation of carbon spheres from glucose, Carbohyd.Res., **346(8)**, 999–1004 **(2011)**.

**[18]** X.D.He, H.T.Li, Y.Liu, H.Huang, Z.H.Kang, S.T.Lee; Water soluble carbon nanoparticles: Hydrothermal synthesis and excellent photoluminescence properties, Colloid. Surface. B, **87**, 326–332 **(2011)**.