



# Cooperative effect of polyethylene glycol and lignin on SiO<sub>2</sub> microsphere production from rice husks

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## HIGHLIGHTS

- Preparation of dispersed SiO<sub>2</sub> microspheres with narrow size distribution from rice husks (RH).
- Optimum synthesis conditions of the employed method were determined.
- Plausible cooperative effect of added PEG and extracted lignin on the formation of SiO<sub>2</sub> microspheres.

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## ABSTRACT

A simple method to the preparation of SiO<sub>2</sub> microspheres from rice husks (RH) was developed. The rice husks were extracted with 2 M NaOH solution to obtain a extract contained silica and lignin, and silica spheres were obtained after addition of distilled water, ethanol, 1 M H<sub>2</sub>SO<sub>4</sub> and PEG (W<sub>silica</sub>:W<sub>PEG</sub> = 2:1) to the extract in a controllable sol–gel process and calcination at 550 °C. The optimum synthesis conditions were, adjustment of the extract to pH 3, a volume ratio of 1:1:2 of ethanol, water, and extract, respectively and a silica to polyethylene glycol weight ratio of 2:1. The SiO<sub>2</sub> microspheres produced under these conditions exhibited a relatively uniform particle size distribution around 500 nm as determined by LPSA, which indicated a cooperative function of added PEG and extracted lignin towards the formation of SiO<sub>2</sub> microspheres.

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## 1. Introduction

With a silica content of about 20%, rice husks (RH) could be a valuable feedstock for the production of porous silica useful for such applications as separation, adsorption, catalysis and thermal insulation (Jullaphan et al., 2009; Li et al., 2011). The conversion of RH, RH char and RH ash into porous silica has already been described (Adam et al., 2008; Chareonpanich et al., 2007; Jullaphan et al., 2009; Li et al., 2011; Sanchez-Flores et al., 2007; Witton et al., 2008, 2009), and the use of rice husks (RH) as sources of lignin and silica for the production of nano-composites by a sol–gel process has also been investigated (Qu et al., 2010).

The hydroxyl groups in lignin can form hydrogen bonds with Si–OH groups (Hayashi et al., 1997; Qu et al., 2010) moreover the three-dimensional network of lignin (Argyropoulos and Mena-chem, 1998) may improve the uniformity and dispersibility of silica spheres. The current research aimed at finding a simple way to make use of RH as sources of lignin and silica to prepare SiO<sub>2</sub> microspheres in situ using a controllable sol–gel process. The influ-

ence of preparation conditions on morphology and size of the microspheres was investigated by changing the pH, the amount of water, ethanol and PEG in the sol–gel reaction system.

## 2. Experimental

### 2.1. Materials and reagents

RH was provided by a rice mill near Dalian, China, in the autumn of 2011. RH was washed thoroughly with distilled water to remove adhering soil and dust, and 0.5 M HCl was used to remove metal impurities. After drying at 100 °C overnight, the samples were porphyzied to 60 mesh. H<sub>2</sub>SO<sub>4</sub> (AR), NaOH (AR), and ethanol (AR) were provided by Tianjin Kernel Co. Ltd. Polyethylene glycol (PEG, molecular weight = 2000, AR) was provided by Aladdin Chemical Co. Ltd. Distilled water was employed in all synthesis and treatment processes.

### 2.2. Preparation of silica spheres

Cleaned RH powder and 2.0 M NaOH were mixed at a ratio of 1:7 (w/v) in a 200-mL three-neck round-bottom flask equipped

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