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Controlled synthesis of magnetic carbon nanoparticles via glycerol/ferrocene co-pyrolysis with magnetic induction

Konrat Kerdnawee[a,b], Noriaki Sano[b,*], Hajime Tamon[b], Tawatchai Charinpanitkul[a,*]

[a] Center of Excellence in Particle Technology, Department of Chemical Engineering, Faculty of Engineering, Chulalongkorn University, Bangkok, Thailand
[b] Department of Chemical Engineering, Graduate School of Engineering, Kyoto University, Kyoto, Japan

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ABSTRACT

We performed a controlled synthesis of magnetic carbon nanoparticles (M-CNPs) via co-pyrolysis of glycerol and ferrocene with magnetic induction. The morphology of the synthesized M-CNPs was confirmed by transmission electron microscopy, and thermogravimetric analysis was used to analyze the carbon and Fe contents. M-CNPs that responded to magnetic stimulation were also examined with an AC-magnetic susceptibility analyzer. Our investigations on the influence of synthesis temperature in the range 700–1000 °C suggested that for an initial glycerol to ferrocene weight ratio of 3:1 and a temperature of 800 °C gave the highest yield of M-CNPs. Comparing the synthesis with and without magnetic induction, the controlled synthesis under the influence of magnetic induction shows promise as a method for producing high quality M-CNPs in high yields.

Introduction

Carbon nanoparticles (CNPs) have been recognized as a promising material that could be applied to many fields that affect our daily life (Iijima, 1991; Iijima et al., 1999; Kroto, Heath, O'Brien, Curl, & Smalley, 1985). Among various types of CNPs, carbon nanotubes (CNTs), which have a cylindrical nanostructure of carbon atoms with diameters in the range of 10–100 nm, was first reported by Iijima (1991). CNTs have attracted the interest of many researchers owing to their unique chemical, mechanical, and electrical properties (Coleman, Khan, Blau, & Gun'ko, 2006; Mutlu et al., 2010; Wu et al., 2004). In general, CNTs can be classified into single-walled carbon nanotubes (SWCNTs) and multi-walled carbon nanotubes (MW-CNTs) based on the number of graphene layers making up their walls. Each type of CNT has certain advantages and drawbacks, which can affect the intended application and have been examined by many research teams (De Volder, Tawfick, Baughma, & Hart, 2013). Many methods have been proposed and developed for the synthesis of CNPs, including arc discharge, chemical vapor deposition, and laser ablation. Chemical vapor deposition is one of the most common methods owing to its scalability and the ability to use various carbon sources. Carbon sources can be decomposed in the vapor phase at a regulated temperature prior to a self-assembly process leading to CNPs. Such CNPs grow on the surface of metal catalysts or specific substrates (Kumar & Ando, 2010; Sinnott et al., 1999). Pyrolysis, which is recognized as a simple CVD method, could be adapted for mass production of CNPs. Recently, it has been reported that co-pyrolysis of metallocenes with carbon precursors is a promising strategy for producing Fe-filled carbon nanoparticles, which exhibit magnetic properties (Kerdnawee et al., 2017; Kuwana & Saito, 2007; Sano, Hirama, & Tamon, 2015). Some experimental variables, such as the ratio of metallocene to the carbon precursor, temperature, reaction time, and pressure, have been regulated to control the formation and structure of carbon nanoparticles via such co-pyrolysis methods (Liu, Zhang, Li, Sun, & Abou-Rachid, 2011; Ma et al., 2014). However, based on a comprehensive literature survey, the formation of CNPs under the influence of magnetic induction has not been thoroughly examined (Yang, Hanna, & Sun, 2012).

Glycerol is a major by-product from the manufacture of bio-diesel, a material of interest for renewable energy research. However, the global supply of refined glycerol has been estimated to be lower than consumption. This oversupply of glycerol derived from bio-diesel conversion has received interest in terms of finding alternative uses for the material. To the best of our knowledge,

* Corresponding authors.
E-mail addresses: sano@cheme.kyoto-u.ac.jp (N. Sano), ctawat@chula.ac.th (T. Charinpanitkul).

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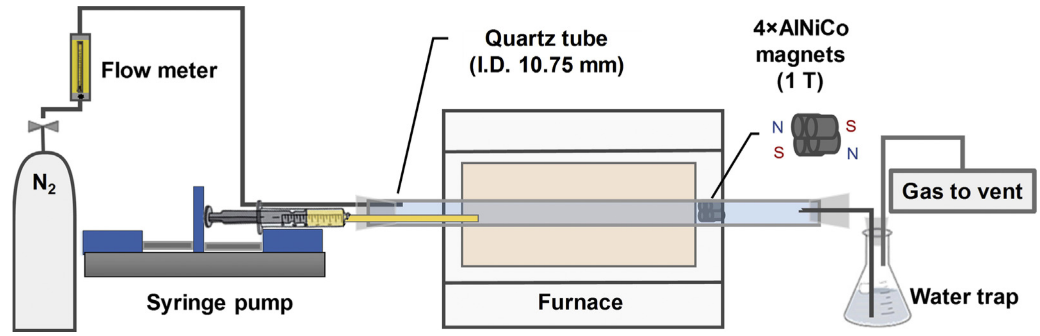

Fig. 1. Experimental apparatus for synthesizing M-CNPs with magnetic collector.

little previous research has examined the direct use of glycerol as a carbon source for synthesis of carbon nanoparticles (Kerdnawee et al., 2017). Therefore, in this work we seek to investigate alternative methods for producing value-added products from glycerol as a carbon source for the synthesis of CNPs. The pyrolysis of glycerol in the presence of ferrocene was used to synthesize magnetic carbon nanoparticles (M-CNPs) under the influence of a magnetic field with the aim of increasing the yield and quality of the resultant M-CNPs. On the basis of experimental investigations with synthesis temperatures in the range of 700–1000 °C, the highest yield of M-CNPs was achieved at 800 °C with an initial glycerol to ferrocene weight ratio of 3:1. Furthermore, AC-magnetic susceptibility analyzer measurements confirmed the strong magnetic induction response of the M-CNPs.

Experimental

Synthesis of M-CNPs with magnetic induction

M-CNPs were synthesized in an experimental set-up consisting of an electrical furnace and quartz tube (10.75 mm internal diameter, 570 mm length). Glycerol (Qrex, 99.5% purity) and ferrocene (Sigma–Aldrich, 98% purity) were used as the precursor and source of carbon and the Fe catalyst, respectively. A 2-mL portion of the

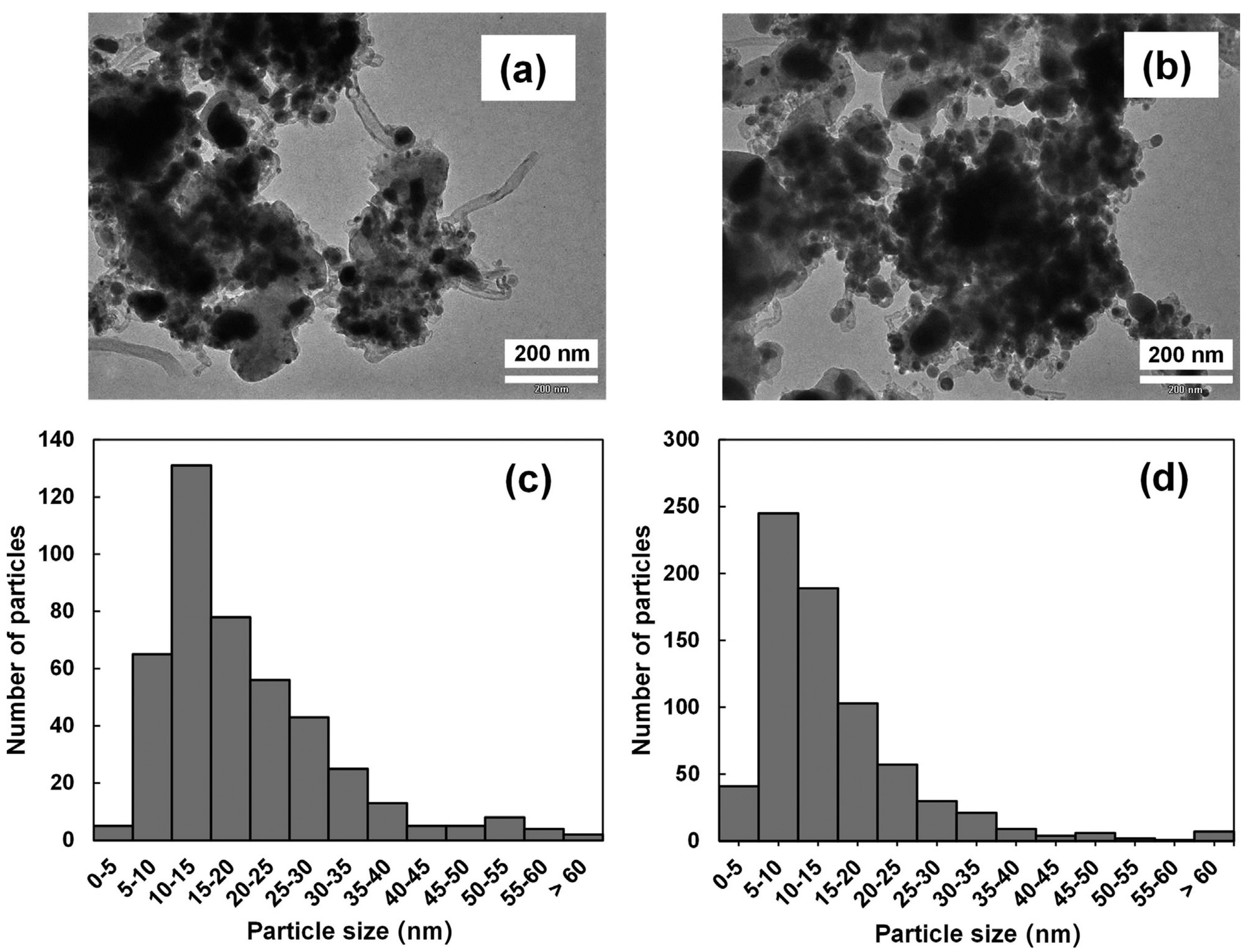

Fig. 2. TEM images of typical sample collected (a) from the inner wall of the quartz tube reactor, and (b) from the surface of the magnetic collectors; with (c) being particle size distribution (PSD) of Fe nanoparticles embedded in sample (a), and (d) the PSD of Fe nanoparticles embedded in sample (b).