Materials Chemistry and Physics 128 (2011) 70–76

Contents lists available at ScienceDirect

Materials Chemistry and Physics

journal homepage: www.elsevier.com/locate/matchemphys

Cobalt/silica nanocomposite via thermal calcination-reduction of gel precursors

Osama A. Fouad[a,*], Salah A. Makhlouf[b], Gomaa A.M. Ali[c], A.Y. El-Sayed[c]

[a] Central Metallurgical Research and Development Institute, CMRDI, P.O. Box 87, Helwan 11421, Egypt
[b] Physics Department, Faculty of Science, Assiut University, Assiut 71516, Egypt
[c] Chemistry Department, Faculty of Science, Al-Azhar University, Assiut branch, Assiut 71524, Egypt

ARTICLE INFO

Article history:
Received 26 August 2010
Received in revised form 11 February 2011
Accepted 16 February 2011

Keywords:
Co/SiO_2 nanostructures
Nanocomposites
Mesoporous
Thermal stability

ABSTRACT

Well dispersed and thermally stable Co/SiO_2 nanocomposite powders have been synthesized successfully via thermal calcination-reduction process of the prepared gel precursors. Co-gelation method for the cobalt and silica sources in the same solution mixture was found to be the best way to prepare these materials. Single (Co_3O_4) and (Co) phase formation could be confirmed by XRD and FTIR techniques for the products after calcination and reduction stages at the optimum process conditions, respectively. Surface analysis and TEM investigations of the reduced sample containing 25 wt.% Co revealed that highly dispersed cobalt nanoparticles embedded in a mesoporous inorganic polymeric silica matrix could be obtained. The silica matrix played a key role in protecting metallic cobalt nanoparticles from oxidation upon heating in air up to 400 °C. Addition of ethylene glycol to the gel mixture during gelation resulted in the formation of an additional organic polymeric protective layer. This layer promotes the formation of a regular pore structure upon its removal through calcinations and reduction steps. The current study could help in understanding the parameters affecting the dispersion and phase formation of cobalt species in silica matrix which indeed affect the activity of Co/SiO_2 nanocomposite as a catalyst in different processes and reactions.

1. Introduction

Magnetic transition metal (TM) nanoparticles have a great affinity to oxygen and form oxides. This property causes spontaneous ignition in atmospheric air and consequently limits their applications in open atmosphere especially at relatively higher temperatures. Capping may provide a protective layer surrounding a magnetic core enhances the resistance of core materials to oxidation [1]. In addition, the protective layer on the surface of a particle may also prevent dipole–dipole interactions between closely spaced magnetic nanoparticles, grain growth and agglomeration during subsequent heat treatment [2]. The process of capping therefore can control grain size and provide a better overall size distribution [1,3].

Nanocomposites of TM species embedded-in or capped-with inorganic oxide matrices are considered effective solution for protecting TM active nanoparticles from oxidation. These composites have gained much attention as promising materials for various applications as it posses the properties of metals, the matrices and the modified composites. It can be applied in high density recording media [4], as catalyst in Fischer–Tropsch synthesis [5], hydrogenation of toluene [6], oxidation of α-pinene and alkenes [7,8], hydroformylation of ethene [9] and as a magnetic separator for specific biological entities in biomedical applications [10]. In addition, it can provide the active materials for humidity [11,12] and gas [13] sensors. Silica (SiO_2) has been regarded as a very effective inorganic polymer matrix for stabilizing the metal nanocrystals against oxidation, tailoring a uniform particle size distribution and controlling the homogeneous dispersion of the ultrafine particles in the matrix [1,2].

Several methods and approaches have been explored for the synthesis of magnetic nanoparticles such as hydrogen arc plasma, reduction of metal salts, polyol process, γ-ray irradiation, metal carbonyl pyrolysis, sol–gel, water-in-oil microemulsion, template synthesis and reversemicelles, ball milling and sonochemical either in powder or thin film forms [14–19]. It is possible to obtain nanocomposite materials at low temperatures through the incorporation of small amounts of different components in the matrix [7]. Cobalt oxide/silica nanocomposite thin films have been prepared by dip-coating technique. Multi cobalt oxide phases and cobalt silicate hydroxide crystalline phase were obtained after calcination at 400–500 °C [20].

In general, there are two main approaches for obtaining dispersed nanoparticles in silica matrix namely: (i) post-synthesis treatment methods which is based on synthesis of cobalt nanoparticles in pre-prepared silica matrix, such as ion-exchange,

* Corresponding author. Tel.: +20 02 25010640; fax: +20 02 25010639.
E-mail address: oafouad@yahoo.com (O.A. Fouad).

0254-0584/$ – see front matter © 2011 Elsevier B.V. All rights reserved.
doi:10.1016/j.matchemphys.2011.02.072