UNIVERSITI TEKNOLOGI MARA

SOL-GEL SYNTHESIS AND CHARACTERIZATION OF IRON-SILICA AEROGEL AND XEROGEL USING RICE HUSK ASH AS SILICA PRECURSOR

KHOMARUL NAFISYAH BINTI MAAMUR

Thesis submitted in fulfillment of the requirements for the degree of
Master of Science

Faculty of Applied Sciences

Candidate's declaration

I declare that the work in this thesis was carried out in accordance with the regulations of Universiti Teknologi MARA. It is original and is the result of my own work, unless otherwise indicated or acknowledged as referenced work. This thesis has not been submitted to any other academic institution or non-academic institution for any other degree or qualification.

In the event that my thesis is found to violate the conditions mentioned above, I voluntarily waive the right of conferment of my degree and agree to be subjected to the disciplinary rules and regulations of Universiti Teknologi MARA.

Name of Candidate	Khomarul Nafisyah binti Maamur
Candidate's ID No.	2006649006
Programme	Master of Science
Faculty	Applied Sciences
Thesis Title	Sol-gel synthesis and characterization of iron-silica aerogel and xerogel using rice husk ash as silica precursor
Signature of Candidate	.....
Date	November 2010

ABSTRACT

Rice husk ash (RHA) has been used as an alternative precursor for silicon alkoxide in preparing porous silica using sol gel process. The use of rice husk has the benefit not only of producing valuable silica powder but also of reducing environmental problem associated with RHA disposal and the high cost of toxic silicon alkoxide precursors. This study is aimed to synthesize iron incorporated silica aerogel and xerogel from rice husk ash as silica precursor. Iron in the form of hydrated iron nitrate with compositions in the range of 3-17wt% ($Fe_2O_3/Fe_2O_3+SiO_2$) were used as iron source. The synthesis of iron doped aerogel was done by supercritical drying at temperature of about 250°C and pressure of about 5.9 MPa (850 psi) while xerogel was formed by drying the aquagel in an oven at 110°C for 24 hours. Characterization of samples were done using BET surface area analyze for pore and surface characteristics, X-Ray Diffractometer (XRD) for phase identification and Scanning Electron Microscope (SEM) for microstructural development. Specific surface area of the prepared silica aerogel and xerogel was 597.9 m^2/g and 340.5 m^2/g respectively. Specific surface area of iron incorporated silica aerogels and xerogels showed a decreasing pattern. The results obtained from SEM demonstrated that the iron particles were successfully incorporated and homogeneously dispersed throughout the silica matrix. After heat treatments, aerogel sample doped with 7.3 wt% iron at temperature 1100°C showed the presence of maghemite (γ-Fe_2O_3) and while other samples showed the presence of magnetite (Fe_3O_4) and hematite (α-Fe_2O_3). Results from FTIR and TGA were found to be consistent with the XRD and SEM. Upon application on column chromatography, silica aerogel prepared from RHA had shown potential as packing material for colour separation of Purple Orchid (*Cattleya bowringiana*) flower. Three different colours; green, blue, and purple were successively parted from the column. Colour separation did not occur with the iron incorporated silica aerogel. Instead, the colour of the sample turned from red to blue possibly due to the chemical reaction taking place between iron and the sample.

TABLE OF CONTENTS

CHAPTER 1

INTRODUCTION

1.1 General Overview

Basic manufacturing processes in powder metallurgy include mixing, compacting and sintering of particulate raw materials. The mixing process is the most important step. It control the particle distribution in composites (Booser, 1994). Generally, ceramic particles fabricated by this technique are prone to cluster during the composite fabrication (Durand, 2008). This is because the present state of the art of mixing by blending does not allow close control, thus making clustering of particles a common problem at this stage (Booser, 1994). It also well recognized that particles tend to agglomerate into large clusters during composite processing even under low loading levels of reinforcement (Durand, 2008).

When clustering occurs, there is a high probability that the mechanical properties of the composites are far below the theoretical values (Durand, 2008). There is also a possibility that little or no matrix powder being incorporated with the cluster of the reinforcement, resulting in a void in the composite and particle-particle contact (Lee, 1992). In this respect, appropriate processing procedures are needed to improve the dispersion of particles (Durand, 2008). One of the techniques that can overcome the problem is the sol-gel technique.