Walnut shell-assisted synthesis of mesoporous alumina

Nasim Abdollahpour[1], Asghar Zamani[2,*], Ahmad Poursattar Marjani[1,*]
[1] Department of Chemistry, Faculty of Science, Urmia University, Urmia 57154, Urmia, Iran
[2] Nanotechnology Research Center, Urmia University, Urmia 57154, Urmia, Iran

ABSTRACT

In the present study high surface area amorphous boehmite and γ-alumina were synthesized using walnut shell as template. This green, simple and useful synthetic protocol was based on the precipitation of aluminum hydroxide as the source of aluminum on biomass in an aqueous phase followed by calcination. The influence of walnut shell was evaluated by varying the calcination atmosphere. Materials were characterized using X-ray diffraction, fourier transform infrared spectroscopy, energy-dispersive X-ray spectroscopy, scanning electron microscopy and N_2 adsorption/desorption porosimetry and the results exhibited high surface area for boehmite and γ-alumina. Furthermore, the pore size and surface area of these mesoporous materials can be adjusted by varying the experimental conditions. Additionally, boehmite was studied as the support of vanadium catalyst for the oxidation of alcohols by hydrogen peroxide. We have found that resulting V-loaded material act as an effective catalytic system for the oxidation of a wide range of alcohols in 1,4-dioxane. The catalyst can be recovered and reused four times without loss of activity.

Keywords: green chemistry, biomass, walnut shell, alumina, boehmite, nanostructures, surface area, textural properties, catalysis, vanadium, oxidation, alcohols

1. INTRODUCTION

Nowadays alumina materials are widely employed in adsorption, separation and catalysis applications, because of its mechanical, chemical and thermal stability and low price [1,2]. However, the control of textural properties especially surface area is important, in order to enhance these applications, particularly in the case of catalysis [3]. While one of the traditional methods to prepare aluminas is thermal treatment of gibbsite $Al(OH)_3$ under pressure in the presence of organic additives [4], some chemists have synthesized alumina and boehmite using co-precipitation method by aluminium salts and basic reactant such as NH_3 [5,6], NaOH, Na_2CO_3, $NaHCO_3$ and $(NH_4)_2CO_3$ [7,8] or urea [9,10]. These methods usually have the disadvantages of the necessity to control pH and considerable waste volume generation.

Recently several papers were published based on the soft templating sol-gel method of mesoporous aluminas, in the presence of a wide range of anionic, cationic and non-ionic structural directing agents and usually using hydrolysis of aluminium alcoholates. Yada et al. reported that alumina with hexagonal structure was prepared by using aluminium nitrate nonahydrate and sodium dodecyl sulfate in water [11]. Mesoporous alumina was also prepared by magnesium stearate and aluminium *iso*-butoxide as an anionic template and an aluminium precursor [12]. *iso*-Butyl alcohol and magnesium nitrate were used as solvent and an additive, respectively. Surface area could be tailored by varying the ratio of magnesium nitrate to surfactant. Lee and co-workers synthesized mesoporous alumina using aluminum *iso*-butoxide and alkyl trimethyl ammonium bromide such as cetyl trimethyl ammonium bromide (CTAB) in *n*-butanol and its surface area could be controlled by changing the amount of water and the chain length of cationic template [13]. In a separate study, Liu et al. described the use of aluminum chloride CTAB in ethanol for the synthesis of nanoleaves gamma alumina [14]. Jaroniec et al. synthesized ordered mesoporous