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# Walnut shell-assisted synthesis of mesoporous magnesia

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**ABSTRACT**

In the present study high surface area amorphous magnesia was synthesized using walnut shell as a template. This green, simple and useful synthetic protocol was based on the precipitation of magnesium nitrate on biomass in an aqueous phase followed by calcination. Materials were characterized using X-ray diffraction, scanning electron microscopy and $N_2$ adsorption/desorption porosimetry and the results exhibited high surface area for magnesium oxide. Furthermore, the pore size and surface area of these mesoporous materials can be adjusted by varying the biomass/magnesium nitrate ratio. Additionally, magnesium oxide was studied as the support of palladium nanoparticles for the aerobic oxidation of alcohols. We have found that resulting Pd-loaded material act as an effective catalytic system for the aerobic oxidation of benzylic and aliphatic alcohols. The catalyst can be recovered and reused three times without loss of activity.

**Keywords:** Green chemistry, Nanostructures, Surface area, Textural properties, Catalysis, Alcohols

## 1. INTRODUCTION

Material chemists are interested in magnesium oxide or magnesia (MgO) because of its outstanding properties, such as lowest solubility among the alkaline earth oxides, easily obtainability and cheapness as well as its textural properties such as high surface area. Nowadays magnesia nanomaterials are widely employed in various areas, such as paint, adsorption, superconductivity, catalysis and toxic waste elimination [1-3].

Several preparative procedures for the synthesis of high surface area magnesia have been reported. While one of the simplest methods to prepare nanoporous MgO with relatively low surface area (97-161 $m^2\ g^{-1}$) is hydrothermal treatment of commercial bulk MgO crystals in water [4] or magnesium salts in mixed solvents of ethylenediamine and water [5] followed by calcination, some chemists have synthesized the magnesium oxide with higher surface area (72-361.5 $m^2\ g^{-1}$) using co-precipitation method by magnesium nitrate and ammonia or urea [6-8]. The other strategy focuses on the synthesis of magnesium bicarbonate $Mg(HCO_3)_2$ by bubbling $CO_2$ into an aqueous suspension of commercial magnesium oxide. By calcination of magnesium carbonate prepared from $Mg(HCO_3)_2$, porous MgO was synthesized [9,10]. Magnesium oxide nanoparticles can be synthesized by hydrolysis of magnesium alkoxide $Mg(OCH_3)_2$ in a toluene-$CH_3OH$ solvent [11-12]. Calcination of the synthesized gel resulted in the formation of magnesia with a surface area of 201-503 $m^2\ g^{-1}$. Evaporation and oxidation of Mg pieces at 913 K and in high vacuum in quartz glass tube resulted in MgO nanocubes with particle size distribution in the range of 10-1000 nm and surface area up to 300 $m^2\ g^{-1}$ [13].

In separate studies, Schüth *et al.* and Yu *et al.* described mesoporous magnesia preparation using a hard-templating method by mesoporous carbon prepared by pyrolization of resorcinol/formaldehyde polymer as a template [14,15]. Nanoporous MgO was also obtained by the mesoporous carbon hard templated method and the corresponding surface area can reach up to 175 $m^2\ g^{-1}$.