Ultrasonics Sonochemistry 31 (2016) 423–428

Contents lists available at ScienceDirect

Ultrasonics Sonochemistry

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

Ultrasound-assisted synthesis of magnesium hydroxide nanoparticles from magnesium

Olga Baidukova *, Ekaterina V. Skorb

Max Planck Institute for Colloids and Interfaces, Am Mühlenberg 1, 14424 Potsdam, Germany

ARTICLE INFO

Article history:
Received 19 November 2015
Received in revised form 25 January 2016
Accepted 28 January 2016
Available online 28 January 2016

Keywords:
Sonochemistry
Cavitation
Nanoparticle
Magnesium hydroxide
Magnesium

ABSTRACT

Acoustic cavitation in water provides special kinetic and thermodynamic conditions for chemical synthesis and nanostructuring of solids. Using cavitation phenomenon, we obtained magnesium hydroxide from pure magnesium. This approach allows magnesium hydroxide to be synthesized without the requirement of any additives and non-aqueous solvents. Variation of sonochemical parameters enabled a total transformation of the metal to nanosized brucite with distinct morphology. Special attention is given to the obtaining of platelet-shaped, nanometric and de-agglomerated powders. The products of the synthesis were characterized by transmission electron microscopy (TEM), electron diffraction (ED), scanning electron microscopy (SEM) and X-ray diffraction (XRD).

1. Introduction

Magnesium hydroxide (brucite) due to its specific properties has found an application in many fields. It is widely used in flame-retardant composite materials due to its ability to endothermically decompose with water release at high temperatures without production of toxic or corrosive substances [1,2], as an acidic waste neutralizer in paper industry and environmental protection field [3,4]. Besides, nanosized MgO products are commonly produced by a thermal decomposition of $Mg(OH)_2$ precursors [5,6]. The microstructure of the hydroxide, i.e. its particle size, shape and agglomeration are crucial in both applications. Among methods which are used for magnesium hydroxide synthesis are hydration of magnesium oxide [7,8], precipitation of a magnesium salt with an alkaline solution [9,10], sol–gel technique [11] and microwave-assisted synthesis [12,13]. However existing methodologies are multistep and require some other chemical presence in solution, e.g. NaOH or NH_4OH. In our case, magnesium hydroxide nanoparticles can be formed by sonochemical oxidation of magnesium in pure solutions. Special attention is given to the obtaining of platelet-shaped, nanometric and de-agglomerated powders.

Thus, in this paper we present a novel method for synthesis of nanosized magnesium hydroxide from pure magnesium by using high intensity ultrasound. The effect of ultrasound on a material is based on acoustic cavitation: generation, growth and collapse of bubbles [14]. Acoustic waves create in liquid a cavitation field of interacting microbubbles [15]. These microbubbles are reaction sites used for chemical synthesis and nanostructuring within which free radicals and excited species are formed due to thermal cleavage of volatile solutes [16]. The interfacial region around the bubbles has unique conditions for synthesis reactions: high temperature (up to 5000 K), high pressure (about 2000 bar) and fast heating and cooling rates [14]. Moreover, collapse of microbubble enables continuous transport of chemicals to the reaction sites [17]. Ultrasound-assisted synthesis and nanostructuring of solids in water is based on physical and chemical effects, i.e. fragmentation of particles and increase of the surface area in the first case and selective etching and oxidation of the metal surface caused by formed during water sonolysis hydroxyl radicals in the second case [18–22] (Fig. 1).

2. Results and discussion

The schematic presentation of ultrasound driven formation of magnesium hydroxide structures in water is schematically shown in Fig. 2. In order to illuminate the ultrasound driven physical and chemical processes in liquid–metal system, we investigated magnesium at different sonication conditions (solvent, concentration of the metal). A mesoporous magnesium sponge-like structure with a thin magnesium hydroxide upper layer and agglomerates of magnesium hydroxide platelets covering the surface were formed by the ultrasound-assisted method at the concentration

* Corresponding author.
E-mail address: baidukova@mpikg.mpg.de (O. Baidukova).

http://dx.doi.org/10.1016/j.ultsonch.2016.01.034
1350-4177/

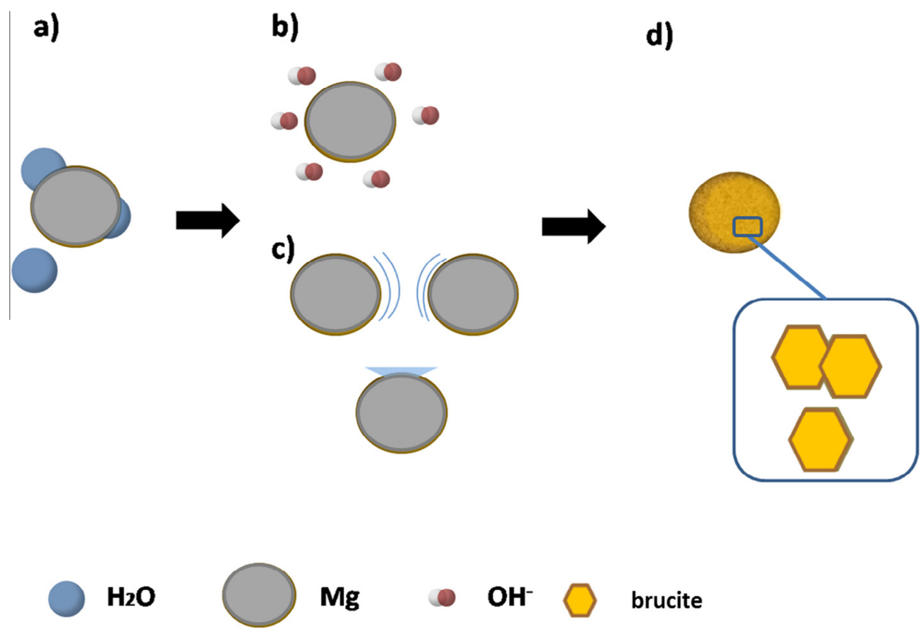

Fig. 1. Sonochemical modification of magnesium particles: (a) initial particles; (b) sonochemical effect: surface oxidation; (c) sonophysical effects: interparticle collisions and surface impinging by high velocity microjets; (d) formation of a mesoporous magnesium structure covered with brucite.

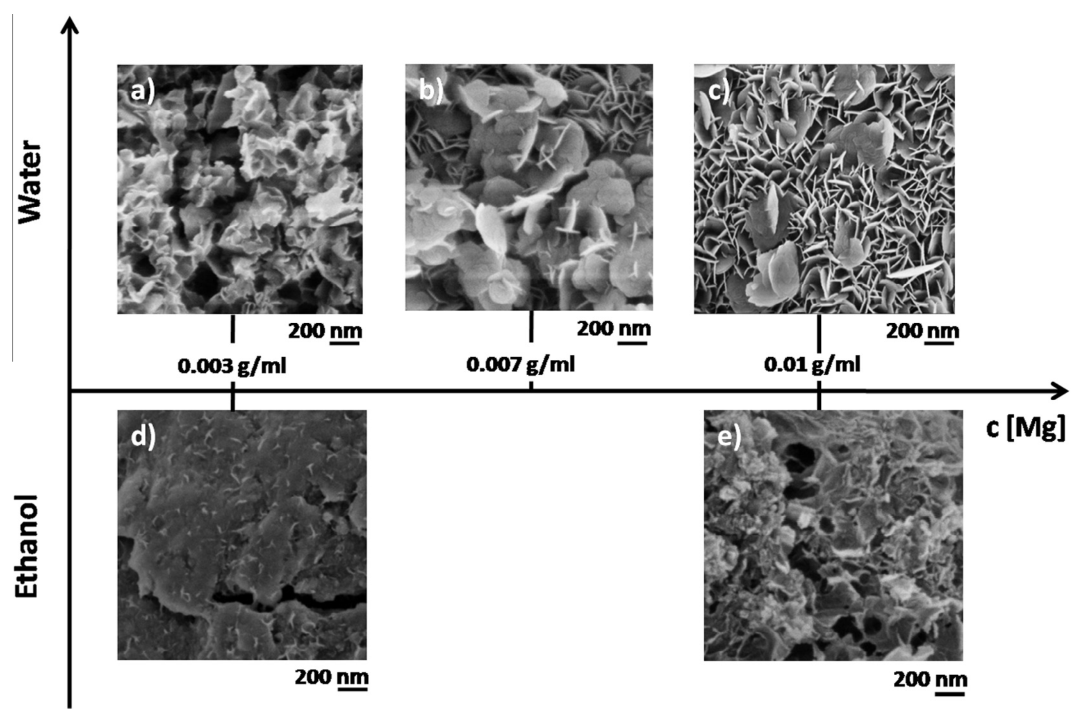

Fig. 2. The schematic presentation of the effect of acoustic cavitation on modification of magnesium particles at different concentrations and in different solvents. A mesoporous sponge-like structure with a thin brucite upper layer and brucite nanosheet-like agglomerates covering the surface were formed by ultrasound-assisted method at the concentration of aqueous magnesium solution 0.01 g/ml (c) and 0.007 g/ml (b), whereas at the lower concentration 0.003 g/ml magnesium exhibits complete transformation into brucite (a). The sonochemical treatment of magnesium in ethanol shows no presence of brucite (d, e).

of aqueous magnesium solution 0.01 g/ml (Fig. 2c) and 0.007 g/ml (Fig. 2b), whereas at the lower concentration 0.003 g/ml a complete transformation of magnesium into magnesium hydroxide was observed (Fig. 2a). In the latter case there is a sufficient amount of hydroxyl radicals per magnesium ion to achieve a quick oxidation of magnesium resulting in the indistinct morphology of the obtained magnesium hydroxide structures. In the meantime, a reduced amount of hydroxyl radicals per magnesium ion slows down the oxidation process and allows the formation of hexagonal platelet-like magnesium hydroxide structures. The sonochemical treatment of magnesium in ethanol shows no presence of magnesium hydroxide (Fig. 2d, e).

The marked diffraction peaks in the XRD pattern of the ultrasonically treated magnesium (Fig. 3) are attributed to magnesium hydroxide of the hexagonal structure with the lattice constants $a = 3.1442$ Å and $c = 4.7770$ Å comparable to the reported data