



CO₂ utilization for fast preparation of nanocrystalline hydrozincite



Erika Turianicová^{a,*}, Mária Kaňuchová^b, Anna Zorkovská^a, Marián Holub^c,
Zdenka Bujňáková^a, Erika Dutková^a, Matej Baláž^a, Lenka Findoráková^a,
Magdaléna Balintová^c, Abdullah Obut^d

^a Institute of Geotechnics, Slovak Academy of Sciences, Watsonova 45, 040 01 Košice, Slovakia

^b Technical University of Košice, Faculty of Mining, Ecology, Process Control and Geotechnology, Park Komenského 19, 040 01 Košice, Slovakia

^c Technical University of Košice, Institute of Environmental Engineering, Faculty of Civil Engineering, Vysokoškolská 4, 042 00 Košice, Slovakia

^d Hacettepe University, Mining Engineering Department, 06800 Beytepe, Ankara, Turkey

ARTICLE INFO

Article history:

Received 21 January 2016

Received in revised form 16 August 2016

Accepted 24 August 2016

Available online 8 September 2016

Keywords:

Mechanochemical processing

CO₂ storage

Mineral carbonation

Nanomaterial

ABSTRACT

In this study, gaseous CO₂, solid ZnO and liquid H₂O were converted to nanocrystalline (13 nm) hydrozincite, i.e. Zn₅(CO₃)₂(OH)₆ via fast one-step mechanochemical process at ambient temperature and pressure. The mechanically induced conversion of the three-phase mixture, studied by X-ray diffraction analysis, Fourier-transform infrared and X-ray photoelectron spectroscopies, completed in 10 min. X-ray diffraction and Fourier-transform infrared spectroscopy indicated the presence of disorder in the crystal structure of mechanothesized hydrozincite. The physico-chemical properties of the mechanothesized hydrozincite, characterized in the view of surface and optical properties, were compared with the commercial hydrozincite. It was found that the Brunauer-Emmett-Teller surface area was almost 2 times higher (56.7 m²/g) than the corresponding value of the commercial material, which is related to the smaller particle size (~230 nm). Both the ultraviolet-visible absorption and photoluminescence emission spectra recorded for the mechanothesized hydrozincite indicated size effect. The weight loss (26.5%) obtained during thermal decomposition of the mechanothesized material is close to the theoretical weight loss value of hydrozincite.

© 2016 Elsevier Ltd. All rights reserved.

1. Introduction

CO₂ is known as the greenhouse gas with the greatest contribution to the global warming. A number of strategies are explored to capture/recycle CO₂ which help to lower its content in the atmosphere. One of the strategies is to use CO₂ as a reactant for producing/synthesizing different inorganic and organic compounds. However, most of these routes involve complicated and multi-step pretreatments (thermal/chemical activation, use of additives, combined high temperature-high pressure, etc.) which enhance the kinetic of the process but restrict their commercial applications [1–8].

Mechanochemical synthesis or mechanochemical synthesis is a non-conventional method considered as one of the cost and time effective methods towards preparation of novel and high-performance nanomaterials [9,10]. It offers several advantages over traditional syntheses routes including (i) one-step approach, (ii)

simple manipulation during preparation, (iii) low-temperature and, (iv) possibility of a large-scale production. Generally, in the field of mechanochemistry the solid-state synthesis is preferred, but the mechanochemical synthesis can proceed also between solid and gas in the presence/absence of liquid medium. In this context, the process of mechanochemical oxidation (milling in air atmosphere) and carbonation (milling in CO₂ atmosphere) can be listed as an examples. Fabian et al. [11] found that the high-energy milling of Zn in air leads to the formation of nanocrystalline ZnO. In our previous work [12], we studied the direct mechanochemical carbonation of celestine (SrSO₄) in the presence of MOH (M: Li or Na). From this study follows that strontium compounds are also exploitable besides the generally used magnesium and calcium compounds (silicates) in mineral carbon dioxide sequestration process [13,14].

Few researcher groups studied the carbonation process of ZnO fine particles, powders or thin films in the presence or absence of H₂O under various conditions (T=5–35 °C; P_{CO₂}=0.01–1 atm; t=0.4 h–100 days) [15–19]. Depending on the conditions, ZnO carbonation leads to the formation of smithsonite (ZnCO₃) or hydrozincite as a predominant phase.

* Corresponding author.

E-mail address: turianicova@saske.sk (E. Turianicová).

Hydrozincite is one of the two known secondary carbonate minerals of zinc which can be formed naturally (geologically and biologically) or synthetically [20–24]. The monoclinic crystal structure with space group $C2/m$ was first estimated in a geologic sample [25]. However, investigation of the structure of different hydrozincite specimens by using X-ray diffraction, Fourier-transform infrared spectroscopy, and solid state nuclear magnetic resonance spectroscopy show some differences [22,26]. Hydrozincite has been studied also as intermediate stage in preparation of porous ZnO architecture with varying morphologies [27–30].

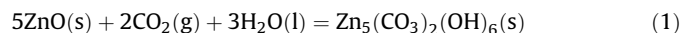
The aim of the present paper is to study, for the first time, the fast interaction between gaseous CO₂, solid ZnO and liquid H₂O leading to hydrozincite formation under high-energy milling conditions, for the purpose of gaseous CO₂ utilization. The evolution of the mechanochemical carbonation process was investigated using X-ray diffraction analysis, Fourier-transform infrared and X-ray photoelectron spectroscopies. Following milling, the physical and chemical properties of the mechano-synthesized product were investigated in detail using different experimental techniques such as N₂ physical sorption, scanning electron microscopy, zeta potential, cross-correlation, ultraviolet-visible absorption and photoluminescence emission spectroscopies, and thermal analysis.

2. Experimental

2.1. Materials

2.1.1. Hydrozincite – mechanochemically synthesized

ZnO (2.5 g, 99.95%, Mikrochem) was milled in a planetary mill Pulverisette 6 (Fritsch) for various times t_M (up to 10 min) in CO₂ atmosphere and in the presence of distilled water (10 mL) at ambient temperature leading to hydrozincite formation according to Eq. (1):



where s – solid, g – gas, l – liquid.

The milling experiments were performed in a laboratory planetary mill Pulverisette 6 (Fritsch) at 550 rpm using a grinding chamber (250 cm³ in volume) and balls (10 mm in diameter) made of tungsten carbide. The reaction chamber was flushed by CO₂ prior to the experiment to ensure a non-air environment. The duration of CO₂ flushing was 10 min at a pressure of 0.5 MPa (gas-flow rate 6 L/min) and the temperature was maintained at $22 \pm 1^\circ\text{C}$.

2.1.2. Hydrozincite – commercially purchased

Zn₅(CO₃)₂(OH)₆ (97%, Zn > 58%, Alfa Aesar (code A14590)) was used as a comparative material to the mechano-synthesized one. The X-ray diffraction pattern (Fig. 1, red line) corresponds to the JCPDS PDF 72-1100. Based on the literature data [31], the average crystallite size of this material was found to be around 19 nm (Table 1).

2.2. Characterization

The powder X-ray diffraction (XRD) data were collected over an angular range $3 < 2\theta < 65^\circ$ with steps 0.025° using the Bruker D8 Advance diffractometer working with the CuK α radiation and scintillation detector in Bragg-Brentano geometry. The diffraction patterns were treated with the Diffrac^{Plus}Eva and Topas programs, using the ICDD PDF2 database for phase analyses and Rietveld analyses, respectively.

Elemental analysis (the total carbon content) was determined using a CHNS Elemental Vario Macro Cube analyser.

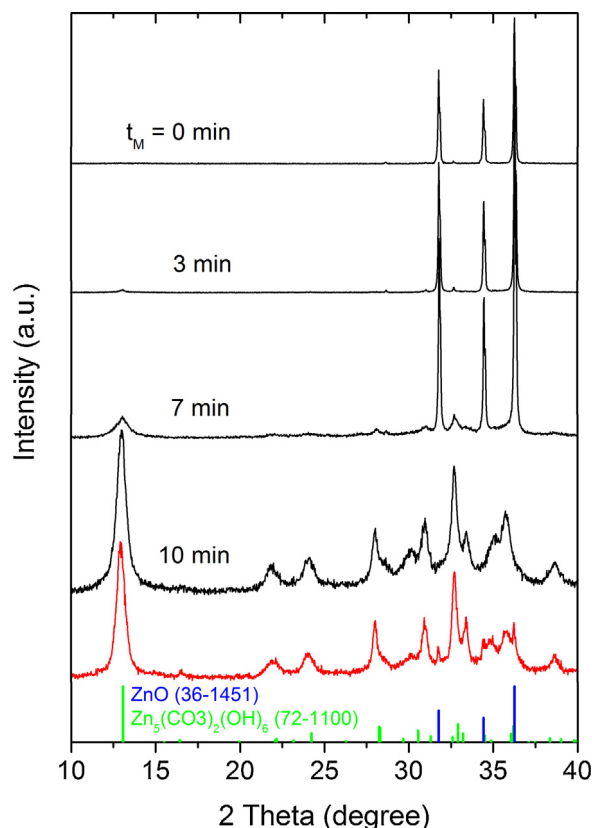


Fig. 1. XRD patterns illustrating the mechanochemical carbonation of ZnO powder in the presence of gaseous CO₂ atmosphere and liquid H₂O for different milling times (t_M , black lines) and the comparative commercial hydrozincite (red line). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

Fourier-transform infrared spectra (FTIR) were measured using the Tensor 29 (Bruker) in the frequency range of 4000–400 cm⁻¹ with the KBr pellet method. KBr was dried before the analysis at 100 °C for 1 h.

The surfaces of the samples were analyzed by the X-ray photoelectron spectroscopy (XPS). XPS measurements were performed using the SPECS instrument equipped with PHOIBOS 100 SCD and non-monochromatic X-ray source. The survey surface spectrum was measured at the transition energy 40 eV and the core spectra at 50 eV at ambient temperature. All spectra were acquired at a basic pressure of 2×10^{-8} mbar with MgK α excitation at 10 kV (150 W). The data were analyzed by SpecsLab2 CasaXPS software (Casa Software Ltd.). A Shirley and Tougaard type baseline was used for all peak fits. The spectrometer was calibrated against silver (Ag 3d). All samples showed variable degrees of charging due to their insulating nature. The problem was resolved by the calibration on carbon.

The specific surface area values were determined and the complete nitrogen adsorption-desorption isotherms were measured by the low-temperature nitrogen adsorption method using NOVA 1200e Surface Area & Pore Size Analyzer (Quantachrome). The samples were outgassed at 50 °C. The specific surface area values were calculated using Brunauer-Emmett-Teller (BET) theory (S_{BET}) and the pore size distribution was calculated using Barret-Joyner-Halenda (BJH) method. For calculations, including the ones for total pore volumes and average pore radii, QuantachromeTM NovaWin software was utilized.

The morphology of the product was analyzed using the scanning electron microscope (SEM) MIRA 3 (TESCAN).

Download English Version:

<https://daneshyari.com/en/article/6529183>

Download Persian Version:

<https://daneshyari.com/article/6529183>

[Daneshyari.com](https://daneshyari.com)