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journal homepage: www.elsevier.com/locate/ijmstPreparation of crystalline $\text{Mg}(\text{OH})_2$ nanopowder from serpentinite mineralV. Sirota^{a,c,*}, V. Selemenev^b, M. Kovaleva^a, I. Pavlenko^a, K. Mamunin^{a,c}, V. Dokalov^a, M. Yapryntsev^a^a Belgorod State National Research University, Belgorod 308015, Russia^b Voronezh State University, Voronezh 394006, Russia^c Belgorod State Technological University named after V.G. Shoukhov, Russia

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ABSTRACT

In this paper we describe a route to produce crystalline $\text{Mg}(\text{OH})_2$ nanopowders from serpentinite ore distributed in the Halilovskiy array (Russia, Orenburg region). An efficient extraction route consisting of treatment on serpentinite in 40% HNO_3 at 80 °C followed by NH_4OH titration for $\text{Mg}(\text{OH})_2$ precipitation was demonstrated. In this study, crystalline $\text{Mg}(\text{OH})_2$ nanopowders have been synthesized by solvothermal reaction method using $(\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O})$ which were obtained from serpentinite, NH_4OH as a precipitator, and hydroxyethylated nonylphenol as surface-active substance. Microstructure and phase composition of samples were investigated employing scanning electron microscopy (SEM) and transmission electron microscopy (TEM), X-ray phase analysis (XRD), and inductively coupled plasma optical emission spectroscopy (ICP-OES). XRD reveals that $\text{Mg}(\text{OH})_2$ nanopowder with high purity has the brucite structure. It was found that crystalline $\text{Mg}(\text{OH})_2$ nanopowders exclusively consist of lamellar-like structures and the sizes of $\text{Mg}(\text{OH})_2$ are 30–265 nm length or width.

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1. Introduction

Magnesium hydroxide ($\text{Mg}(\text{OH})_2$) is a popular inorganic compound because of a wide range of its important applications. It plays an important role in many fields, such as flame retardant in polymers, special ceramics, wastewater treatment and fillers in bleaching agent [1–6]. Besides, $\text{Mg}(\text{OH})_2$ is one of the most important precursors of magnesium oxide.

The chemicals magnesium nitrate, magnesium chloride hexahydrate, and magnesium acetate are the commonly used raw materials [7]. However, other sources of $\text{Mg}(\text{OH})_2$ production include minerals with high content of magnesia such as dolomite ($\text{CaMg}(\text{CO}_3)_2$), hydromagnesite ($\text{Mg}(\text{CO}_3) \cdot 4\text{H}_2\text{O}$), brucite ($\text{Mg}(\text{OH})_2$), and serpentinite ($\text{Mg}_3\text{Si}_2\text{O}_5(\text{OH})_4$). Sea water, underground salt deposits of brines and salt beds where magnesium hydroxide is processed are also sources for the production of magnesia [8–12].

Serpentinite is a potential magnesium source for synthesis of magnesium hydroxide. Serpentinite mineral ($\text{Mg}_3\text{Si}_2\text{O}_5(\text{OH})_4$) is a hydrous magnesium-rich silicate mineral which generally occurs in three types: antigorite, chrysotile, and lizardite. It commonly contains 32–38% MgO and 35–40% SiO_2 with minor amounts of Fe, Al, Ca, Cr, and Ni [13]. Currently, serpentinite has mainly been

used as: the flux in blast furnaces in the iron and steel industry, a road construction material, foundry sand, fertilizer, and for soil amendment etc. [14,15].

Several studies were concerned with investigation of industrial chemical utilization of serpentinite rocks as a stock material for magnesium hydroxide [16–18].

In recent years, the advances in imaging, engineering and manipulating system at the nanometer scale have led to numerous research studies dealing with synthesis of $\text{Mg}(\text{OH})_2$ nanoparticles or nanosurfaces [19,20]. In literature, many methods were proposed to produce nano-sized $\text{Mg}(\text{OH})_2$ [21,22].

$\text{Mg}(\text{OH})_2$ with different morphologies such as rods, tubes, needles, and lamina have been synthesized by hydrothermal reaction using different magnesium sources such as magnesium powder, MgSO_4 , and $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ [19].

But producing of $\text{Mg}(\text{OH})_2$ from Mg-silicates is not straightforward. Extraction of Mg from Mg-silicate minerals, specifically serpentinite rock and its subsequent conversion to $\text{Mg}(\text{OH})_2$ suffer from setbacks arising from slow kinetics, low conversion, high energy requirements and chemical costs [23–25]. In this paper a novel method of producing of crystalline $\text{Mg}(\text{OH})_2$ nanopowders from serpentinite which addresses some of these drawbacks is presented.

The aim of this study is to investigate a novel method of producing of crystalline $\text{Mg}(\text{OH})_2$ nanopowders by solvothermal reaction

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method using $(\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O})$ which was obtained from serpentinite.

2. Experimental

2.1. Materials

The serpentinite ore distributed in the Halilovskiy array (Russia, Orenburg region) was selected for this study. The provided quantity (~100 g) was sampled according to the usual method of sampling [26]. It was ground by a vibrating ball mill to a median diameter 22.5 μm (size distribution 1–130 μm). Samples were analyzed using X-ray diffraction (XRD) with $\text{Cu K}\alpha$ radiation (Rigaku Ultima-IV X-ray diffractometer).

The chemical composition of the serpentinite samples which were used in the experiments is shown in Tables 1 and 2. The chemical composition was investigated using SEM Quanta 200 3D.

The serpentinite consisted mostly of magnesium, silicon and iron in the form of serpentinite $(\text{Mg}_3\text{Si}_2\text{O}_5(\text{OH})_4)$; lizardite (Fig. 1).

2.2. Procedure

Previous research has shown that mineral acids are suitable solvents for extracting magnesium from serpentinite: all magnesium were extracted from serpentinite in 1–2 h using 2 M solutions of HCl, HNO_3 or H_2SO_4 at 70 °C [25,27].

In this study nitric acid was used in the experiments. A schematic summarizing the procedure for the production of $\text{Mg}(\text{OH})_2$ and crystalline nanopowders of $\text{Mg}(\text{OH})_2$ from the serpentinite is shown in Fig. 2.

The procedures for the preparation of $\text{Mg}(\text{OH})_2$ from the serpentinite were as follow: serpentinite with the particle sized of 1–130 μm were dissolved in 40% nitric acid solution (273.88 mL) at 80 °C for 2.5 h in a glass reactor [28,29]. The temperature (80 °C) selected in this study was determined based on the previous studies [30–32].

The reactor was equipped with a tap water-cooled condenser to avoid solution vapour from escaping. When the temperature had stabilized at 80 °C, 100 g of serpentinite was added to the solution. Two and half hours after the addition of the serpentinite, the solution was removed from the reactor and filtered. Fe, Si and other elements present (though in minute amounts) in serpentinite were extracted during the reaction of serpentinite and nitric acid. However, the amounts of Si extracted may be a low estimate. Teir et al. [27] noted that the silicon concentrations in the filtrate may not be accurate because part of the silica can precipitate as gel on the filter thereby reducing the silicon concentration in the filtrate. The remaining residues SiO_2 were separated by filtration, washed with distilled water, and dried at 120 °C.

The $(\text{Mg}(\text{NO}_3)_2)$ -rich filtrate was transferred to a glass beaker with 25 g of MgO and 80 mL of distilled water. Then, the mixture was placed in an ultrasonic bath for 90 min and 90 °C. The mixture was well mixed using a magnetic stirrer set to 600–700 r/min.

Simultaneously pH of the reaction mixture was measured and recorded by pH-meter. Addition of MgO to the $(\text{Mg}(\text{NO}_3)_2)$ -rich filtrate rapidly changes its pH from acidic (pH < 6) to slightly alkaline (pH = 8), while also changing the colour of solution from colourless to dark green. The dark green precipitate after being filtered, upon

exposure to air and drying at 120 °C (atmospheric pressure), quickly turns dark brown in colour. This material was identified as of iron oxide hydroxide (mineral goethite – FeOOH). The precipitated solid product ($\text{FeO}(\text{OH})$) is regarded as the valuable products. There is an ongoing study [18] on utilization of the Fe-rich by-product and integration of this process with the iron-and steel-making industry. $\text{Mg}(\text{NO}_3)_2$ solution was obtained after the ions like Fe^{3+} and Fe^{2+} were transformed into hydroxide precipitates, and the precipitates were separated by filtration.

Adding hydrated magnesium carbonate ($\text{MgCO}_3 \cdot 3\text{H}_2\text{O}$) to the $(\text{Mg}(\text{NO}_3)_2)$ -rich solution obtained after filtering the precipitated $\text{FeO}(\text{OH})$ -rich by-product leads to precipitation of CaCO_3 .

$(\text{Mg}(\text{NO}_3)_2)$ -rich solution was transferred to a glass beaker for evaporation of the solution. The solution started boiling at 80 °C and was boiled for 4 h in order to evaporate most of the solvent. The residue of hydrated magnesium nitrate was cooled to room temperature and dissolved in distilled water (4 L).

Magnesium nitrate ($\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$), NH_4OH as a precipitator (5 g), and hydroxyethylated nonylphenol as surface-active substance (10 mg) have been used to produce crystalline $\text{Mg}(\text{OH})_2$ nanopowders. Chemical precipitation of magnesium hydroxide was performed in 1 M solution of magnesium nitrate in distilled water containing 0.01% (by weight) of nonylphenol hydroxyethylated by ammonia solution. Value for pH for the $\text{Mg}(\text{OH})_2$ precipitation was 1 L. The obtained precipitation was washed to get rid of nitrates. Complete conversion of $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ to $\text{Mg}(\text{OH})_2$ is possible at this stage. The concentration of nitrates after flushing must not be higher than 10 mg/L. The formed suspension of milk color was subjected to centrifugation at the rate of 2000 r/min to increase the percentage composition of the bulk material. The obtained gel was loaded into a supercritical reactor R-401-5L (South Korea), where solvothermal reaction was performed at 220 °C and 180 atm. The reaction time was 45 min while achieving operation parameters. The solvothermal decomposition of powders was carried out in isopropyl alcohol as buffering agent. The reactor inner pressure control is performed by changing the heater temperature and by the reactor tube water cooling system. The obtained powder was dried in a drying oven at 60 °C for 15 min.

2.3. Characterization

Analysis of surface morphology and chemical composition of the samples of serpentinite, magnesium nitrate and magnesium hydroxide was performed by a scanning electronic microscope Quanta 200 3D (accelerating voltage of 20 kV) equipped with an X-ray detector of the PEGASUS 2000 system and multitype ICP emission spectrometer ICPE-9000 (SHIMADZU). The particle size was measured by the Analysette 22 NanoTec laser diffractometry. The microstructure of $\text{Mg}(\text{OH})_2$ nanopowders was carried out using a JEM 2100 (JEOL Ltd., Tokyo, Japan) transmission electron microscope (TEM) equipped with an INCA energy-dispersive X-ray spectrometer (EDS; Oxford Instruments, Oxfordshire, U.K.) with an acceleration voltage of 200 kV. The TEM specimens are prepared by method for the preparation of micrometer-sized powder particles described in [33]. The phase composition of the samples was analyzed by XRD with $\text{Cu K}\alpha$ radiation. A Rigaku Ultima IV X-ray powder diffractometer was used. Crystalline phases were identified by the ICDD PDF-2 (2008) powder diffraction database.

Table 1
Chemical composition of serpentinite ore studied as elements (wt%).

Elements	Mg	Si	Al	Fe	Ca	O
Content	23.61	26.33	1.02	7.58	0.36	41.09

Table 2
Chemical composition of serpentinite ore studied as oxides (wt%).

Oxide	MgO ^a	SiO ₂ ^a	Al ₂ O ₃	Fe ₂ O ₃ + FeO	CaO	Other
Content	36.64	51.22	1.76	9.33	0.46	0.59

^a Assuming all Mg as MgO, all Si as SiO₂.

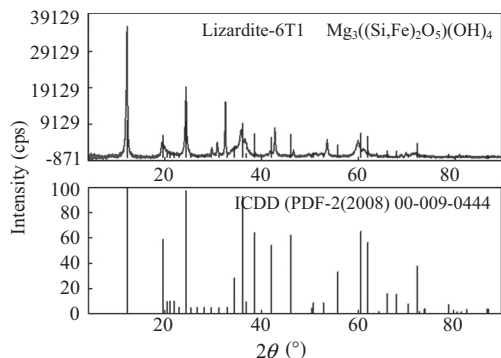


Fig. 1. XRD pattern of the serpentinite used in this study.

3. Results and discussion

The chemical composition (energy-dispersive X-ray spectroscopy (EDS)) of the magnesium nitrate and magnesium hydroxide which were obtained in the experiments is shown in **Table 3**.

Analysis of the phase composition of the magnesium nitrate ($\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$) (**Fig. 3**) shows that according to the ICDD data catalog, the powder consists of $\text{Mg}(\text{NO}_3)_2(\text{H}_2\text{O})_6$ with a monoclinic lattice with $P121/c1$, unique- b , cell-1 space group ($a = 6.238 \text{ \AA}$, $b = 12.712 \text{ \AA}$, $c = 6.611 \text{ \AA}$); $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ with a monoclinic lattice with $P121/c1$, unique- b , cell-1 space group ($a = 6.192 \text{ \AA}$, $b = 12.707 \text{ \AA}$, $c = 6.599 \text{ \AA}$).

Table 3
Chemical analysis of the magnesium nitrate and crystalline $\text{Mg}(\text{OH})_2$ nanopowder studied as elements (wt% (dry) (EDS)).

Element	$\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$	$\text{Mg}(\text{OH})_2$
Mg	17.76	50.63
Ca	0.47	0.29
O	67.62	49.08
N	14.62	

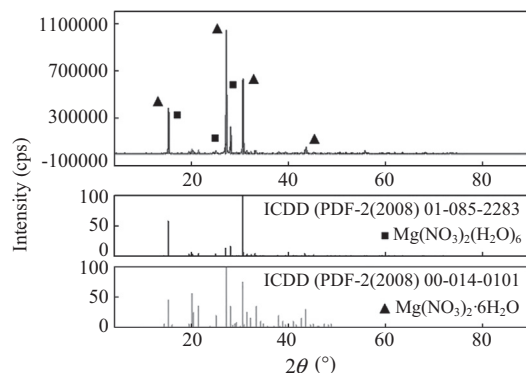


Fig. 3. XRD pattern for $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ produced from serpentinite.

Analysis of the phase composition of crystalline $\text{Mg}(\text{OH})_2$ nanopowder (**Fig. 4**) shows that according to the ICDD data catalog, it is one-phase material $\text{Mg}(\text{OH})_2$ (brucite) and had trigonal lattice

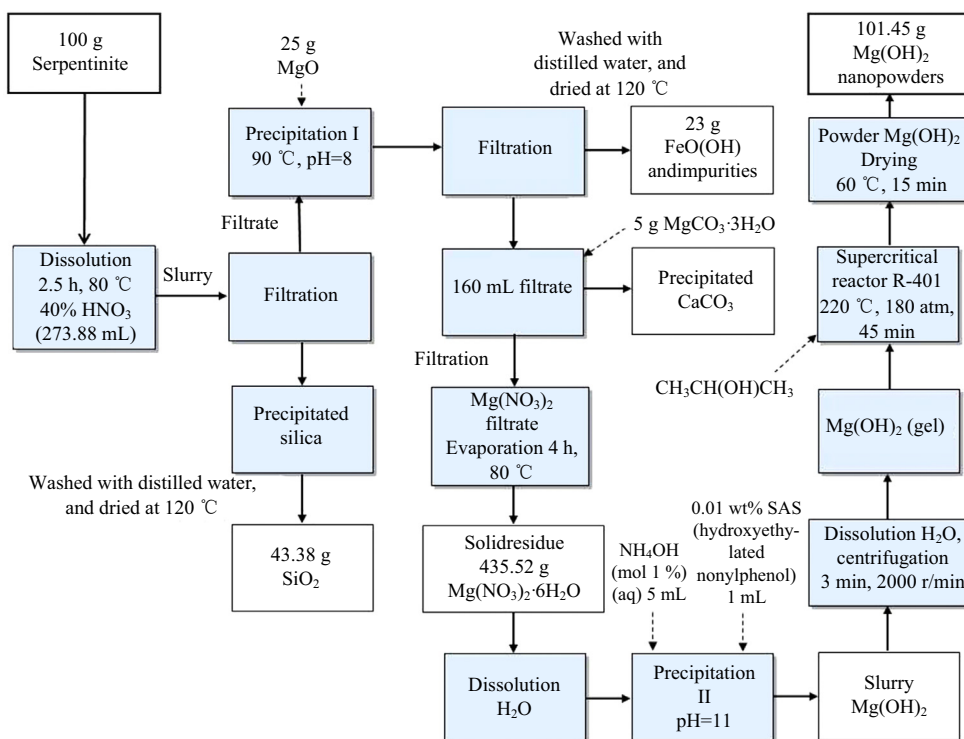


Fig. 2. Schematic of the staged route for crystalline $\text{Mg}(\text{OH})_2$ nanopowders production from the serpentinite.

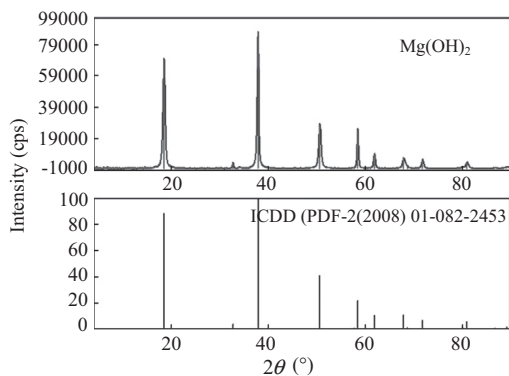


Fig. 4. XRD pattern for crystalline $\text{Mg}(\text{OH})_2$ nanopowder produced from serpentinite.

with P-3 mL space group ($a = b = 3.148 \text{ \AA}$, $c = 4.785 \text{ \AA}$). No diffraction peaks representing other phase were detected in Fig. 4, which indicated a high purity of the brucite.

The chemical composition of crystalline $\text{Mg}(\text{OH})_2$ nanopowder was confirmed by inductively coupled plasma optical emission spectroscopy (ICP-OES) (Table 4). A Multitype ICP Emission Spectrometer ICPE-9000 (SHIMADZU) was used for analysis. The analysis of the chemical composition by analytical quantitative technique like the ICP-OES is important because it establishes exactly how much impurities can be incorporated into $\text{Mg}(\text{OH})_2$ nanopowder. ICP-OES analysis shows the major phases to be $\text{Mg}(\text{OH})_2$ with few impurities (Table 4).

ICP-OES and XRD analysis performed on the samples showed that high quality $\text{Mg}(\text{OH})_2$ was produced.

The morphological and structural features of crystalline $\text{Mg}(\text{OH})_2$ nanopowder, shown in Fig. 5, were characterized with transmission electron microscope (TEM) which shows the crystalline $\text{Mg}(\text{OH})_2$ nanoparticles.

It can be seen from the TEM images of $\text{Mg}(\text{OH})_2$ (Fig. 5) that crystalline $\text{Mg}(\text{OH})_2$ nanopowders exclusively consist of lamellar-like structures and the sizes of $\text{Mg}(\text{OH})_2$ are 30–265 nm length or width. They have hexagonal shape. Some $\text{Mg}(\text{OH})_2$ particles were found standing straight on the copper grid, which demonstrates these lamellae have a thickness of about 11–19 nm. The above values are comparable with literature data [21,34–36].

4. Conclusions

The test results suggest that the serpentinite ore could be used as a source material for production of crystalline $\text{Mg}(\text{OH})_2$ nanopowders. The XRD patterns show that the obtained magnesium hydroxide with high purity has the brucite structure. TEM images, it can be seen that the sizes of $\text{Mg}(\text{OH})_2$ are 30–265 nm length or width and 11–19 nm thickness. They have very homogeneous structure without any observable pores. We believe that the present work will promote further experimental studies on the

Table 4
Weight percent of impurities in crystalline $\text{Mg}(\text{OH})_2$ nanopowder (ICP-OES).

Element	Content
Mg	Base
Ca	0.187
Fe	0.009
Si	0.410

Note: The content of Cr, Cu, S, Na, Mn, Zn, K is in minor amounts.

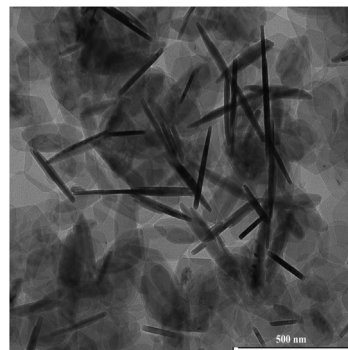


Fig. 5. TEM images of crystalline $\text{Mg}(\text{OH})_2$ nanoparticles obtained from serpentinite.

Table 5

The price of crystalline $\text{Mg}(\text{OH})_2$ nanopowder.

	Amount (kg)	Price (US\$/kg)
Serpentinite (s)	0.100	0.02
40% HNO_3 (aq)	0.330	0.97
MgO (s)	0.025	4.79
$\text{MgCO}_3 \cdot 3\text{H}_2\text{O}$ (s)	0.005	2.32
Hydroxyethylated nonylphenol (0.01 wt%) (aq)	0.00001	15.38
NH_4OH (mol 1%) (aq)	0.005	1.05
$\text{Mg}(\text{OH})_2$ nanopowder	0.101	4.68

physical properties and the applications of $\text{Mg}(\text{OH})_2$ nanomaterials in the fields such as high-density ceramics, additives in bactericide, refractory products.

Nitric acid used as reagent for obtaining $\text{Mg}(\text{OH})_2$ from serpentinite is relatively cheap (Table 5). Apart from the relatively cheap nitric acid reagent, the prospect of recovery and use of by-products of the process: silica, $\text{FeO}(\text{OH})$, magnesium nitrate presents significant benefits. The Fe-rich compound produced in significant amounts from the process may be a useful raw material in the iron and steel industry.

Interestingly, smaller volumetric amounts of ammonia solution are needed in the precipitation process (Table 5). ($\text{MgCO}_3 \cdot 3\text{H}_2\text{O}$) salt used as reagent for extracting Mg from the minerals is relatively cheap, and is a product and by-product of several chemical processes.

It was found that the process for the utilization of serpentinite ore is feasible and provides a potential usage of serpentinite waste in the future. It is essential for the economic viability of the method that the cost of crystalline $\text{Mg}(\text{OH})_2$ nanopowders is 4.68 US\$/kg (Table 5).

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