

ISSN 2010-0264



# IJESD

International Journal of Environmental Science and Development

Vol.8, No.1, January 2017

[www.ijesd.org](http://www.ijesd.org)

# International Journal of Environmental Science and Development (IJESD)

Vol. 8, No. 1, January 2017  
ISSN: 2010-0264

Chief Editor:  
Prof. Richard Haynes



# Industrial Waste as Raw Material for Producing Synthetic Wollastonite in Russia

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**Abstract**—In this paper we present the results of analysis of the scientific and technical literature concerning the ways of obtaining wollastonite from various calcium and silicon-containing technogenic wastes accumulated on the territory of the Russian Federation. We proposed a method for producing autoclave needle-like wollastonite from waste of boric acid (borogipsa) containing both calcium and silicon constituents. The phase composition and morphology of the products autoclaving borogipsa were studied. It is found that the initial product has a crystalline phase of unreacted calcium sulfate anhydrous, tobermorite and xonotlite. After firing at 1000 °C, the amorphous calcium hydrosilicates are converted to a crystalline phase of wollastonite triclinic modification. The effect of firing temperature on phase composition and morphology of the compounds obtained by autoclave treatment were described. It is shown that with increase of the firing temperature up to 1200 °C along with a phase change, the composition variation of the particle morphology occurs. It is found that the processing borogipsa to obtain needle-like wollastonite recommended firing temperature should not exceed 1000 °C.

**Index Terms**—Industrial waste, wollastonite, borogypsum.

## I. PROBLEM REVIEW

Main processing properties of wollastonite  $\text{Ca}_6\text{Si}_6\text{O}_{18}$  are high chemical resistance in different media, small specific gravity, unique dielectric properties and low thermal conductivity, as well as environmental friendliness and safety. This fact explains the widespread usage of wollastonite in the production of building materials, paper, paints, plastics, polymer composites and sintered materials, and sorbents for the purification of water. This mineral is an effective substitute for a number of materials like asbestos, kaolin, chalk, talc, titanium dioxide in the production of decorative materials, products on the basis of asbestos-cement, acoustic and thermal insulating materials etc. Wollastonite is widely used as a material of various additives in order to increase their strength, heat resistance, chemical resistance and durability, and to improve the electrical insulating

characteristics, reduce the duration of processes in their manufacture, and processing temperature reduction [1], [2]. The wollastonite market report performed by the research group "InfoMine", noted that the first place has got China in terms of production, consumption and export of wollastonite, where production of wollastonite exceeds 300 thousand tons a year. India takes the second place in the world in terms of production of wollastonite, the third – the USA, where production of wollastonite has been going on for over 60 years [3].

In the Russian Federation, despite the growing demand of wollastonite from the number sectors of domestic industry and explored deposits of this mineral, its production is practically absent. In this case the "wollastonite issue" repeatedly mentioned scientists working in the field of materials science, chemical engineering, geology, and economics [4]–[7].

However, the Russian Federation, has a large number of so-called "technogenic deposits" in addition to natural deposits of wollastonite which can be counted as a promising source of raw material for production of synthetic wollastonite. Analysis of ongoing research and existing scientific papers covering the period from the 1970s till today has shown that Russian and former Soviet Union scientists have been actively pursuing and carried out research related to the use of calcium and silicon technogenic waste for wollastonite. They have developed plenty of methods for producing synthetic wollastonite, such as the waste phosphogypsum, belite (nepheline) sludge, phosphoric slag, and waste production of aluminum fluoride. Moreover, in a number of works for the synthesis of wollastonite, two or more kinds of man-made wastes have been used. Some kinds of calcium and silicon waste, and links to a number of scientific works devoted to obtaining wollastonite using them, are given in the Table I.

As it can be seen from the Table I, there is a certain large inventory of technogenic raw material sources for wollastonite production. There is an example below illustrating the basic idea of the report, presented the results of studies on the production of synthetic wollastonite on the basis of waste production of boric acid, whose number in Russia exceeds 50 mln tons.

Since early 2007, the Institute of Chemistry of Far Eastern Branch of the Russian Academy of Sciences in cooperation with the Vladivostok State University of Economics and Service, Far Eastern Federal University and Armenian scientists actively carried out work related to the development of physical and chemical bases of complex processing of boric acid production waste – borogypsum – to give calcium hydrosilicates, wollastonite and potassium

Manuscript received March 28, 2016; revised July 5, 2016.

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fertilizers. Borogypsum main components are calcium sulfate dihydrate and amorphous silica. Borogypsum characterized by the following content of the main components, weight percentage: SiO<sub>2</sub> – 26–28; CaO – 26–28; SO<sub>4</sub><sup>2-</sup> – 38–40; Fe<sub>2</sub>O<sub>3</sub> – 1.8–2; Al<sub>2</sub>O<sub>3</sub> – 0.6–0.8; B<sub>2</sub>O<sub>3</sub> – 0.7–1.2; MnO – 0.2; MgO – 0.1–0.2. It is from 5 to 5.5 tons of borogypsum can be obtained from 1 ton of boric acid. Borogypsum is of particular interest because its composition contains both calcium and silicon components in an optimum ratio to obtain wollastonite, and therefore requires no additional sources of raw materials for blending initial mixture.

TABLE I: SOME TYPES OF "TECHNOGENIC DEPOSITS" USED FOR THE PRODUCTION OF SYNTHETIC WOLLASTONITE STUDIES SINCE 1970

No item	Waste type	Reference
Lime containing waste		
1	Phosphogypsum	[1], [8]–[14]
2	Soda ash waste	[15]
3	Waste of carbide calcium separation	[16]
4	A mixture of Cretaceous rocks (overburden)	[17]
Silicon-containing waste		
5	Aluminum fluoride production waste (silica gel)	[8]–[13], [18]
6	Waste from ferroalloy industry (microsilica)	[19]–[21]
7	Ash waste from the combustion of brown coal	[22]
8	Calx (crashed glass)	[23]
Waste containing calcium and silicon		
9	Belite (nepheline) slurry	[21], [24]–[30]
10	Phosphoric slag	[31]–[34]
11	Slag of metallurgical production	[35]
12	Borogypsum	[36]–[39]

As a result of research work, the kinetic characteristics of ultrasonic, microwave and autoclave synthesis of calcium silicates from borogypsum were analyzed [37]–[39]. Later studies have been conducted on the effect of additives on the basis of wollastonite obtained in autoclaved borogypsum on the functional properties of fine-grained concrete. Obtained data showed 3.5% additive based on wollastonite enables to increase the limit of fine-grained concrete compressive strength to 35% for pressure and up to 50% for bending, reduce water absorption and increase frost resistance [40].

Following upon researches obtained wollastonite by autoclave method with needle-shaped particles, which is an important aspect to consider when selecting field of application.

## II. EXPERIMENTAL PART

### A. Synthesis of Calcium Silicates

Borogypsum was mixed with a solution of potassium hydroxide in stoichiometric ratio. Synthesis was carried out in autoclave 4848, Parr Instrument Company (USA) at 220 °C for 3 hours. After specified time, the resulting mixture was removed from the autoclave, precipitate was washed with distilled water, heated to 60–70 °C, then was separated from the solution by filtration through a paper filter "blue tape" and dried at 85 °C for several hours.

### B. Analysis Methods

X-ray diffraction patterns were collected on a D8 Advance automatic diffractometer (sample rotation, CuK $\alpha$  radiation).

Phase compositions were determined using EVA search software and PDF 2 data.

The IR spectra of samples were recorded in the region of 400–2000 cm<sup>-1</sup> using a Shimadzu Prestige-21 FTIR Fourier-transform IR spectrometer at room temperature. Samples for recording were ground in an agate mortar to a finely dispersed state and deposited onto a KRS-5 substrate as a suspension in mineral oil.

The morphological characteristics of the samples were studied with a Hitachi S5500 high-resolution scanning electron microscope equipped with an attachment for scanning transmission microscopy and a Thermo Scientific energydispersive spectrometer.

## III. RESULTS AND DISCUSSION

According to the X-ray diffraction, a part of the synthesized sample detected crystalline phases unreacted anhydrous calcium sulfate CaSO<sub>4</sub>, tobermorite Ca<sub>5</sub>(OH)<sub>2</sub>Si<sub>6</sub>O<sub>16</sub>·4H<sub>2</sub>O and xonotlite Ca<sub>6</sub>[Si<sub>6</sub>O<sub>17</sub>](OH)<sub>2</sub>. After sample firing, there was a transition of amorphous calcium hydrosilicates into a crystalline phase of wollastonite of triclinic modification as identified by X-ray diffraction analysis of the sample after calcination at 1000 °C for 1 hour (Fig. 1). The precipitate obtained by firing at 1000 °C represents a sample composed of wollastonite phase (with a crystal unit cell parameters a=7.92580; b=7.32020; c=7.06530;  $\alpha$ =90.055;  $\beta$ =95.217;  $\gamma$ =103.426).

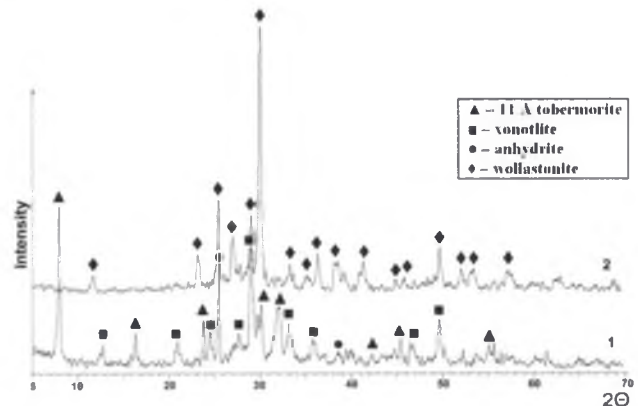


Fig. 1. X-ray diffraction patterns of the synthesized sample: 1 – before firing (initial sample); 2 – after firing at 1000 °C for 1 h.

Fig. 2 shows the IR spectra of synthesized sample before and after firing at 1000 °C for 1 h.

According to [41]–[43], it can be seen from the given IR spectra that synthesized calcium silicates are characterized by an intense absorption band produced by the asymmetric vibrations of bridging Si–O–Si bonds and the asymmetric and symmetric vibrations of terminal Si–O bonds in the region of 850–1100 cm<sup>-1</sup>. When the firing temperature is increased to 1000 °C, amorphous calcium silicates (tobermorite and xonotlite) is observed to transition into the crystalline wollastonite phase (spectrum 2). A set of bands in the region of 550–750 cm<sup>-1</sup> is assigned to the symmetric vibrations of bridging Si–O–Si bonds in [SiO<sub>4</sub>] tetrahedra. The absorption bands in the low-frequency region of 400–550 cm<sup>-1</sup> are due to the bending vibrations of terminal O–Si–O bonds and the vibrations of Ca–O bonds in [CaO<sub>6</sub>]

octahedra [41]–[43]. The absorption band at  $1600\text{ cm}^{-1}$  is due to the bending vibrations of crystallization water [44].

According to the scanning electron microscope, the resulting wollastonite consists of agglomerates of nanoscale particles having predominantly needle-like shape (Fig. 3). Anisotropy factor ( $L/d$ ) varies from 70 to 140.

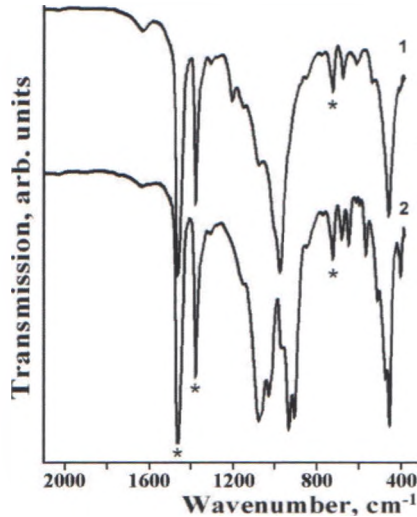


Fig. 2. IR spectra of the synthesized sample: 1 – before firing (initial sample); 2 – after firing at  $1000\text{ °C}$  for 1 h.

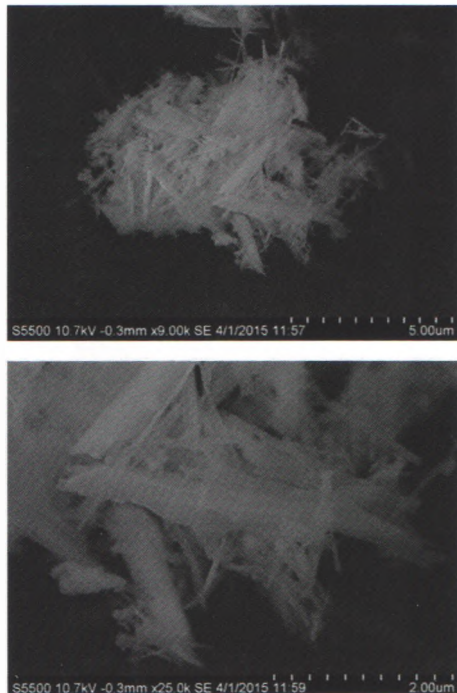


Fig. 3. SEM image of microparticles of wollastonite, the resulting autoclave synthesis from borogypsum.

Table II presents the data on the phase composition of calcium hydrosilicates samples depending on the temperature. This will give specific recommendations in selecting areas of application of the compounds in a variety of industries. This Table also shows the phase composition of the samples as a function of firing temperature.

As seen from the Table II, by firing the samples obtained at  $1000\text{ °C}$  and  $1100\text{ °C}$ , phase composition is characterized by the wollastonite triclinic modification, but different crystal lattice parameters, and after firing at  $1200\text{ °C}$ , the formation of high temperature polymorph modification of wollastonite

(pseudowollastonite  $\alpha\text{-CaSiO}_3$ ) occurs.

TABLE II: THE PHASE COMPOSITION OF THE SAMPLES AS A FUNCTION OF FIRING TEMPERATURE

Firing temperature, $^{\circ}\text{C}$	Phase composition	Crystal unit cell parameters
1000	Wollastonite ( $\text{CaSiO}_3$ )	PDF-2, 01-084-0654; a-7.92580; b-7.32020; c-7.06530; $\alpha=90.055$ ; $\beta=95.217$ ; $\gamma=103.426$
1100	Wollastonite ( $\text{CaSiO}_3$ )	PDF-2, 00-010-0487; a-7.88000; b-7.27000; c-7.03000; $\alpha=90.000$ ; $\beta=95.217$ ; $\gamma=103.200$
1200	Pseudo- wollastonite ( $\text{CaSiO}_3$ )	PDF-2, 01-089-6463; a-11.83220; b-6.86240; c-10.52970; $\alpha=90.000$ ; $\beta=111.245$ ; $\gamma=90.000$

As described above, wollastonite is formed as a result of firing at  $1000\text{ °C}$ , consisting mainly of agglomerates of nanoscale particles having a needle-like shape (Fig. 3).

Fig. 4 shows a sample image of the microparticles after firing at  $1100\text{ °C}$ .

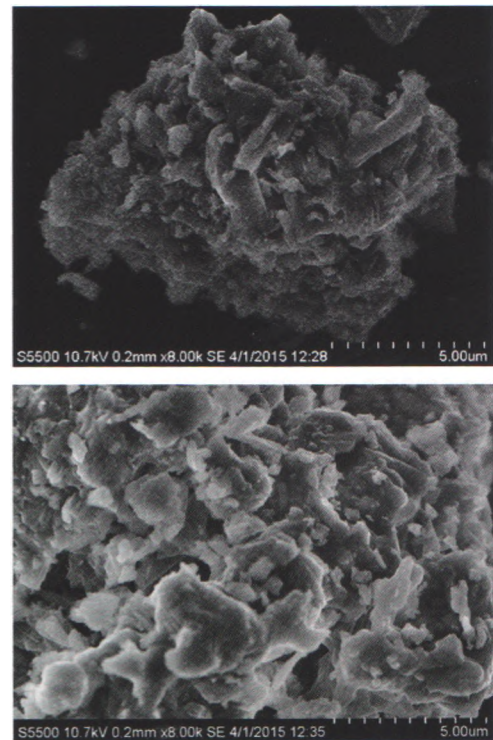


Fig. 4. SEM images of microparticles of wollastonite, the resulting synthesis autoclave, after firing at  $1100\text{ °C}$ .

As can be seen from Fig. 4, with an increase in the firing temperature to  $1100\text{ °C}$ , there is a change in particle morphology. In particular dense aggregates of particles of various shapes are formed, and the needle-like particles get disappeared.

Fig. 5 shows SEM images of microparticles of a sample, obtained by synthesis autoclave, after firing at  $1200\text{ °C}$ . As can be seen from the Fig. 5, as a result of the firing of the sample at  $1200\text{ °C}$ , the formation of mostly flat particles with distinct edges occurs. Particle size also increases. Needle-like

particles was not found.

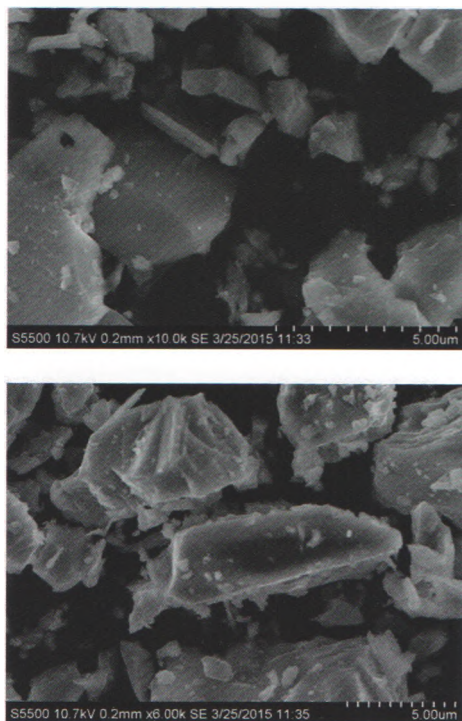


Fig. 5. SEM images of wollastonite microparticles, the resulting synthesis autoclave, after firing at 1200 °C.

This suggests that an increase in the firing temperature to 1200 °C along with a phase change, the composition variation of the particle morphology occurs. The main features of these changes are the disappearance of the needle-like particles, the appearance of dense particle aggregates of various shapes and particles enlargement. Thus, the borogipsa processing to obtain acicular wollastonite recommended firing temperature should not exceed 1000 °C.

#### IV. CONCLUSIONS

Thus, this study provides an overview of the scientific and technical literature, related to the problem of obtaining wollastonite from industrial waste in Russia. In addition, the authors of the work are presented results of own research on the synthesis of wollastonite from industrial waste. The possibility of obtaining needle-like wollastonite on the basis of boric acid production waste, the amount of which in the Far Eastern region of Russia exceeds 50 mln tons, was shown.

The study is advisable to make a conclusion that there are a number of backgrounds in Russia for the production of synthetic wollastonite:

- a wide range of calcium and silicon-containing industrial waste accumulated on the territory of the Russian Federation (nearly in every region) in large quantities;
- the results of carried out extensive research with the involvement of a industrial waste in the process of obtaining wollastonite;
- scientific research results allowing to obtain high quality wollastonite simultaneously from two or more kinds of anthropogenic waste containing calcium and silicon

components in an optimum ratio.

All of these conditions contribute to the reduction of Russia's demand for wollastonite raw materials and eliminate the need to import at the same time solving the problem of disposal of waste deposits.

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