



Utilization of serpentinite waste for the production of magnesium compounds

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Abstract. The article presents the results of research on the complex processing of powdered man-made serpentinite waste formed during the enrichment of chrysotile raw materials in order to obtain high-purity magnesium compounds and assess the potential of their use in the production of building materials. The relevance of the work is due to the need to dispose of accumulated waste and expand the raw material base for the construction industry within the framework of the principles of circular economy. The developed technology includes the stages of acidic leaching of magnesium from PTW with sulfuric acid, neutralization and purification of the solution using thermally activated PTW (TA-PTW) and subsequent precipitation of the target products. It was found that thermal activation of waste at 750 °C leads to dehydroxylation and the formation of highly reactive phases, forsterite and periclase, which significantly increases their sorption activity. The optimal leaching regime is recognized as the use of 0.7 stoichiometrically normal amount of H₂SO₄, which makes it possible to extract 82.5% of magnesium from the amount of acid introduced into the solution. The combined use of the initial and thermally activated PTW provides a degree of magnesium extraction of 52.3% of its total content in the system and effective purification of the magnesium sulfate solution from impurities of iron, aluminum, chromium and nickel. Sequential precipitation from the purified solution made it possible to obtain high-purity magnesium hydroxide with a calcium content of 0.0110%, and its subsequent calcination - magnesium oxide with a calcium content of 0.0187%. Special attention is paid to the prospects of practical application of synthesized compounds in the construction industry. It is shown that magnesium sulfate can be used as a modifying additive in cements and as a sealer for magnesia binders. Magnesium hydroxide is an effective flame retardant filler, and magnesium oxide is the main component for the production of flame-resistant and moisture-resistant magnesia binders and plates such as glass-magnesia sheets. Thus, the work demonstrates not only the technical feasibility of highly efficient processing of serpentinite waste, but also the significant resource potential of the resulting magnesium compounds for creating modern building materials with improved performance characteristics.

Keywords: serpentinite, magnesium sulfate, magnesium hydroxide, magnesium oxide, thermal activation, fire protection, building materials

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1. INTRODUCTION

Serpentine processing waste, which contains a significant amount of magnesium, represents an important secondary resource. The rational utilization of this waste not only contributes to the reduction of environmental impact, but also enables the production of valuable products for the chemical, agricultural, and metallurgical industries.

Technogenic serpentine waste from the mining industry typically contains 40–45 wt.% Mg and 35–45 wt.% SiO₂, along with minor minerals containing iron, aluminum, chromium, and other elements [1]. Given their high magnesium content, such wastes can be considered an alternative raw material for the production of industrially significant magnesium compounds due to their availability and low cost. Such magnesium compounds include magnesium salts (MgSO₄, MgCl₂, Mg(NO₃)₂, Mg(OH)₂, and MgO), which are widely used in the production of construction materials, as well as in the chemical and agrochemical industries, metallurgy, and other sectors. In Kazakhstan, where an operating chrysotile production enterprise exists, the amount of such waste tends to increase. The increasing accumulation of industrial waste from chrysotile production, in particular serpentine tailings, represents both an environmental problem and an opportunity for resource utilization. The need for resource diversification, environmental protection, and socio-economic development justifies the search for efficient methods to extract valuable magnesium compounds from serpentine, as well as for the development of innovative processing approaches. For investors, a key criterion in evaluating the applicability of any new technology is the ability to extract the magnesium-containing fraction from serpentine in the form of high-quality (especially high-purity) and cost-effective magnesium compounds. To date, numerous methods for the technological processing of serpentine have been developed and proposed (hereinafter referred to simply as “serpentine,” as the composition of serpentine waste slightly varies depending on the specific deposit). Most of these methods focus on the extraction of magnesium-containing components from minerals with the general formula Mg₃Si₄O₁₀(OH)₂, whose composition mainly includes chrysotile, antigorite, and lizardite [2]. Most published studies aimed at identifying effective methods for magnesium extraction have focused on acid treatment of serpentine [3–6]. The differences between these studies and their respective process schemes lie in the type of acid used, the conditions under which the processes are carried out, and the methods of purifying the resulting solutions from impurity metal ions. It should be emphasized that all currently known schemes and methods for acid processing of serpentine have not yet been widely implemented in practice. Although each method has its advantages, they also present certain limitations that hinder their application. Examples of industrially significant magnesium compounds obtained from serpentine can be found in the literature: magnesium sulfate [7, 8], magnesium hydroxide [9, 10], and magnesium oxide [11]. However, the fact that the acid leaching of serpentine has been actively studied over the past decades once again demonstrates that serpentinites and technogenic wastes from chrysotile raw material processing are accessible, promising, and alternative raw materials for the production of important magnesium compounds. Acid leaching makes it possible to transfer magnesium into a soluble form, accompanied by the formation of a silica gel (SiO₂·nH₂O), which is the main problem in the production of silicate-containing materials. In many cases, concentrated mineral acid solutions are employed to maximize magnesium extraction from serpentine [12]. However, the use of acid concentrations exceeding the stoichiometrically required level further intensifies the adverse effects of this acid decomposition factor. Despite the widespread application of acid decomposition, the applicability limits of the surface-chemical model and the diffusion regime through product layers remain the subject of ongoing scientific debate [13]. Another important issue is that leaching is accompanied by the transfer of impurity metals into the solution, which reduces the quality of the final product. Effective purification is required to obtain magnesium compounds with

high purity. This fact is particularly relevant when the processed raw material is serpentinite tailings from chrysotile production. The stage of neutralization and purification of the acid leaching solution is critical, especially for ensuring the production of magnesium hydroxide and magnesium oxide with high purity indicators based on magnesium salts. The approach proposed in this work, involving the use of thermally activated PTW for the neutralization and purification of the productive magnesium sulfate leach solution, was determined by the following characteristic physicochemical properties: (1) enhanced alkalinity acquired as a result of the chemical transformation of brucite components in the serpentine structure $[\text{Mg}(\text{OH})_2(\text{MgOH})_2\text{Si}_2\text{O}_5]$ into MgO during calcination at 750°C . MgO exhibits higher alkalinity and greater solubility in water [14]; (2) higher sorption activity [15, 16] compared to non-thermally activated serpentinite.

In this study, powdered technological waste (PTW) from chrysotile beneficiation at JSC “Kostanay Minerals” (Kazakhstan) was used as a source of magnesium for the production of magnesium compounds.

Based on the above, the objective of the present work is to investigate the effect of the stoichiometrically required amount of sulfuric acid and thermally activated powdered technological waste (TA-PTW) on the degree of magnesium leaching and on the purity of the magnesium compounds obtained according to the following sequence: $\text{PTW} \rightarrow \text{MgSO}_4 \rightarrow \text{Mg}(\text{OH})_2 \rightarrow \text{MgO}$.

2. METHODS AND MATERIALS

2.1 Effect of SNQ H_2SO_4 on Magnesium Leaching from PTW

The chemical and mineralogical composition of PTW differs little from that of the original chrysotile feed. It is a bluish-gray loose powder without lumps or hard particles. PTW is formed in the baghouse dust collection systems of production facilities, where crushing and fractionation of chrysotile raw material take place. The chemical composition of PTW is presented in Table 1.

Table 1. Elemental composition of PTW.

Element (symbol)	C	O	Mg	Al	Si	S	Ca	Fe	Total
PTW elemental composition, wt. %	2.58	51.00	25.00	0.54	17.45	n.d.	0.50	2.93	100.00
Note: n.d. – not detected.									

A 20 g PTW sample was ground and sieved, and the fraction with a particle size of <0.14 mm was chosen for the experiments, with a weight of 10.0 g.

The stoichiometric amount of sulfuric acid required for the interaction with PTW was calculated according to the following reaction:



The mass and volume of 200 mL of sulfuric acid solution containing the stoichiometric amount were calculated using the following equation:

$$m = C \cdot M_r \cdot V \cdot 100 / 92. \quad (2)$$

Solutions containing the stoichiometric amount of sulfuric acid were prepared from 92% H_2SO_4 of analytical grade. The calculations for preparing the solution with the required molar concentration were carried out according to the following equations:

$$m = C \cdot M_r \cdot V \cdot 100/92 = C \cdot 21.304, \quad (3)$$

$$V = m/d = C \cdot 21.304/d, \quad (4)$$

where m is the mass of H_2SO_4 (92%) required to prepare a solution of the specified molar concentration; C is the molar concentration; M_r is the molar mass; V is the volume; and d is the density of 92% H_2SO_4 . All experiments on magnesium leaching from 10 g of PTW were carried out using 118 mL of solution, prepared separately, containing (0.1–1.0) SNQ of H_2SO_4 .

The final concentration of H_2SO_4 (mol/L) after each experiment (based on the interaction between SNQ H_2SO_4 and PTW) was determined by titration with NaOH solution and calculated according to the formula:

$$C_{H_2SO_4} = C_{NaOH} \cdot V_{NaOH} / 2V_{H_2SO_4}. \quad (5)$$

The amounts of initial and final H_2SO_4 (mol) in the solution were calculated using the formula:

$$n_{H_2SO_4} = C_{H_2SO_4} \cdot V_{H_2SO_4} / 1000 \quad (6)$$

The amount of H_2SO_4 (mol) consumed for the dissolution of PTW was determined according to the formula:

$$n_{consum.} = n_{init.} - n_{final.} \quad (7)$$

In percentage terms, it was calculated by the following equation:

$$H_2SO_{4consum.} (\%) = n_{consum.} \cdot 100 / n_{init.} \quad (8)$$

Samples (10 g of PTW) were dissolved in separate 118 mL aqueous solutions with concentrations ranging from 0 to 1.0 SNQ H_2SO_4 , with an increment of 0.1 SNQ, at a liquid-to-solid ratio (L:S) of 10:1, at 80 °C for 10 minutes in a sealed Erlenmeyer flask equipped with a reflux condenser. Experiments were conducted at Mg(PTW):0.1–1.0 SNQ (H_2SO_4) ratios. The PTW suspension in H_2SO_4 solution was stirred at 350 rpm. After the reaction time elapsed, the suspensions were immediately filtered (blue filter paper). The filtrate and the insoluble residue were subjected to elemental analysis using a JSP-6490LV (JEOL, Japan) equipped with an INCA Energy 350 energy-dispersive microanalysis system. The final products $MgSO_4$, $Mg(OH)_2$, and MgO were analyzed by inductively coupled plasma mass spectrometry (ICP-MS, Thermo iCAP-Q, Thermo Scientific, Germany). The acidity of the reaction medium (pH) was measured using an I-160MI pH/ion meter.

The extraction degree of Mg (P_{Mg}) and other elements (R_E) from 10 g of PTW by H_2SO_4 solutions was calculated as the ratio of the analytical amount of the element in the filtrate to its amount in the initial PTW sample, according to the formula:

$$P_{Mg} = \frac{m \cdot Mg, \%}{2.538} \quad (9)$$

where P_{Mg} is the mass of extracted magnesium; m is the mass of the dry residue of the filtrate (g); $Mg, \%$ is the magnesium content in the dry filtrate residue according to chemical analysis; 2.538 is the magnesium content in the initial 10 g PTW sample according to chemical analysis. The extraction degrees of Fe, Ca, and Si were determined in a similar manner.

2.2 Thermal treatment

The thermal treatment of PTW, used as a reagent for the neutralization and purification of the sulfate leach solution, was carried out at 750 °C for one hour. Thermal treatment of PTW resulted in finer particle disintegration, with no fractions smaller than <0.9 mm present. The main fraction (94%) was in the range of 0.14 ÷ 0.08 ÷ 0.07 mm. In the experiments, this fraction was used without additional grinding.

2.3 X-ray diffraction (XRD)

X-ray diffraction (XRD) patterns were recorded using a D8 Advance diffractometer (Bruker AXS GmbH, Karlsruhe, Germany) operating with Cu-K α radiation at 40 kV and 40 mA. Diffraction data were processed using the EVA software package, and phase identification was carried out using the Search/Match function based on the PDF-2 Powder Diffraction File (JCPDS-ICDD).

3. RESULTS AND DISCUSSION

3.1 X-ray Diffraction Analysis of Products Obtained after Acid and Thermal Treatment of PTW

The monitoring of changes in the phase composition of PTW subjected to acid and thermal treatment was carried out using X-ray diffraction (XRD) analysis of the products obtained after these processes. XRD analysis makes it possible to trace mineralogical transformations occurring during the leaching of PTW and the thermal transformations during its heat treatment. This method also provides the opportunity to identify the formation of secondary phases and the amorphous Si-rich layer.

In Fig. 1, the diffraction pattern of the initial PTW (dried at 105 °C) is presented. The diffractogram of the untreated PTW clearly shows the interplanar spacing (d/n) peaks of chrysotile $\text{Mg}_6[\text{Si}_4\text{O}_{10}](\text{OH})_8$ at d/n values of 7.38, 4.619, 3.661, 2.487, 2.141, and 1.53 Å, which represent the main phase, as well as the interplanar spacing peaks of a considerable amount of brucite $\text{Mg}(\text{OH})_2$ at d/n = 4.77, 2.365, and 1.794 Å.

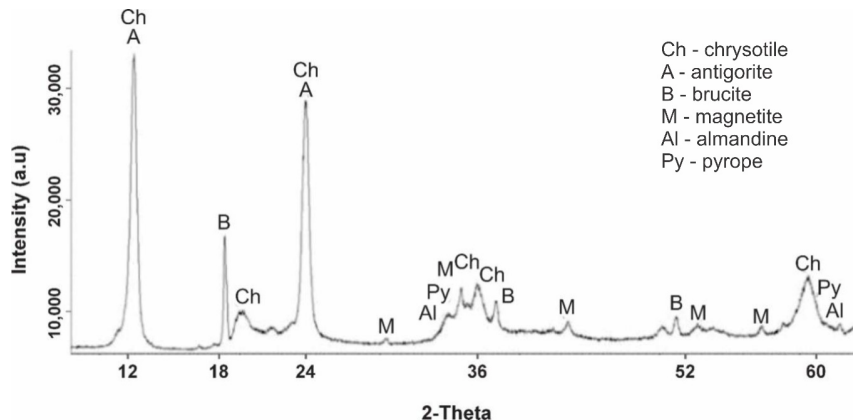


Fig. 1. Diffractogram of the initial PTW (dried at 105 °C).

The diffractogram also shows overlapping interplanar spacing (d/n) peaks corresponding to a minor amount of antigorite at d/n = 7.30, 3.63, and 2.52 Å, a small amount of magnetite $\text{Fe}[\text{Fe}_2\text{O}_3]$ at d/n = 2.99, 2.541, 2.098, 1.710, and 1.612 Å, as well as very weak peaks of pyrope $\text{Mg}_3\text{Al}_2[\text{SiO}_4]_3$ at d/n = 2.92, 2.69, and 1.50 Å, and almandine $\text{Fe}_3\text{Al}_2[\text{SiO}_4]_3$ at d/n = 2.90, 2.60, and 1.51 Å. Thus, according to X-ray diffraction analysis, six phases are present in the initial PTW.

For the acid treatment, the concentration of sulfuric acid was selected as 1.0 SNQ H_2SO_4 (relative to the Mg content in the PTW sample) at a liquid-to-solid ratio of 10:1 and a temperature of 80 °C. The diffractogram of PTW obtained after sulfuric acid treatment shows that, out of the six phases, the interplanar spacing (d/n) peaks of brucite $\text{Mg}(\text{OH})_2$ at 4.77, 2.365, and 1.794 Å disappear, while the intensity of the chrysotile peaks at d/n = 7.38, 3.661, 2.487, and 1.53 Å, as well as the antigorite peaks at d/n = 7.30, 3.63, and 2.52 Å, decreases (Fig. 2).

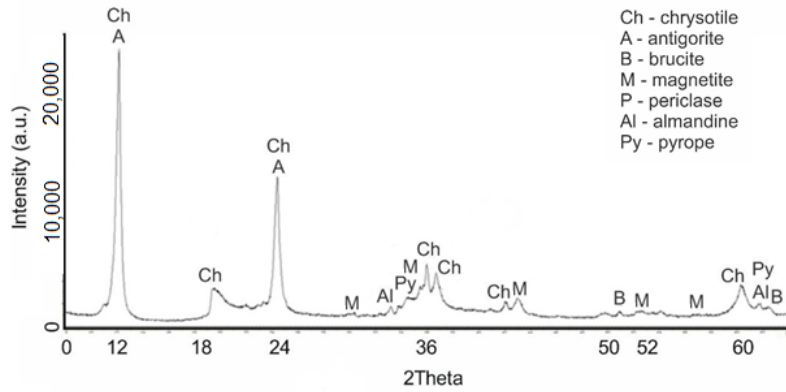


Fig. 2. Diffractogram of the initial PTW after acid treatment (1.0 SNQ H₂SO₄).

The disappearance of brucite peaks is clearly associated with its participation in acid–base interactions with sulfuric acid. A decrease in the peaks of serpentine group minerals (chrysotile and antigorite) is also observed, apparently due to the formation of amorphous silica during the dissolution of PTW in H₂SO₄.

For the thermal activation of PTW, a calcination temperature of 750 °C for 1 h was selected, since at this temperature the structure of serpentine minerals [Mg₃Si₂O₅(OH)₄] undergoes complete decomposition [17] through dehydroxylation. In the proposed method, thermally activated PTW acts as a neutralizing agent. Therefore, XRD studies were conducted to monitor phase transformations both during thermal treatment (at 750 °C) and during acid treatment with sulfuric acid solution. Upon calcination of PTW at 750 °C, the XRD patterns (Fig. 3) show the disappearance of the diffraction characteristics of chrysotile and brucite, with the formation of forsterite ($d/n = 3.875, 3.47, 2.753, 2.497, 2.441$ Å), magnetite ($d/n = 2.99, 2.541, 1.710$ Å), diopside ($d/n = 2.99, 2.894, 2.56, 2.04$ Å), periclase ($d/n = 2.431, 2.108, 1.48$ Å), d-tridymite ($d/n = 4.39, 4.12, 3.73$ Å), pyrope ($d/n = 2.92, 2.69, 1.50$ Å), and almandine ($d/n = 2.92, 2.69, 1.50$ Å). The predominant phases are forsterite, magnetite, and periclase.

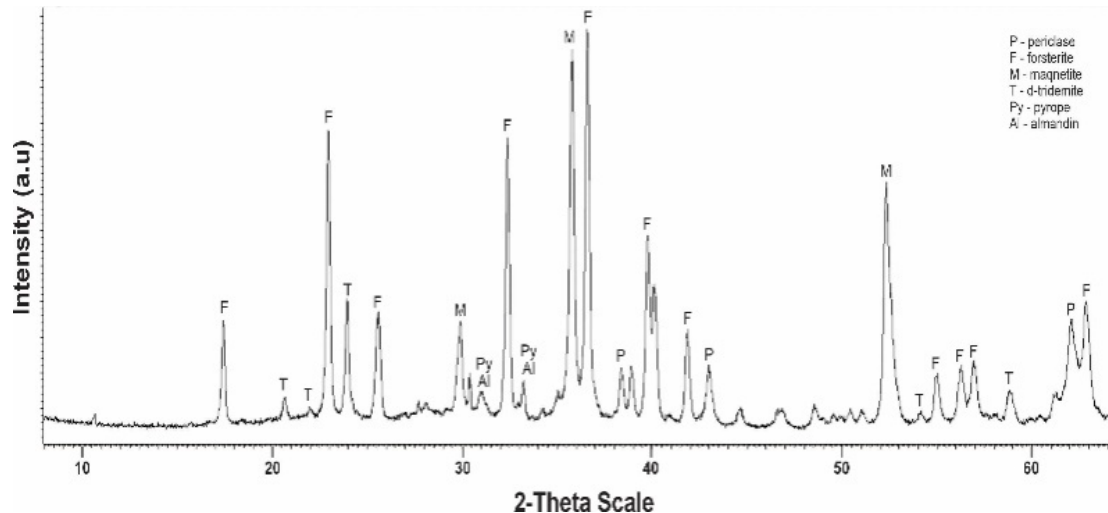


Fig. 3. Diffractogram of PTW calcined at 750 °C for 1 h.

The diffractogram of PTW calcined at 750 °C (Fig. 4) after acid treatment is characterized by the absence of interplanar spacing (d/n) peaks of diopside. The intensity of pyrope peaks at $d/n = 2.92, 2.69, \text{ and } 1.50$ Å and almandine peaks at $d/n = 2.92, 2.69, \text{ and } 1.50$ Å increases significantly. From the diffraction characteristics of d-tridymite with interplanar spacings of $d/n = 4.39, 4.12, 3.73, 2.49, 1.69,$

and 1.52 Å, only the peaks at $d/n = 1.69$ and 1.52 Å could be identified due to broad humps and overlapping shoulders of the diffractogram in the 5–3 Å region. The peaks of forsterite ($d/n = 3.875$, 3.47, 2.753, 2.497, 2.441 Å) and periclase ($d/n = 2.431$, 2.108, 1.48 Å) are significantly reduced, which is associated with their participation in acid–base interactions during the treatment of thermally activated PTW with sulfuric acid.

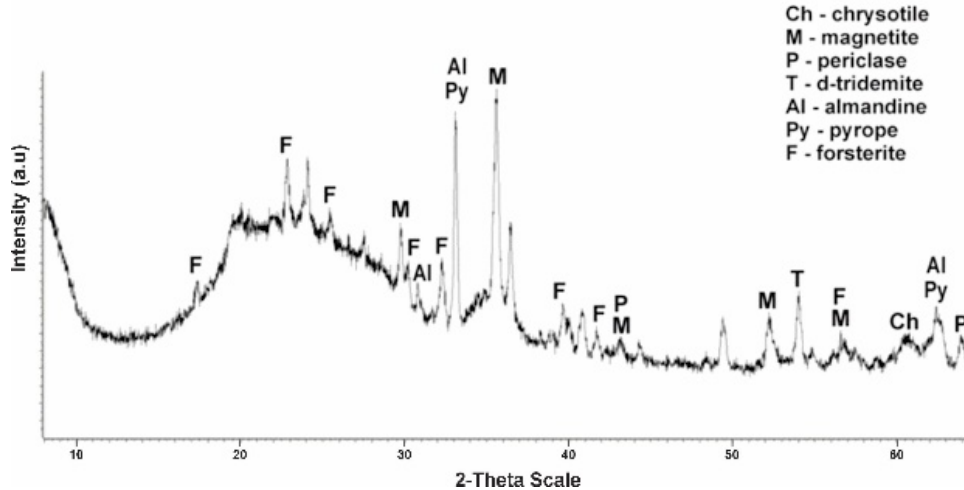


Fig. 4. Diffractogram of PTW (750 °C, 1 h) obtained after acid treatment (1.0 SNQ H₂SO₄).

Thus, during the thermal treatment of PTW at 750 °C, the serpentinite structure Mg₆Si₄O₁₀(OH)₈ is completely destroyed. It can be assumed that the products of its dehydrosilication formed at 750 °C are subjected to acid attack, which significantly affects the dissolution behavior in sulfuric acid. Table 2 presents the results of magnesium distribution in the dissolution products of the initial PTW and thermally activated TA-PTW.

Table 2. Distribution of magnesium in the dissolution products of PTW and TA-PTW in sulfuric acid, C(H₂SO₄) = 1.0 SNQ, t = 80 °C, V = const (100 mL).

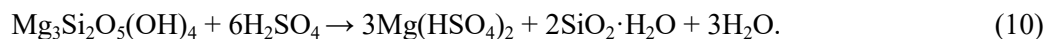
Sample No.	Sample	Amount of MgO in sample, g	MgO transferred into solution		MgO in insoluble residue	
			g	%	g	%
1	PTW (initial)	4,16	1,95	47,0	2,18	52,2
2	TA-PTW	4,66*	4,08	87,5	0,57	12,2

Note: * – the amount of MgO in the sample increases due to water loss during calcination.

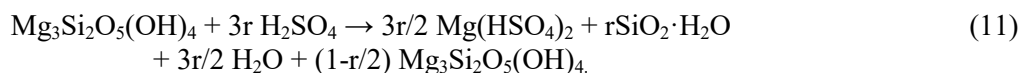
During thermal activation (TA-PTW), the amount of MgO transferred into solution from PTW increases significantly (from 47% to 87.5%), which indicates the higher reactivity of TA-PTW toward the acidic medium.

3.2 Study of Interaction Processes in the “PTW–H₂SO₄” System

The investigation of the quantitative interaction between magnesium (Mg) in PTW and sulfuric acid, as well as the molar ratio between the consumed H⁺ ions and the extracted Mg²⁺ ions in solution, makes it possible to determine the effective stoichiometry of the process and to establish material balances. The overall reaction of PTW with sulfuric acid can be described as follows:



For 1 mol of Mg, the formation of $\text{Mg}(\text{HSO}_4)_2$ requires 2.0 mol of H_2SO_4 . Consequently, if r mol of H_2SO_4 is supplied per 1 mol of Mg, the fraction of reacted magnesium will be $f = r/2$, or equivalently r mol of H_2SO_4 per 1 mol of Mg. With a gradual increase in r (0.1; 0.2; 0.3; ... 1.0 SNQ H_2SO_4), the amount of sulfuric acid is sufficient only for interaction with the equivalent amount of Mg in PTW. Thus, for 1 mol of $\text{Mg}_3\text{Si}_2\text{O}_5(\text{OH})_4$ (containing 3 mol of Mg), $3r$ mol of acid is supplied, and the reaction equation can be expressed as follows:



Such an equation would imply that the fraction of the mineral (PTW) that fully reacts to form $\text{Mg}(\text{HSO}_4)_2$ and $\text{SiO}_2 \cdot \text{H}_2\text{O}$ is equal to $r/2$, while the remaining portion $(1 - r/2)$ remains unchanged. The balance under this representation is maintained for any value of $0 \leq r \leq 2$. The hydration of SiO_2 does not affect the yield of Mg, but it significantly alters the mass and properties of the Si-rich layer. To study the dissolution processes of serpentinite in this context, experiments on the quantitative interaction in the “PTW– H_2SO_4 ” system were conducted.

Fig. 5 presents the results of the experimental determination of Mg extraction from (Mg)PTW using solutions containing 0.1 to 1.0 SNQ H_2SO_4 . In the range of 0.1–0.3 SNQ H_2SO_4 , a linear dependence is observed, which characterizes the equivalence of the extracted amount of Mg^{2+} relative to the applied SNQ H_2SO_4 , with the Mg^{2+} yield into solution approaching 100%. However, in the range of 0.4–0.5 SNQ H_2SO_4 , a noticeable deviation (plateau) from the equivalent extraction of Mg is observed, which may be associated with the appearance of silicate ions in the system.

Table 3 shows the results of Mg and Si analyses obtained during the study of their dissolution degree as a function of the amount of H_2SO_4 . It can be seen that in the range of 0.3–0.5 SNQ H_2SO_4 there is a significant increase in the Si concentration in solution. At the same time, the formation of acidic forms (HSiO_4^{3-} , HSiO_3^{2-}) also depends on the concentration of $[\text{H}^+]$ ions.

Table 3. Concentrations of Mg, Si, and pH values in solutions during PTW dissolution as a function of H_2SO_4 content (0.1–1.0 SNQ H_2SO_4).

Elements and pH	Concentration, mol/L									
	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9	1.0
Mg	10.46	19.62	30.33	33.79	35.08	43.66	53.50	56.33	52.22	54.50
Si	1.24	3.24	3.12	9.23	14.30	14.65	10.68	15.17	23.90	29.45
pH	6.71	3.98	2.79	1.87	1.36	1.24	1.06	0.92	0.81	0.72

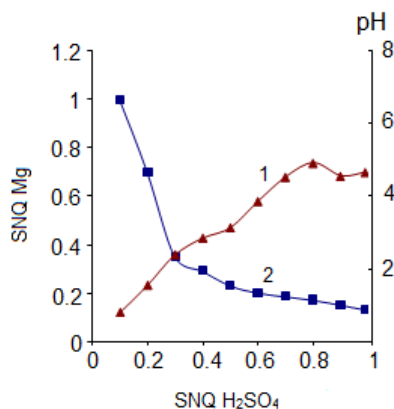


Fig. 5. Correlation between the extracted equivalent amount of Mg into solution from PTW upon treatment of 10 g of PTW with H₂SO₄ solutions containing 0.1–1.0 SNQ H₂SO₄ (1), and the change in pH (2).

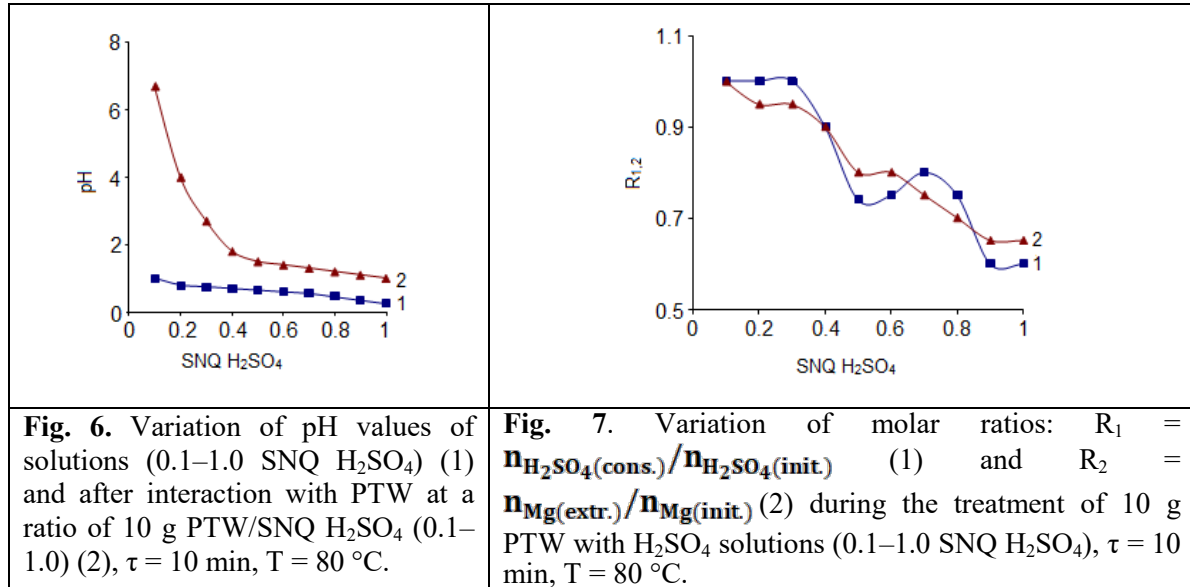
Apparently, in this acid concentration range, several competing reactions involving H⁺ ions occur, which is confirmed by the change in pH at the intersection point when using 0.3 SNQ H₂SO₄. The structure of serpentine Mg₃Si₂O₅(OH)₄ can be conveniently represented as Mg(OH)₂ – (MgOH)₂Si₂O₅. From this structure, it follows that 1/3 of the magnesium in serpentine is present in the form of Mg(OH)₂, while the remaining 2/3 are in the form of (MgOH)₂Si₂O₅. The analysis of the molar ratio H⁺/Mg²⁺ makes it possible to determine the controlling stage. In the range of 0.1–0.3 SNQ H₂SO₄, the amount of extracted magnesium increases linearly with acid concentration (equivalence); however, signs of diffusion limitation begin to appear thereafter. The classical framework for kinetic analysis of serpentine dissolution in acids includes the Shrinking Core Model (SCM) and its modifications [18]. Nonlinear transitions to mixed control due to the growth of the product layer [19], as well as the significant influence of pH/H⁺ activity, have been reported in [20] and [21]. The results obtained from the study of quantitative interactions in the “PTW–H₂SO₄” system (Fig. 5) allow, to some extent, quantitative evaluation of the contributions of the surface reaction [chemical control: $1 - (1 - x)^{1/3} = k_r t$] and the diffusion regime [diffusion control through the product layer: $1 - 3(1 - x)^{2/3} + 2(1 - x) = k_d t$], where k_r and k_d are the rate constants of the reaction and diffusion, respectively, and x is the fraction of extracted Mg during PTW dissolution in H₂SO₄. These findings make it possible to define the criteria for transition between regimes for practical applications.

In this context, the stoichiometric extraction of Mg at varying acid amounts within the range of 0.1–0.3 SNQ H₂SO₄ per 0.1 mol of Mg is governed by the material balance. An increase in the number of H⁺ equivalents almost proportionally increases the amount of extracted Mg, since the properties of the product shell (porosity) change only slightly within this range. A quasi-linear section of the dependence “Mg yield – [H₂SO₄]” is observed, where the reaction order n is close to unity [22]. In the range of 0.5–0.8 SNQ H₂SO₄, a second quasi-linear section is also observed; however, in this interval, the Mg yield is not stoichiometric, and the extraction degree does not exceed 70%. Similar patterns have been reported in studies of acid leaching of serpentine and laterites [18], where linear growth of extraction degree was observed within narrow acid concentration ranges under diffusion control.

It can be assumed that stoichiometric Mg extraction is typical of the brucite layer (tetrahedral), while under mixed and diffusion control the extraction of Mg mainly occurs from the [Mg(OH)₂Si₂O₅] structural component of serpentine (from the octahedral sublayer of the tetrahedral–octahedral layered pair). At concentrations of 0.7–1.0 SNQ H₂SO₄, an Si-rich surface layer apparently forms on PTW particles, preventing further increase in Mg extraction from (Mg)PTW. In separate experiments using H₂SO₄ concentrations >1.0 SNQ (1.2–1.4), it was not possible to achieve Mg extraction degrees higher than 76%. Thus, the contribution of diffusion control can be estimated in that the formation of an Si-rich layer may reduce the overall leaching degree by ~25%.

The study of pH variations in the range of 0.1–1.0 SNQ H₂SO₄ before and after the PTW–H₂SO₄ interaction showed that, at the end of the process, a certain amount of free H⁺ ions always remains

(Fig. 6), unreacted with PTW. The relationship between sulfuric acid consumption and the extracted amount of Mg from (Mg)PTW is also evident from Fig. 7, where the amount of unreacted acid correlates with the value of free acid in the solution after interaction, determined by NaOH titration. These data served as the basis for selecting 0.7 SNQ H₂SO₄ as the most optimal concentration in terms of rational utilization for acid leaching of Mg from PTW.



3.3 Production of High-Purity Magnesium Sulfate from PTW Using 0.7 SNQ H₂SO₄ and TA-PTW

To evaluate the efficiency of 0.7 SNQ H₂SO₄ for leaching magnesium from PTW and thermally activated TA-PTW in the production of high-purity magnesium sulfate, separate experiments (in triplicate) were carried out. PTW with the composition given in Table 1 was used for leaching. A 100 g portion of PTW was leached with a solution containing 70% of SNQ H₂SO₄ at 80 °C. The liquid-to-solid ratio (L:S) was 2.78:1. The leaching process lasted 30 minutes, selected based on the results of kinetic studies [14]. The total mass of the working suspension was 441.82 g. From the resulting light-blue acidic suspension (after leaching), a 20 mL sample was taken for analysis. The sample was filtered, washed, dried at 105 °C, and the dry residue amounted to 5.502 g. After the leaching step, TA-PTW was gradually added to the suspension, while monitoring the pH after each addition (every 1–2 min), maintaining the L:S ratio at 3:1 by adding hot water. The addition of TA-PTW was stopped when pH = 8.3 was reached. The total consumption of TA-PTW was 30 g. From the neutralized suspension, a 20 mL sample was also taken for analysis, yielding a dry residue of 4.883 g. The main portion of the suspension was filtered (blue-ribbon filter) and washed with distilled water by decantation. As a result, a colorless transparent filtrate with a volume of $V = 515.86$ mL was obtained. Table 4 presents the results of filtrate analyses after acid leaching and subsequent neutralization with TA-PTW, determined using an ICP-MS instrument (Thermo iCAP-Q), except for silicon.

Table 4. Analytical data of the initial PTW, thermally activated TA-PTW, and the averaged results of filtrate analyses obtained after acid leaching and subsequent neutralization with TA-PTW.

Samples and filtrates	Mg	Fe	Si*	Ca	Cr	Al	Ni
PTW (initial)	25.04	4.46	17.80	0.30	0.34	0.42	0.13
TA-PTW	28.17	4.52	19.67	0.21	0.27	0.53	0.11
Filtrate (after acid leaching)	11.89	1.31	0.21	0.76	0.06	0.31	0.08
Relative error	± 0.2	± 0.1	± 0.04	± 0.1	± 0.01	± 0.05	± 0.02
Filtrate (after neutralization with TA-PTW)	13.92	n.d.	n.d.	0.23	n.d.	n.d.	n.d.
Relative error	± 0.4	n.d.	n.d.	± 0.05	n.d.	n.d.	n.d.

*Note: The silicon data were obtained using a JSP-6490 LV scanning electron microscope equipped with an INCA Energy 350 microanalyzer.

From Table 3 it can be observed that the acid leaching solution is significantly purified from impurity metal ions, except for calcium (which decreases only by approximately three times).

Calculations based on the analysis of the acid leachate filtrates show that the magnesium (Mg) yield relative to its content in the initial PTW is 58%. The yield relative to the amount of sulfuric acid (0.7 SNQ H_2SO_4) used for leaching is 82.5%. The results of the analysis of the neutralized filtrate (to pH = 8.3 with TA-PTW) and the calculations based on them showed a magnesium yield of 52.3% relative to the total magnesium content in [PTW + TA-PTW]. The decrease in leaching efficiency is apparently associated with the low residual concentration of H^+ in the acid leachate solution (Figure 6) and the relatively high consumption of TA-PTW.

3.4 Production of Magnesium Hydroxide and Magnesium Oxide

Since acid leaching of PTW with 0.7 SNQ H_2SO_4 solution followed by purification with TA-PTW makes it possible to obtain a high-purity MgSO_4 solution, subsequent application of well-known precipitation operations [23] enables the production of magnesium hydroxide and magnesium oxide with high purity. In this case, magnesium hydroxide was obtained from the magnesium sulfate solution as follows: into 250 mL of a 19.5% (195 g/L) magnesium sulfate solution, 131.5 mL of a 25% NaOH solution was gradually added under moderate stirring. When pH = 9.5 was reached, the solution became turbid. At the end of the process, the suspension reached pH = 12.5. The suspension was kept for 1 h under mild stirring and then allowed to stand for 2 h to thicken the magnesium hydroxide precipitate and cool the suspension. The precipitate was filtered and washed twice by decantation with 0.01% NaOH and distilled water. The mass of $\text{Mg}(\text{OH})_2$ after drying at 105 °C to constant weight was 23.09 g. The yield of $\text{Mg}(\text{OH})_2$ relative to the magnesium content in the solution was 98%. Analysis of magnesium hydroxide (ICP-MS, Thermo iCAP-Q) confirmed the high purity of the $\text{Mg}(\text{OH})_2$ precipitate, with only minor calcium contamination (0.0110%).

Magnesium oxide was obtained from magnesium hydroxide by programmed high-temperature calcination. The process of MgO production consisted of the thermal decomposition of magnesium hydroxide into magnesium oxide within the temperature range of 20–1100 °C. Calcination was performed in a muffle furnace under a programmed heating regime. Overall, the calcination of 20 g of magnesium hydroxide required 3–5 h. The yield of magnesium oxide was 13.6 g, corresponding to 98.6% of the theoretical value.

The periclase obtained by calcination (based on ICP-MS, Thermo iCAP-Q analysis) was practically free of impurity elements, except for calcium (0.0187%).

3.5 Prospects of application of the obtained magnesium compounds in building materials

High purity of magnesium compounds obtained according to the developed scheme – magnesium sulfate (MgSO_4), magnesium hydroxide ($\text{Mg}(\text{OH})_2$) and magnesium oxide (MgO) – allows them to be considered as valuable raw materials for the production of a wide range of building materials. The use

of man-made serpentinite waste for their production not only solves environmental problems, but also complies with the principles of circular economy in the construction industry.

Magnesium sulfate can be used as a modifying additive in cement systems and as a component for magnesia binders. Its use makes it possible to regulate the rate of setting and hardening, as well as to increase the strength characteristics of composites in the early stages [24]. As a sealer for magnesia cements, magnesium sulfate, in comparison with magnesium chloride, makes it possible to obtain materials with reduced hygroscopicity and a tendency to fade [25]. The requirements for sulfate-based additives are regulated in the GOST 24640-91.

Magnesium hydroxide is a highly effective flame retardant agent. When heated to 300-400 °C, it decomposes endothermically with the release of water, absorbing a significant amount of heat and preventing the spread of flame. This property makes it a valuable non-toxic filler for flame-retardant paints, coatings for metal and wooden structures, as well as for polymer composites used in construction (cable insulation, roofing membranes) [26]. Magnesium hydroxide is a highly effective flame retardant agent. When heated to 300-400 °C, it decomposes endothermically with the release of water, absorbing a significant amount of heat and preventing the spread of flame. This property makes it a valuable non-toxic filler for flame-retardant paints, coatings for metal and wooden structures, as well as for polymer composites used in construction (cable insulation, roofing membranes) [26]. Its use is regulated by standards for flame retardants, in particular, GOST R 53295-2009.

Magnesium oxide is a key component in the production of so-called magnesia binders (Sorel cements), which are characterized by high bending strength, good adhesion to organic fillers (wood chips, sawdust), high fire resistance and low thermal conductivity [27]. Magnesium oxide and chloride or magnesium sulfate are used to produce magnesia boards (glass–magnesium sheets - MGOP), which are widely used as an alternative to drywall and particle board for interior and exterior decoration, partitions and suspended ceilings. The main regulatory document for a binder is GOST 12367, and STB 1212-2009.

Compounds obtained in the work, especially $Mg(OH)_2$ and MgO with a low calcium content (0.0110% and 0.0187%, respectively) meet the requirements for raw materials for the production of high-quality building materials (Table 5), where impurities can negatively affect durability and performance.

Table 5. Comparative characteristics of the use of magnesium compounds in construction and building materials.

Connection	Main functions and applications	Key advantages	Regulatory and technical documentation (examples)
Magnesium Sulfate ($MgSO_4$)	1. Modifying additive in cements and concretes. 2. Filler for magnesia binders (alternative to $MgCl_2$).	1. Regulation of hardening kinetics. 2.Reduction of hygroscopicity and corrosion activity compared to chloride closures.	GOST 24640-91 "Additives for cements. Classification" M.: IPK Publishing House of Standards, 2004. 6 p.
Magnesium Hydroxide ($Mg(OH)_2$)	1. Flame retardant filler in polymer composites, paints, coatings. 2.Additive to building mortars to increase fire resistance.	1. High flame retardant efficiency (endothermic decomposition with heat absorption). 2.Environmental safety (halogen-free).	GOST R 53295-2009 "Flame retardants for steel structures. General requirements. Methods for determining fire-retardant effectiveness." Moscow: Standartinform, 2010. 16 p.
Magnesium oxide (MgO)	1. The main component of magnesia binders (Sorel cement). 2. Production of magnesia plates (MGOP, glass-magnesium sheets). 3.Flame retardants and plasters.	1. High bending strength. 2. Fire resistance and moisture resistance. 3.Good adhesion to organic fillers.	GOST 12367-85 "Magnesia binders. Technical specifications" M.: IPK Publishing House of Standards, 2002. 7 p. STB 1212-2009 "Magnesia-chipboard. Technical specifications" Minsk: Gosstandart, 2009. 24 p.

Thus, the developed technology for processing serpentinite waste makes it possible to obtain magnesium compounds in demand in the construction industry that meet the requirements of the current regulatory framework and contribute to the creation of building materials with improved operational and environmental characteristics.

4. CONCLUSIONS

Based on the conducted research, the following conclusions can be drawn:

1. A comprehensive technology for processing powdered technogenic serpentinite waste to produce high-purity magnesium compounds: magnesium sulfate, magnesium hydroxide and magnesium oxide has been developed and investigated. It was found that the use of 0.7 CHQ H₂SO₄ for acid leaching and subsequent neutralization and purification of the solution with thermally activated powdered technogenic waste (TA-PTW) at 750 °C make it possible to achieve magnesium extraction of 82.5% of the amount of sulfuric acid and 52.3% of the total magnesium content in the system [PTW + TA-PTW].

2. X-ray diffraction methods have confirmed that thermal activation of PT at 750 °C leads to the complete destruction of the serpentine structure with the formation of highly reactive phases – forsterite and periclase, which significantly increases their neutralizing and sorption capacity with respect to impurity ions (Fe, Al, Cr, Ni) in a solution of magnesium sulfate.

3. It is shown that sequential precipitation of magnesium hydroxide with a purified solution of magnesium sulfate and its subsequent calcination make it possible to obtain high-purity products: Mg(OH)₂ with a calcium content of 0.0110% and MgO with a calcium content of 0.0187%.

4. The principal possibility of targeted application of the obtained compounds in the production of building materials has been established:

- *Magnesium sulfate* can be used as a modifying additive in cements and as a sealer for magnesia binders regulated by GOST 12367-85.

- *Magnesium hydroxide* is an effective flame retardant filler for polymer composites and coatings that meet the requirements of GOST R 53295-2009.

- *Magnesium oxide* serves as the main component for the production of fire-resistant and moisture-resistant magnesia binders and plates (glass-magnesia sheets), regulated by STB 1212-2009.

5. The implementation of the proposed technological scheme helps to solve environmental problems associated with the accumulation of serpentinite waste, and allows creating a resource-saving, closed production chain within the framework of a waste-free production strategy, providing the construction industry valuable and competitive raw materials.

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