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TECHNICAL COMMUNICATION

Removal of Sulfate and Metals from Wastewater of a Mining Enterprise by a Dual Sorbent System: A Case Study

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Abstract

This paper presents the results of wastewater treatment at a mining enterprise developing a complex ore deposit in the Murmansk region, Russia. The main pollutants are sulfate, manganese, and strontium. For this complex wastewater treatment, a two-stage scheme was proposed and tested using two sorbents (at a pH of 6): an aminated peat biosorbent to remove sulfate and a commercial sorbent based on the mineral brucite to remove metals. Optimal conditions were established for purifying the wastewater of sulfate and manganese and strontium ions to the level of maximum available concentrations in water bodies for fishery purposes.

Keywords Biosorbent · Brucite · Strontium · Manganese · Adsorption

Introduction

The mining industry contributes meaningfully to the pollution of ground and surface waters (Maree et al. 2004; Wolkersdorfer 2014; Younger et al. 2006). The urgency of the wastewater treatment problem is due to the large volume of discharged wastewater and the multielement pollutant composition. The Murmansk region is one of the most urbanized and industrially developed regions of the Arctic zone of the Russian Federation. A large amount of waste is generated by the mining activities in the region: uneconomical ore material, overburden, tailings of various concentrations of ore, and wastewater. The total volume of solid waste is about 8 billion tonnes, with more than 200 million tonnes added annually (Masloboev et al. 2016; Ministry of Natural Resources and Ecology of the Murmansk Region 2020). The sectoral discharge of polluted wastewater by the "mining" type of economic activity in the reporting year 2019 in the Murmansk region amounted to 192.74 million m^3 (Ministry of Natural Resources and Ecology of the Murmansk Region 2020). Specific pollutants of the water bodies of the Kola Peninsula are metal compounds (for example, copper, nickel, manganese, iron, and molybdenum), as well as nitrogen, fluorides, sulfate, phosphate, dithiophosphate, petroleum products, and anionic synthetic surfactants (Ministry of Natural Resources and Ecology of the Murmansk Region 2020).

This study focuses on wastewater from a mining enterprise in the Murmansk region. This mine site has a complex ore deposit, with elevated concentrations of sulfate, manganese, and strontium. It is important to note that, in the Russian Federation, the maximum available concentration (MAC) for waters is divided into two categories—fisheries and drinking. The sulfate MAC for fishery waters is only 100 mg/L, whereas

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the MAC for drinking water is 500 mg/L (Ministry of Agriculture of Russia 2016).

Several wastewater treatment methods are available, such as chemical precipitation, ion exchange, adsorption, membrane filtration, coagulation–flocculation, flotation, and electrochemical methods (Anirudhan and Sreekumari 2011; Hegazi 2013; Kadirvelu 2001). Recently, treatment schemes based on a bioplateau (Vymazal 2010, 2014; Wu et al. 2015; Zhang et al. 2014) have been actively developed. However, the need to purify multimillion volumes of water hinders the use of most of the proposed methods in mining enterprises.

The most effective and inexpensive technique for wastewater treatment is the sorption method (Dubrovskay et al. 2017; Pohl 2020). Although many number of studies have reported on the treatment of synthetic solutions using sorbents, there is limited literature on the use of these products for the treatment of mine wastewater. In addition, there is a need to develop inexpensive and sustainable sorbents from local waste materials. Effective sorbents can be produced from bio-based materials, such as sawdust, and peat, (Gogoi et al. 2019, 2021b; Keränen et al. 2016) as well as brucite, which is a readily available magnesium hydroxide-based sorbent. For example, it is possible to obtain a brucite-containing reagent from technogenic waste, which is available in sufficient quantities in the Murmansk region (Krasavtseva et al. 2021). Presently, wastewater treatment at the enterprise occurs by the gravitational sedimentation of suspensions in a settling tank system, but the results do not satisfy either the managers of the enterprise or the supervisory authorities. Better treatment facilities are being planned and adsorption is being considered as an option for wastewater treatment.

The aim of the study was to determine the optimal conditions for purification of this wastewater by a combination of a synthesized biosorbent (Gogoi et al. 2019), as well as by a commercial mineral-based sorbent, brucite. The biosorbent has been reported to exhibit high sulfate removal capacity under acidic conditions (pH 2, 189.5 ± 2.7 mg/g) (Gogoi et al. 2019, 2021a). The main functional groups providing binding sites for sulfate uptake onto the biosorbent are the amine and quaternary ammonium groups. Brucite can remove metal ions, arsenic, and organic pollution effectively (Akinwekomi et al. 2016; Bochkarev and Pushkaryova 2009; Bochkarev et al. 2010, 2014; Makarov et al. 2009; Masliy et al. 2012; Orekhova et al. 2021; Pushkaryova 1999; Sulaiman et al. 2018). For both, the removal mechanism is based on the principle of ion exchange. This can be represented by the exchange of the crystal lattice magnesium ions with metal ions present in water (Krasavtseva et al. 2021).

Materials and Methods

Wastewater Sampling

This study focused on the wastewater of a mining enterprise developing a complex ore deposit in the Murmansk region (Russia), which had a high content of sulfate, manganese, and strontium. Wastewater for this study was collected in November 2019 from the wastewater storage pond. Clarified waters routed to cleaning in the settling pond from the tailing dump accounted for up to 80% of the water volume in the storage pond, while surface runoff and the adjacent wetland system accounted for the rest. Table 1 presents the content of targeted contaminants in the studied wastewater and the MAC.

Characteristics of the Sorbents

Aminated peat biosorbent, which was developed in the study by Gogoi et al. (2019), was selected for removing sulfate from the wastewater. The peat raw material was obtained from Stora Enso Veitsiluoto pulp mill. The properties of the peat can be found in the study by Leiviskä et al. (2018). The biosorbent was prepared by modifying raw peat using a unique combination of two chemicals at the optimal conditions reported by Gogoi et al. (2019): branched polyethylenimine (PEI, 0.26 mmol/g peat) and glycidyltrimethylammonium chloride (GTMAC, 0.0447 mol/g peat). The synthesized biosorbent has quaternary ammonium and N–H groups on its surface and is referred to as PG-Peat.

Ground brucite, a commercial reagent (AgroMag), was chosen as a sorbent for the purification of wastewater from metal ions. Finely dispersed magnesium hydroxide powder (particle size < 300 μm) was produced from selectively mined natural magnesium hydroxide (brucite), by grinding and grading. The active brucite content was (in weight %): $MgO/Mg(OH)_2$– 60.0/87.0, CaO – 3.0, SiO_2 – 3.0, and Fe_2O_3 – 0.5. The sorption properties of brucite can be intensified at high temperature (Gumerova 2012; Krasavtseva et al. 2020). Therefore, the brucite samples were baked in a muffle furnace at temperatures of: 450, 550, and 600 °C for two hours.

Table 1 Concentration of pollutants and MAC in the studied wastewater (pH 8.5)

	SO_4^{2-}	Mn	Sr
Concentration in wastewater, mg/L	335	0.12	1.154
MAC, mg/L	100	0.01	0.4

Wastewater Treatment Experiments

The work was done in two parts. First, we examined the feasibility of using the PG-Peat on wastewater obtained from a local mine site, which is characterized by low levels of sulfate (335 mg/L). The experiments were carried out with continuous stirring. The sorbent was added to the wastewater samples, varying the PG-Peat dosage and the initial pH. Based on a previous study (Gogoi et al. 2019), varying dosages of PG-Peat were selected: 0.1–0.2 g per 100 mL/water (1–2 g/L), while the contact times for the batch experiments were chosen to be 5 min, 10 min, 1 h, 3 h, and 5 h. Then the effect of PG-Peat dosage (1–5 g/L) on sulfate removal was investigated at pH 8.5 (1 h contact time).

We then attempted to optimize the combined system for treating the mine wastewater to MAC values step by step, using both the PG-Peat and brucite sorbents. First, the aminated peat sorbent (4 g/L) was added to the original wastewater, varying the pH (2–6) of the solution, with an interaction time of 1 h. Then, weighed portions of brucite were added to the resulting solutions, varying the dosage (1–3 g/L) and the type (unbaked and baked at selected temperatures) of the brucite sample. At the end of each stage, part of the water sample was filtered and sent for chemical analysis.

Physico-chemical Analysis

The x-ray photoelectron spectroscopy (XPS) analysis of fresh and recovered PG-Peat samples were performed using a Thermo Fisher Scientific ESCALAB 250Xi with a monochromatic Al Kα source (1486.6 eV). Analysis of the obtained XPS data was conducted using Avantage software, where the Shirley function was used for background subtraction. The binding energy of adventitious carbon was set to 284.8 eV for charge correction. The C1s, N1s, and S2p spectra were fitted using a Shirley background and Gaussian–Lorentzian sum function.

The pH before and after the sorption experiment was determined using an I-160-MI ionomer. The sulfate concentration was analyzed by ion exchange chromatography using a Waters HPLS liquid chromatograph with a Waters 432 conductivity detector. The concentrations of metals (Sr and Mn) were determined by atomic absorption spectrometry with an AA-7000 Shimadzu atomic absorption spectrophotometer and an ICE-3300 atomic absorption spectrometer (INEP KSC RAS).

Results and Discussion

Removal of Sulfate Using Only the PG-Peat Biosorbent

Figure 1 shows how the amount of sulfate removal by the reagent depends on interaction time (initial $SO_4^{2-} = 335$ mg/L, PG-Peat dosage = 2 g/L, initial pH 2). There was a rapid decrease in sulfate (≈100 mg/L) from the contact times of 5–10 min to 60 min. Gogoi et al. (2019) reported that the main mechanism for sulfate uptake by the PG-Peat was ion exchange, which results in relatively quick equilibrium.

The PG-Peat removed sulfate more efficiently at low pH. The results for a three hours of contact time are shown in Table 2. Acidifying the wastewater sample to pH 2 with HCl and applying a PG-Peat dosage of 2 g/L resulted in a final sulfate concentration of 82.24 mg/L (initial $SO_4^{2-} = 335$ mg/L), which was below the MAC of 100 mg/L. The higher sulfate removal efficiency at a pH of 2 was due to the presence of protonated amine groups under acidic conditions, in addition to the positively charged quaternary ammonium groups (which are cationic throughout the pH range) (Gogoi et al. 2019). Therefore, more binding sites were available at a pH of 2 and thus a higher amount of negatively charged sulfate ions was removed, thus resulting in a more rapid sorption and lower residual sulfate concentration in the treated solution.

The XPS analysis of the recovered PG-Peat samples after the treatment confirmed that the biobased anion exchanger had bound the sulfate onto the surface. The surface elemental composition of the raw peat and recovered PG-Peat samples is presented in Table 3. The biosorbent mainly contained carbon, oxygen, and nitrogen. The presence of chloride in the fresh samples was due to the GTMAC modification, but the absence of this peak in the recovered sorbent could be due to the release of most of the chloride ions to the treated solution in exchange for the sulphate ions onto the binding sites of the biosorbent (Gogoi et al. 2019). A slight decrease in the nitrogen content (6.6–6.7 at.%) was observed compared to the fresh PG-Peat samples (7.3 at.%). Figure 2 shows the S2p high resolution spectrum of the recovered PG-Peat. The doublet with a binding energy difference of 1.4 eV could be seen at 168 eV for $S2p_{3/2}$ and at 169.4 eV for $S2p_{1/2}$, which can be attributed to sulfate (Gogoi et al. 2019; Virpiranta et al. 2022), thus confirming the binding of sulfate ions onto the PG-Peat during the ion exchange process.

Next, the effect of PG-Peat dosage on sulfate removal was investigated over a larger range (Fig. 3) at a pH of 8.5 (without adjustment). The sulfate concentration could only be decreased to the MAC level by using a PG-Peat dosage of 5 g/L, due to the lower efficiency at higher pH values. It is difficult to recommend such a high dosage because of the huge volumes and multielement composition of the wastewater requiring treatment.

Dual Sorbent System

In the second part of the work, the possibility of using both PG-Peat and brucite was investigated. The first step in the

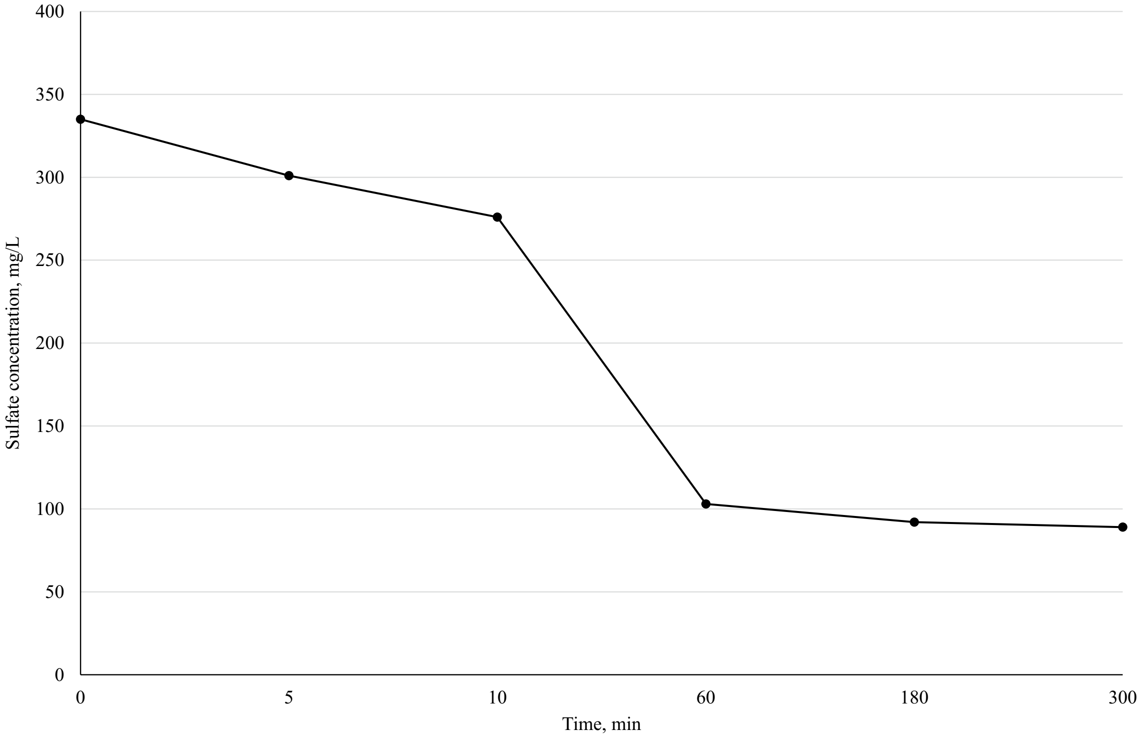


Fig. 1 Purification efficiency depending on the time interaction (SO_4^{2-} initial: 335 mg/L, PG-Peat dosage 2 g/L, initial pH 2)

Table 2 Results of wastewater treatment from sulfate ions using PG-Peat (contact time 3 h)

Sample №	Dosage PG-Peat, g/L	Initial sulfate concentration, mg/L	Initial pH	SO_4^{2-}, mg/L	Final pH
1	1	335	8.9	248.5	8.3
2	1		2.0	172.6	2.2
3	2		8.9	197.2	8.6
4	2		2.0	82.2	2.4

Table 3 Surface composition of fresh and recovered PG-Peat samples used for treatment of wastewater (SO_4^{2-}initial: 335 mg/L, PG-Peat dosage 2 g/L, initial pH 2)

Element	Fresh PG-Peat (at.%)	Recovered PG-Peat (at.%)
C	78.1	73.2
O	12.1	16.7
N	7.3	6.7
Cl	2.6	1.1
S	0.0	1.3
Si	0.0	1.1

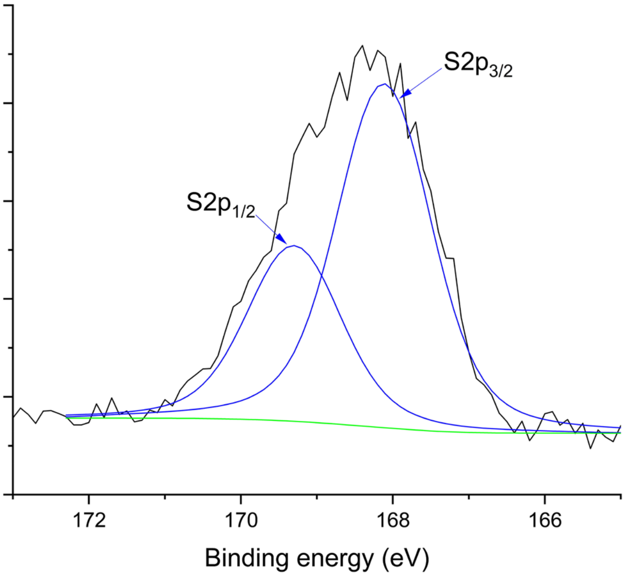


Fig. 2 S2p high-resolution XPS spectrum of recovered PG-Peat (SO_4^{2-} initial: 335 mg/L, PG-Peat dosage 2 g/L, initial pH 2)

study was to carry out the sulfate removal at varying pH and constant PG-Peat dosage of 4 g/L (Fig. 4). The MAC was reached with a dosage of 4 g/L at all studied pH values (2, 4, and 6). As stated earlier, the presence of protonated amine groups, apart from the positively charged quaternary

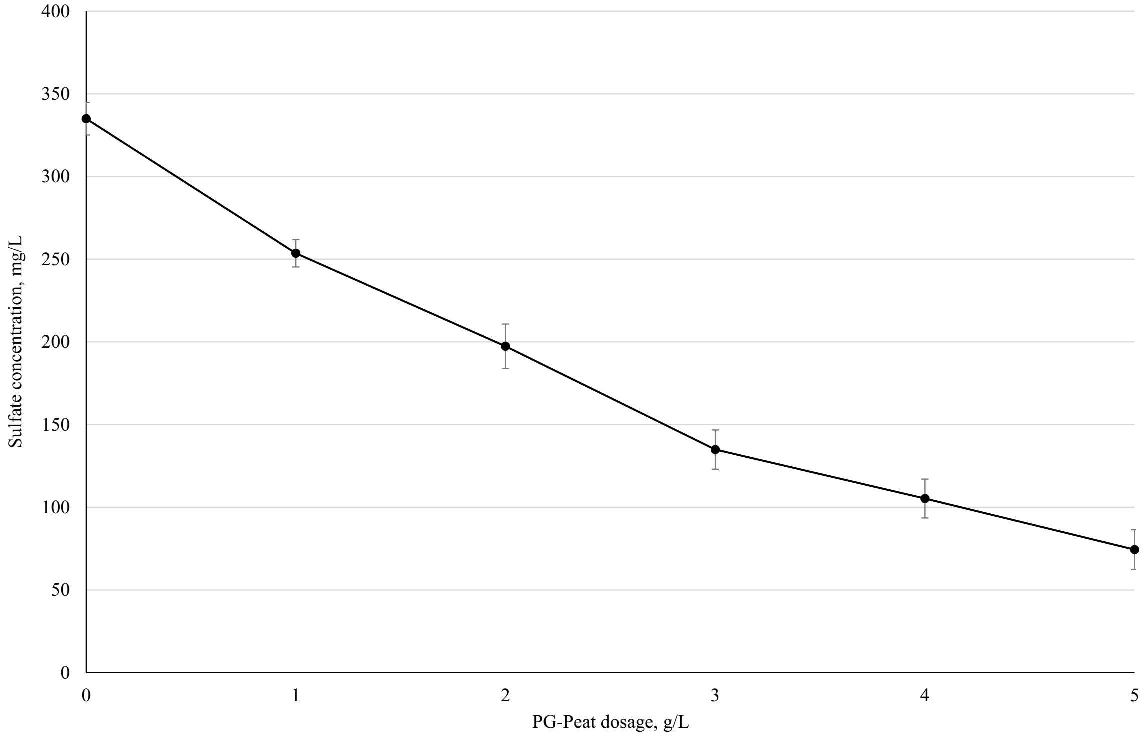


Fig. 3 Purification efficiency depending on the consumption of PG-Peat sorbent (initial sulfate concentration: 335 mg/L, pH 8.5, contact time 1 h, temperature 20 °C)

ammonium groups, improved the uptake of the anionic sulfate species (Gogoi et al. 2019). A similar trend can be seen in Fig. 4. However, decreasing the pH to as low as 2 is not practical unless the wastewater already has a low pH. It should also be noted that the PG-Peat did not effectively remove the manganese and strontium in these experiments.

Next, pre-treated wastewaters were treated with unbaked brucite and brucite baked at different temperatures (450, 550 and 600 °C). Unbaked brucite showed poor results for Mn and Sr removal. None of the dosages of unbaked brucite were able to approach the MAC standard (Figs. 5 and 6). The increase in the Mn concentration in the water sample treated with the PG-Peat sorbent at a pH of 2 is explained by the fact that manganese in waste and natural waters is present, for the most part, in the form of suspended solids. With a decrease in pH, the manganese dissolves, due to the reduction of Mn (IV) to Mn (II) (Ogryzkova et al. 2014).

The removal of metal ions from wastewater using brucite can be carried out by the parallel processes of ion exchange adsorption and chemisorption: adsorption on the surface of the mineral with the formation of bulk aqua- and hydrocomplexes with OH groups, and the replacement of Mg^{2+} ions by ions of the sorbed metal (Calugaru et al. 2016; Korolev et al. 2016; Orekhova et al. 2021). At the same time, the sorption of strontium occurs with the formation of strontianite ($SrCO_3$) on the brucite surface (Bochkarev and Pushkaryova 2009).

The following series of experiments was performed with baked brucite. It has previously been shown that heating natural brucite at 400–600 °C leads to the formation of periclase (MgO) (Bochkarev and Pushkaryova 2009). This improves the sorption capacity of the modified mineral by increasing the specific surface area by up to 10 times and creating a “defective” crystal structure (Pushkaryova 2000). A dosage of 1 g/L was selected due to the increased cost of the sorbent because of the heating process. The results are shown in Figs. 7 and 8. Manganese and strontium concentrations were the lowest when brucite treated at 600 °C was used.

The results obtained are in complete agreement with the results reported by Pushkaryova (2000); the sorption capacity of the thermally modified brucite significantly increases due to the "loosening" of its crystal structure during firing (Bochkarev and Pushkaryova 2009). It is known that the efficiency of sorption treatment with brucite is directly proportional to the pH; therefore, the decreased efficiency in solutions at initial pH values of 2 and 4 is explained by the established optimal solution pH range of 7.5–9 (Bochkarev and Pushkaryova 2009). At the same time, once dissolved,

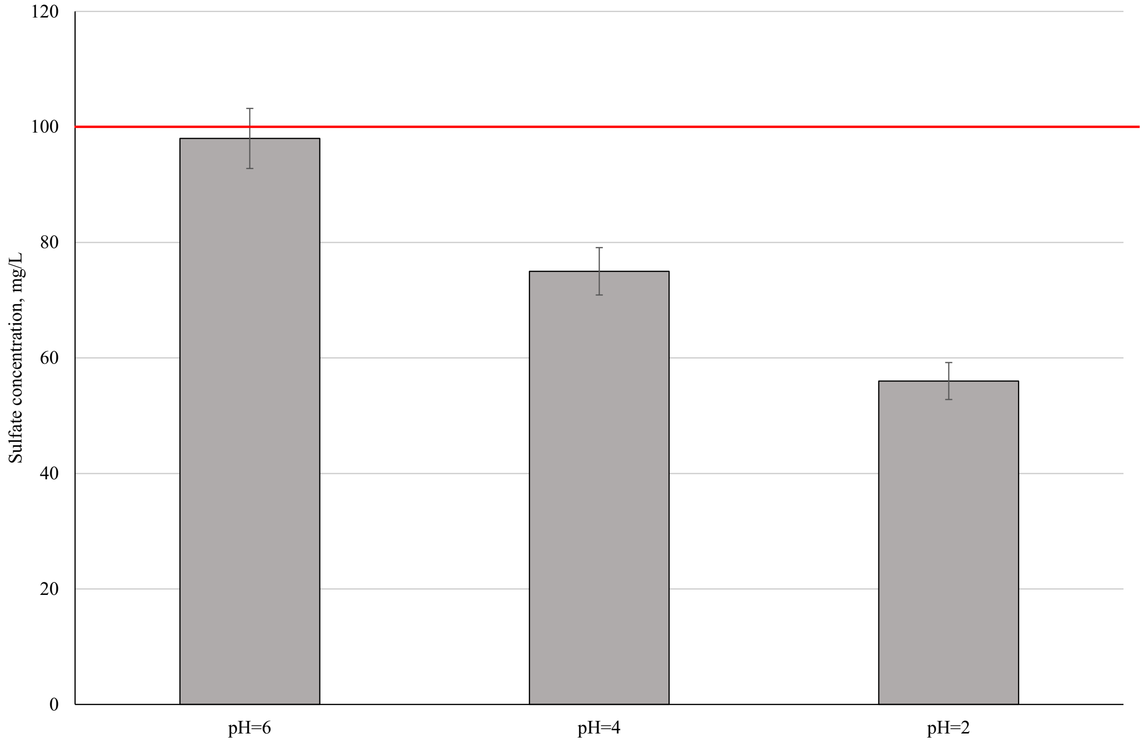
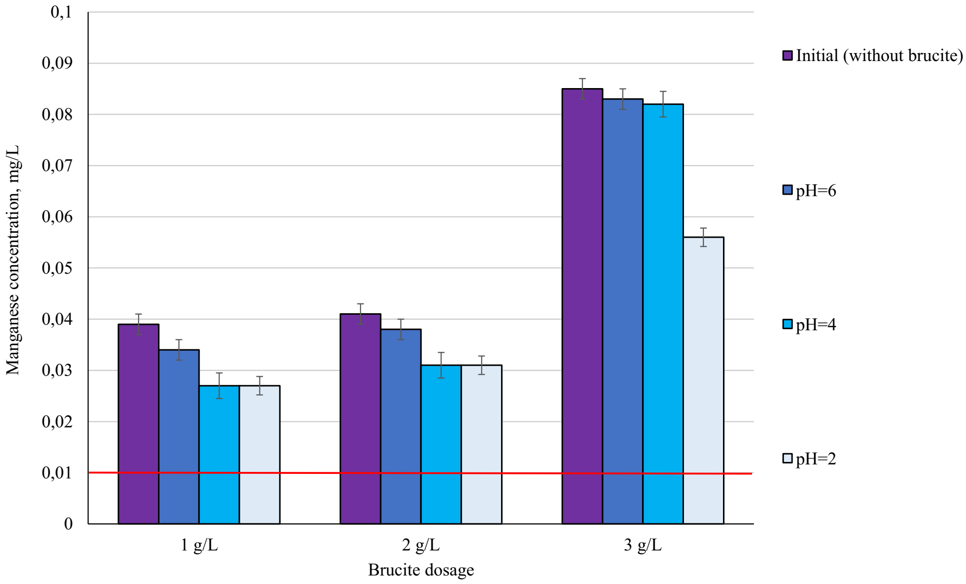


Fig. 4 Residual sulfate concentrations in PG-Peat-treated water at different pH values (PG-Peat dosage—4 g/L). Red line—MAC (100 mg/L)

Fig. 5 Manganese concentrations during treatment with unbaked brucite at different reagent dosages in wastewater pretreated with PG-Peat sorbent (dosage 4 g/L). Red line—MAC (0.01 mg/L)



brucite is capable of alkalinizing water, which is why it is used to neutralize acidic mine waters (Korolev et al. 2016).

The optimal parameters for the purification of wastewater from a mining enterprise from sulfates and ions of manganese and strontium are as follows: preliminary pH adjustment of wastewater to pH = 6, treatment with 4 g/L of PG-Peat sorbent, and subsequent introduction of thermally treated 1 g/L of brucite. Since, at the initial pH of 6, there was no significant difference in the intensification of sorption properties, from the point of view of economic feasibility, a firing temperature of 450 °C for the brucite was judged sufficient.

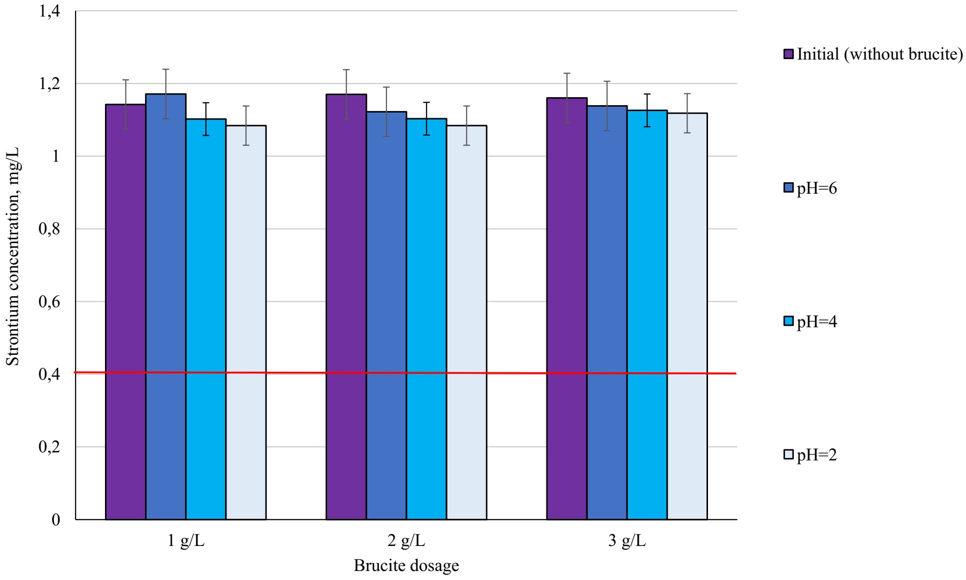


Fig. 6 Strontium concentrations during treatment with unbaked brucite at different reagent dosages in wastewater pretreated with PG-Peat sorbent (dosage 4 g/L). Red line—MAC (0.4 mg/L)

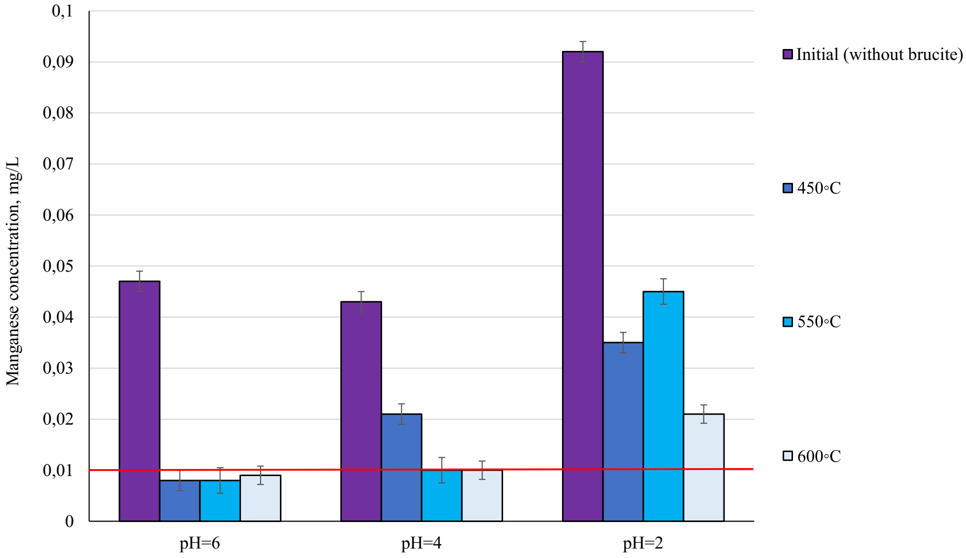


Fig. 7 Manganese concentrations during the treatment with baked brucite (dosage 1 g/L) at different heating temperatures in wastewater pretreated with PG-Peat sorbent (dosage 4 g/L). Red line—MAC (0.01 mg/L)

Conclusions

This study presented the results of laboratory studies on the treatment of wastewater from a mining enterprise. The prospect of removing sulfate ions from wastewater as well as the ability to treat a multielement effluent stream containing a combination of contaminants was explored using two sorbents: one synthesized from peat (PG-Peat) and a commercial mineral-based product (brucite) that exhibit different sorption principles. Based on the results of the proposed treatment with the use of two sorbents, the concentration of sulfate ions can be significantly decreased, while attaining the MAC standard for manganese and strontium. Thus, the results obtained indicate a real prospect for developing a sorption technology for purifying wastewater from mining enterprises using a combination of sorbents. Further research should be conducted in larger pilot-scale tests and with a wider list of contaminants to assess the possibility of producing PG-Peat sorbent for industrial use and to assess the prospects for the regeneration of the used sorbents.

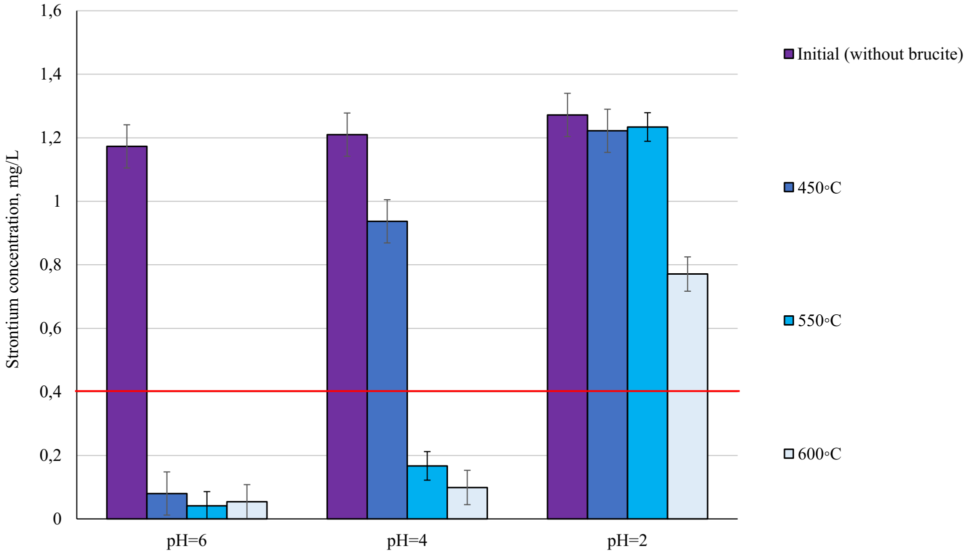


Fig. 8 Strontium concentrations during the treatment with baked brucite (dosage 1 g/L) at different heating temperatures in wastewater pretreated with PG-Peat sorbent (dosage 4 g/L). Red line—MAC (0.4 mg/L)

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Data availability Data will be made available on request.

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