

Article

Sustainable Resort Infrastructure: Wastewater Utilization in Thermal Spa Complexes

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Abstract

A modern approach to the organization of wastewater disposal systems ensures the sustainable development of resort areas. Warm mineral springs (WMSs) located in suburban areas are not only recreational facilities for the population, but also sources of used mineral and communal wastewater. Preventing or reducing the discharge of pollutants in this type of liquid waste can reduce the anthropogenic impact on the environment. The article proposes a method for the chemical precipitation of ammonium ions in a mixture of mineral and domestic wastewater, and the production of struvite, a complex agricultural fertilizer. Three series of experiments were conducted: (1) on a model solution of ammonium ions; (2) on a mixture of a model solution and real mineral water; and (3) on real wastewater and spent mineral water taken from a thermal resort. As a result of an experiment with water samples at an initial concentration of no more than 85 mg/L, optimal doses of reagents were identified: 5.67 g/L of sodium hydrophosphate and 3.21 g/L of magnesium chloride to achieve a residual concentration of ammonium ions in purified water of no more than 1.5 mg/L at pH = 9–10. In the case of mixing of domestic wastewater and used mineral water, it was possible to reduce the use of magnesium chloride by 80%. At the same time it was possible to obtain the amount of sediment containing struvite, which is a valuable agricultural fertilizer. With the introduction of this technology, it is possible to reduce the expenses of reagents and reduce the damage caused by the discharge of liquid waste from a thermal resort, while the profit earned through the sale of fertilizer can partially offset the cost of wastewater disposal.

Keywords: mineral water; wastewater; ammonium ion; chemical precipitation; struvite; sustainability of resort areas



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1. Introduction

Mineral springs are waters flowing out of the earth's crust that contain various mineral particles corresponding to the composition of the rocks and soil from which these waters flow. Mineral waters (MWs) are complex solutions in which substances are contained in the form of ions, undissociated molecules, gases, colloidal particles, and even isotopes [1–3]. Mineral water sources have been considered very useful for humans since ancient times; the ancient Romans used mineral water in thermal baths to treat wounds, relieve fatigue, and revitalize the whole body [4]. Nowadays, mineral waters also have an important balneological value, and they are widely used in sanatorium treatment. Worldwide, millions of people consume bottled mineral water every day [5]. Additionally, sources of mineral water are very attractive for tourists [6].

Mineral waters in the Tyumen region (Russia) are widespread. Like mineral waters all over the world, they are characterized by specific features in terms of geological conditions of occurrence, chemical and gas compositions, temperature and other indicators, as well as their purpose and use [2,3,5–7]. Thermal springs are underground waters with a temperature of more than 20 °C that come to the surface. Thermal springs are also often referred to as springs where the water temperature is higher than the annual average for a given area. Warm mineral water sources (WMSs) are divided into warm (water temperature less than 37 °C), hot (37–50 °C) and very hot (more than 50 °C) [8,9]. The main objects of exploitation are deposits from the Cretaceous period (from 145 to 66 million years ago) and the Jurassic Period (about 200 million years ago), which are distributed almost throughout the entire territory of the south of the Tyumen region [1,10]. It is believed that this region stands on an underground mineral sea. A total of 76 wells have been discovered here, of which less than half are currently in operation. The most popular sources (17 equipped facilities) are concentrated in cities and suburban areas (Figure 1). The depth of such waters varies from 950 to 2500 m. For example, almost anywhere in the city of Tyumen and its suburban area has access to hot mineral water at a depth of over 1.3 km [10].

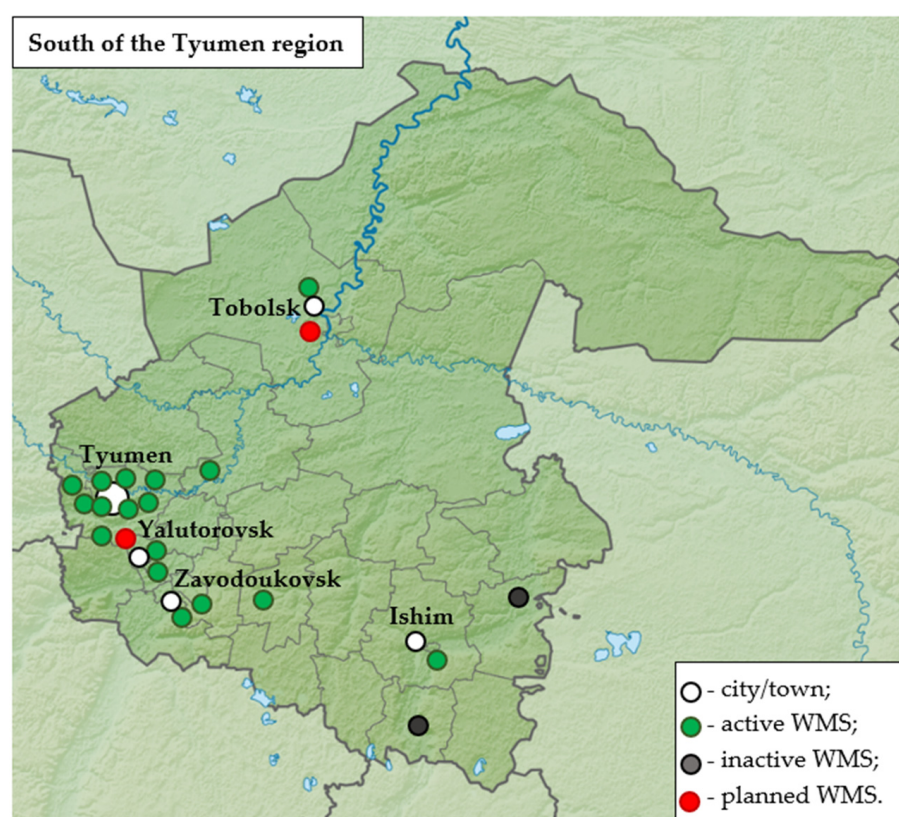


Figure 1. Map of the location of hot mineral springs (WMSs) in the south of the Tyumen region.

The chemical composition of the water is sodium chloride, with mineralization ranging from 6–7 to 15–20 g/L. The biologically active components are iodine, bromine and boron. The composition of dissolved gases is predominantly nitrogen–methane. The waters are thermal, the temperature in the reservoir varies from 15–20 °C to 40–45 °C or more, and the concentration of dissolved salts reaches 75 g per liter of water [1–3]. The chemical composition of some sources located within the city of Tyumen and its suburbs is shown in Table 1.

Table 1. The chemical composition of the water from hot mineral springs.

Water Quality Indicators	Thermal Resorts and Spa Complexes				
	“Sosnovy Bor”	“Sovetsky”	“Volna”	“Marcial”	“Verkhny Bor”
Mineralization, g/L	13.2	No data available	No data available	8–9	6–7.5
Chlorides, mg/L	8156	1100	4964	5360	420
Bicarbonates, mg/L	183	366	220	131	300–350
Bromides, mg/L	38.5	30	29.75	25	25–30
Iron, mg/L	0.85	0.5	8.14	0.9	No data available
Sodium, mg/L	442	2571	3023	3254	2500
Calcium, mg/L	314	120.8	208	164.5	1250
Magnesium, mg/L	77	31.2	62	40.1	25
Potassium, mg/L	24	37.7	30	13	38
Iodine, mg/L	0.85	3	1.76	4.3	2.5–5
Boron, mg/L	9.12	No data available	No data available	7.6	35
Fluorides, mg/L	No data available	12	0.93	0.69	0.76
Ammonium, mg/L	No data available	10	13	No data available	No data available
pH	6.9	7.4	8.04	7.7–7.9	No data available

According to Table 1, it can be seen that the MW of different WMSs has a different chemical composition, a high degree of neutralization, a pH index close to neutral, and an increased content of chlorides, bicarbonates, magnesium, calcium, ammonium, and other chemical elements. After use in swimming pools, usually in a running mode, this water must be cleaned and disposed of. The discharge of spent MW into the environment causes serious environmental violations.

As a rule, modern thermal resorts and spa complexes are built in the area of explored mineral water sources for the recreation of the urban population. In addition to the main pools with MW, toilets, shower rooms, and treatment rooms are mandatory in the infrastructure of such complexes. The resort usually includes small hotels, restaurants and laundries. All of these facilities generate liquid municipal waste, namely domestic wastewater (WW), which must be collected and treated [11–13]. In addition, a small village for staff accommodation is usually located next to the thermal resort, where WW also accumulates. The qualitative composition of such WWs corresponds to the domestic wastewater of settlements with a drainage rate of 100–180 Liters per person per day. At the same time, it is not always possible to connect communal WMS infrastructure facilities to the centralized sewerage system of a nearby city; therefore, an autonomous sewerage is often provided.

Two main problems related to the accumulation and disposal of liquid communal waste at such recreation and recreation facilities are recognized: (1) used mineral water (10–30% of the total volume of recreational pools), which is drained into a surface fresh-water body adjacent to the WMS territory (pond, lake, or river). This causes a significant change in water quality in such reservoirs, and as a result, overall mineralization increases,

flora and fauna become poorer, fish disappear, and soil salinization occurs [14]. (2) Household wastewater accumulates in special tanks, which must be periodically pumped out and transported to drainage stations that transport wastewater to wastewater treatment plants (WWTPs) [15]. This is not always economically beneficial, so there is a practice of unauthorized discharge of untreated wastewater into ponds, swamps and onto the terrain. This is especially true for small, decentralized facilities.

There is a practice of building small-capacity WWTPs that can accept WW from decentralized facilities for cleaning. Biological purification is usually recognized as the traditional method of removing nitrogen-containing elements [16]. However, for small autonomous facilities and settlements, the installation of a biological treatment plant is not always economically justified. In addition, such WWTPs are characterized by low efficiency, especially in winter in conditions of low temperatures typical for this region [17]. Ammonium ions are most difficult to remove from the water, and subsequently cause active eutrophication of reservoirs [18]. And if spent mineral waters are also taken to such WWTPs and municipal wastewater is diluted with them, this leads to a gradual degradation of activated sludge due to the high mineralization of MW. The sludge flakes become fine, the overall efficiency of water purification decreases (less than 70%), and sludge is additionally carried into the purified water. In this case, the concentration of suspended solids reaches more than 50 mg/L, although high-quality disinfection of wastewater with ultraviolet radiation occurs at a suspension concentration of no more than 10 mg/L [19].

A number of authors [20–23] propose physico-chemical methods for cleaning WW, which partially or completely exclude the stage of biological treatment of wastewater with activated sludge. For example, existing treatment schemes include coagulation, flocculation, flotation, electroflotation, chemical oxidation, mechanical and sorption filtration and other methods. Unfortunately, all these schemes do not meet the regulatory requirements established in Russia for the content of ammonium ions in purified water (no more than 1.5 mg/L) for WWTPs of low and ultra-low power (or a daily capacity of 1000 m³/day or less) [24].

There is evidence in the public domain that chemical precipitation of ammonium ions from water using magnesium and phosphorus-containing reagents can produce insoluble magnesium–ammonium phosphate or struvite, an excellent agricultural fertilizer [25,26]. For these purposes, sodium hydrophosphate Na₂HPO₄, magnesium chloride MgCl₂ and sodium hydroxide NaOH are required to provide the required pH [27]. It has been found that the maximum degree of wastewater purification from ammonium ions is achieved at pH = 8.5–10 and a slight excess of the amount of precipitating reagents (by about 5%) [25]. The optimal mode of struvite deposition is the direct instantaneous supply of reagents, with solutions of Na₂HPO₄ and NaOH being added to the wastewater first, and then the MgCl₂ solution [25–27]. In addition, it was found [28] that struvite is mainly formed when the concentration ratio of [Ca²⁺]: [Mg²⁺] ions in wastewater is no more than 0.25:1. However, it must be taken into account that the proposed technology is always associated with the cost of reagents and the maintenance of the reagent equipment. But unfortunately, at this stage of global research, there is still no universal method of struvite formation for all types of wastewater. The best chemical deposition conditions, optimal doses of reagents, pH values, the sequence of reagent administration, the interfering influence of other types of pollutants, and other factors have not yet been determined. In each individual case, a precipitate containing from 20 to 75% pure struvite can be obtained, and this reduces the stability of the results obtained.

In recent years, there has been growing concern globally about the depletion of exploited phosphorus reserves [29,30], which has led to a decrease in the production of phosphorous ore in some countries and ultimately led to a sharp increase in prices for raw

materials for phosphorous fertilizers. Due to the above-mentioned problems, increasing attention is being paid to the study of struvite ($\text{NH}_4\text{MgPO}_4 \cdot 6\text{H}_2\text{O}$), its precipitation and the concomitant extraction of phosphorus from various types of wastewater [26,31–35].

Experiments have already been conducted on the isolation of struvite in salt water. For example, in seawater recirculation systems in aquaculture facilities, spontaneous struvite formation may occur due to the natural presence of magnesium in the water, as well as ammonia and phosphorus compounds from the decomposition of fish feces and/or fish feed. Moreover, when anaerobic digestion is used for the precipitation of such salty WW rich in nitrogen, phosphorus, and magnesium, an intensification of struvite deposition is observed [36]. The authors in [37] conducted a study on the crystallization of struvite in marine brackish aquaculture. It was found that struvite was formed in an anaerobic reactor due to the natural presence of magnesium in seawater and ammonia and phosphates released by aquaculture at $\text{pH} = 7.7$. The average size of sediment crystals in a salty environment decreases with increasing pH and increases with increasing water temperature. The high content of ammonium ions enhances the formation of struvite.

As noted above, the mineral water of Tyumen WMSs is rich in ammonium and magnesium, and domestic wastewater is saturated with ammonium and phosphate ions. In this case, when WW and MW are mixed, theoretically a necessary and sufficient symbiosis arises to produce struvite. This makes it possible to implement low-waste WMSs wastewater treatment technology for two types of wastewater. Additionally, the production of struvite makes it possible to compensate for the cost of reagents and reduce the cost of water purification. In this scientific work, the aim is to find out whether it is possible to obtain struvite by mixing spent mineral water and household wastewater generated at thermal resorts and spa complexes. It is also necessary to determine the optimal parameters of chemical struvite deposition from a mixture of WW and MW, the consumption of reagents, the amount of precipitate produced, and the composition of the treated water.

2. Materials and Methods

2.1. Chemical Composition of the Source Water

Five variants of the initial samples were used for the study: (1) a model solution of ammonium ions (MSA) for two specified initial concentrations of 53.5 mg/L and 84.8 mg/L; (2) used mineral water (MW); (3) a mixture of a model solution of ammonium and mineral water (MSA + MW); (4) real wastewater (WW); and (5) a mixture of real wastewater and mineral water (WW + MW). A model solution of ammonium ions of a given concentration was prepared in the laboratory as follows: ammonium chloride NH_4Cl was added to distilled water. A sample of the used mineral water was taken at the “Verkhny Bor” thermal resort (13 km from Tyumen). The real wastewater was taken from the WWTP of Tyumen at the stage after rough cleaning and primary precipitation. The chemical composition of the source water samples after laboratory measurement (initial concentrations) is shown in Table 2.

Table 2. Chemical composition and degree of dilution in the source water samples.

Water Indicators	Unit	MSA	MW	MSA + MW *	WW	WW + MW ***
pH	-	6.71 ± 0.01	7.71 ± 0.01	6.7–7.5	7.12 ± 0.01	7.1–7.6
Ammonium ion	mg/L	53.5 ± 1.13 84.8 ± 1.52	5.6 ± 0.4	64.7 ± 1.5	82.9 ± 3.2	82.9–24.9
Magnesium	mg/L	-	65.6 ± 2.1	0–49.2	17.6 ± 1.2	17.6–53.6

Table 2. Cont.

Water Indicators	Unit	MSA	MW	MSA + MW *	WW	WW + MW ***
Phosphates	mg/L	-	0.11 ± 0.01	0–0.083	18.5 ± 1.3	18.5–4.63
Mineralization	mg/L	190 ± 16	6407 ± 95	190–4853	670 ± 23	670–4973
Chlorides	mg/L	124.1 ± 8.6	425 ± 34	124–350	102 ± 8.0	102–344
COD	mg/L	-	-	-	450 ± 22	-
Temperature	°C	23.5 ± 0.5	33 ± 0.5 **	23–24	16.5 ± 0.5 **	23–24

* The values were calculated depending on the degree of dilution of MSA:MW from 0 to 0.75. ** At the time of sampling, the water was then cooled/heated to room temperature. *** The indicators were determined by calculation depending on the degree of dilution of WW:MW from 0 to 0.75.

2.2. Chemical Reagents, Instruments and Methods of Determination

The following reagents were used in the experiments: An 11.91% solution of sodium hydrophosphate (Na_2HPO_4 , designation R1), a 6.75% solution of magnesium chloride (MgCl_2 , designation R2), and a 10% solution of sodium hydroxide (NaOH , designation R3) were used to stabilize the pH index.

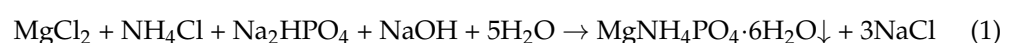
The following criteria were used as benchmarks for changes in water quality during the experiment: pH index, concentration of ammonium ion NH_4^+ (hereinafter referred to as NH_4), magnesium, chlorides Cl^- (hereinafter referred to as Cl), and phosphates PO_4^{3-} (hereinafter referred to as PO_4), total mineralization, precipitate volume, and water temperature. The benchmarks, methods of determination and the devices used are presented in Table 3.

Table 3. Benchmarks, detection methods, and instruments.

Water Indicators	Unit	Method	Device
pH	-	PND F 14.1:2:3:4.121-97 (Russia)	pH-Meter “pH-150MI” (Measuring technology, Russia)
Ammonium ion	mg/L	PND F 14.1:2:3:4.262-10 (Russia)	Spectrophotometer PE-5400vi (Ecos, Russia)
Magnesium	mg/L	PD 52.24.403-2018 (Russia)	Titrimetry equipment
Chlorides	mg/L	PND F 14.1:2:3.96-97 (Russia)	Titrimetry equipment
Phosphates	mg/L	PND F 14.1:2:4.112-97 (Russia)	Spectrophotometer PE-5400vi (Ecos, Russia)
Mineralization	mg/L	Test	Digital Water quality Meter EZ-9909SP (Noyafa, China)
Precipitate Volume	mL, cm^3	Volumetric	Measuring Beaker
Temperature	°C	PND F 12.16.1-10 (Russia)	pH-Meter “pH-150MI” (Measuring technology, Russia)

2.3. The Method for Obtaining Struvite

Chemical precipitation of ammonium ions in an aquatic environment by adding reagents containing phosphate ions (sodium hydrophosphate) and magnesium ions (magnesium chloride) is accompanied by the production of magnesium–ammonium phosphate, or struvite ($\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$). This process is carried out according to the following mechanism [25]:



To obtain struvite, an optimal ratio of elements in water by molar mass is required: $\text{Mg:NH}_4:\text{PO}_4 = 1:1:1$.

Struvite is a fine white powder that is obtained after drying the precipitate of a chemical reaction. Struvite is highly effective due to its three main nutrients: nitrogen, phosphorus and magnesium, and it promotes plant growth. For example, the weight gain rate of tomatoes that have been treated with this fertilizer can increase by four times or more. One study [26] states that the main advantage of this technology is that the product is hardly soluble and does not go into the ground during irrigation or heavy rains. Therefore, the fertilizer “works” during the entire agricultural period.

Two main schemes have been tested in previous research: the production of struvite from domestic wastewater or a model solution of ammonium ions (Figure 2), and the production of struvite from a mixture of mineral water and domestic wastewater or a model solution of NH_4 (Figure 3). Domestic wastewater is the main source of ammonium ions, and mineral water is rich in magnesium ions, phosphates, and chlorides. The second scheme (Figure 3) allows you to save on reagents, reduce the total mineralization of water, and find a use for spent mineral waters from WMSs.

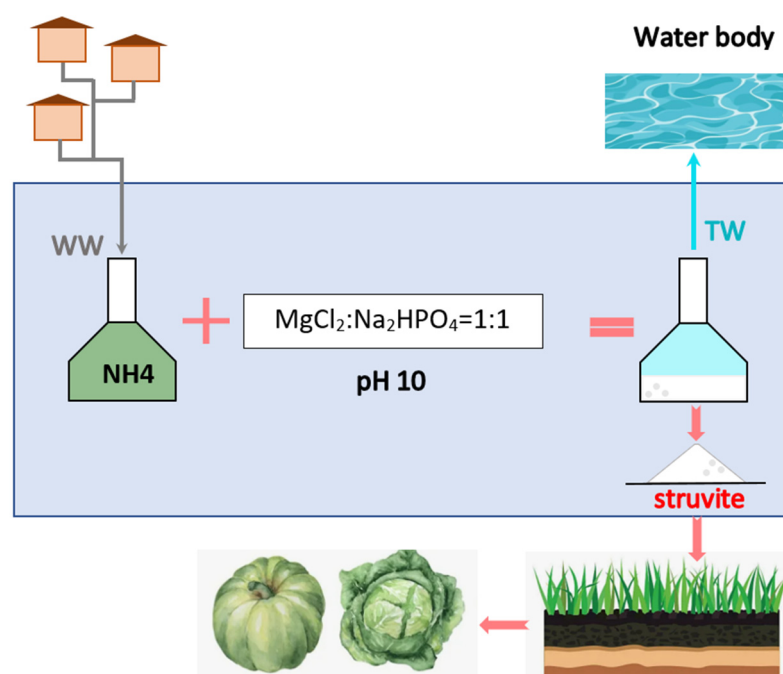


Figure 2. Struvite extraction from domestic wastewater (WW): treated water (TW) can be discharged into a water body.

2.4. Experimental Conditions and Limitations

The task was performed at a stable laboratory room temperature of 22–23 °C. In one article, the authors [27] considered lower water temperatures, as it was shown that the physico-chemical treatment of domestic wastewater is not significantly affected by a decrease in temperature. At the same time, the typical temperature of domestic wastewater is 16–18 °C, while spent mineral water has a temperature of up to 33 °C. The temperature of the mixture of the two waters was in the range of 23–24 °C.

Doses of reagents R1 (sodium hydrophosphate), R2 (magnesium chloride) and R3 (sodium hydroxide) were calculated using molar masses for the average concentration of ammonium ions, which are often found in domestic wastewater (the calculation is given in the Supplementary Materials, Section S1). Optimization of reagent doses was

carried out experimentally. The reagents were fed into the water in a certain order and mixed for 5 min at a speed of 200 rpm.

The pH value of 10 ± 0.5 in the water sample was determined based on an experiment conducted using the optimization method, depending on the residual concentration of NH_4 (Supplementary Materials, Section S4).

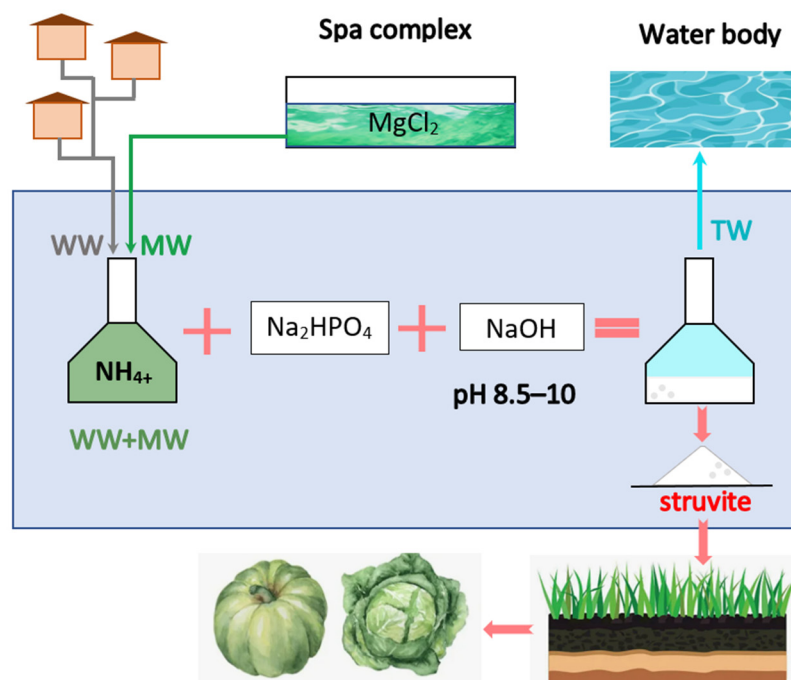


Figure 3. Struvite extraction from a mixture of mineral water (MW) and domestic wastewater (WW): the treated water (TW) can be discharged into a water body.

After settling, the supernatant fluid was carefully drained and the precipitate was filtered on a paper filter and dried in natural conditions at a temperature of $22\text{--}23\text{ }^{\circ}\text{C}$. Other types of pollutants are present in the composition of real wastewater, which in practice can affect the quality of sediment, while the content of pure struvite in it ranges from 20 to 75%. Section S2 (Supplementary Materials) provides an example of calculating the theoretical amount of pure struvite as a function of the initial concentration of ammonium ions. To confirm that struvite is present in the composition of the obtained sediment, an X-ray phase analysis was performed (the results are presented in Supplementary Materials, Section S3).

2.5. Determination of Measurement Error

For each experiment, three parallel measurements were performed. To estimate the error, standard methods were used to account for the error of direct measurements (Formulas (2)–(5)). The measurement error is written as

$$X = \bar{X} \pm \Delta; \quad (2)$$

where $\bar{X}$ is the average value of the measured indicator; Δ is the absolute measurement error.

The average value of the indicator is determined using the formula

$$\bar{X} = \frac{1}{n} \cdot \sum_{i=1}^n X_i; \quad (3)$$

where n is the number of measurement results.

The uncertainty of indirect measurements is estimated using the standard deviation of the results and Student's t-value:

$$S(\bar{X}) = \sqrt{\sum_{i=1}^n \left(\frac{\partial f}{\partial x_i} \right)^2 \cdot S^2(\bar{x}_i)}; \quad (4)$$

$$\Delta = S(\bar{X}) \cdot t; \quad (5)$$

where $s^2(\bar{x}_i)$ is the average square deviation of the random errors of the measurement result of the x_i -th argument; t is Student's t-value, depending on the confidence probability $p = 0.05$ and the number of measurements $n = 3$. The relative error is determined as a percentage (%) of the average value, which is accounted for by the absolute error Δ .

3. Results

3.1. Production of Struvite from a Model Ammonium Solution

At the beginning of the experiment, a model ammonium solution with a concentration of 60 mg/L was prepared (a detailed calculation is provided in Supplementary Materials, Section S1): based on the calculation, 0.178 g of ammonium chloride (NH_4Cl) was added to distilled water with a volume of 1 L. Next, solutions of 11.91% sodium hydrophosphate solution (R1) and 6.75% magnesium chloride solution (R2) were prepared. A 10% solution of sodium hydroxide (R3) was used to maintain a constant pH = 10. A model solution of ammonium (MSA) was added to five tall laboratory beakers with a working volume of 1 L each, then, with rapid mechanical stirring; the reagents were added in the following order: R1, R2 and R3. In each experiment, solutions of reagents R1 and R2 were added in the same volumes (10, 20, 30, 50 and 60 mL). The temperature of the water samples was in the range of 23–24 °C. Further, each sample was subjected to gravitational settling, during which a white precipitate was released.

In the initial and clarified (after struvite deposition) water samples, the concentrations of ammonium ions (NH_4), phosphates (PO_4), chlorides (CL), pH index, and volume of the formed sediment were measured. The measurement results are shown in Table 4. Figure 4 shows the dependence of the content of residual ammonium ions in clarified water on the applied fraction (%) of reagents R1 and R2, with 100% being the maximum volume of each reagent solution of 60 mL per 1 L of sample. The experiment to increase the volume of reagent solutions continued until the minimum value of the residual concentration of ammonium ions, not exceeding 1.5 mg/L, was reached. This meets the requirements for discharge into category B water bodies according to the standards for CBS with a capacity of 100–1000 m³/day [24].

Table 4. The results of the chemical precipitation of struvite in a model solution of ammonium.

Addition of the Main Reagents, %	Volume of R1 Solution, mL	Volume of R2 Solution, mL	NH4 Concentration, mg/L	CL Concentration, mg/L	PO4 Concentration, mg/L
0	0	0	53.50 ± 1.32	124,1 ± 8.6	0
16	10	10	15.51 ± 1.25	350 ± 15	0.71 ± 0.05
33	20	20	9.72 ± 0.82	615 ± 22	0.88 ± 0.05
50	30	30	7.29 ± 0.66	850 ± 22	1.06 ± 0.08
83	50	50	3.51 ± 0.41	1170 ± 26	1.22 ± 0.08
100	60	60	1.08 ± 0.09	1200 ± 30	1.35 ± 0.11

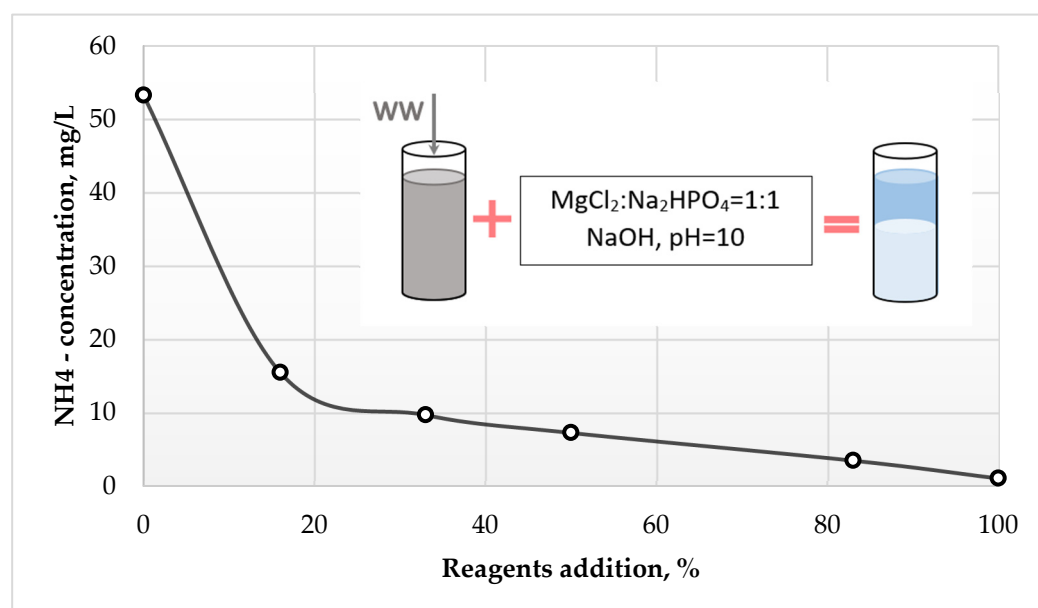


Figure 4. The change in the concentration of ammonium ions in the solution after the addition of reagents.

The maximum dose of R1 reagent was 5.67 g/L, and the dose of R2 reagent was 3.22 g/L. The concentration of chlorides was monitored in all samples: it increased with the addition of magnesium chloride to the water sample and ranged from 124 to 1200 mg/L. The residual phosphate concentration (PO_4) was also under control, and it ranged from 0.71 to 1.35 mg/L. For the maximum concentration of chlorides, the standard for discharge into a water body has not been established. For phosphates, the norm is 1–1.5 mg/L, and it is established when discharged into water bodies of category B for WWTPs of low productivity (1000–100 m^3/day). For WWTPs with a capacity of 10–100 m^3/day , the concentration of phosphorus phosphates in water can reach 5 mg/L [24].

As a result of each experiment, a white precipitate with a clear phase separation boundary was formed in the laboratory beaker. The precipitate was compacted for several hours. The maximum degree of compaction and, consequently, a decrease in the interface were achieved after 6 h. Figure 5 shows the dependence of the sludge volume on the duration of compaction. The initial volume of precipitate was 49.1 cm^3 , and after compaction—27.5 cm^3 . The moisture content of the sediment immediately after chemical deposition was 97.8%. After 6 h of compaction, it was 96.07%, and the average rate of precipitate moisture reduction was 0.29% per hour. If such a precipitate is dried at a constant temperature, a white powder is eventually obtained. In this case, after drying at room temperature 23 °C, 5.35 g of powder was obtained. The obtained product is sludge including the valuable mineral fertilizer struvite or $\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$, applied to the soil in dry form (confirmed by X-ray phase analysis; Supplementary Materials Section S3).

Thus, an experiment with a model MSA solution showed the possibility of obtaining struvite by chemical precipitation of ammonium ions, while the minimum residual concentration of NH_4 ions in a water sample was 1.08 mg/L. The amount of sludge containing struvite obtained from 1050 mL of the sample was 5.35 g in dry weight. The content of pure struvite in this sediment does not exceed 1.1 g, which corresponds to the published data [37].

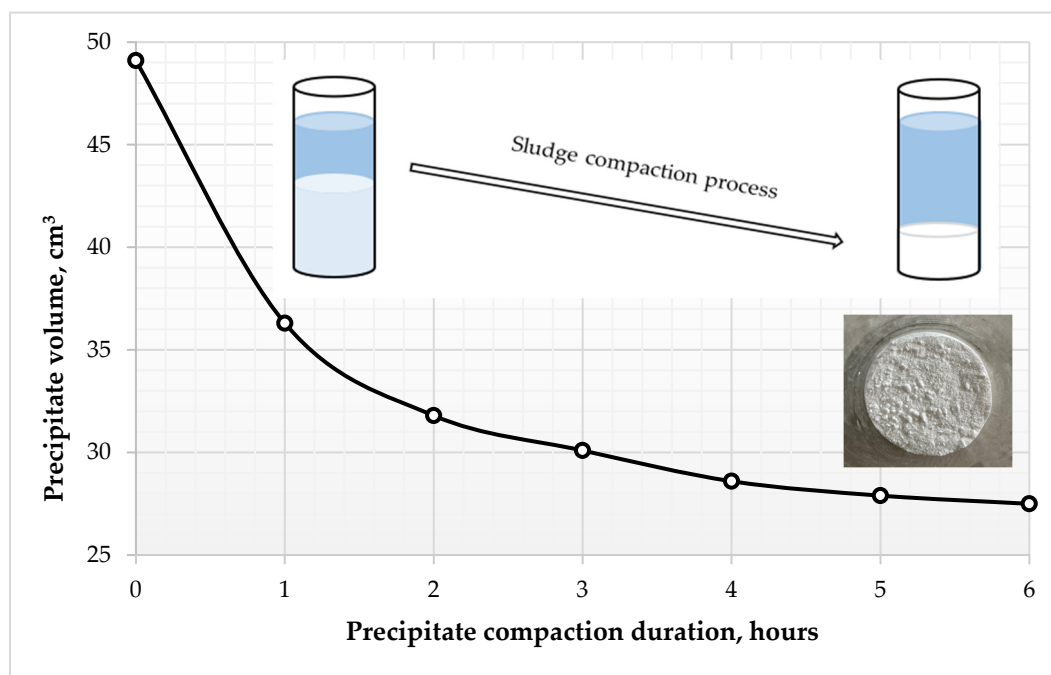


Figure 5. Precipitate compaction process.

3.2. Production of Struvite from a Mixture of a Model Solution and Mineral Water

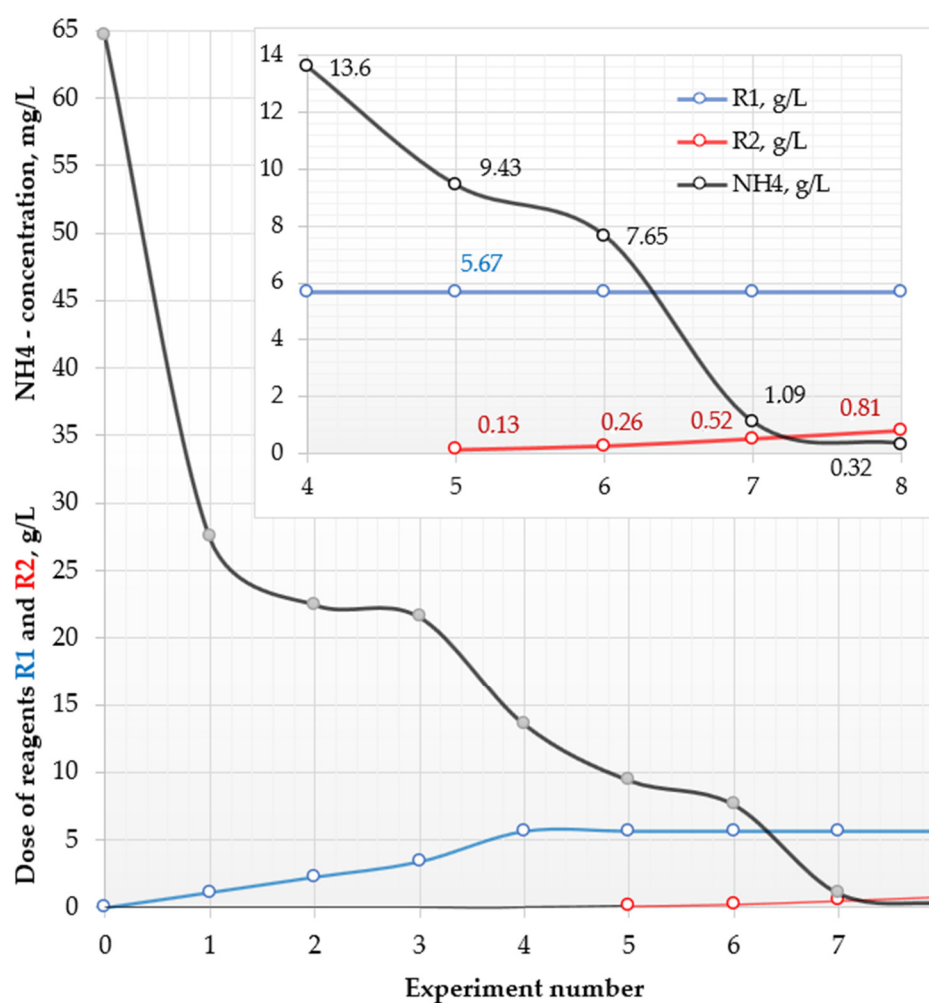
In the second stage of the experiment, the object of research was a mixture of a model solution of ammonium ions and mineral water (MSA + MW) saturated with magnesium and chlorides. The initial NH_4 concentration in the model solution was 82.8 mg/L; the NH_4 concentration of the MSA + MW mixture was 64.7 mg/L. In this series of experiments, the R2 reagent (magnesium chloride) was replaced with spent mineral water taken from the “Verkhny Bor” hot spring. Different degrees of dilution MSA:MW were considered, 0.25, 0.5, and 0.75, in order to increase the concentration of magnesium in the mixture of the model solution and mineral water. A model solution was added to laboratory tall beakers with a working volume of 1 L; then, with rapid mechanical stirring, MW mineral water was added in the required dilution. Then, the reagents were added in the following order: R1 and R3. In each experiment, the R3 reagent solution was applied drop by drop until the pH index reached 10 at a water temperature of 23–24 °C. After applying the reagents, each sample was settled until struvite was obtained as a precipitate. In the samples after struvite deposition, the pH was measured, which tended to decrease by an average of 5–7%.

When only sodium hydrophosphate (R1) was added to the MSA + MW mixture, the magnesium contained in mineral water reacted with other chemical elements and allowed for the reduction in NH_4 content, as well as the formation of struvite. In this experiment, it was possible to reduce the content of ammonium ions to a concentration of 13.6 mg/L, but this is not enough to achieve the established norm of 1.5 mg/L [24]. The experiment showed that to reduce the ammonium concentration below 1.5 mg/L, the addition of R2 reagent (magnesium chloride) to the MSA + MW mixture was still required, although in smaller quantities (0.52–0.81 mg/L) than was carried out in the previous series of experiments with a pure model MSA solution (Section 3.1). Table 5 shows the results for diluting MSA with mineral water by 75%; the relative error did not exceed 10%.

Figure 6 shows the dynamics of the decrease in the concentration of ammonium ions depending on the doses of the R1 and R2 reagents. Figure 7 shows photographs of the laboratory production of struvite from a mixture of MSA + MW.

Table 5. Reagent doses and residual contaminant concentrations after struvite deposition in a mixture of a model solution and mineral water at a dilution of 75%.

The Dose of the R1 Solution, g/L	The Dose of the R2 Solution, g/L	NH ₄ Concentration, mg/L	pH After Precipitation	Mineralization, mg/L	Sediment Volume, cm ³
1.13	0	27.5	9.03	4935	170
2.27	0	22.5	9.60	5021	180
3.41	0	21.6	9.65	5196	190
5.67	0	13.6	9.85	5273	200
5.67	0.13	9.43	10.01	5345	220
5.67	0.26	7.65	9.96	5419	230
5.67	0.52	1.09	9.33	5563	280
5.67	0.81	0.32	8.66	5725	300
Initial solutions:					
MSA	-	84.8 ± 1.52	6.7	190 ± 16	-
MW	-	5.6 ± 0.4	7.7	6407 ± 95	-
MSA + MW (75%)	-	64.7 ± 1.5	7.3	4853 ± 57	-

**Figure 6.** Dynamics of the decrease in the residual concentration of ammonium NH₄ ions depending on the doses of R1 and R2 reagents.

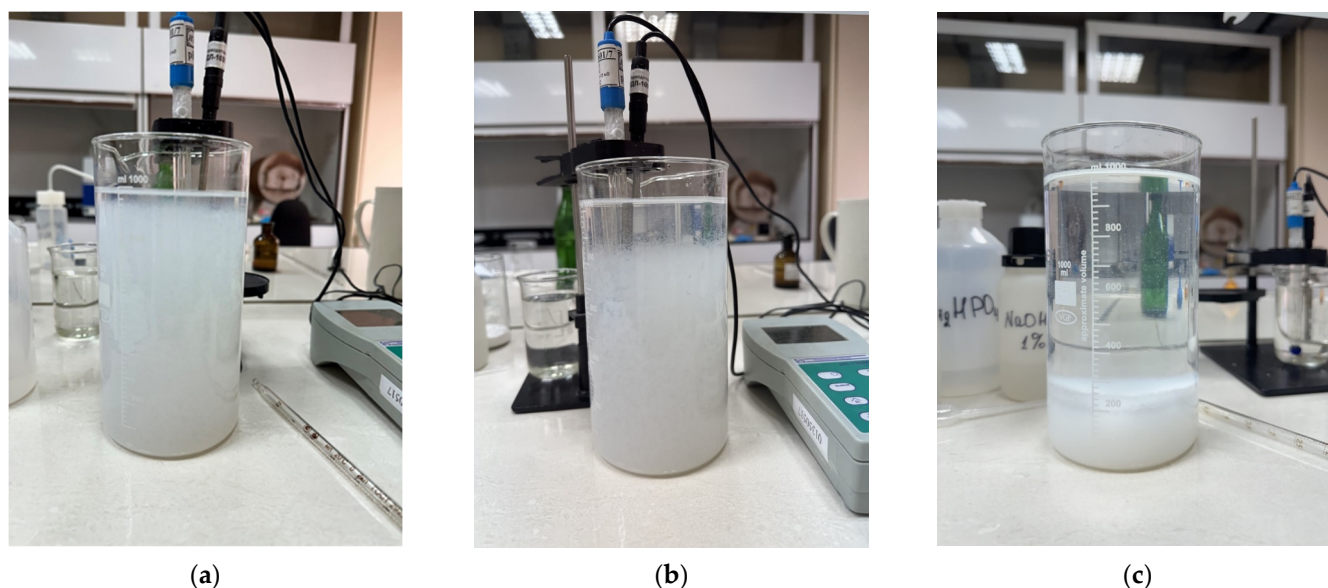


Figure 7. Struvite deposition in the laboratory (photos) from a mixture of MSA and MW: (a) the initial stage of coagulation; (b) formation of the phase interface; (c) formed sediment (not compacted).

Thus, when mixing a model solution of ammonium ions and mineral water, it was possible to reduce the consumption of R2 reagent by almost four times. At the same time, the residual concentration of ammonium ions was 0.32 mg/L, and the total mineralization in the MSA + MW mixture (75% dilution with mineral water) after settling of struvite did not exceed the initial value in MW (5725 mg/L < 6407 mg/L). The initial volume of precipitate in the sample was 300 mL. A decrease in the pH index from 10 to 8.66 was observed after settling. The results obtained confirm the fact that magnesium compounds in mineral water from natural WMSs can participate in the formation of struvite crystals [36].

3.3. Obtaining Struvite from a Mixture of Real Wastewater and Mineral Water

The process of struvite formation in a mixture of real domestic wastewater (WW) and mineral water (MW) was studied under conditions of varying degrees of dilution: without dilution, and with the addition of MW in volumes of 25%, 50% and 75%. The real WW was taken from the primary settling tank at the Tyumen sewage treatment plant (the composition is presented in Table 2). During the experiment, a decrease in the consumption of magnesium chloride (R2) was expected, since mineral water contains magnesium and its compounds in a sufficiently high concentration (65.6 mg/L). Table 6 shows the dose values of R1 and R2 reagents, concentrations of NH_4 , mineralization and PO_4 after the chemical deposition of struvite. Figure 8 shows a reduction in the use of R2 reagent and the concentration of residual ammonium NH_4 for different dilutions of wastewater and mineral water content in wastewater. The residual ammonium in all samples did not exceed the standard value (1.5 mg/L). Phosphates in the process also decreased from 8.22 to 4.83 mg/L, and as a result did not exceed the norm of 5 mg/L [24]. Presumably, this also occurred due to the use of part of the PO_4 in the WW to produce struvite.

The results showed that in this experiment, the use of real wastewater did not affect the result as a whole: with similar doses of R1, R2 and R3 reagents (Section 3.1), it was possible to reduce the concentration of NH_4 to the established standards. When diluting WW with mineral water, it was possible to reduce the dose of magnesium chloride (R2) fivefold. At the same time, the concentration of NH_4 remained acceptable for discharge into a water body. The total mineralization as a whole increased from 3380 to 5460 mg/L; this is due to an increase in the proportion of mineral water and the yield of chlorides

in solution. However, overall, the mineralization was 14.8% lower than in the original mineral water. The pH index ranged from 9 to 10. The volume of precipitate ranged from 100 to 200 cm³; after drying at a constant temperature in room conditions, it was possible to obtain 4.75 ± 0.3 g of dry precipitate.

Table 6. Reagent doses and residual contaminant concentrations after struvite deposition in a mixture of real wastewater and mineral water.

Degree of Dilution WW: MW, %	The Dose of R1 Solution, g/L	The Dose of the R2 Solution, g/L	NH ₄ Concentration, mg/L	Mineralization, mg/L	PO ₄ Concentration, mg/L
0	5.67	3.22	0.74 ± 0.11	3380 ± 22	8.22
25	5.67	2.57	1.29 ± 0.16	4430 ± 35	7.54
50	5.67	1.29	0.94 ± 0.44	4640 ± 38	6.63
75	5.67	0.64	1.21 ± 0.20	5460 ± 52	4.83
Initial solutions					
WW	-	-	82.9 ± 3.2	670 ± 23	18.5 ± 1.3
MW	-	-	5.6 ± 0.4	6407 ± 95	0.11 ± 0.01

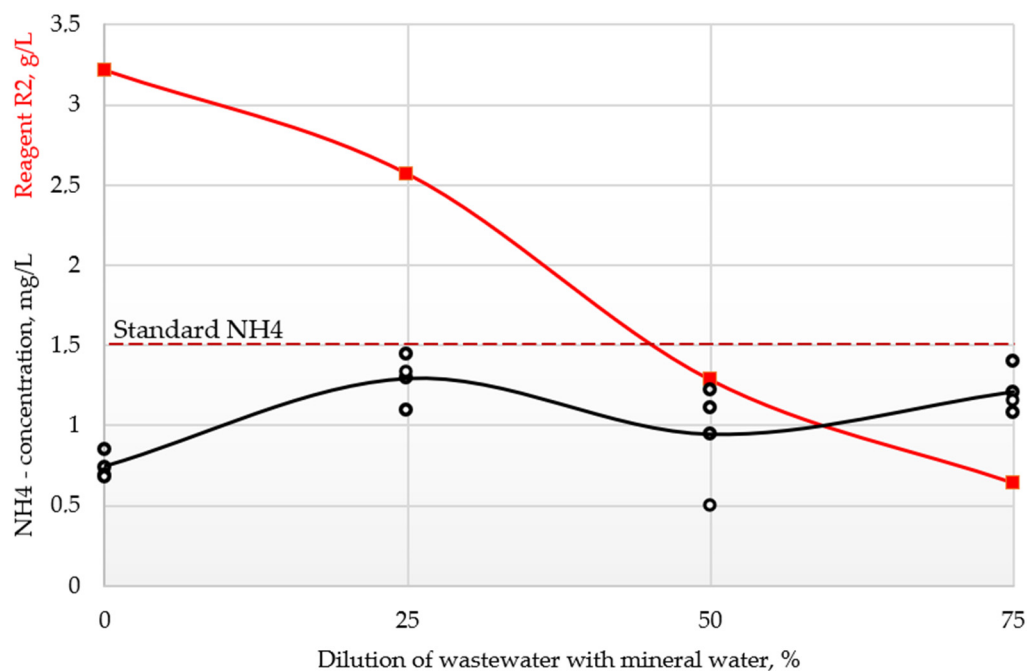


Figure 8. Change in the dose of magnesium chloride (R2) and residual ammonium (NH₄) in the sample depending on the degree of dilution of the wastewater with mineral water.

4. Discussion

4.1. The Mechanism of Struvite Production

Struvite crystallizes in the orthorhombic system in the shape of pyramidal crystals from white to yellowish or brownish-white in color or in the shape of lamellar mica. The mechanism of formation of struvite crystals is still not fully understood. It is assumed that the stability of crystals is based on the electrostatic interaction of ions: the formal charge (+1) on nitrogen in NH₄ will be attracted to one of the (−1) formal charges on oxygen in PO₄, and the magnesium ion absorbs some of the free electrons on the other two oxygens. Then, H₂O forms strong hydrogen bonds with a total dipole moment. According to [38], the struvite crystal structure consists of PO₄ tetrahedra, Mg·6H₂O octahedra, and NH₄ groups held together by hydrogen intermolecular bonds. Figure 9 shows the following scheme of

the mechanism for producing struvite from an aqueous solution of ammonium ions. The scheme shows how groups of NH_4 ions, PO_4 tetrahedra, and $\text{Mg} \cdot 6\text{H}_2\text{O}$ octahedra form struvite crystals ($\text{NH}_4\text{MgPO}_4 \cdot 6\text{H}_2\text{O}$) that then precipitate.

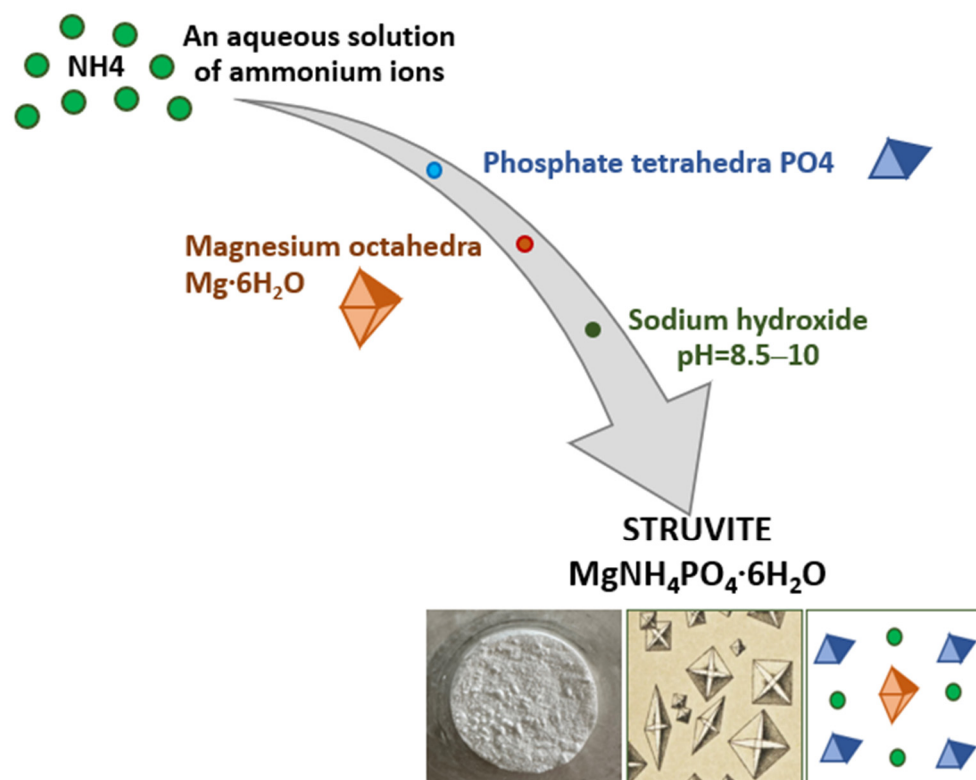


Figure 9. Scheme of the mechanism for obtaining struvite crystals.

Based on the results of these studies, it can be concluded that experiments with real waters and model solutions will differ in the doses of reagents and the amount of precipitation obtained due to the presence of ions of various substances in wastewater that are absent in solutions prepared with distilled water—calcium, sodium, potassium, iron, sulfates, nitrites, and nitrates, as well as higher concentrations of phosphates and magnesium. All this leads to differing quality in the resulting struvite. For example, ref. [39] discusses in detail how iron impurities affect the structure of struvite. The section on X-ray phase analysis shows how the diffractogram changes: when Fe^{3+} is added to the solution, the pure struvite phase is first preserved, and when a certain concentration is exceeded, peak broadening and a shift towards larger angles can be observed, indicating compression of the crystal lattice due to the substitution of Mg^{2+} ions by Fe^{3+} ions. In addition, the authors in [40] proved that on average, 94–97% of the product obtained from brines was struvite; the remaining 3–6% was hazenite ($\text{MgNaPO}_4 \cdot 4\text{H}_2\text{O}$) (both struvite and hazenite are potentially suitable as fertilizers). They determined that the struvite yield depended on the molar ratio N:P: with increasing N:P 0.2:1 to 1.2:1, the proportion of struvite increases from ~52% to ~93%.

The amount of fertilizer that can be obtained depends on the concentration of NH_4 in the source water. When using domestic wastewater, the content of pure struvite in the sediment can range from 20 to 75%. Additionally, this is influenced by the active use of industrial reagents, which sometimes reaches as little as 50%. Moreover, in real wastewater, the interfering effects of other types of pollutants increase. This significantly affects the quality of struvite and the increase in reagent consumption. In addition, domestic wastewater must be pre-treated by sedimentation, meaning that up to 90% of pathogenic

organisms (larvae and eggs of helminths) precipitate together with suspended solids to reduce the likelihood of biological contamination.

4.2. Controversial Issues and Promising Areas for Research

When using chemical methods for the removal of ammonium ions from domestic wastewater, the following controversial issues arise: (1) economic expediency of using reagents; (2) increased salinity of water after treatment; and (3) formation of secondary waste (chemical sewage sludge). Undoubtedly, biological purification remains a priority for large cities and towns as the main method of decomposing ammonium ions in the processes of nitrification and denitrification. Recent studies [41–44] have shown that adjusting the parameters of the aeration tank, supplementing physical effects (for example, magnetic field, electrodialysis, microwave irradiation, or others), introducing granular activated sludge [45,46], and other methods can help achieve high levels of purifying wastewater (WW) through the removal of ammonium ions (NH_4) and phosphates (PO_4). In this case, we consider small objects that can occupy a decentralized position in relation to large cities that provide biological WWTPs. It is not always possible to build and operate your own biological treatment facilities. In addition, at spa resorts of mineral water, there are large amounts of used mineral water containing large volumes of unclaimed useful minerals. The simple discharge of this MW into the natural environment entails irreversible consequences for the soil, flora and fauna [14].

Chemical precipitation of ammonium ions is one of the methods that can be used to completely abandon the biological treatment of domestic wastewater. Mixing domestic wastewater and used mineral water makes it possible to reduce the cost of reagents, as well as reduce environmental damage from the discharge of mineral water into water bodies and onto the terrain. In addition, it becomes possible to obtain a secondary useful product—mineral fertilizer for the soil. And as practice shows, the operation of physico-chemical treatment plants is much easier and can be performed completely automatically.

In this case, the problem of increasing the mineralization of domestic wastewater has been partially eliminated: the residual mineralization in the treated WW + MW mixture is 15–30% lower than in the initial mineral water. A solution to this may be the introduction of ion-exchange filters or desalination membranes at the last stage of water treatment.

In future studies, the following issues need to be examined in more detail: the quality of the struvite produced; bacterial contamination of struvite; the influence of other factors (for example, the quality of domestic and mineral wastewater, the quality of reagents, temperature, and others) on the quality of struvite. It is also required to check the germination of plant seeds using this secondary product obtained from wastewater. In addition, a more detailed feasibility study is required to substantiate the economic feasibility of using this technology in practice, taking into account all factors (costs of reagents and organization of water purification processes, reduction in damage to the environment, sale of a secondary product, and others). It is also required to conduct industrial tests at a real facility to confirm in practice all of the theoretical results obtained.

5. Conclusions

Hot mineral springs are distinguished by two types of liquid municipal waste: domestic wastewater and waste mineral water. During the study, it became clear that it is possible to simultaneously purify water containing ammonium ions and obtain a valuable agricultural fertilizer, struvite. This requires three types of reagents: sodium hydrophosphate, magnesium chloride, and sodium hydroxide to stabilize the pH. In this case, the concentration of ammonium ions can be reduced to the standard value (1.5 mg/L) or lower. Mineral water can also participate in this process and compensate for reagent consumption

(i.e., reduce magnesium chloride consumption by five times) due to its natural chemical composition. In laboratory tests, it was possible to obtain a precipitate containing struvite, which is saturated with nitrogen and phosphorus compounds. This makes it possible not only to purify wastewater by removing ammonium but also to achieve economic efficiency through the implementation of a secondary water treatment product.

Nevertheless, unresolved problems remain: increased water mineralization after struvite sedimentation, including due to chloride release; instability and diversity of the quality of domestic wastewater and, as a result, the influence of other types of pollutants on reagent costs; and other factors affecting the quality and quantity of struvite. However, solving these issues in the future will serve as a guarantee for the sustainable development of resort areas and the conservation of natural resources.

Supplementary Materials: The following supporting information can be downloaded at <https://www.mdpi.com/article/10.3390/su18168049/s1>, Section S1: Calculation of the concentration of the model solution; Section S2: Theoretical calculation of the amount of struvite; Table S1: Approximate values; Section S3: The results of the XRD Struvite study; Figure S1: Sediment–struvite diffractogram; Figure S2: XRD—analysis of the struvite crystal; Figure S3: XRD sediment analysis; Figure S4: The structure of struvite under a microscope; Section S4: Optimization of the pH of chemical precipitation of ammonium ions; Figure S5: The dependence of the residual concentration of ammonium ions in water on the pH value; Table S2: The results of the study of the residual concentration of ammonium ions in the model solution depending on the pH change.

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Abbreviations and Designations

The following abbreviations and designations are used in this manuscript:

WMSs	Warm Mineral Springs
WW	Wastewater
MSA	Model Solution of Ammonium
MW	Mineral Water
WWTP	Wastewater Treatment Plant
TW	Treated Water
NH ₄	ammonium-ions NH ₄ ⁺
PO ₄	phosphate-ions PO ₄ ^{3−}
CL	chloride-ions Cl [−]
R1	sodium hydrophosphate Na ₂ HPO ₄
R2	magnesium chloride MgCl ₂
R3	sodium hydroxide NaOH

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