

ADDITIONAL TREATMENT OF BIOLOGICALLY TREATED WASTEWATER USING ENVIRONMENTALLY FRIENDLY SORBENTS

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Abstract. Biological wastewater treatment is effective for the removal of organic matter; however, residual nitrogen and phosphorus often remain in the treated effluent, contributing to eutrophication of receiving water bodies. Small and decentralized wastewater treatment plants are particularly vulnerable to fluctuating influent loads and operational constraints, which can result in elevated concentrations of ammonium and phosphate in the final effluent. This study evaluates environmentally friendly zeolite-containing sorbents as tertiary filtration media for polishing biologically treated domestic wastewater. Column filtration experiments were conducted using wastewater after biological treatment, and the performance of four sorbents was assessed by monitoring phosphate phosphorus (PO₄-P), ammonium nitrogen (NH₄-N), nitrate nitrogen (NO₃-N), and pH. The results demonstrate that zeolite-based sorbents can effectively reduce nutrient concentrations, with notable differences in removal efficiency among the tested materials. The findings indicate that sorbent-based tertiary treatment represents a sustainable and low-energy option for reducing nutrient loads from small-scale wastewater treatment facilities, contributing to improved water quality and resource-oriented wastewater management.

Keywords: wastewater, nutrient removal, adsorption, phosphate, ammonium, nitrate, tertiary treatment.

1. Introduction

Nutrient pollution in water often originates from a specific discharge point, such as a wastewater treatment plant (WWTP). Nitrogen and phosphorus concentrations in human waste and wastewater streams from domestic sources are around 50 and 10 mg/l, respectively (Nikpour et al., 2020). Approximately one quarter of all water quality degradation is due to nutrient-related causes. Harmful algae blooms begin in water bodies, nutrients and oxygen become scarce, ammonia is released, biological integrity is compromised, and turbidity increases (Arias et al., 2025; Brenckman et al., 2025). As concerns about pollution increase, much attention is being paid to the removal and capture of nitrogen and phosphorus compounds in WWTP. Biological wastewater treatment technologies are fundamental and are constantly being improved. In wastewater treatment plants, nitrogen removal mainly occurs in nitrification-denitrification processes, but nitrogen is not completely removed during these processes. The total nitrogen removal efficiency using conventional biological methods is only 28–30% (Ye et al., 2008) or 59% (Wang et al., 2008). Using modern

modified systems, the total nitrogen removal efficiency increases to 79% (Nikpour et al., 2020). Biological nitrogen removal has disadvantages. Wastewater treatment is costly and requires long reaction times, especially when treating wastewater with high nitrogen content, where its effectiveness is limited (Ye et al., 2008).

Phosphorus-accumulating microorganisms (PAOs) are important in the removal of phosphorus from wastewater, as they accumulate phosphate as extracellular polyphosphate and remove phosphorus from the liquid phase. This biological method does not effectively remove phosphorus from wastewater. According to Troiani et al. (2011) and Ye et al. (2008), phosphorus removal using conventional activated sludge or anaerobic oxidative precipitation process is only 62% of total P (Troiani et al., 2011; Ye et al., 2008). The phosphorus removal efficiency in the membrane bioreactor process modified with anaerobic oxidative precipitation process is also low, only about 28–63% (Saby et al., 2003).

The biological phosphorus removal process is sensitive to environmental and operating conditions, and its efficiency can be significantly affected by changes in temperature, pH and dissolved oxygen (Abdoli et al.,

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2024; Li et al., 2013). Biological phosphorus removal is believed to be less effective in colder climates (Liu et al., 2019). The operation of biological wastewater treatment plants can be erratic, resulting in repeated discharges of phosphorus and poor separation of sludge from treated wastewater, increasing its turbidity (Gebremariam et al., 2011). In practice, when removing phosphorus from wastewater, the biological method is combined with the chemical method. Chemical phosphorus removal typically involves precipitating influent phosphorus with an iron or aluminum salt. Disadvantages of this method include storage requirements and sludge production increases of 20% to 30%.

Recently, in the search for new methods to more effectively remove nitrogen and phosphorus from wastewater, much attention has been paid to sorbents (Usman et al., 2022). Sorption or retention of nitrogen compounds from wastewater occurs through ion exchange, ligand exchange, physisorption, chemisorption and chemisorption (Ai et al., 2023; Sahu et al., 2024). Sorption or retention of phosphorus compounds from wastewater occurs due to electrostatic attraction, surface complexation, hydrogen bonding and precipitation (Gao et al., 2025). Sustainable applications of mineral adsorbents are possible (Wei et al., 2024). Natural zeolite can be modified to improve its sorption properties. For example, MnO₂-coated zeolite granules demonstrated superior cadmium adsorption in simulated 'soil solution' (Bondar et al., 2025).

Nitrogen and phosphorus are two of the leading causes of eutrophication and water degradation when discharged in excess to water bodies from municipal and industrial wastewater streams. Their effective removal and potential recovery therefore are critical for environmental protection and also for advancing resource sustainability. In recent years, sorbent-based technologies using natural, modified and engineered materials have attracted more and more attention as potential methods for nutrient removal and recycling, with some of these systems showing potential in the production of value-added fertilizer products (Gray et al., 2014; Mažeikienė & Šarko, 2023). Natural mineral sorbents like zeolite (clinoptilolite), bentonite and glauconite have been widely studied for the removal of ammonium and phosphate from aqueous solutions. Among these materials, clinoptilolite-based zeolites have a high affinity for ammonium ions due to their ion-exchange capacity, lead to reported NH₄⁺ removal efficiencies up to 76–90% and sorption capacities up to 42.5 mg/g for nano-sized materials. Phosphate removal by natural zeolites generally is not high; nevertheless, surface modification with iron, lanthanum, alkali or surfactants can significantly improve sorption performance Fe³⁺ greater than those achieved with unmodified materials (Cyrus & Reddy, 2010; Manikandan & Subramanian, 2017; Mažeikienė & Šarko, 2023; Moharami & Jalali, 2015). Similar enhancement effects have been reported for bentonite, where

Fe³⁺ having modification capacity of phosphate removal to more than 4 times (Moharami & Jalali, 2015). Other natural sorbents such as biochar and glauconite have also shown potential for removing nutrients although their performance is highly dependent on the feedstock, conditions used in the preparation of the sorbent and actual modification strategy.

Beyond natural materials, several commercial and engineered sorbents have been produced in order to obtain greater nutrient removal efficiencies. Ion exchange resins such as Amberlite IRA-958 and hybrid metal loaded resins have shown phosphate removal up to 95% achieved in both lab and pilot study, and partial nutrient recovery achievable via chemical regeneration (Bottini & Rizzo, 2012; Liu et al., 2016; Mei et al., 2013). Nano-structured sorbents such as magnetic composites, metal-organic framework (ZIF)-based materials and lanthanum-modified biochars have shown extremely high phosphate sorption capacity with simultaneous removal efficiency of ammonium and phosphate contents approaching 95%. Hybrid composite materials using mineral and nano scale have also demonstrated increased performance and improved recyclability (Sahu et al., 2024; Usman et al., 2022; Yang et al., 2025).

In addition to the conventional sorbents, due to the low cost and the potential for circular economy, industrial wastes-based materials like fly ash, red mud, slag and ceramsite, derived from sludge or fly ash, have gained attention. Modified fly ash based zeolites have been reported to have a phosphate sorption capacity up to 250 mg/g (Barca et al., 2022; Cheng et al., 2018; Hermassi et al., 2020). Red mud based and gypsum based materials have shown good removal of phosphorus, while possessing good hydraulic properties. Ceramsite materials with alkali or lanthanum modification have been proven to have a nitrogen and phosphorus recovery function at the same time and can be suitable for use as slow-release fertilizer products (Shao et al., 2022).

The mechanisms that control nutrient sorption vary, depending on the sorbent composition and modification. Ammonium removal is controlled mainly by ion exchange processes, especially in the case of materials and ion exchange resins based on zeolites. In contrast, phosphate removal is dominated by chemisorption and precipitation reactions, which are facilitated by presence of metal oxides such as iron, lanthanum, magnesium and aluminum. Sorption kinetics may be characterized by pseudo-second-order models, whereas equilibrium behaviour may be characterized by Langmuir or Freundlich isotherms. However, competing ions, pH and wastewater matrix complexity may have significant effects on sorption performance (Hu et al., 2025; Huo et al., 2012).

Although several studies have demonstrated high nutrient removals efficiencies with the help of sorbents, the performance in real wastewater is generally lower than

in synthetic systems. In municipal and domestic wastewater, natural and modified sorbents generally have ammonium removal efficiencies in the range of 58–76%, nitrate removal ranging from 14–15% and phosphate removal of 9–45% with modified materials usually exhibiting higher performance than their natural counterparts (Mažeikienė & Šarko, 2023). Ion exchange resins are able to keep high removal efficiencies of phosphate from real sludge liquors although there is a potential decrease of regeneration efficiency due to organic fouling. Industrial waste-based sorbents have also shown interesting results in the phosphate removal from real effluents, with also the potential nutrient recovery (Barca et al., 2022; Shao et al., 2022).

By contrast, in experiments performed based on synthetic wastewater, higher nutrient removal capacities are often reported because of controlled conditions and the absence of competing which may lead to overestimation of performance under practical operating conditions. Consequently, there is still a need for systematic testing of sorbent-based nutrient removal under the condition of biologically treated domestic wastewater in continuous flow condition that is more appropriate for real applications (Cinta Vincent-Vela et al., 2024; Nagabalaaji et al., 2019).

Natural minerals containing zeolite have different impurities that affect the adsorption process. In this work, four zeolite-containing materials were tested for the treatment of wastewater (after biological treatment) from nitrogen and phosphorus compounds. The chemical (elemental) composition of these materials was investigated and their effectiveness in reducing the concentrations of phosphate phosphorus, ammonium nitrogen and nitrate nitrogen in filtered wastewater was evaluated. Typically, adsorbents are tested by removing one pollutant at a time from wastewater. This work differs from others in that ammonium nitrogen, nitrate nitrogen, and phosphate phosphorus were removed from wastewater in the same experiment, during a single filtration.

2. Methodology

2.1. Biologically treated wastewater and experimental stand

For this study, domestic wastewater after treatment in individual facilities was brought to the VILNIUS TECH laboratory. The temperature, pH, conductivity, ammonium nitrogen, nitrate nitrogen, and phosphate phosphorus concentrations of the delivered wastewater were measured there. In total, wastewater was delivered 6 times. The wastewater was poured into a 5-liter container, from which it was supplied by a pump to 4 columns mounted on a stand (Figure 1). The diameter of the columns was 2.5 cm, and the height of the sorbent filling was 15 cm.

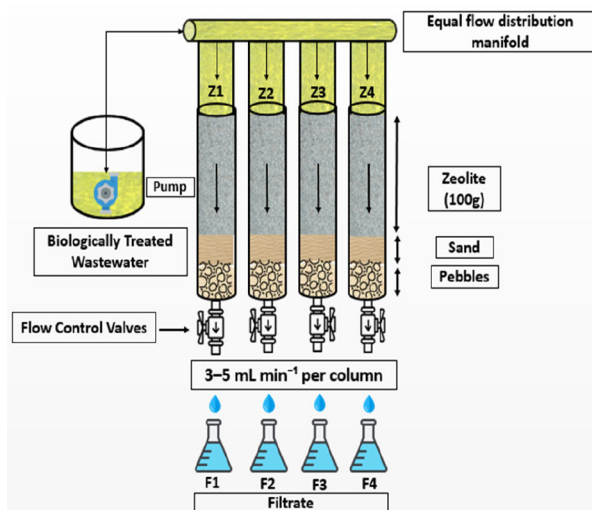


Figure 1. Schematic diagram of the experimental stand used for tertiary treatment of biologically treated wastewater

2.2. Column fillers

The aim was to find the best adsorbent that simultaneously removes nitrogen and phosphorus compounds from wastewater, and 4 zeolite-containing materials were selected for the research:

1. Zeolite mining tailings grains, obtained from Ukraine (Z1). The fraction (0.6–1.0 mm) was obtained by sieving the zeolite grains through sieves.
2. Soil Substrate ZeoSand2 (Z2). ZeoSand product is intended for lawn care or ventilation. Improves H₂O retention in the soil and reduces nutrient loss. Produces a fine fraction (0.5–1.0 mm).
3. Zeolite primer for water filtration in aquariums “Zeolayer” (Z3). It is a non-toxic, 100% natural mineral material without dyes, additives and chemical components. Zeolite very fine 1–3 mm.
4. Zeolite-rich tuff, the black zeolite from Ukraine (Z4). It is a naturally occurring volcanic rock composed primarily of zeolite mineral and hydrothermally altered volcanic ash deposits. The tuff consists mainly of clinoptilolite (65–90 wt%) along with associated minerals such as montmorillonite, quartz, feldspar, calcite, biotite, muscovite, and chlorite. This tuff is characterized by high porosity, selective ion exchange, and strong adsorption (Costafreda et al., 2024).

The selected materials were washed with tap water, dried (2 hours at 105 °C) and poured into 4 filtration columns (all of the same mass – 100 g each). Under each sorbent filler was a supporting layer of small pebbles and quartz sand (2 cm high).

2.3. Measurement methods

Temperature and pH for the wastewater samples were determined immediately after sampling in accordance

with standard laboratory procedures by calibrated portable probes.

The concentration of phosphate phosphorus ($\text{PO}_4\text{-P}$) was determined by the MERCK Spectroquant® test kits. The measurement range of the test was 0.50–30.0 mg/L $\text{PO}_4\text{-P}$, and absorbance measurements were carried out by transferring the prepared test solutions in 10 mm optical glass cuvettes (Hellma, Germany) and measuring the absorbance at the wavelength of 410 nm by Genesys 10 UV-Vis spectrophotometer (Thermo Fisher Scientific, USA). A control sample was prepared in the same way using distilled water in the place of test sample. The phosphate phosphorus concentration was determined by multiplying the measured amount of absorbance by a factor of 18.0, as described by the manufacturer of the test.

Ammonium nitrogen ($\text{NH}_4\text{-N}$) concentration was also determined using MERCK Spectroquant® test kits, with a measurement range of 2.0–75.0 mg/L $\text{NH}_4\text{-N}$. After a reaction time of 15 mins, absorbance was measured at 690 nm and measured with the same UV-Vis spectrophotometer and type of cuvette. Control samples were prepared analogously using distilled water. The concentration of ammonium nitrogen was determined by multiplying the measured amount of absorbance with a factor of 27.8.

Nitrate nitrogen ($\text{NO}_3\text{-N}$) concentration was determined using MERCK Spectroquant® test kits with measurement ranges of 0.10–25.0 mg/L $\text{NO}_3\text{-N}$ and 0.4–110.7 mg/L NO_3^- . Absorbance readings were taken after a reactivity time of 10 minutes at 340 nm using 10 mm cuvettes and the Genesys 10 UV-Vis spectrophotometer. Control samples were prepared by using distilled water in the same manner. Nitrate concentration was calculated by multiplying the measured absorbance by a factor of 86.8.

The elemental composition of the materials containing zeolite was analyzed by wavelength-dispersive X-ray fluorescence (WD-XRF). Measurements were carried out using a ZSX Primus IV spectrometer (Rigaku Corp., Japan) following standard analytical procedures.

All physical and chemical analyses were performed in triplicate. The results are presented as mean values, and the associated variability is expressed as standard deviation (SD).

3. Results

3.1. Phosphate phosphorus removal

Three independent experiments were performed on the removal of phosphate phosphorus ($\text{PO}_4\text{-P}$) at 0.5, 2, and 3 h. The effluent concentrations and efficiencies of removal are represented in Figures 2 and 3. The influent concentration phosphate in all sets of the experiments was approximately between 6.5 and 17.9 mg/L. Each of the zeolite-containing sorbents was able to effectively

decrease phosphate contents compared to the influent.

The phosphate removal efficiencies of all sorbents in the first set of experiments were above 82% and Z2, Z3 and Z4 had efficiencies of over 90% throughout the filtration. The removal efficiencies reduced a little in the second set of experiments and it was approximately in between 65 and 82% but Z4 performed better with longer time of filtration. In the third experimental set, there was more variation in the performance; however, Z2 showed the excellent phosphate removal of as high as 98% at the end of the 2 h filtration period.

Considering all three experimental sets together, Z2 exhibited the most stable and effective phosphate removal, followed by, Z4 whereas Z1 and Z3 experienced earlier breakthrough and higher sensitivity with an influent variation. All experiments had a phosphate removal efficiency of between about 65–99%, thus indicating that the zeolites based sorbents can be used to obtain high phosphorus removal under continuous-flow.

3.2. Ammonium nitrogen removal

Three independent experiments were performed on the removal of ammonia nitrogen ($\text{NH}_4\text{-N}$) at 0.5, 2, and 3 h. The effluent concentrations and efficiencies of removal are represented in Figures 4 and 5. Across all

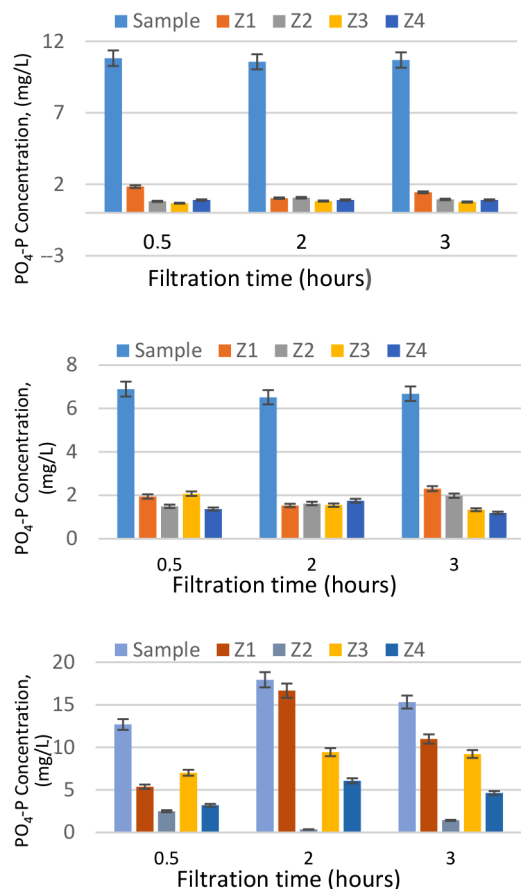


Figure 2. $\text{PO}_4\text{-P}$ removal concentrations (mg/L)

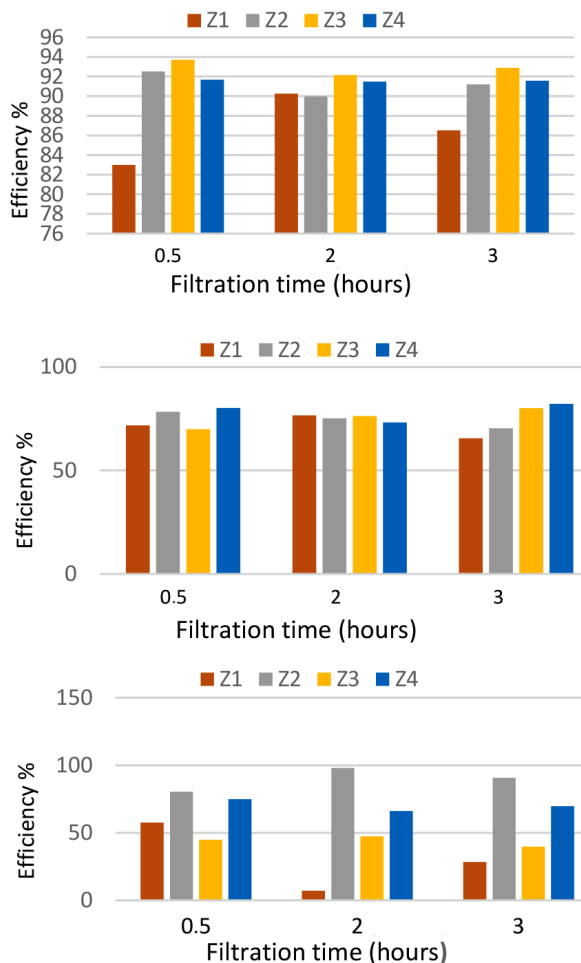


Figure 3. PO₄-P removal efficiency graph

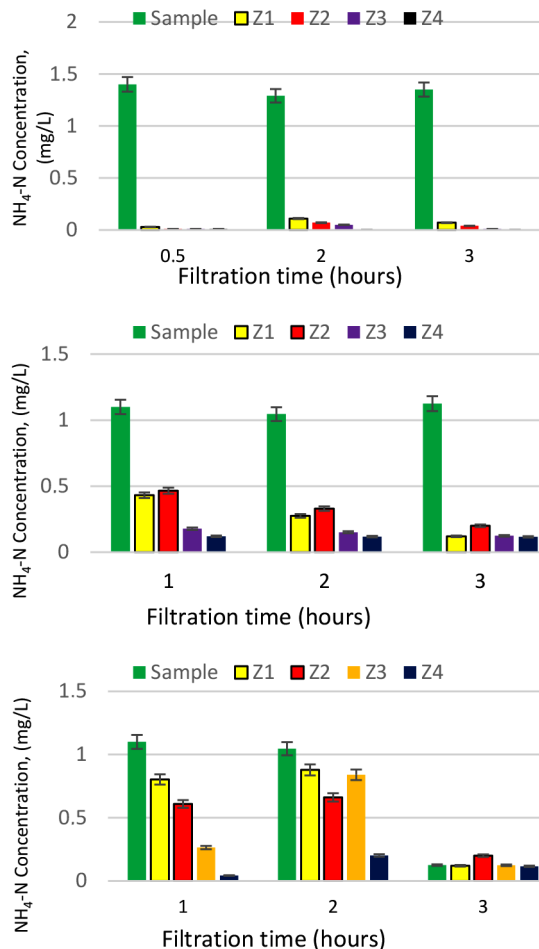


Figure 4. NH₄-N removal concentrations (mg/L)

experimental sets, influent ammonium concentrations ranged between approximately 1.0 and 1.40 mg/L, implying moderate variation in the treated wastewater. All zeolite-containing sorbents had the potential to lower the level of ammonium concentration, though it was observed that the level and the consistency of removal was uneven across the materials as well as across the experimental runs.

In the first experimental set, high ammonium removal efficiencies were obtained for all sorbents. The removal efficiency of NH₄-N was greater than 94% for Z1–Z3, while Z4 achieved complete 100% removal. In the second experimental set, removal efficiency is approximately between 57% to 89%. In the third experimental set, bigger variation was observed in removal of NH₄-N. At 0.5 h the initial removal efficiencies varied between 27% and 96% and a negative trend was noted in removal efficiencies at longer filtration time, with removed efficiencies dropping below 10% after 3 h of operation with all the sorbents.

When considering all three experimental sets together, Z4 showed the best and the most stable experiment, often achieving zero effluent levels of NH₄-N. Z3 was also found to be strong in ammonium removal as

compared to Z1 and Z2, which were sensitive to the variants in the influent and time of filtration. Generally, the efficiency of all experiments in removing ammonium was between about 27 and 100%, which meant that under the good operating conditions, the zeolite containing sorbents would be able to deliver useful application in ammonium reduction although the performance can also vary based on the wastewater composition and stability of the operation.

3.3. Nitrate nitrogen removal

Three independent experiments were performed on the removal of nitrate nitrogen (NO₃-N) at 0.5, 2, and 3 h. The effluent concentrations and efficiencies of removal are represented in Figures 6 and 7. Across the experimental sets, influent nitrate concentrations showed considerable variation, ranging from approximately 0.6 to 2.5 mg/L, indicating variations in the composition of the biologically treated wastewater. Compared to ammonium and phosphate, removal of nitrates was usually less and more fluctuated across all the tested sorbents.

The first set of the experiments had less efficiency of nitrate removal, approximately between 5% and 40%

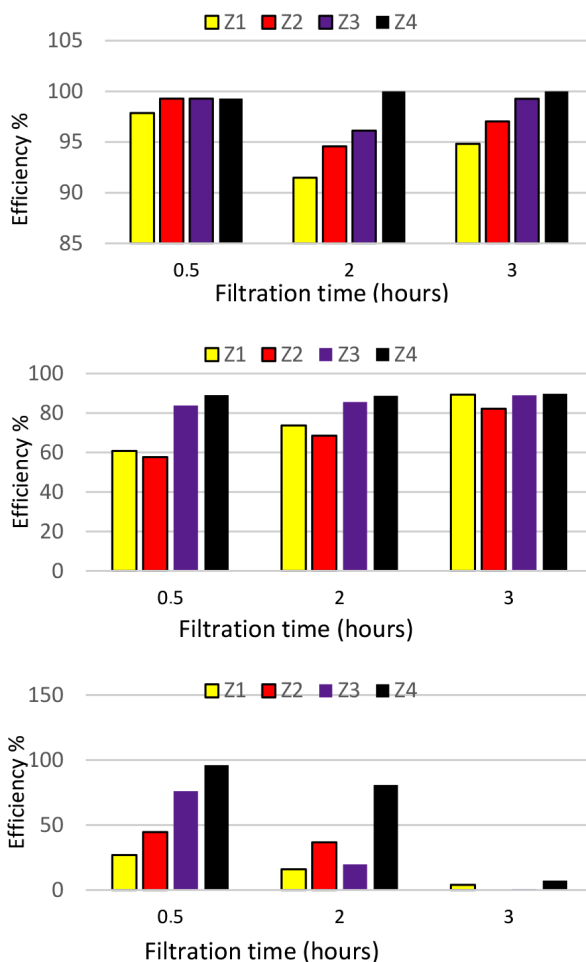


Figure 5. NH₄-N removal efficiency graph

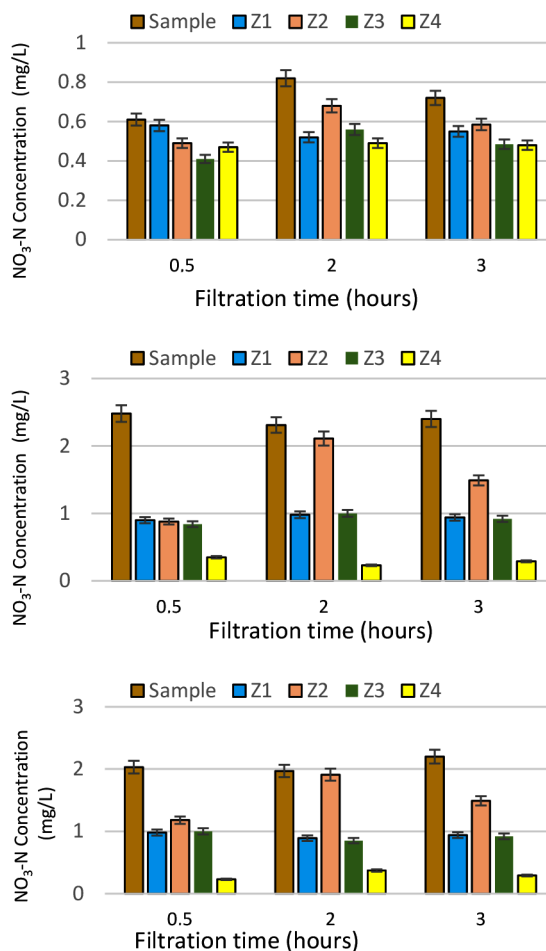


Figure 6. NO₃-N removal concentrations (mg/L)

based on the sorbent and filtration time. Among the other sorbents, Z4 has the highest rate of nitrate removal. Greater nitrate removal efficiencies were experienced in the second set of the experiment. The performance of Z4 was good and the removal efficiencies were above 85% at all the time of filtration. The trends obtained in the first and second set were similar in the third experimental set as well. Z4 recorded best removal of nitrates with efficiencies up to 88% and the rest of the samples, Z1 and Z3 recorded moderate removal efficiencies. Z2 was the least efficient amongst the sorbents.

Considering all three experimental sets, Z4 demonstrated the most consistent and good removal of the nitrate, and Z1 and Z3 provided the average type of removal of the nitrate but Z2 was the least and the highly fluctuating. In general, the efficiencies of nitrate removal in the experiments were approximately between 3% and 90%, which fully verified that by zeolite-based sorbents, it was possible to perform nitrate removal, although it heavily relied on the properties of the sorbents, the influent composition, and the operating conditions.

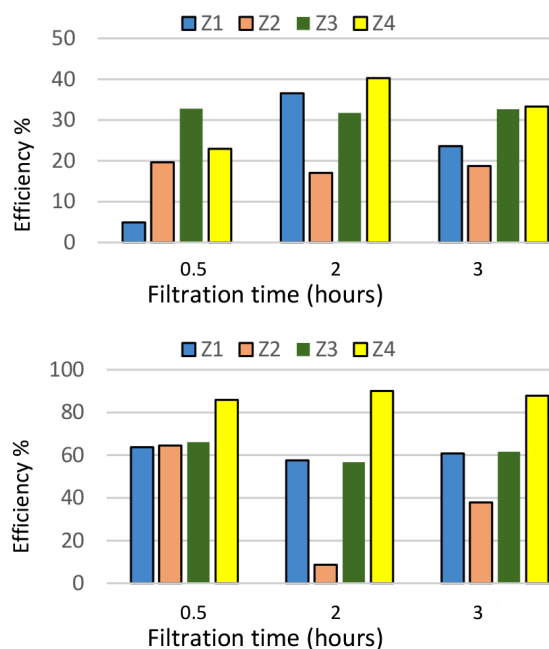


Figure 7. To be continued

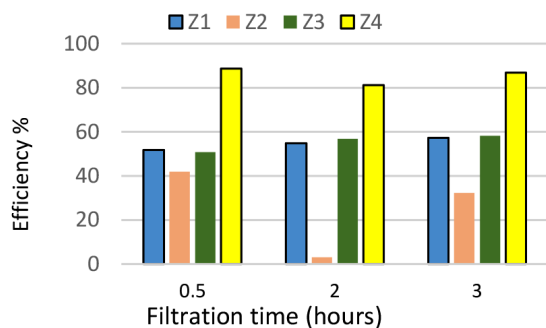
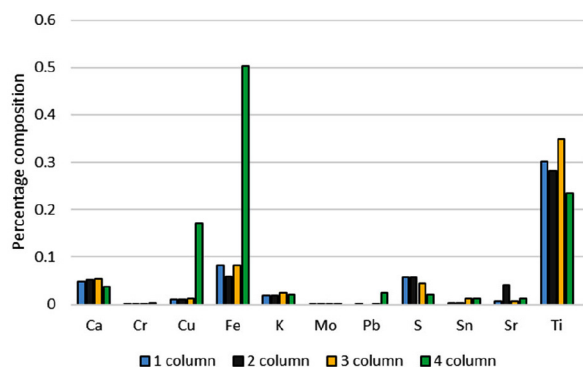


Figure 7. NO₃-N removal efficiency graph

3.4. Composition of sorbents

The chemical elemental composition of the tested samples is presented in Figure 8:



Note: Al, Si, Mg concentrations were not measured in the samples.

Figure 8. The chemical elemental composition: 1 column – Z1, 2 column – Z2, 1 column – Z3, 1 column – Z4

The Figure 7 shows that all zeolite samples contain Ca, Cu, Fe, K, S and Ti. Z4 contains more Cu, Fe, Pb and Zn than the others.

Summarizing the results of the study, it can be seen that all three column fillers reduced the concentrations of phosphate phosphorus, ammonium nitrogen, and nitrate nitrogen in the filtered wastewater. The concentrations of phosphorus and nitrogen compounds in wastewater were probably reduced not only due to adsorption (physical and chemical adsorption could have occurred), but also due to sedimentation and bacterial activity. Real wastewater was filtered after biological treatment, and it contained activated sludge microorganisms that entered the wastewater samples during their collection. It is assumed that bacterial enzymes contributed to the reduction of nitrate concentrations in wastewater, as observed in previous studies (Mažeikienė & Šarko, 2023). Typically, the efficiency of nitrogen compound removal from wastewater is higher at higher initial concentrations. In this study, the initial concentrations of ammonium nitrogen and nitrates were low, so their reduction efficiency is significant. Although the tested sorbents appear to be suitable for reducing the nitrogen and phosphorus

contamination of filtered wastewater, more studies should be conducted in the future before these sorbents can be recommended for practical use. In this study, the full sorption capacity of the tested materials was not utilized and material regeneration was not performed. In the future, detailed studies of these promising materials are planned not only on a laboratory scale.

4. Conclusions

This study showed that all 4 zeolite-containing materials removed phosphorus and nitrogen compounds from wastewater. All experiments had a phosphate removal efficiency of between about 65–99%. Phosphorus concentration was most effectively reduced around (93–98%) by Z2 and Z3 respectively. The concentrations of nitrate nitrogen and ammonium nitrogen were most effectively reduced approximately in between (96–99%) by the Z4. The results were influenced by the chemical composition of the zeolitic materials and the properties of the filtered wastewater. It is noteworthy that these zeolitic materials removed phosphate phosphorus, ammonium nitrogen, and nitrate nitrogen from wastewater at the same time by filtering the wastewater through flooded columns. This indicates that zeolite-based sorbents can be used for additional wastewater treatment to achieve high removal of phosphorus and nitrogen compounds during continuous flow.

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