

APPLICATION OF NANO ZERO-VALENT IRON PARTICLES FOR THE REMOVAL OF PHOSPHATE FROM RIVER WATER

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Abstract. Nano zero-valent iron (nZVI) particles have attracted much attention due to their ability to eliminate phosphate ($\text{PO}_4\text{-P}$) from water. In this study, samples of river water with a high concentration of $\text{PO}_4\text{-P}$ were collected from Laukupė River, near the treated municipal wastewater drain outlet. The concentration of $\text{PO}_4\text{-P}$ in the sampled river water was 1.1 mg/L (bad ecological status). Adsorption experiments were carried out to determine the suitability of nZVI to remove $\text{PO}_4\text{-P}$ from actual river water. The applied dose of nZVI ranged from 0.05 g/L to 3.2 g/L, and contact time ranged from 5 to 120 min. The results have shown that the 0.4 g/L dose of nZVI was sufficient to reduce the $\text{PO}_4\text{-P}$ concentration below 0.05 mg/L after 5 min of contact time to reach a high ecological status of river water with respect to phosphate.

Keywords: kinetic modelling, adsorption, river water, nanomaterials, phosphate.

Introduction

Pollution of water with nitrogen (N) and phosphorus (P) compounds is constantly increasing. Phosphorus compounds enter wastewater treatment plants as a result of the use of chemicals that contain P in homes, industry, communal services, and other areas (Mažeikienė et al., 2021). Phosphate compounds, such as sodium tripolyphosphate, are commonly added to many washing and cleaning products to improve their performance by binding hardness-giving salts and preventing the formation of deposits (Comber et al., 2013).

Wastewater treatment plants typically remove a significant amount of nutrients, including N and P, from wastewater prior to releasing it into natural water bodies. However, some residual compounds may still be present in the treated effluent and can enter the environment. When these nutrients enter natural waters, they can contribute to eutrophication and reduce the ecological status of water bodies (Mažeikienė & Šarko, 2021). Therefore, the removal of P compounds from surface waters is crucial. In Lithuania, to reach a high ecological status of river water in terms of phosphate, the concentration of $\text{PO}_4\text{-P}$ must be below 0.05 mg/L (Ministry of Environment..., 2010). Chemical removal of P is a reliable and practically proven method for

removing P from water. Most often, three types of metals are used in this method: iron (Fe), aluminium (Al), and calcium (Ca) (Arias et al., 2001). In recent years, engineered nanomaterials have begun to be used for environmental clean-up applications after scientists discovered that small amounts of nanomaterials can efficiently eliminate various pollutants from ground or surface water. One example of those nanomaterials is nanoscale zero-valent iron (nZVI). Many studies have been conducted on the removal of various pollutants, such as heavy metals and phosphates ($\text{PO}_4\text{-P}$) from artificially contaminated water samples by nZVI (Danila & Šerevičienė, 2021; Gil-Díaz et al., 2021; Maamoun et al., 2021; Vasarevičius et al., 2019; Wen et al., 2014). Several studies have successfully applied nZVI for the removal of phosphate from real wastewater (Arshadi et al., 2018; Liu et al., 2022). However, little research has been done on the removal of $\text{PO}_4\text{-P}$ from actual river water.

The purpose of the work was to determine the effect of contact time and nZVI dose on the efficiency of $\text{PO}_4\text{-P}$ removal from river water. Three different adsorption kinetic models were applied to study the reaction rate controlling mechanism. The best-fit model was determined by comparing statistical measures such as the reduced chi-square error and residual sum of squares for each model.

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1. Materials and methods

1.1. Sample collection and analysis

Laukupė River is a river in north-eastern Lithuania that flows through the municipality of Rokiškis district. The length of the river is 24 km and the catchment area is 60 km². The river flows into the Nemunėlis River at 176 km from its mouth. The water samples from the Laukupė River were collected near the treated municipal wastewater drain outlet (Lithuanian coordinates X598185; Y6203885). The sampling was carried out according to the ISO 5667-1:2020 standard (Water Quality – Sampling). The samples were taken directly into dark 2 litre bottles. The samples taken were stored at 4 °C. The pH of the water samples was measured using the Mettler Toledo pH metre. The methods to measure the concentrations of nitrates (NO₃-N), nitrites (NO₂-N), ammonium nitrogen (NH₄-N), total N, total P, and PO₄-P are presented in Table 1.

Table 1. Methods for measuring ecological parameters of river water samples

Parameter	Research method
pH	LST ISO 10523:2012. Water quality. Determination of pH.
Total nitrogen, mg/L	Measurements with a total carbon analyser Shimadzu TOC-V.
Nitrates, mg/L	LAND 65-2005. Determination of nitrate. Spectrometric method using sulfosalicylic acid.
Nitrites, mg/L	LAND 39-2000. Water quality. Determination of nitrite. Molecular absorption spectrometric method.
Ammonium nitrogen, mg/L	LAND 38-2000. Determination of ammonium. Manual spectrometric method.
Total phosphorus, mg/L	LAND 58:2003. Water quality. Determination of phosphorus. Ammonium molybdate spectrometric method.
Phosphates, mg/L	LAND 58:2003. Water quality. Determination of phosphorus. Ammonium molybdate spectrometric method.

Note: LAND: Lithuanian Environmental Protection Normative.

In this work, all of the chemicals used were of analytical grade and all the solutions were prepared using deionised water.

1.2. Nano zero-valent iron

The nZVI powder used in this study was commercially available from Nano Iron s.r.o. (Czech Republic) under the trade name NANO FER STAR. The powder contains surface-stabilized Fe(0) nanoparticles that are air-stable due to a thin layer of Fe oxides coating the surface. This layer of Fe oxides serves to prevent immediate oxidation

upon exposure to atmospheric oxygen. The obtained nanoparticles were in the form of agglomerates; therefore, the nanoparticles were activated by the preparation of an aqueous suspension from this powder (1 part of nZVI powder was mixed with 4 parts of deionised water using an ordinary blender). The weight content of Fe (0) in suspension was approximately 18%.

1.3. Adsorption kinetics

Batch adsorption experiments were conducted to determine the suitability of nZVI to remove PO₄-P from actual river water. The experiments were carried out in 100 ml glass bottles, in which 100 ml of river water samples were poured. The applied doses of nZVI were 0.05, 0.10, 0.20, 0.40, 0.80, 1.6 and 3.2 g/L. The mixtures were shaken for 5, 10, 30, 60, and 120 min using a Gerhardt RS12 shaker at room temperature. The mixtures were then filtered using 0.45 μm membrane filters.

The removal efficiency (%) of PO₄-P was calculated as follows:

$$R = \frac{C_0 - C_e}{C_0} \times 100, \quad (1)$$

where: C_0 is the initial concentration of PO₄-P in river water sample (mg/L), C_e is the equilibrium concentration of PO₄-P (mg/L).

The removal capacity (mg/g) at a certain contact time was calculated as follows:

$$q_t = \frac{(C_0 - C_t) \times V}{m}, \quad (2)$$

where: C_t is concentration of PO₄-P at certain time interval (mg/L), V – is the volume of the treated river water sample (L), m – is the mass of the sorbent (g).

Three kinetic models were used to interpret the adsorption experiments: the pseudo-first order kinetic model, the pseudo-second order kinetic model, and the intraparticle diffusion model. The pseudo-first order model assumes that the rate of adsorption is proportional to the number of unoccupied sites on the solid surface, and that the concentration of adsorbate in the fluid phase decreases exponentially with time (Rodrigues & Silva, 2016). The pseudo-first order model can be expressed by non-linear equation form:

$$q_t = q_e \left(1 - e^{-k_1 t}\right), \quad (3)$$

where: q_e is the PO₄-P removal capacity at equilibrium (mg/g), k_1 is pseudo-first-order rate constant (1/min), t is time (min).

The pseudo-second order kinetic model assumes that the rate-limiting step in the adsorption process is the chemisorption and the rate of adsorption is proportional to the number of active sites available on the adsorbent surface (Nayak & Pal, 2019). The pseudo-second order adsorption kinetic model can be expressed by the

following equation:

$$q_t = \frac{q_e^2 k_2 t}{q_e k_2 t + 1}, \quad (4)$$

where k_2 is pseudo-second order rate constant (g/mg min).

The intraparticle diffusion model assumes that the rate of adsorption is controlled by intraparticle diffusion, which is the diffusion of adsorbate molecules from the bulk solution to the interior of the adsorbent particles. The intraparticle diffusion equation is expressed as:

$$q_t = k_i t^{0.5} + C, \quad (5)$$

where: k_i is the intraparticle diffusion rate constant (mg/g min^{0.5}) and C is the intercept.

1.4. Error analysis

The experiments were carried out in triplicate. The average values and standard deviations were calculated. The parameters of the kinetic models were calculated using the OriginPro software. The goodness-of-fit for kinetic models was assessed by the adjusted coefficient of determination (adj. R^2), reduced Chi-square error (χ^2) and residual sum of squares (RSS).

2. Results and discussion

2.1. Chemical parameters of river water samples and PO₄-P removal efficiency by nZVI

The concentrations of N and P compounds in the river water, along with the pH value, are presented in Table 2. The river water had a slightly alkaline pH of 7.85±0.05. Based on the concentrations of NO₃-N and NH₄-N, the ecological status of the river was classified as “good” and “poor”, respectively. The concentrations of PO₄-P and total P exceeded the limit values established for the river’s “very bad” ecological status classification by approximately 2.8 and 3 times, respectively. Therefore, in this study, nZVI was used to reduce the concentration of PO₄-P and improve the ecological status of the river.

The initial pH of river water samples and initial concentration of PO₄-P were kept constant during adsorption studies. HPO₄²⁻ is the predominant form of phosphate at pH values ranging between 7.2 and 12.7 (Ma et al., 2020). Figure 1 illustrates the effect of nZVI dose on PO₄-P removal efficiency and adsorption capacity. As shown in Figure 1, the efficiency of PO₄-P removal by

nZVI increased as the dose of nZVI increased. The efficiency of PO₄-P removal increased from 62.4% to 100% when the dose of nZVI increased from 0.05 to 3.20 g/L. The increase in sorbent dose led to an increase in the surface area and number of active adsorption sites available for PO₄-P adsorption (Edet & Ifelebuegu 2020; Wu et al., 2013). The adsorption capacity of PO₄-P decreased as the dose of nZVI increased. The highest adsorption capacity of PO₄-P was 13.7 mg/g when the dose of nZVI was the lowest. With an increasing dose of nZVI, the adsorption capacity of PO₄-P by nZVI gradually decreased to 0.34 mg/g. The decrease in PO₄-P adsorption capacity with an increased dose of nZVI could be attributed to the fact that a larger number of available sites on the nZVI surface were not occupied by phosphate molecules.

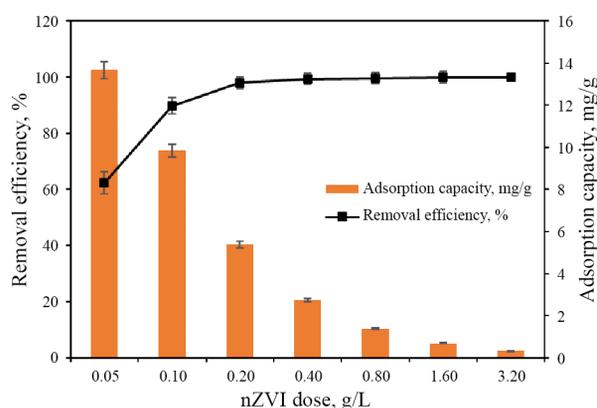


Figure 1. Effect of the dose of nZVI on PO₄-P removal efficiency and adsorption capacity (contact time: 120 min)

Significantly higher adsorption capacities of PO₄-P were observed in other studies where nZVI was used to remove phosphate from artificially contaminated water samples. For example, Wen et al. studied the removal of PO₄ from aqueous solution using nZVI and found that the maximum adsorption of PO₄ was 245.65 mg/g (Wen et al., 2014). Maamoun et al. studied P adsorption from aqueous solutions using nZVI. The authors found that the dose of nZVI of 1 g/L could remove 76.8 mg of P (Maamoun et al., 2021). In the other study, Eljamal et al. found that maximum PO₄-P adsorption capacity by nZVI was 28 mg/g (Eljamal et al., 2016). It can be assumed that the lower adsorption capacity of PO₄-P by nZVI obtained in this study was due to the lower concentration of PO₄-P in river water compared to that in aqueous solutions that were used in other studies. The lower dose of nZVI and the lower concentration of PO₄

Table 2. Chemical parameters of river water

Parameters						
pH	Total nitrogen, mg/L	Total phosphorus, mg/L	PO ₄ -P, mg/L	NH ₄ -N, mg/L	NO ₃ -N, mg/L	NO ₂ -N, mg/L
7.85±0.05	3.72±0.09	1.45±0.04	1.10±0.04	0.98±0.03	1.62±0.07	0.93±0.04

could decrease the probability of nZVI and pollutant collision in treated river water samples.

2.2. Adsorption kinetics

The kinetic modelling used nZVI doses of 0.05–0.80 g/L. The experimental data was analysed using the pseudo-first and pseudo-second order kinetic models, with the corresponding curves and parameter values presented in Figure 2a and 2b and Table 3. The results showed that

longer contact time increased PO₄-P adsorption capacity, with rapid removal observed at the initial reaction time. For instance, at a 0.05 g/L nZVI dose, 75% of the maximum PO₄-P adsorption capacity was achieved after 5 minutes of contact time, after which the reactions slowed due to saturation of active surface sites on nZVI. 0.4 g/L nZVI dose was sufficient to reduce the PO₄-P concentration below 0.05 mg/L after 5 minutes of contact time, reaching a “high” ecological status of river water with respect to phosphate.

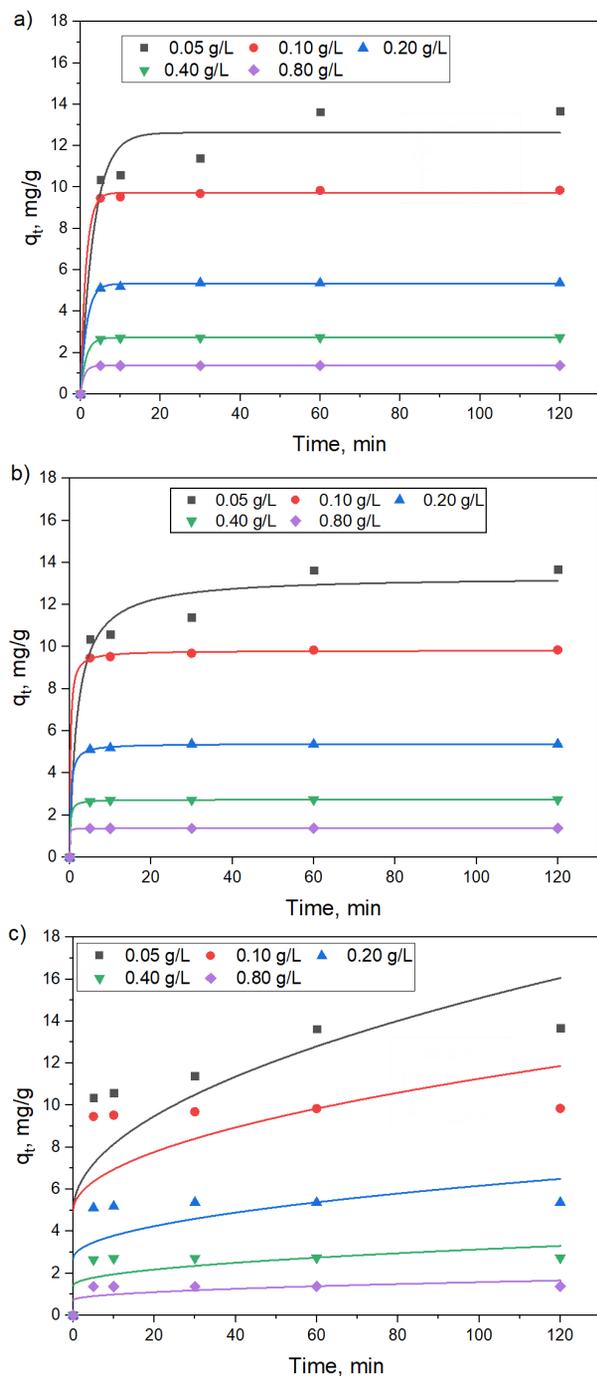


Figure 2. Non-linear curves of pseudo-first (a); pseudo-second (b); and intraparticle diffusion (c) kinetic models for PO₄-P adsorption on nZVI

Table 3. Kinetic parameters of pseudo-first and pseudo-second order kinetic models and intraparticle diffusion model

nZVI dose, g/L	Pseudo first order	Pseudo second order		Intraparticle diffusion		
	Parameters					
	$q_{e, cal1}$, mg/g	k_1 , 1/min	$q_{e, cal2}$, mg/g	k_2 , g/mg min	k_p , mg/g min ^{0.5}	C
0.05	12.6	0.29	13.3	0.041	1.01	4.94
0.10	9.73	0.72	9.81	0.481	0.631	4.95
0.20	5.33	0.63	5.39	0.619	0.347	2.69
0.40	2.72	0.71	2.74	2.22	0.173	1.40
0.80	1.37	1.04	1.37	20.2	0.085	0.717

To identify the most appropriate kinetic model, we compared the calculated equilibrium values (Table 3) with the values obtained experimentally. Our analysis revealed that there were only minor discrepancies between the calculated ($q_{e,cal}$) and experimental ($q_{e,exp}$) adsorption equilibrium values. The pseudo-second order model showed better agreement between the two sets of values, indicating its superior ability to describe the experimental kinetics accurately.

Table 4 presents the values of three statistical parameters, namely reduced chi-square error, residual sum of squares, and adjusted R^2 , which were obtained from the fits of the kinetic models used to describe the PO₄-P adsorption kinetics. The calculated statistical parameters indicate that both the pseudo-first and pseudo-second order kinetic models provided a good fit to the experimental data, as evidenced by their high adjusted R^2 values (>0.94). The reduced chi-square error and residual sum of squares are commonly used measures to assess the goodness of fit between the model and the experimental data, where lower values indicate a better fit. By comparing the statistical parameters obtained from different kinetic models, it was found that the pseudo-second order model exhibited the best fit to the experimental data.

The intraparticle diffusion model was also applied (Figure 2c) to further examine the rate control mechanism of PO₄-P adsorption. During intraparticle diffusion, PO₄-P anions could pass through the particles of the adsorbent and diffuse to the internal surface. As determined by statistical analysis (Table 4), adj. R^2 values

Table 4. Statistical parameters of the kinetic models

nZVI dose, g/L	Pseudo first-order			Pseudo second-order			Intraparticle diffusion		
	Parameters								
	Reduced chi-square error	Residual sum of squares	Adjusted R ²	Reduced chi-square error	Residual sum of squares	Adjusted R ²	Reduced chi-square error	Residual sum of squares	Adjusted R ²
0.05	1.48	5.91	0.9428	0.742	2.97	0.9713	11.8	47.4	0.540
0.10	0.0162	0.065	0.9990	0.00467	0.019	0.9997	11.6	46.5	0.255
0.20	0.00595	0.0238	0.9987	0.00108	0.00434	0.9998	3.43	13.7	0.262
0.40	$1.29 \cdot 10^{-4}$	$5.16 \cdot 10^{-4}$	0.9999	$4.22 \cdot 10^{-5}$	$1.69 \cdot 10^{-4}$	0.9999	0.929	3.72	0.238
0.80	$4.61 \cdot 10^{-6}$	$1.84 \cdot 10^{-5}$	0.9999	$4.25 \cdot 10^{-7}$	$1.70 \cdot 10^{-6}$	1	0.244	0.976	0.216

for the intraparticle diffusion model ranged from 0.216 to 0.540. The lower adj. R^2 values were for higher doses of nZVI. In addition, the values of reduced chi-square error and residual sum of squares obtained for the intraparticle diffusion model were considerably higher than those for pseudo-first and pseudo-second order kinetic models. Therefore, based on the statistical parameters, it can be concluded that the intraparticle diffusion model was not suitable for the description of the experimental data and that $\text{PO}_4\text{-P}$ diffusion through the pores did not occur.

Based on the experimental and modelling results of $\text{PO}_4\text{-P}$ adsorption by nZVI, it can be stated that the adsorption rate control mechanism involves two steps. Firstly, $\text{PO}_4\text{-P}$ ions diffuse from the bulk solution to the interface between the sorbent and liquid. Secondly, the ions are adsorbed onto the surface of nZVI. There are two possible mechanisms for this adsorption process, physical sorption and chemisorption. The point of zero charge for nZVI is around 8, which means that the surface of the nZVI is mainly positively charged at a pH less than 8 (Jiemvarangkul et al., 2011; Sun et al., 2007). As a result, negatively charged phosphate ions are attracted to the positively charged surface of nZVI through electrostatic forces, which is physical adsorption (pH of the river water was 7.85). In addition, chemisorption can occur as the phosphate ions react with Fe^{2+} or Fe^{3+} ions to form iron phosphate compounds such as FePO_4 or $\text{Fe}_3(\text{PO}_4)_2$, which precipitate on the surface of nZVI (Amiri et al., 2022; Maamoun et al., 2021).

Conclusions

Batch adsorption experiments were conducted in this study to investigate the removal of $\text{PO}_4\text{-P}$ from river water using commercially available nZVI. Results showed that the addition of 0.4 g/L of nZVI reduced the $\text{PO}_4\text{-P}$ concentration from 1.1 mg/L to 0.042 mg/L, achieving a removal efficiency of 96% within 5 minutes. Increasing the dose of nZVI and the contact time improved the removal efficiency of $\text{PO}_4\text{-P}$. The data obtained from the experiments were fitted with both the pseudo-first and pseudo-second order kinetic models, indicating that

both physical and chemical sorption mechanisms contributed to the removal of phosphate from river water. In summary, nZVI was proven to be an effective treatment method for phosphate-contaminated river water.

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Contribution

Vaidotas Danila: Investigation, Writing – original draft, Methodology. Tomas Januševičius: Supervision, Resources, Writing – review & editing.

Disclosure statement

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