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Original Article

Determination of Nitrogen and Phosphorus Removal Performance of Sand, Rice Husk and Zeolite Mixture

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ABSTRACT

Growing population, urbanization, and agricultural activities are causing an increase in nitrogen and phosphorus loads in water resources, leading to eutrophication and water quality issues. In this study, the nitrogen and phosphorus removal performance of a mixture of sand, rice husks, and zeolite—which could be used as an alternative filter material in constructed wetland systems—from synthetic wastewater was investigated using laboratory-scale column experiments. The filter material mixture, prepared in equal volumes, was placed in columns; synthetic wastewater containing nitrogen and phosphorus, prepared at different concentrations (50 to 100 mg L^{-1}) under laboratory conditions, was applied at different flow rates (1 and 2.3 mL min^{-1}). Throughout the experiments, samples were collected at different retention times (3, 6, 12, 24, and 48 hours), and analyses of pH, electrical conductivity (EC), total nitrogen (TN), and total phosphorus (TP) were performed. The obtained pH (8.07–9.52) and EC (470.4–1284 µS cm^{-1}) values were found to be compliant with relevant discharge standards. The highest phosphorus removal efficiency (99%) was achieved at a concentration of 50 mg L^{-1} and a flow rate of 1 mL min^{-1}, resulting in an effluent concentration of 0.50 mg L^{-1}, while the lowest removal efficiency (62%) was observed at a concentration of 100 mg L^{-1} and a flow rate of 2.3 mL min^{-1}, resulting in an effluent concentration of 38.0 mg L^{-1}. For nitrogen removal, the highest performance (90.1%) was achieved at a concentration of 100 mg L^{-1} and a flow rate of 2.3 mL min^{-1}, resulting in an effluent concentration of 9.99 mg L^{-1}, while the lowest removal performance (4%) was measured at a flow rate of 1 mL min^{-1}, resulting in an effluent concentration of 68.34 mg L^{-1}. In conclusion, the study demonstrated that the mixture of sand, rice husks, and zeolite provides high efficiency in nitrogen and phosphorus removal and can be used as a sustainable, low-cost, and environmentally friendly alternative filter material in constructed wetland systems. However, it was determined that increasing concentration and retention time did not produce the expected positive effect, particularly in phosphorus removal. Furthermore, it was found that low flow rates and appropriate pH conditions enhance adsorption efficiency, and it was assessed that pH optimization is necessary, particularly in wastewater with high pH values. It is believed that the findings obtained at the laboratory scale will make significant contributions to the applicability of natural filter materials for nutrient removal in constructed wetland technologies if supported by long-term and pilot-scale studies.

Key words: Adsorption, Column Tests, Treatment

Kum, Çeltik Kavuzu ve Zeolit Karışımının Azot ve Fosfor Giderim Performansının Belirlenmesi

ÖZ

Artan nüfus, kentleşme ve tarımsal faaliyetler, su kaynaklarında azot ve fosfor yüklerinin artmasına neden olmakta ve buna bağlı olarak ötrofikasyon ile su kalitesi problemlerini beraberinde getirmektedir. Bu çalışmada, yapay sulak alan sistemlerinde alternatif filtre materyali olarak kullanılabilecek kum, pirinç kabuğu ve zeolit

karışımının sentetik atık sulardan azot ve fosfor giderim performansı laboratuvar ölçekli kolon deneyleri ile araştırılmıştır. Eşit hacimlerde hazırlanan filtre materyali karışımı kolonlara yerleştirilmiş; laboratuvar koşullarında farklı konsantrasyonlarında (50 ile 100 mg L^{-1}) hazırlanan ve azot ile fosfor içeren sentetik atık sular, farklı debilerde (1 ve 2.3 mL dak^{-1}) uygulanmıştır. Denemeler boyunca farklı bekleme sürelerinde (3, 6, 12, 24 ve 48 saat) alınan numunelerde pH, elektriksel iletkenlik (EC), toplam azot (TN) ve toplam fosfor (TP) analizleri gerçekleştirilmiştir. Elde edilen pH (8.07–9.52) ve EC (470.4–1284 µS cm^{-1}) değerlerinin ilgili deşarj standartlarıyla uyumlu olduğu belirlenmiştir. Fosfor gideriminde en yüksek performans (%99), 50 mg L^{-1} konsantrasyon ve 1 mL min^{-1} debide 0.50 mg L^{-1} çıkış konsantrasyonu ile elde edilirken, en düşük giderim performansı (%62), 100 mg L^{-1} konsantrasyon ve 2.3 mL dak^{-1} debide 38.0 mg L^{-1} çıkış konsantrasyonu ile belirlenmiştir. Azot gideriminde ise en yüksek performans (%90.1), 100 mg L^{-1} konsantrasyon ve 2.3 mL dak^{-1} debide 9.99 mg L^{-1} çıkış konsantrasyonu ile elde edilirken, en düşük giderim performansı (%4), 1 mL dak^{-1} debi koşullarında 68.34 mg L^{-1} olarak ölçülmüştür. Sonuç olarak, kum, pirinç kabuğu ve zeolit karışımının azot ve fosfor gideriminde yüksek verimlilik sağladığını ve yapay sulak alan sistemlerinde sürdürülebilir, düşük maliyetli ve çevre dostu alternatif bir filtre materyali olarak kullanılabileceğini göstermiştir. Bununla birlikte, artan konsantrasyon ve bekleme süresinin özellikle fosfor gideriminde beklenen düzeyde olumlu bir etki oluşturmadığı belirlenmiştir. Ayrıca düşük debi ve uygun pH koşullarının adsorpsiyon verimini artırdığı tespit edilmiş olup, özellikle yüksek pH değerine sahip atık sularda pH optimizasyonunun gerekli olduğu değerlendirilmiştir. Laboratuvar ölçeğinde elde edilen bulguların, uzun süreli ve pilot ölçekli çalışmalarla desteklenmesi durumunda doğal filtre materyallerinin yapay sulak alan teknolojilerinde besin elementi giderimine yönelik uygulanabilirliğine önemli katkılar sağlayacağı düşünülmektedir.

Anahtar kelimeler: Adsorpsiyon, Arıtma, Kolon Denemeleri

INTRODUCTION

The world population is increasing every day. In addition, industrialization and water consumption are increasing worldwide. Consequently, waste in solid, liquid, and gaseous forms is reaching levels that disrupt the balance of life and threaten human existence.

The reckless use of human natural resources, particularly available water resources, increases water consumption and leads to water waste. Water resources are among the most essential resources necessary for sustaining live on earth. Water resources, which are essential for the life of every living being, are becoming increasingly polluted (Kılıç, 2008). Freshwater resources, which are extremely limited in the world, determine the future of both people and countries. Therefore, one of the most important fundamental issues is to have good organization in every area, from the protection of water resources to their operation, and from the distribution of water to its removal. In fact, the protection of water resources within a holistic structure should also be considered an indicator of the integrity of nature (Soylu et al., 2006). It is of great importance to protect water resources and pass them on to future generations. Domestic and industrial wastewater are the main factors causing pollution of water resources, which have many different applications. Owing to legal regulations aimed at protecting the environment and the increasing demand for water in businesses, minimizing wastewater generation, recovering valuable materials, and reusing water have become increasingly important (Murathan et al., 2013).

Türkiye, a country not rich in water resources, will increasingly feel this pressure due to its growing population. Especially in large cities, unplanned growth and increasing population density require water-related problems to be addressed not with short-term and superficial solutions, but with long-term and strategic ones (Aksungur and Firidin, 2008). Türkiye has a total area of approximately 78 million hectares. Of this area, 24 million hectares are cultivated for agriculture, 21.5 million hectares are pastures and meadows, 23 million hectares are forests and scrublands, and the remainder consists of unused water surfaces and rocky terrain. The total is 112 billion m^3 per year, consisting of an average of 94 billion m^3 of surface water and 18 billion m^3 of groundwater (DSI, 2025). More than 70% of the world's water is used for agricultural irrigation. Therefore, although it only covers a portion of irrigation water needs, there is great potential for the use of treated wastewater in irrigation (WWAP, 2003). Agricultural activities are one of the most significant sources of water pollution in Türkiye. Fertilizers and pesticides are used intensively in agricultural areas to increase production. The fertilizers and pesticides used are carried by rain or irrigation water, polluting surface waters and groundwater resources (Olhan and Ataseven, 2009). The use of treated wastewater in agriculture has many advantages. First, it contributes to increased production. This is because treated wastewater contains plant nutrients. The nutrient richness of treated wastewater has led to an increase in its reuse for agricultural purposes (Filibeli et al., 2017).

Many risk factors associated with the reuse of water in agricultural irrigation have been identified. Some risk factors have short-term effects, while the severity of the effects of other risk factors varies depending on the

potential for contact with animals, humans, or the environment. Other risk factors have effects that increase over time and with the continuous use of treated water (e.g. as soil salinity and the effects of toxic chemicals) (Toze, 2006). Dissolved nitrogen in water is one of the main parameters that cause pollution in surface and groundwater. Although nitrate is the most common form of dissolved nitrogen found underground, it can also be found in the form of ammonium, nitrite, nitrous oxide, and organic nitrogen. Bacteria found in aerobic environments convert ammonia into nitrite or nitrate ions (Akosman and Özdemir, 2018). Nitrogen fertilizers commonly used in agricultural applications, industrial waste, and waste from livestock operations increase the nitrogen content of soil, water, and produced goods, causing environmental pollution (Sünal and Erşahin, 2012).

In addition to high-cost advanced biological treatment plants for wastewater treatment, less expensive constructed wetlands are being built for use in rural areas. Constructed wetlands provide habitats for many organisms and have the capacity to utilize solar energy and self-renew. They contribute to the natural balance of the atmosphere by consuming CO_2 and producing O_2 in the environment. They can remove organic matter, suspended solids, toxic substances, heavy metals, and biological elements from wastewater and have high treatment capacities (Bütünoğlu, 2018). Constructed wetlands are systems that enable the regular control of water flow, retention time, and wastewater level by placing porous materials such as stones, gravel, and sand within an impermeable clay layer (Esmeray and Armutcu, 2020). Constructed wetlands perform physical, chemical, and biological treatments through interactions between the fill material, plants, and air space, aided by microorganisms present in the soil structure (Dutta and Mia, 2010).

Constructed wetlands are constructed by filling impermeable ground with bedding materials such as sand or gravel that can support rooted plants. In these systems, water does not come into contact with the atmosphere because it flows through the filling materials (Demirörs, 2006). In constructed wetlands, natural materials such as soil, clay, and sand, processed materials such as zeolite or pumice, and waste materials such as fly ash or blast furnace slag can be used as filter materials. Depending on the basic pollutant parameters and pollutant concentrations in wastewater, these materials are often used in combination with each other (Gökalp and Taş, 2018).

The addition of zeolite to water treatment systems increases sludge activity while also improving the removal efficiency of suspended solid particles and the phosphate precipitation effect of trivalent cations. Zeolites are highly absorbent porous minerals composed primarily of silicon and aluminum. Similar to sponges that absorb water, zeolites are useful for their ability to capture and retain various unwanted materials (Miteva and Stoyanova, 2020). With their high ion exchange and heavy metal removal efficiencies, these materials have been used to improve the physical, chemical, and biological properties of soil and are evaluated for fertilization applications. Thus, zeolite enables the controlled release of nutrients into the soil. Furthermore, the production of zeolite-based fertilizers results in lower carbon dioxide emissions into the environment (Jarosz et al., 2022).

Rice constitutes half of the food supply for 1.6 billion people worldwide. Rice production results in excessive amounts of rice husks as waste material (Pourshakiba, 2012). Rice is obtained by processing paddy. During the separation of the husks, which are a waste product of rice production, from the grains, two types of husks are formed. The first husk is a thin membrane surrounding the rice grain, known as the bran. It is rich in nutrients and is used as animal feed. The second husk is the outermost shell of rice grain. This husk is also called the hull or husk (Sadıç, 2008). The composition of rice husks contains up to 46.9% organic carbon. The nitrogen content is 1.6% and the moisture content is 9.3% (Çakirer, 2015).

Fertilizers derived from industrial sources are commonly used to address phosphorus deficiency in soil. While plants consume the phosphorus applied during the year, the remainder accumulates in the soil as “residual phosphorus.” Only a small portion of this residual phosphorus dissolves in water as phosphate, while an estimated 80% reaches freshwater sources and the ocean in granular form. Industrial and domestic waste contains high levels of phosphorus (Kılıç and Korkmaz, 2012). All organisms involved in the biological processes of wastewater treatment require phosphorus for reproduction and the synthesis of new cells. In this regard, domestic wastewater contains much more phosphorus than the amount necessary for the stabilization of the organic matter it contains. This situation can be understood by looking at the amount of phosphorus in the effluent of biological wastewater treatment plants. In Türkiye, according to the urban wastewater treatment regulation, at least 80% of total phosphorus must be removed in discharges to sensitive water bodies with low water circulation (Kaya, 2013).

Ammonia-based fertilizers are used to meet the nitrogen requirements of plants. However, the use of these fertilizers has caused environmental problems has caused. While some of the ammonia applied as fertilizer is used by plants, part of it is used by bacteria in the soil for cellular respiration, resulting in the release of nitrite. Nitrite, which is oxidized by other microorganisms, is converted into nitrate and, owing to its chemical properties, can easily mix with groundwater. Groundwater carries nitrates to sea and oceans, causing water pollution (Serin, 2014).

Recent studies have focused on various filter materials such as peat soil, sand, gravel, blast furnace slag, coal furnace slag, limestone, rice husk, pumice, zeolite, and vermiculite for nitrogen and phosphorus removal from wastewater (Gökalp and Kanarya, 2018; Uzun et al., 2021; Atıcı, 2023; Aşık and İrik, 2025; Demir et al., 2025; İrik and Kanarya, 2025).

In previous studies, the removal performance of various wastes has generally been investigated using the materials in their pure forms. This study was conducted to determine the performance of a homogeneous mixture of sand, rice husk, and zeolite materials in removing nitrogen and phosphorus from wastewater.

MATERIALS AND METHODS

The study was contained in the form of column tests, in which filter materials were subjected to artificially generated wastewater. The filter materials consisted of sand, rice husk, and zeolite (50 cm^3 each), mixed to form a total of 150 cm^3 of material. Before being transferred to the columns, the materials used in the study were sieved through screens with pore diameters of 1 and 2 mm, and the materials retained between the two screens were used.

The columns contain pores with a pore diameter of 1 mm to prevent clogging caused by the filter material. This study was conducted on a pilot scale in the form of column tests representing constructed wetlands to avoid harming the environment.

Sand is a granular material composed of fragmented rock and mineral particles. It is finer than gravel and coarser than silt. Sand can also refer to a textural soil or soil type. That is, it is a soil containing more than 85% sand-sized particles by mass. The composition of sand varies depending on rock sources and conditions. In inland environments and non-tropical coastal environments, the most common component of sand is silica (SiO_2), usually in the form of quartz (Apaydın, 2007).

Rice husks are bio-waste materials used as adsorbents in studies. While rice husks exhibit good and economical adsorbent properties, they pose significant environmental problems in the management and disposal of agricultural waste (Kalderis et al., 2008; Asadi et al., 2008). Rice husks are insoluble in water, have a silica-cellulose structure, are woody, abrasive, and contain 20% silica material. The important substances in their structure are cellulose, hemicellulose, lignin, hydrated silica, and ash. The chemical components of rice husks are SiO_2, H_2O, Al_2O_3, Fe_2O_3, K_2O, Na_2O, CaO, and Mg (Foo and Hameed, 2009).

Zeolite is a hydrated aluminum silicate with a crystalline structure originating from volcanic rock, which has micro-pores and can be widely used as an adsorbent and catalyst. There are 42 different types of zeolites according to their compositions (Altan et al., 1998). Zeolites are used in agriculture as soil conditioners, fertilizer additives, water retainers, and for aeration purposes; in plant production as a growing medium; in landscaping and garden design; in animal husbandry as feed additives and bedding material; in purification for mechanical and chemical filtration; in the retention of toxic waste; as filter material in pools and spas, as aquarium material, cat litter, cleaning materials, in the textile industry, paper and energy sectors, as toxin binders in the health sector, as catalysts in aquaculture and chemistry, as well as in waste and wastewater treatment (Çağın et al., 2006).

Columns made of glass material with a standard inner diameter of 3 cm and a length of 60 cm were used in this study. A LongerPump brand 12-channel peristaltic pump was used in the experiment to ensure that a specified amount of liquid was transferred into the columns within a specified time, and the apparatus used in the experiment is shown in Figure 1. Nine columns were used at a time in the experiments, and each study was conducted with three replicates.

In the study, synthetic wastewater was prepared using potassium nitrate (KNO_3) and potassium dihydrogen phosphate (KH_2PO_4) at a concentration of 1000 mg L^{-1} as a stock solution. Synthetic wastewater was prepared at concentrations of 50 and 100 mg L^{-1} using the prepared stock solution. The synthetic wastewater prepared under laboratory conditions using a peristaltic pump was applied at flow rates of 1 and 2.3 mL min^{-1}.

In order to better determine the effectiveness of the materials used, retention was performed at 3, 6, 12, 24, and 48 hours after the system was started. After measuring the pH and EC of the samples taken at different times, they were filtered through filter paper with a pore size of 0.45 µm to determine the total phosphorus and nitrate content.

Samples collected throughout the study were filtered through filter paper. pH and EC analyses were measured using a Hanna brand HI-5522 model pH and EC meter. Phosphorus content was determined using a Hanna brand phosphorus analyzer. Ten milliliters of each sample was taken, 10 drops of Hanna brand HI93717A-0 model reagent were added, and one packet of Hanna brand HI93717B-0 model powder was added, and a reading was taken from the device. The phosphorus content was determined based on the findings. For nitrate determination, 10 ml of the samples were taken, 0.2 ml ISA (Ionic Strength Adjuster) was added to them, and

they were mixed. Nitrate analysis was performed using the Hanna HI-4113 ion probe on the Hanna HI-5522 ion meter device. The findings obtained were divided by 4.43 to determine the nitrogen content in the nitrate.

Figure 1. The system used in the study

Using the findings obtained in the study, the nitrogen and phosphorus removal efficiencies of the materials were determined as percentages. At this stage, Equation 1 was used.

$$RP = \frac{C_{in}-C_{out}}{C_{in}} x100 \quad (1)$$

Where; RP: Removal Performance (%); C_{in}: Concentration of influent (mg L^{-1}); Cout: Concentration of effluent (mg L^{-1}).

RESULTS AND DISCUSSION

Change in pH Value During the Treatment Process

The pH values of solutions prepared at different concentrations (50 and 100 mg L^{-1}) were determined to be 5.03 and 4.99. As a result of the study, the pH values of the effluent water varied between 8.07 and 9.52 depending on the different concentrations loaded with phosphorus and nitrogen mixtures and different retention times. The Water Pollution Control Regulation published by the Ministry of Environment, Urbanization, and Climate Change states that pH values must be between 6 and 10 for wastewater to be discharged into receiving environments (SKKY, 2004). According to the findings, it was determined that this criterion was met (Table 1, Figure 2).

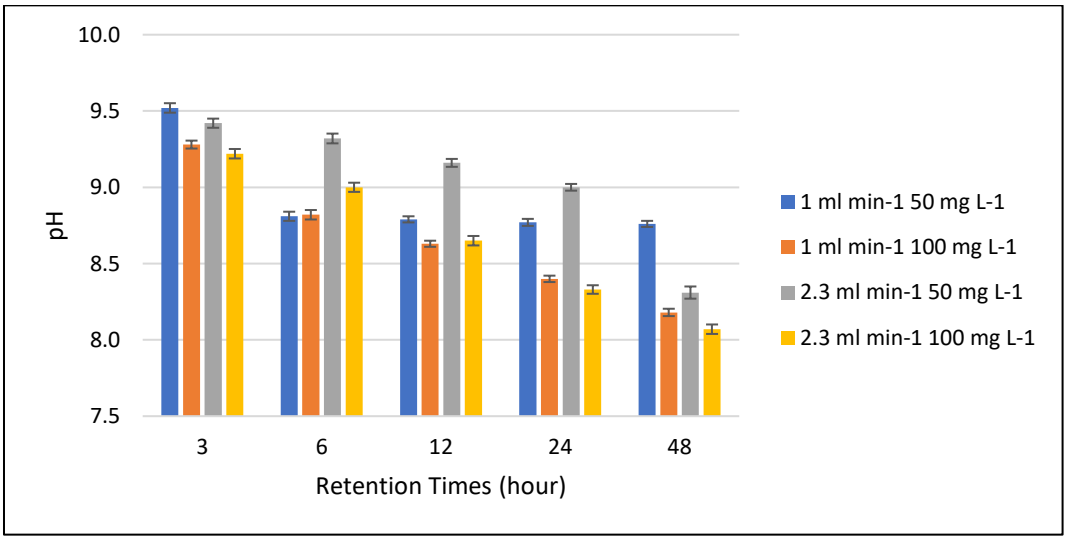

Figure 2. pH changes during study

As shown in Figure 2, the effect of nitrogen and phosphorus-containing solutions applied at different concentrations on pH values varied depending on the retention times. These changes increased initially because the pH values of the materials were higher after contact with the prepared solutions and then tended to

decrease. During the study, decreases and increases in pH values were observed due to the material was washed. The pH data obtained at different flow rates and after different retention times are presented in Table 1.

Table 1. pH changes during study

Flow Rate (mL min^{-1})	Conc. (mg L^{-1})	Retention Times (hour)				
		3rd	6th	12th	24th	48th
1	50	9.52±0.031	8.81±0.03	8.79±0.02	8.77±0.023	8.76±0.02
	100	9.28±0.026	8.82±0.031	8.63±0.02	8.4±0.021	8.18±0.024
2.3	50	9.42±0.03	9.32±0.032	9.16±0.026	9.00±0.22	8.31±0.04
	100	9.22±0.031	9.00±0.03	8.65±0.031	8.33±0.028	8.07±0.031

During the study, pH values ranged from 9.22 to 9.52 at the end of the 3rd hour, 8.81 to 9.32 at the end of the 6th hour, 8.63 to 9.16 at the end of the 12th hour, 8.33 to 9.00 at the end of the 24th hour, and 8.07 to 8.76 at the end of the 48th hour. The lowest pH value was observed in the sample taken at 48 hours from the material treated with a solution at a flow rate of 2.3 mL min^{-1} and a concentration of 100 mg L^{-1}, while the highest pH value was observed in the sample taken at 3 hours from the material treated with a solution at a flow rate of 1 mL min^{-1} and a concentration of 50 mg L^{-1}. The pH analysis results obtained in the study show that the values decrease with increasing concentration and time. The pH values complied with the criteria of the relevant regulation for the discharge of treated wastewater into receiving environments.

Demir et al. (2025), in their column test study, used zeolite, vermiculite, and mixtures of both materials in different ratios as filter materials and did not observe a standard change in pH values. Atıcı (2023) used zeolite, sawdust, and mixtures of both materials in different ratios as filter materials in column tests. It was observed that pH values decreased depending on the concentration of the applied solution and did not show significant change over time.

Change in EC (µS cm^{-1}) Value During the Treatment Process

The EC values of the solutions prepared at different concentrations (50 and 100 mg L^{-1}) were determined to be 436.89 and 1016.42. The analyses conducted in the study revealed that EC values ranged between 470.4 and 1300 µS cm^{-1}. The relevant ministry stated in its announcement that the ideal EC value should be below 2000 µS cm^{-1} (SKKY, 2004). The findings indicate that this criterion was met (Table 2, Figure 3).

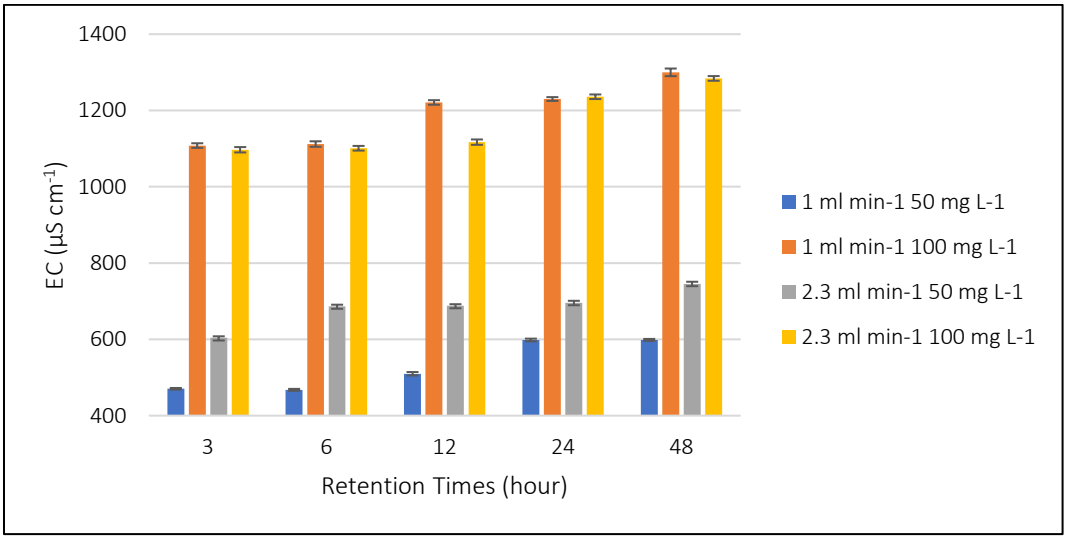

Figure 3. EC (µS cm^{-1}) changes during study

As can be seen from Figure 3, it was determined that the effect of nitrogen and phosphorus-containing solutions applied at different concentrations on EC values varied depending on the retention times. These changes increased because the EC values of the materials were higher after the prepared solutions encountered the materials. The EC data obtained at different flow rates and after different retention times are presented in Table 2.

Table 2. EC (µS cm^{-1}) changes during study

Flow Rate (mL min^{-1})	Conc. (mg L^{-1})	Retention Times (hour)				
		3rd	6th	12th	24th	48th
1	50	470.4±2.12	467.6±2.55	509.8±4.51	598.4±3.55	598.6±2.52
	100	1108±6	1112±7.21	1221±6	1230±5	1300±10
2.3	50	604±4	686.3±4.59	688.5±3.61	695.5±5.57	745.5±5.51
	100	1097±7	1101±6.11	1117±7.02	1236±6	1284±6.11

During the study, EC values (µS cm^{-1}) ranged from 470.40 to 1108.00 at the end of the 3rd hour, from 497.60 to 1112. 00 at the end of the 6th hour, between 509.80 and 1221.00 at the end of the 12th hour, between 598.40 and 1236.00 at the end of the 24th hour, and between 598.60 and 1300.00 at the end of the 48th hour. The lowest EC value was observed in the sample taken at the 3rd hour of the material treated with a solution at a concentration of 50 mg L^{-1} at a flow rate of 1 mL min^{-1}, while the highest EC value was observed in the sample taken at the 48th hour of the material treated with a solution at a concentration of 100 mg L^{-1} at a flow rate of 1 mL min^{-1}. The EC analysis results obtained in the study show that the values increase over time. The EC values were found to comply with the criteria of the relevant regulation for the discharge of treated wastewater into receiving environments.

In their column tests, Demir et al. (2025), determined that different materials did not affect EC levels in pollution but had an effect on the retention process and observed that EC results increased over time depending on the retention period. Atıcı (2023), in column tests, evaluated the EC analysis results and found that the values were proportionally close to each other with the change in materials and that the results obtained were in compliance with the regulation. The data we have obtained within the discharge limits specified in the Water Pollution Control Regulation published by the Ministry of Environment, Urbanization, and Climate Change does not indicate any problems with wastewater discharge.

Change in Total Phosphorus (mg L^{-1}) Value During the Treatment Process

After applying solutions prepared at different concentrations (50 and 100 mg L^{-1}) to the columns, the analyses revealed that the total phosphorus values ranged between 0.50 and 38.0 mg L^{-1}. The relevant ministry's published regulation states that the TP value of treated wastewater must be below 10 mg L^{-1} (SKKY, 2004). According to the findings, it was determined that this criterion was mostly met (Table 3, Figure 4).

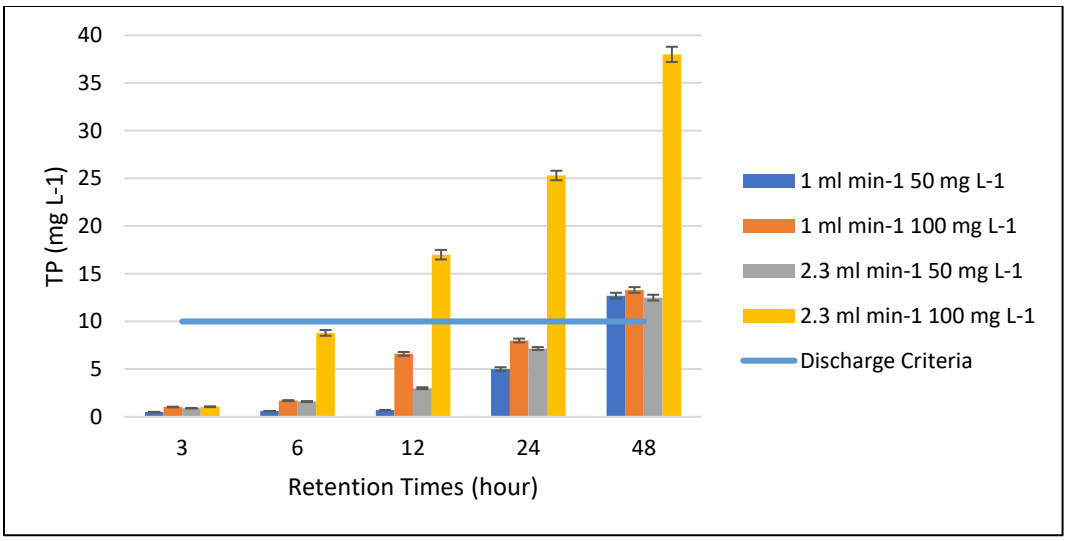

Figure 4. Changes in TP (mg L^{-1}) during the study

As can be seen in Figure 4, it was determined that phosphorus-containing solutions applied at different concentrations varied according to their retention times. After the prepared solutions met the material, the TP values increased due to the materials reaching saturation over time. The TP values obtained at different flow rates and after different retention times are given in Table 3.

Table 3. Changes in TP (mg L^{-1}) during the study

Flow Rate (mL min^{-1})	Conc. (mg L^{-1})	Retention Times (hour)				
		3rd	6th	12th	24th	48th
1	50	0.5±0.02	0.6±0.02	0.7±0.02	5±0.2	12.7±0.305
	100	1.03±0.031	1.7±0.05	6.6±0.2	8±0.2	13.3±0.3
2.3	50	0.9±0.031	1.6±0.05	3±0.1	7.16±0.153	12.5±0.3
	100	1.06±0.04	8.8±0.3	17±0.5	25.3±0.5	38±0.8

During the study, TP values (mg L^{-1}) ranged from 0.50 to 1.06 at the end of the 3rd hour, from 0.60 to 8.80 at the end of the 6th hour, from 0.70 to 17.00 at the end of the 12th hour, 5.00 to 25.30 at the end of the 24th hour, and 12.50 to 38.00 at the end of the 48th hour. The lowest TP value was observed in the sample taken at 3 hours from the material treated with a solution at a concentration of 50 mg L^{-1} at a flow rate of 1 mL min^{-1}, while the highest TP value was observed in the sample taken at 48 hours from the material treated with a solution at a concentration of 100 mg L^{-1} at a flow rate of 2.3 mL min^{-1}. The TP analysis results obtained in the study show that the values increase over time. It was observed that TP values complied with the relevant regulation criteria for the discharge of treated wastewater into receiving environments up to 24 hours at a flow rate of 1 mL min^{-1}, 12 hours at a flow rate of 2.3 mL min^{-1} with a concentration of 50 mg L^{-1}, and 6 hours with a concentration of 100 mg L^{-1}. As the flow rate, the concentration of the applied solution, and the retention time increased, the limit was approached, and it was determined that the criterion could not be met thereafter. The total phosphorus change obtained as a result of the study was similar to the results obtained by Uzun et al. (2021) and Demir et al. (2025).

Change in Total Nitrogen (mg L^{-1}) Value During the Treatment Process

Following the application of solutions prepared at different concentrations (50 and 100 mg L^{-1}) to the columns, analyses revealed that total nitrogen values ranged between 9.99 and 68.34 mg L^{-1}. The relevant ministry's published regulation states that the TN value of treated wastewater must be below 40 mg L^{-1} (SKKY, 2004). According to the findings, it was determined that this criterion was mostly met (Table 4, Figure 5).

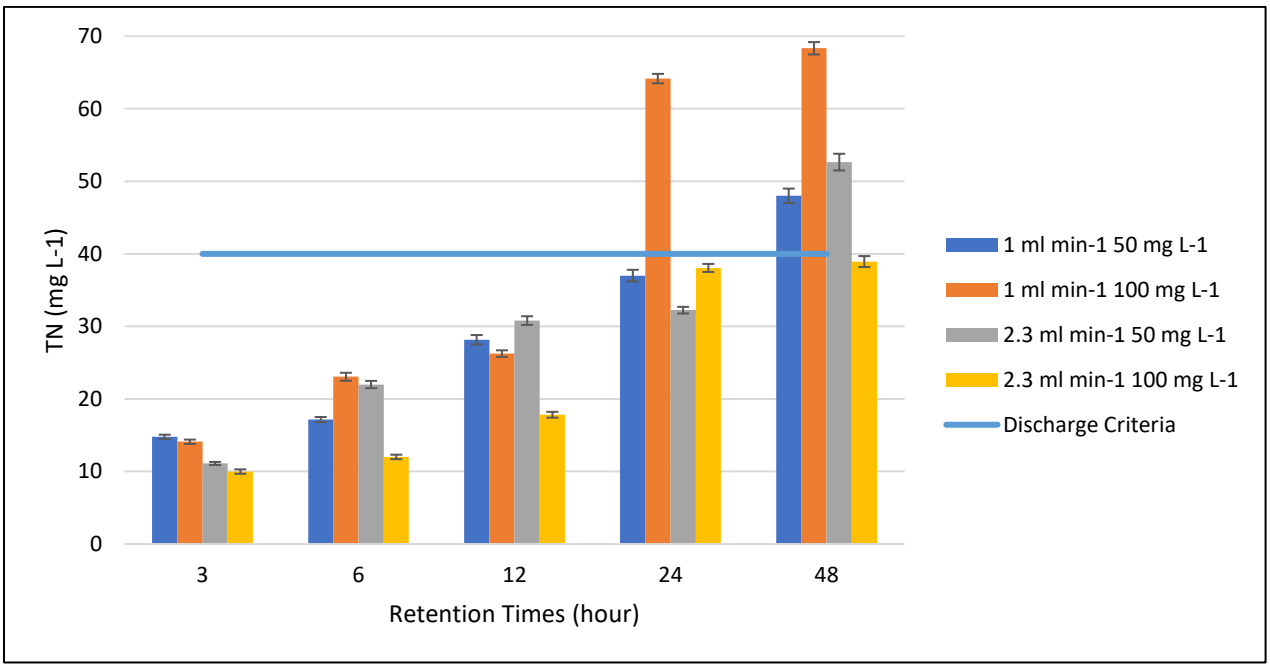

Figure 5. Changes in TN (mg L^{-1}) during the study

As can be seen in Figure 5, it was determined that nitrogen-containing solutions applied at different concentrations varied according to their retention times. After the prepared solutions met with the material, the values increased due to the materials reaching saturation over time. The TN values obtained at different flow rates and after different retention times are given in Table 4.

Table 4. Changes in TN (mg L^{-1}) during the study

Flow Rate (mL min^{-1})	Conc. (mg L^{-1})	Retention Times (hour)				
		3rd	6th	12th	24th	48th
1	50	14,77±0,306	17,16±0,351	28,16±0,651	37±0,8	48±1
	100	14,1±0,3	23,06±0,551	26,25±0,451	64,16±0,651	68,34±0,854
2.3	50	11,11±0,208	21,99±0,503	30,8±0,6	32,24±0,458	52,65±1,153
	100	9,99±0,306	12,01±0,306	17,82±0,404	38,06±0,551	38,94±0,755

During the study, TN values (mg L^{-1}) were 9.99 to 14.77 at the end of the 3rd hour, 12.01 to 23.06 at the end of the 6th hour, 17.82 to 30.80 at the end of the 12th hour, 32.24 to 64.16 at the end of the 24th hour, and 38.94 to 68.34 at the end of the 48th hour. The lowest TN value was observed in the sample taken at the 3rd hour of the material treated with a solution at a flow rate of 2.3 mL min^{-1} and a concentration of 100 mg L^{-1}, while the highest TN value was observed in the sample taken at the 48th hour of the material treated with a solution at a flow rate of 1 mL min^{-1} and a concentration of 100 mg L^{-1}. The TN analysis results obtained in the study show that the values increase over time. For treated wastewater to be discharged into receiving environments, TN values must comply with the criteria of the relevant regulation at a flow rate of 1 mL min^{-1} at a concentration of 50 mg L^{-1} up to 24 hours and at a concentration of 100 mg L^{-1} up to 12 hours. At a flow rate of 2.3 mL min^{-1}, compliance with the relevant regulation criteria was observed at the end of the 24th hour. As the flow rate, the concentration of the applied solution, and the retention time increased, the limit was approached, and it was determined that the criterion could not be met thereafter. The total nitrogen change obtained as a result of the study was found to be similar to the results obtained by Aşık and İrik (2025) and Demir et al. (2025).

CONCLUSION

The use of soil or filter materials is recommended for nitrogen and phosphorus removal in natural treatment systems (Mann, 1990). Therefore, this study was conducted in the form of column tests in the laboratories of the Department of Biosystems Engineering, Faculty of Agriculture, Erciyes University, to investigate the performance of different filter materials in removing nitrogen and phosphorus pollution. Using a homogeneous mixture of sand, rice husk, and zeolite materials, samples were taken after 3, 6, 12, 24, and 48 hours of retention time with synthetic wastewater containing nitrogen and phosphorus produced under laboratory conditions at two different concentrations (50 and 100 mg L^{-1}). The solutions prepared at concentrations of 50 and 100 mg L^{-1} were applied to the filter materials at flow rates of 1 and 2.3 mL min^{-1} for 48 hours, and pH, EC, total nitrogen, and total phosphorus analyses were performed on a total of 135 samples. The obtained pH (8.07–9.52) and EC (470.4–1284 µS cm^{-1}) values were determined to meet the relevant discharge standards.

The highest phosphorus removal performance (99%) was achieved at a concentration of 50 mg L^{-1}, a flow rate of 1 mL min^{-1}, and 0.50 mg L^{-1} at 3 hours. The lowest phosphorus removal performance (62%) was detected at a concentration of 100 mg L^{-1}, a flow rate of 2.3 mL min^{-1}, and 38.0 mg L^{-1} at 48 hours. The highest performance in nitrogen removal performance (90.1%) was achieved at a concentration of 100 mg L^{-1}, a flow rate of 2.3 mL min^{-1}, and 9.99 mg L^{-1} at 3 hours; the lowest removal performance (4%) was measured at a flow rate of 1 mL min^{-1}, a concentration of 100 mg L^{-1}, and 68.34 mg L^{-1} at 48 hours.

The results show that the mixture of sand, rice husk, and zeolite had high nitrogen and phosphorus removal efficiencies and can therefore be used as an alternative filter material in constructed wetlands. Since the study was conducted on a laboratory scale, it is thought that applications conducted over longer periods and at different concentrations will reveal the material's performance in more detail. It has been determined that applying wastewater at low pH and low flow rates increases adsorption efficiency, and pH adjustment is recommended, especially for wastewater with high pH values. Furthermore, using natural filter materials mixed in appropriate ratios will increase phosphorus and nitrogen removal efficiency in constructed wetlands.

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Declaration of interests

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Author Contributions

***Kübra BERKKAN*:** Data curation; formal analysis
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