



Research article

Adsorption and desorption of ammonium from treated wastewater by zeolite filled columns: An experimental study at the water resource recovery facility of Palermo University – Italy

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ABSTRACT

Water scarcity and mineral fertilizer depletion are becoming recognised environmental challenges worldwide. Treated wastewater (TWW) could be a potential resource for reusing water and nutrients, such as nitrogen (N), for fertilizers. This study explores the possibility of adopting columns filled with zeolite to recover ammonium (NH_4^+) from real TWW. Specifically, this study aimed to evaluate zeolite's adsorption capacity with different particle sizes arranged in columns and various flow rates to determine the most efficient way of NH_4^+ adsorption from a real wastewater treatment plant's effluent. The same zeolite with two different size diameters (0.5–1.0 mm and 2.0–5.0 mm) was tested using three different flow rates (1.2, 1.6 and 2.4 L h^{-1}) to evaluate their NH_4^+ adsorption capacity. After the adsorption test, a desorption trial assessed the zeolite's desorption ability. The results showed that the highest flow rate increased the adsorption capacity of both zeolites by about 29% more than the lowest flow rate. Moreover, the 0.5–1.0 mm zeolite adsorbed approximately 60 mg more NH_4^+ than the 2.0–5.0 mm zeolite, highlighting the influence of particle size on adsorption capacity. Furthermore, the zeolite was characterised by a rapid NH_4^+ release since 44–78% of the adsorbed NH_4^+ was released in the first 30 min. The desorption test with the lowest flow rate achieved the highest amount of desorbed NH_4^+ , up to 123–148% more than the higher flow rates. Results have shown that due to its adsorption capacity, zeolite can be used to recover NH_4^+ from treated wastewater (TWW) and potentially recycle resources in the agriculture field, contributing to the circular economy.

1. Introduction

In recent years, population growth has required increased agricultural productivity to meet the growing demand for food (Beltran-Peña et al., 2020). In response to this problem, farmers have increasingly turned to intensive application of fertilizers, with nitrogen (N) as one of the principal constituents (Penuelas et al., 2023). Indeed, this element is classified as a macronutrient for plants due to its uptake in large quantities and plays different roles, such as supporting the rapid growth of the plant body, improving leaf quality and size, and promoting seed and fruit development (Leghari et al., 2016). Additionally, N is essential in forming amino acids and is involved in numerous biochemical processes as a catalytic agent (Adnan et al., 2021). Furthermore, fertilizer

production is currently using a large amount of energy. Indeed, the N fertilizer production process, the Haber–Bosch method, consumes more than 1% of the global energy supply and emits significant greenhouse gases (Du et al., 2023). A significant percentage of N introduced into crops as fertilizer is leached, subsequently contributing to the eutrophication of various aquatic environments (Ding et al., 2022). This phenomenon enriches groundwater, rivers, lakes, and coastal and marine ecosystems with nutrients that alter the ecological balance and cause disturbances in the affected areas (Huang et al., 2017).

A promising solution for these issues is recovering these essential elements from treated wastewater (TWW), characterized by its elevated nutrient content (Angin et al., 2005; Al-Suhaibani et al., 2021), particularly in N (Pérez-Legaspi et al., 2023). Wastewater treatment has

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recently gained significant attention in the European Union, supported by the recently proposed Urban Wastewater Treatment Directive, which aligns with the circular economy approach. This initiative encourages the production of high-quality wastewater suitable for agricultural reuse, while introducing stricter limits on N and P concentrations in effluent from wastewater treatment plants (WWTPs) (Mannina et al., 2022; Pérez-Legaspi et al., 2023). As a result, wastewater treatment plants must be revamped to incorporate advanced water and nutrient recovery technologies, ensuring conformity to these more stringent standards and promoting sustainable resource management (Angin et al., 2005; Al-Suhaibani et al., 2021). Chemical, biological, and physical techniques can recover N as ammonium (NH_4^+) from TWW (Ye et al., 2018; Zhang and Liu, 2021; De Magalhães et al., 2022). Physical adsorption-based processes stand out among these methods since they are based on low-cost adsorbent materials that are environmentally sustainable and recyclable (Sengupta et al., 2015), such as zeolite (Kithome et al., 1999; Demir et al., 2002; Ivanova et al., 2010; Muscarella et al., 2023b). Zeolite is a highly porous aluminosilicate mineral, and it possesses a high cation adsorption capacity attributed to the isomorphic substitution of silicon ions (Si^{4+}) with aluminium ions (Al^{3+}) (Hedström, 2001; Dyer, 2007), resulting in a net negative charge. These negative charges are balanced by various cations, exhibiting a strong affinity for NH_4^+ (Cruz et al., 2018; Tatlier et al., 2018; Muscarella et al., 2021). For example, Khosravi et al. (2014) carried out a batch test to demonstrate the NH_4^+ adsorption capacity of zeolite. To do this, they used Western Azerbaijan zeolite to recover NH_4^+ from a monocomponent solution with different concentrations of NH_4^+ ($10\text{--}200\text{ mg L}^{-1}$). The results showed a maximum adsorption of 43.5 mg g^{-1} . Du et al. (2005) conducted a batch test in which zeolite was placed in contact with an NH_4^+ -rich solution for 4 h. They observed that zeolite adsorbed the highest amount of NH_4^+ in the first 15 min of contact with NH_4^+ -rich solution. Moreover, NH_4^+ -enriched zeolites could be employed in agriculture to enhance soil physical and chemical properties and serve as a source of N for the absorption of this element by plants within a circular economy framework (Cosenza et al., 2022; Mannina et al., 2022; Muscarella et al., 2023a; Paliaga et al., 2024a, 2024b). In addition, zeolite can be regenerated to recycle the used material. Zeolite regeneration is an essential process to restore the ion exchange and adsorption capacity of zeolite, making it effective again for applications such as removing pollutants from water or for nutrient absorption (Wang et al., 2006; Zhou et al., 2021; Chen et al., 2023). Until now, studies conducted for the recovery of ammonium through zeolite have been carried out at a laboratory scale or in pilot plants, often using synthetic effluent. This study aims to provide information on ammonium recovery from real TWW by zeolite-filled columns cost-effectively and efficiently through a sustainable physical method. This focus is significant because ammonium recovery from wastewater could reduce pollution and recycle resources in agriculture. The TWW adopted in this work was collected from the domestic wastewater treatment line of the Water Resource Recovery Facility at the University of Palermo (Mannina et al., 2022). Columns filled with zeolite were applied to recover NH_4^+ in the TWW. The adsorption capacity of zeolite was determined as a function of the particle size of the material (0.5–1.0 mm and 2.0–5.0 mm) and the flow rate of the column adopted (1.2 L h^{-1} , 1.6 L h^{-1} and 2.4 L h^{-1}). A desorption test was carried out to validate the material's recycling potential.

2. Materials and methods

2.1. Adsorption test

The experiments were conducted at the Water Resource Recovery Facility (WRRF) of Palermo University (Italy) built within the Wider Uptake H2020 project (Mannina et al., 2021a, 2021b). The polymethylmethacrylate columns used in the experiment had an internal diameter of 5 cm and a length of 20 cm. The two diameters of the zeolite

used were 2.0–5.0 mm and 0.5–1.0 mm, respectively, coming from the same raw material (ZEOWATER ZN, Zeocel Italia). Zeolite tetrahedral structure consists of tetrahedral units of SiO_4^{4-} and AlO_4^{5-} . It is constituted by 85% clinoptilolite, 8% cristobalite, 4% illite, 3–4% plagioclase and a Si/Al ratio of 4.8–5.5 and has a bulk density of 0.98 g cm^{-3} , the surface area of $40\text{ m}^2\text{ g}^{-1}$, pH of 7.6. K^+ and Ca^{2+} were the predominant exchangeable cations. Three different flow rates were considered to evaluate three water retention times with zeolite inside the column. The assessed values were 1.2, 1.6 and 2.4 L h^{-1} , corresponding to retention times of 20 min, 15 min, and 10 min, respectively. Three columns (C1, C2 and C3) were filled with 0.5–1.0 mm particle size zeolite, around 225 g per column, and three columns (C4, C5 and C6) with 2.0–5.0 mm zeolite, around 239 g per column. The six columns were connected to a pump (Watson Marlow Qdos 30) with a specific flow rate that pumped the TWW from the effluent of the pilot wastewater treatment plant (Mannina et al., 2021a; Mofatto et al., 2024). The characteristics of TWW are reported in Table 1. The TWW leaving the plant was accumulated in 3 tanks of 1 m^3 each to maintain a constant NH_4^+ concentration at the inlet. The adsorption tests were conducted for 11 days until the accumulated water in the tanks was exhausted and the maximum absorption capacity of the zeolite was achieved. The experimental design is reported in Fig. 1.

2.2. Analytical methods

The sampling of the inlet and outlet of water was taken after 1, 3, 6, and 9 h in the first 24 h and subsequently every 24 h until the eleventh day. One sample was taken at the inlet of the columns to verify that the NH_4^+ concentration remained constant, and one sample at the outlet for each column. Once collected, the samples were filtered with $0.45\text{ }\mu\text{m}$ syringe filters and stored in the refrigerator at $4\text{ }^\circ\text{C}$. The samples were analysed using an ion chromatograph (Thermo Scientific Dionex Easion) equipped with CS12 and AS12 IC columns to determine the concentration of cations (Na^+ , K^+ , Ca^{2+} , Mg^{2+} , NH_4^+) and anions (NO_2^- , NO_3^- , F^- , and Cl^-).

2.3. Desorption test

After the adsorption test, a desorption test was conducted on the NH_4^+ -enriched material. The test aimed to recover the ammonia adsorbed by the zeolite to regenerate the material. The same experimental design was used for the desorption test (Fig. 2). Instead of TWW, a 1 M NaCl solution was used, and the test was carried out for 24 h. The samples were analysed using the Berthelot method (Mulvaney, 1996) and a spectrophotometer (UVmini-1240, Shimadzu Italia srl, Milan, Italy) to determine extractable NH_4^+ ions.

3. Results

3.1. Influence of flow rate and particle size on ammonium adsorption

Considering the columns with a particle diameter of 0.5–1.0 mm, column 3 (C3) showed the highest NH_4^+ adsorption capacity, as shown in Fig. 3. Adopting a flow rate of 2.4 L h^{-1} , it adsorbed 2078 mg of NH_4^+ for the total grams in the column, 632 mg more than C2, working with a flow rate of 1.6 L h^{-1} . C2 adsorbed 1446 mg of NH_4^+ , slightly lower than C1, which had a flow rate of 1.2 L h^{-1} and adsorbed 1612 mg of NH_4^+ , 416 mg less than C3. A similar trend was observed for higher particle diameter columns (2.0–5.0 mm), C4–C6. The highest flow rate (2.4 L h^{-1}) adopted in C6 resulted in the highest amount of NH_4^+ adsorbed, namely 2010 mg. Following C6, C5 (flow rate of 1.6 L h^{-1}) adsorbed 378 mg less than C6, while C4, adopting a flow rate of 1.2 L h^{-1} , adsorbed 407 mg of NH_4^+ less than C6.

Comparing the columns with the same flow rate, the lowest zeolite particle diameters slightly increased the amount of NH_4^+ adsorbed. C3 adsorbed 68 mg more than the corresponding C6, while C1 adsorbed

Table 1
Characteristics of TWW used in the column study.

U.M.	NH ₄ ⁺ -N	Ca ²⁺	Mg ²⁺	K ⁺	Na ⁺	F ⁻	Cl ⁻	NO ₂ ⁻ -N	NO ₃ ⁻ -N	PO ₄ ³⁻	pH
	mg L ⁻¹										
	22	31	59	18	125	0.4	189	0.1	2	2	7

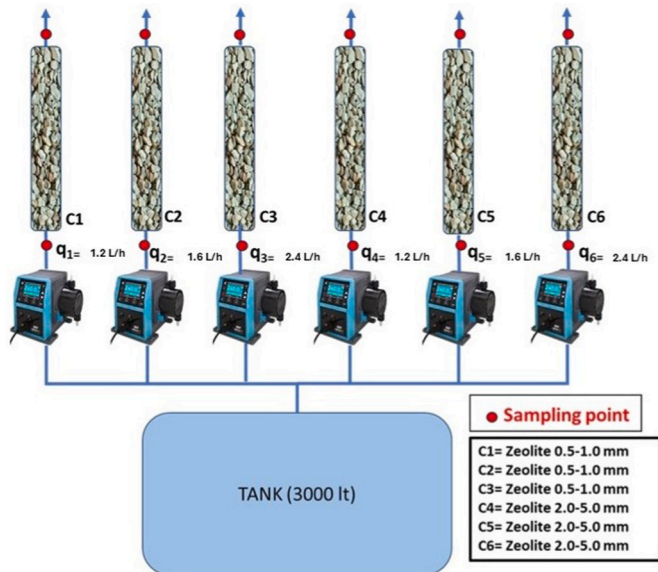


Fig. 1. Schematic layout of the experimental design.



Fig. 2. Representation of the experimental setup, showing the six columns filled with zeolite.

around 100 mg more than C4. However, C2 adsorbed 150 mg of NH₄⁺ less than C5, in contrast to the other flow rates. Nevertheless, as reported by the mass balances in Fig. 3, the columns overall reported the same adsorption efficiencies, meaning that the material's particle size can be considered to have a minor effect corresponding to, at maximum, 150 mg of adsorbed NH₄⁺. Furthermore, ion chromatography analysis was carried out to exclude interference or preference towards other cations. As reported in Table 3 for cations and Table 4 for anions, the species concentrations remained relatively constant throughout the experiments. This stability highlights the affinity of zeolite in the selective adsorption of NH₄⁺ (Muscarella et al., 2023a). Some of these results found during the experiment in which absorption capacities increase

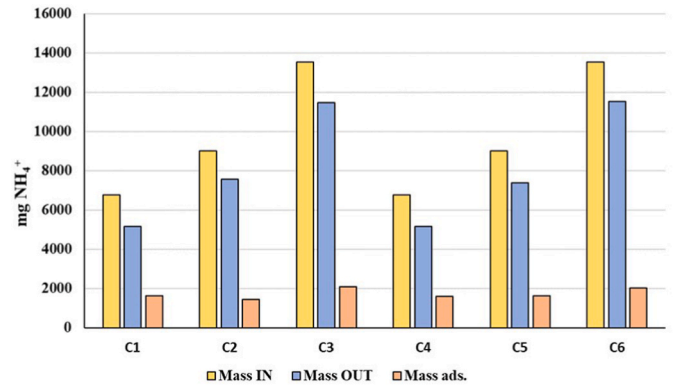


Fig. 3. Mass balance calculated for the six columns.

Table 2
Comparison of different studies found in bibliography.

Material	Ammonium concentration	Type of solution	type of experiment	Reference
natural zeolite and alkaline treated zeolite	1000 mg L ⁻¹	synthetic solution	batch	Muscarella et al. (2021)
natural zeolite	3.8–10 mg L ⁻¹	real TWW	batch	Al-Suhaibani et al. (2021)
natural and synthetic zeolite	NH ₄ ⁺ -N of 0.7 meq NH ₄ ⁺ -N/L	real TWW	batch	Guida et al. (2020)
natural zeolite	5–50 mg L ⁻¹	greywater	batch	Widiastuti et al. (2011)
zeolite 13X	5–400 mg L ⁻¹	synthetic wastewater	batch	Zheng et al. (2008)
Thermal treated zeolite	50 mg L ⁻¹	synthetic solution	batch	(Lei et al., 2008)
natural zeolite modernite	200 mg L ⁻¹	synthetic solution	batch	Zhou et al. (2021)
natural zeolite	0.3 M NH ₄ ⁺ Cl	synthetic solution	batch	Malekian et al. (2011)
natural zeolite	70–2000 mg L ⁻¹	synthetic solution	batch	Kithome et al. (2008)
natural zeolite	20–300 mg L ⁻¹	synthetic solution	batch	He et al. (2019)
zeolite from volcanic ash	10–40 mg L ⁻¹	deionized water vs. secondary effluent wastewater	batch and column	Gagliano et al. (2022)
Natural and modified zeolites	500 mg L ⁻¹	swine wastewater	batch and column	Cyrus and Reddy, 2011
natural zeolite	263.2–1363.6 mg L ⁻¹	wastewater	batch and column	Temel et al. (2021)
natural zeolite	0.2–300 g NH ₄ -N m ⁻³	wastewater	column	Nguyen et al. (1998)
natural zeolite	60–800 mg L ⁻¹	wastewater	column	Sayavedra et al. (2024)

Table 3

Average concentration of cations at the inlet of the columns (Pre-column) and the outlet of columns C1, C2, C3, C4, C5, and C6.

Column	Na ⁺	Ca ²⁺	Mg ²⁺	K ⁺
	mg L ⁻¹			
Pre-Column	124 ± 46	31 ± 6	59 ± 14	17 ± 3
C1	163 ± 90	35 ± 13	55 ± 21	17 ± 5
C2	152 ± 74	38 ± 9	60 ± 15	16 ± 6
C3	134 ± 44	39 ± 10	65 ± 9	16 ± 5
C4	156 ± 79	36 ± 11	59 ± 16	18 ± 5
C5	151 ± 73	36 ± 9	61 ± 15	17 ± 5
C6	149 ± 70	38 ± 10	59 ± 15	17 ± 5

Table 4

Average concentration of anions at the inlet of the columns (Pre-column) and the outlet of columns C1, C2, C3, C4, C5, and C6.

Column	F ⁻	Cl ⁻	NO ₂ ⁻	NO ₃ ⁻
	mg L ⁻¹			
Pre-Column	0.4 ± 0.1	188 ± 12	0.06 ± 0.02	1.9 ± 1.2
C1	0.5 ± 0.3	184 ± 17	0.07 ± 0.05	1.8 ± 1.04
C2	0.4 ± 0.2	190 ± 14	0.06 ± 0.01	1.7 ± 1.1
C3	0.5 ± 0.2	190 ± 14	0.06 ± 0.02	1.4 ± 1.1
C4	0.4 ± 0.2	189 ± 14	0.06 ± 0.01	1.5 ± 1.1
C5	0.5 ± 0.2	189 ± 13	0.06 ± 0.02	1.7 ± 1.1
C6	0.4 ± 0.1	188 ± 12	0.06 ± 0.02	1.6 ± 1.2

with decreasing particle diameter and increasing flow rate coincide with some results found in the literature (Table 2).

3.2. Efficiency of ammonium desorption

To assess the possibility of regenerating zeolite and recovering the adsorbed NH₄⁺, desorption tests have been carried out over 24 h. The amount of NH₄⁺ recovered during the tests is reported in Table 5. The desorption trend was not constant over the 24 h since all the columns desorbed 44–78 % of the overall adsorbed NH₄⁺ in the first 30 min. C1 and C2 desorbed a similar amount of NH₄⁺ in the first 30 min, 121 mg and 118 mg, respectively. C4 is the column that desorbed the most quantity of NH₄⁺ during this period (174 mg), while C5 desorbed 108 mg in the first 30 min. C3 and C6 desorbed 41 and 41 mg of NH₄⁺ respectively. After 24 h, the amount of NH₄⁺ desorbed decreased to 0.5–0.6 mg. Considering the total amount of NH₄⁺ desorbed, C1, C2 and C3 achieved 159, 150 and 71 mg, respectively. Conversely, C4, C5, and C6 desorbed 228, 180 and 92 mg of NH₄⁺. Despite only small differences reported in the adsorption rates, zeolite's particle diameter highly affected the desorption efficiency. The columns with the highest particle diameters were desorbed 20%–43% more than the smaller ones. Furthermore, it was observed that the lowest flow rates achieved a considerably higher amount of NH₄⁺ desorbed than the higher ones. Adopting the flow rate of 1.2 L h⁻¹ resulted in 4–27% more NH₄⁺ desorbed, compared to the 1.6 L h⁻¹ flow rate, while it desorbed 123–148% more compared to the highest flow rate adopted of 2.4 L h⁻¹. Small particle diameter zeolites, higher flow rates in the adsorption tests, and low flow rates in the

Table 5

Desorption test.

TIME (h)	C1	C2	C3	C4	C5	C6
	mg NH ₄ ⁺					
0.5	121	118	41	174	108	41
1	26	21	9	47	56	18
2	7	6	14	2	9	20
3	4	4	6	2	6	9
6	0.7	0.7	0.2	2	0.4	1
9	0.9	0.3	0.3	0.8	0.7	0.8
24	0.5	0.5	0.6	0.6	0.5	0.6

desorption test showed the best operational conditions to adsorb and recover the highest amount of NH₄⁺.

4. Discussion

The results show that zeolite exhibits a strong affinity for absorbing NH₄⁺ ions. This explanation agrees with the basic principles of how zeolites, particularly natural ones such as clinoptilolite, work in adsorbing cations such as NH₄⁺. The substitution of Si⁴⁺ with Al³⁺ in the structure of zeolites creates a negative charge balanced by cations (Widiastuti et al., 2011). Furthermore, zeolites can exchange their cations with those in the surrounding environment, such as NH₄⁺, without altering their crystal structure. Zhou et al. (2024) tested the adsorption and desorption capacities of a natural zeolite and a synthetic zeolite treated with sodium acetate. The results indicate that approximately 80% of the NH₄⁺ adsorption and desorption process, in their case, is determined by ion exchange capacity. In addition, it has been shown that zeolite treated with sodium acetate has a higher ion exchange capacity and is, therefore, more suitable for NH₄⁺ adsorption than untreated natural zeolite. These properties make zeolites effective in water treatment, agriculture, and aquatic systems (Han et al., 2021). A characteristic of the material that can influence the ion exchange capacity of NH₄⁺ with zeolite is the particle size (Table 2). When smaller particle sizes are used, NH₄⁺ has a higher exchange capacity, probably caused by a higher mass transfer in the zeolite (Hedström, 2001; Guida et al., 2020). The results indicate that zeolites with a particle size of 0.5–1.0 mm exhibit a slightly higher adsorption capacity for NH₄⁺ ions than those with a larger 2.0–5.0 mm particle size. This difference is attributed to zeolites with smaller particle diameters possessing a higher specific surface area (Taddeo et al., 2017; Uygur et al., 2019). This increased specific surface area provides a larger contact zone for ions, enhancing adsorption capacity due to the higher number of available sites for interaction with NH₄⁺ ions (He et al., 2019). This result is in line with the literature. For example, Wen et al. (2006) compared two zeolites with different particle diameters, 1.0–3.2 mm and 8.0–15.0 mm, and observed that the cation exchange capacity of zeolite increased with decreasing particle diameter. Langwaldt (2008) conducted a column study testing the absorption capacities of 5 zeolites with particle diameters 0.9–2.5 mm. The test was carried out with a flow rate of 0.3 L h⁻¹ and a synthetic influent with an NH₄⁺ concentration of 4.5 mg L⁻¹. Results highlighted the higher efficiency of zeolite chabazite compared to clinoptilolite despite having a higher particle size, thus pointing out the importance of the mineralogical nature of the material. Among the chabazite, the one with the smallest particle diameter (1.3 mm) adsorbed 46 mg g⁻¹ of NH₄⁺, the highest quantity reported. This result also aligns with a recent study by Muscarella et al. (2024). In their column study, the authors tested the same zeolite with different particle diameters of 0.5–1.0 mm and 2.0–5.0 mm. Zeolite with the smallest particle diameter (0.5–1.0 mm) showed the highest adsorption rate by adsorbing 49% of the influent NH₄⁺ and 22% more than 2.0–5.0 mm zeolite. In another study, Pinelli et al. (2022) discovered that the mixture of chabazite and phillipsite, characterized by a smaller diameter than other studied sorbent materials, had a high capacity to recover NH₄⁺ from municipal wastewater. Cyrus and Reddy (2011) noted an increase in the absorption capacity of zeolite with a smaller particle diameter (1 mm mesh) compared to that with a larger size (2–4 mm mesh). The smaller zeolite adsorbed around 0.60 g N kg⁻¹ more than the others.

The adsorption tests indicated that increasing the flow rate increases the amount of NH₄⁺ adsorbed by the columns. Indeed, C3 and C6, with a 2.4 L h⁻¹ flow rate, showed the highest adsorption capacity. This is attributed to the fact that with a higher flow rate, a greater volume of NH₄⁺-containing solution passes through the column within a given time interval. This increase in the available NH₄⁺ concentration results in more NH₄⁺ ions having contact with the available adsorption sites in the zeolite. Wang and Peng (2010) in their study compared the efficiency of removing NH₄⁺ from aqueous solutions using natural zeolites at different flow rates. The results showed that an increase in flow rate increases the

total volume of treated NH_4^+ , thus improving the overall adsorption capacity of the column. Canellas-Garriga (2018) compared three different types of zeolites at different flow rates. In their case, an increase in NH_4^+ capacity from 13.6 to 21.5 mg g^{-1} was reported for a change in flow rate from 0.11 to 0.54 L h^{-1} . As reported by the literature mentioned above, a higher flow rate means higher NH_4^+ ions availability, which leads to an earlier achieved saturation point (Sundhararasu et al., 2022). However, Temel et al. (2021) tested three different flow rates and obtained a higher absorption with the lowest flow rate (2.5 ml min^{-1}) due to the fact that by reducing the flow rate, the contact time of the absorbent material with the NH_4^+ solution used has increased. Nevertheless, it should be noted that in this study the concentration of NH_4^+ used was high, around 1200 ml L^{-1} .

Results reported in Table 3 also highlight the zeolite's high affinity for NH_4^+ ions, mainly because of the ion size that fits perfectly within the zeolite pores (Kithome et al., 1998). This dimensional compatibility is crucial since many other cations, such as sodium, potassium and calcium, may be too large or small to be adsorbed with the same effectiveness. Moreover, zeolites have a crystal structure with negative charges due to the isomorphous substitution of Si^{4+} with Al^{3+} . NH_4^+ ion, being a monovalent cation, is strongly attracted by these negative charges (Muscarella et al., 2021). Additionally, the NH_4^+ ion has relatively low hydration energy compared to other cations such as Na^+ and Ca^{2+} . This means that the NH_4^+ ion is less likely to interact with water molecules in solution and more likely to interact with active zeolite exchange sites (Shi et al., 2013).

Regarding the desorption test, rapid desorption was observed in the first half hour with a decrease until reaching around 0.5 mg NH_4^+ desorbed at 24 h. Such a fact can be attributed to a combination of factors: a high concentration of sodium ions, an efficient ion exchange process, accessibility of surface exchange sites, a strong concentration gradient and initially rapid diffusion (Muscarella et al., 2021; Widiastuti et al., 2011) (Table 2). Canellas-Garriga (2018) performed a desorption test using NaCl, achieving a desorption of 30% in the first cycle of regeneration and 10% in subsequent cycles. Zhang et al. (2022) conducted a study comparing NaOCl and NaCl efficiency for zeolite regeneration. Although NaOCl resulted in faster desorption, using NaCl has proved to be the most suitable method. The results also showed that desorption can continue for more than 24 h, though more slowly, until the exchange sites for Na^+ ions run out.

5. Conclusions

The results observed in the present study demonstrate the high efficiency of zeolite as NH_4^+ adsorbent material from real TWW, which can be regenerated and reused through desorption processes. Moreover, two critical parameters that significantly influence the adsorption capabilities of zeolite, namely flow rate and particle size, were thoroughly tested. The findings related to these parameters are consistent with existing literature, reinforcing the established understanding of how these factors affect zeolite performance. Specifically, the highest flow rates (2.4 L h^{-1}) and smallest particle sizes (0.5–1.0 mm) were associated with the highest adsorption efficiency, reaching up to 2078 mg of NH_4^+ adsorbed. The desorption test further validated the adsorption capabilities of zeolite by demonstrating its high potential for reuse after regeneration. Specifically, the greatest particle diameter columns desorbed 20%–43% more than the smallest ones. Regardless of particle size, a lower flow rate (1.2 L h^{-1}) resulted in a 123–148% higher desorption rate than the highest. Incorporating zeolite to recover nutrients in long-term water treatment processes presents a viable solution for escalating resource scarcity and high pollution from excessive fertilizer use. Beyond the sustainability and cost-efficiency of this approach, the findings highlight the effectiveness of zeolite in facilitating tertiary TWW. This process enables nutrient recovery, with zeolite potentially acting as an N source for plants, and produces water with reduced N concentrations, suitable for agricultural reuse. Establishing a nutrient

recovery pathway within wastewater treatment systems, such as those implemented in a university setting in this study, and the potential application of NH_4^+ -enriched zeolite as an N source for plants enhance the circular economy framework. This strategy offers a sustainable, actionable approach to repurposing treated TWW while supporting agricultural productivity's environmental and economic sustainability.

CRedit authorship contribution statement

Pedro Tomas Bulacio Fischer: Writing – original draft, Investigation, Conceptualization. **Daniele Di Trapani:** Writing – review & editing, Writing – original draft, Supervision. **Vito Armando Laudicina:** Writing – review & editing, Supervision, Conceptualization. **Antonio Mineo:** Writing – review & editing, Investigation. **Sofia Maria Muscarella:** Writing – review & editing, Writing – original draft, Supervision, Conceptualization. **Giorgio Mannina:** Writing – review & editing, Supervision, Conceptualization.

Declaration of competing interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: Giorgio Mannina reports financial support was provided by European Union. If there are other authors, they declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Data availability

Data will be made available on request.

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