https://doi.org/10.15255/KUI.2025.028
KUI-8/2026
Original scientific paper
Received July 21, 2025
Accepted September 26, 2025

Performance Evaluation of Nanofiltration and Reverse Osmosis Membranes for Atrazine Removal from Water

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5th ZORH CONFERENCE

Abstract

Pesticide contamination from agricultural activities has become a growing environmental concern since pesticides can migrate across environmental compartments and accumulate on undesirable surfaces and in water bodies. Given their high toxicity to living organisms and resistance to degradation, developing effective removal strategies is essential. This study investigates the removal of pesticide atrazine from a binary solution using commercially available nanofiltration (NF) and reverse osmosis (RO) membranes, with molecular weight cut-offs of 150–300 Da and 100–200 Da, respectively. The experimental study was conducted in a laboratory-scale RO/NF system with six cells connected in parallel over a duration of 3 h. Removal efficiency was determined by analysing all samples (feed and permeate) using liquid chromatography-tandem mass spectrometry. The results showed that the atrazine removal efficiency ranged from 16.0 to 84.9 % with NF membranes, and from 64.0 to 93.1 % with RO membranes, indicating that size exclusion was the main removal mechanism.

Keywords

Pesticides, atrazine, nanofiltration, reverse osmosis, size exclusion

1 Introduction

The increasing global population has led to a rise in food production, intensifying agricultural practices and increasing pesticide usage.[1] The World Health Organization defines pesticides as substances used to protect crops from pests such as insects, fungi, and weeds.[2] However, only around 1 % of applied pesticides reach the target plant tissue, while the remainder disperses into the environment. Aerial spraying of pesticides can pollute surrounding areas with micro- and macro-droplets, while irrigation facilitates the spreading of pesticides through runoff or leaching, contaminating nearby soils and groundwater systems.[1,3] There is a growing interest in the environmental fate of pesticides and their impact on soil and water quality, particularly due to their chemical complexity and low biodegradability, which also poses risks to human health.[4,5] To address this, the European Union adopted a groundwater directive in 2006 to protect groundwater from pollution. The directive sets quality standards for groundwater and drinking water at a concentration of 0.1 $\mu g\ l^{-1}$ for individual pesticides and their relevant metabolites, and 0.5 $\mu g\ l^{-1}$ for the total sum of pesticides.[6]

Removing pesticides from water remains a major challenge. Conventional water treatment technologies such as filtration, coagulation-flocculation, and sedimentation are generally ineffective.[7,8] Other technologies such as advanced oxidation processes, photodegradation, and similar techniques also present drawbacks, such as the formation of hazardous by-products.[9–11] Pressure-driven membrane processes for the removal of various pollutants are gaining attention primarily due to their cost-effectiveness.[8]

However, the variability in pesticide structures and the wide pH range of contaminated water, from highly acidic to alkaline, pose additional challenges where membrane separation may offer viable solutions.[12] The success of membrane-based separation processes such as nanofiltration (NF) and reverse osmosis (RO) for pesticide removal largely depends on the type of membrane selected. Key factors in selecting a suitable membrane include molecular weight cut-off (MWCO), porosity, surface charge, degree of salt rejection, hydrophobicity, and composition.[13] Pesticide removal efficiency is also influenced by the physicochemical properties of the pesticides themselves, such as molecular weight and size, polarity, hydrophobicity/hydrophilicity (log $K_{O/W}$), and acid dissociation constant (pK_a).[13,14] These properties collectively influence the mechanisms governing pesticide-membrane interactions.[13] According to previous studies, size exclusion is the primary mechanism of pesticide removal. *Musbah et al.*[15] and *Mukherjee et al.*[8] both concluded that the most important parameters for the rejection of organic molecules are molecular weight and size, with the shape of the molecule also playing an important role in rejection. *Fini et al.*[16] tested the removal of different pesticides with NF and RO membranes. Since the difference in rejection correlated with the MWCO of the membranes, the overall mechanism appeared to be

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Note: The investigations in this paper were presented at the 5th International ZORH conference of scientists, professionals and students – "*Environmental Protection, Sustainable Production and Examples of Best Practice*" held on April 9–11, 2025 at the Faculty of Chemistry and Technology University of Split, Croatia.

size exclusion. *Bodalo et al.*[17] observed higher atrazine rejection with membranes of lower MWCO, which also indicated size exclusion. *Tan et al.*[18] showed that, with various effects of solutes such as hydrophobicity, dipole moment, and adsorption, size exclusion still played the dominant role in the rejection of the pesticides atrazine and dimethoate.

Atrazine is a triazine herbicide with a molecular weight of 215.69 g mol^{-1}, commonly used to control weeds in corn and sugarcane cultivation.[19] Although its use has been banned in the EU under Commission Decision 2004/248/EC, it is still used, and can be found in groundwater and surface waters due to its persistence.[20,21] Studies have shown that atrazine is one of the most frequently detected herbicides in various groundwater, surface water, and tap water monitoring sites, with concentrations ranging from 0.44 ng l^{-1} to 5.0 µg l^{-1}.[22–24] This study investigates the removal efficiency of atrazine from a binary solution using three NF and three RO membranes.

2 Experimental

2.1 Membranes and compounds

The commercially available membranes used in this study included NF membranes HL and DK from Suez (France), and NF from Dow-Filmtec (USA), as well as RO membranes XLE from Dow-Filmtec (USA), AP from Suez (France), and ESPA4-LD from Hydranautics (Japan). The membrane characteristics, as provided by the manufacturers, are summarised in Table 1.

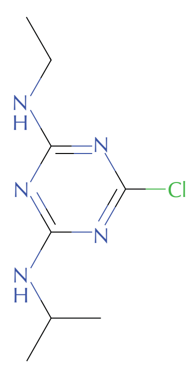
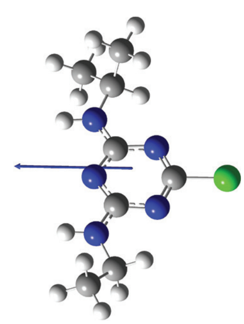
The pesticide atrazine (Thermo Fischer Scientific, USA), with a high purity (> 95 %) was used in this study. A summary of its physicochemical properties is provided in Table 2. A binary solution (100 µg l^{-1}) of standard was initially prepared in methanol (1 g l^{-1}) due to its low solubility in water and subsequently diluted with demineralised water. Since the proportion of methanol in the solution was less than 5 %, it was considered aqueous. The pH value of the binary solution was 6.90.

Table 1 – Membrane characteristics as provided by the manufacturer
Tablica 1 – Karakteristike membrana od proizvođača

Type Tip	Membrane Membrana	Maximum operating pressure / bar Maksimalni radni tlak / bar	pH range pH raspon	R^a / %
Nanofiltration Nanofiltracija	NF	41	3–10	98.0 ($MgSO_4$)
	DK	41	3–9	98.0 ($MgSO_4$)
	HL	41	3–9	95.0 ($MgSO_4$)
Reverse osmosis Reverzna osmoza	ESPA4	41	2–10	99.0 (NaCl)
	XLE	41	2–11	97.0 (NaCl)
	AP	13	2–11	92.0 (NaCl)

[a] Concentrations of $MgSO_4$ were 2000 mg l^{-1}, and NaCl 500 mg l^{-1} and 2000 mg l^{-1} (XLE).

Table 2 – Physicochemical characteristics of atrazine. The characteristics were obtained from EPI SUITE™ v4.11, except where indicated by superscript letters a, b, and c.
Tablica 2 – Fizikalno-kemijske karakteristike atrazina. Karakteristike su dobivene iz EPI SUITE™ v4.11, osim u situacijama koje su označene eksponentima a, b i c.

		Molecular structure[a] Molekulska struktura[a]	Direction of dipole moment Smjer dipolnog momenta
CAS no.	1912-24-9		
molecular weight M_w, g mol^{-1} molekulska masa M_w, g mol^{-1}	215.69		
solubility in water, mg l^{-1} topljivost u vodi, mg l^{-1}	34.7		
log $K_{O/W}$	2.61		
pK_a	1.60		
dipole moment μ, D [b] dipolni moment μ, D	3.8915		
width, nm [c] širina, nm height, nm [c] visina, nm length, nm [c] duljina, nm	0.616 0.439 0.970		

[a] Molecular structure obtained from Chemspider; [b] Dipole moment calculated by Gaussian[25]; [c] Calculated using HyperChem 8.0.

Table 3 – Physicochemical characteristics of membranes
Tablica 3 – Fizikalno-kemijske karakteristike membrana

Type Tip	Nanofiltration Nanofiltracija			Reverse osmosis Reverzna osmoza		
Membrane Membrana	NF	DK	HL	ESPA4	XLE	AP
contact angle / ° kontaktni kut / °	34.95	52.91	49.85	40.08	65.32	83.74
zeta potential pH 7 / mV zeta potencijal pH 7 / mV	−18.33	−14.58	−32.39	−30.83	−23.02	−25.10

2.2 Methods

The removal of atrazine from a binary solution (standard dissolved in demineralised water) was tested in a laboratory-scale RO/NF system, as described in detail by *Dolar et al*.[26] The experiment was conducted at a working pressure of 10 bar, with a membrane area of 11 cm^2. Before the removal of atrazine, the membranes were pressurised at 12 bar until the membrane flux stabilised (membrane compaction), after which the pure water flux was measured under working conditions. Treatment with the binary pesticide solution lasted 3 h in batch circulation mode, where both permeate and retentate were re-circulated into the feed stream. Feed and permeate samples were collected after 1 h, 2 h, and 3 h. The membrane contact angle was measured using the Ossila Contact Angle Goniometer (UK). A 2 µl droplet of room-temperature demineralised water was manually dispensed using a micropipette and placed onto the surface of the membrane sample. An image of the droplet was captured using the instrument's built-in camera. Three measurements were taken for each sample, and the mean value was used. Zeta potential of the membranes was measured using a SurPASS Electrokinetic Analyzer-type A (Anton Paar, Austria). The membrane samples were fixed to the sample holder with the Adjustable Gap Cell using double-sided adhesive tape. The pH dependence of the zeta potential was determined across the range of pH 2.5–9.

A Bruker Vertex 70 (Germany) Fourier transform infrared (FTIR) spectrometer with a Platinum ATR single reflection diamond (n = 2.4) crystal-based module in the mid-IR range (400–4500 cm^{-1}) was used to characterise the membrane surfaces. The membrane spectra were recorded with a resolution of 4 cm^{-1} and 32 scans.

2.2.1 Chemical analysis

Atrazine was quantified using an ACQUITY Ultra Performance Liquid Chromatography H-Class System (Waters, USA), coupled with a Xevo TQ-S micro mass spectrometer (Waters, USA), equipped with an electrospray ionisation source, operated in positive ion mode. Chromatographic separation was performed at 60 °C on an XBridge Premier BEH C18 column (100 × 2.1 mm i.d., 2.5 µm particle size; Waters, USA). The injection volume was 1 µl. The mobile phase consisted of 0.1 % v/v formic acid in water (A) and 0.1 % v/v formic acid in methanol (B), with a flow rate of 400 µl min^{-1} and the following gradient programme: 0–3 from 45 % B to 95 % B; 3–4 min, 95 % B; and 4–5 min 45 % B. Instrument parameters were as follows: capillary voltage 3.0 kV, cone voltage 25 V, source temperature 150 °C, desolvation temperature 450 °C, desolvation gas flow 1000 l h^{-1}, and cone gas flow 50 l h^{-1}. The MRM transitions of 216→**174**; 104; and 96 were used for atrazine detection (quantification trace in bold). The limit of quantification was 0.6 µg l^{-1}.

3 Results and discussion

3.1 Membrane characterisation

The pristine membranes were characterised in terms of hydrophobicity, charge, and FTIR spectra for easier determination of the atrazine removal mechanisms. Hydrophobicity is relevant for assessing the adsorption of organic compounds onto the membrane matrix, while surface charge defines electrostatic attraction/repulsion. FTIR analysis was employed to detect any new interactions between atrazine and the membranes.[27,28] The results are presented in Table 3. Zeta potential measurements confirmed that all membranes used in this study were negatively charged under the working pH value. Nevertheless, variations were observed, with nanofiltration HL membrane and the reverse osmosis ESPA4 membrane exhibiting the lowest zeta potential values. Contact angle measurements, used as a relative measurement of surface hydrophobicity, showed that all membranes had contact angles below 90°, classifying them as hydrophilic. Nevertheless, the contact angles for XLE and AP membranes were greater than 60°, indicating a relatively hydrophobic character, according to *Akin* and *Temelli*.[29]

3.2 Removal of atrazine

The removal of atrazine from the binary solution during the three-hour treatment using the NF and RO membranes is presented in Table 4. Rejection (R) of atrazine was calculated as:

$$R = \frac{c_f - c_p}{c_f} \cdot 100\ \% \qquad (1)$$

where, c_f (μg l^{-1}) and c_p (μg l^{-1}) are concentrations of atrazine in feed and permeate stream, respectively.

Since sampling was performed hourly, the removal mechanism is initially discussed for the first hour of treatment. At first glance, a difference between reverse osmosis and nanofiltration membranes could be observed.

As expected, RO membranes exhibited higher atrazine removal than NF membranes, with the exception of AP membrane. Due to the MWCO of RO membranes (100–200 Da) and the higher molecular weight of atrazine (215.69 g mol^{-1}), size exclusion was probably the dominant removal mechanism. This was further supported by the dipole moment (indicating the molecule's orientation toward the membrane), suggesting that the length of the atrazine molecule (0.970 nm) must be taken into account, as it is larger than the pores of the RO membranes, ranging from 0.3 to 0.9 nm.[30]

According to the manufacturer, the AP membrane is an RO membrane characterised by high flux and relatively high sodium chloride rejection at a typical operating pressure of 4.8 bar. In this study, however, all membranes were operated at 10 bar. The lower removal efficiency observed for the AP membrane may be attributed to this elevated pressure. The higher working pressure caused a much higher flux for the AP membrane (140.2 LMH) compared to the XLE (52.5 LMH) and ESPA4 (19.4 LMH) membranes, which resulted in a higher passage of atrazine through the AP membrane.

In the case of nanofiltration membranes, removal efficiencies were much lower for the NF (31.4 %) and HL (29.3 %) membranes, whereas the DK membrane achieved 89.9 % removal, comparable to RO membranes. Considering the MWCO of NF membranes (150–300 Da) and the molecular weight of atrazine (215.69 g mol^{-1}), it is reasonable to assume that atrazine molecules can pass through these membranes. Moreover, atrazine is neutral at pH 7,[31] meaning that additional electrostatic repulsion was not involved. For the DK membrane, the removal was higher due to the denser structure of this membrane, which was confirmed by the relatively low flux of 23.4 LMH.

Table 4 – Removal of atrazine by RO/NF membranes
Tablica 4 – Uklanjanje atrazina pomoću RO/NF membrana

Type Tip	NF			RO		
Membrane Membrana	NF	DK	HL	ESPA4	XLE	AP
R_{1h}/%	31.4	89.9	29.3	95.7	89.0	56.5
R_{2h}/%	25.8	86.4	20.1	93.8	88.3	58.8
R_{3h}/%	19.5	84.9	16.0	93.1	88.8	64.0

It is important to note that atrazine removal decreases over time for most membranes, with the exception of XLE (which remained constant) and AP (which showed an increase). Based on the physicochemical characteristics of atrazine, primarily its log $K_{O/W}$, the observed decrease in rejection for the NF, HL, and DK membranes can be attributed to adsorption at the beginning of treatment. Atrazine, with a log $K_{O/W} > 2$, can be considered hydrophobic, with a tendency to adsorb onto the polymeric matrix of the membrane. Over time, this can lead to diffusion through the membranes, resulting in a decrease in rejection rates.[32] FTIR analysis (Fig. 1) reveals minor structural changes on the membranes after analysis, but confirms the adsorption of atrazine onto the membrane. Slight changes were observed around 3600 and 3700 cm^{-1}, corresponding to OH stretching, and around 2300 cm^{-1}, associated with carbon dioxide stretching, both of which suggest the possibility of diffusion of molecules to the permeate.[32]

The slight changes in the FTIR spectra for the NF, DK, and HL membranes can be attributed to the dipole moment of atrazine. Since the dipole moment is not significantly higher than 3 D, the probability of the molecule entering the pores and interacting with the membrane surface is much lower. Moreover, the direction of the dipole moment determines the position of the molecule in the solution with respect to the membrane. According to the structure shown in Table 1, atrazine is parallel to the membrane surface and aligned with the pore, meaning that it should penetrate the pore more easily.[33] However, since the dipole moment is aligned with the branched alkyl groups attached to nitrogen atoms, the dominant factor becomes the length of the molecule. This may hinder the likelihood of the molecule penetrating through the pore and interacting with the membrane surface. This depends on the pore size of the membranes; NF membranes have pores between 1 and 2 nm, while RO membranes have pore sizes smaller than 0.8 nm.[30] Since the length of the atrazine molecule is 0.970 nm, it is still possible for the molecule to enter the pore and adsorb onto the polymer matrix of the membrane, as evidenced by changes observed in the FTIR spectra.

4 Conclusion

Reverse-osmosis membranes demonstrated good overall removal efficiency, as the molecular weight of atrazine exceeds the MWCO of all membranes used. This confirmed that size exclusion was the primary removal mechanism in RO membranes. For the nanofiltration membranes, atrazine removal ranged from 16 to 89 %, indicating that atrazine could permeate through the membrane due to their higher MWCO. Additionally, adsorption of atrazine, indicated by changes in the FTIR spectra, led to reduced atrazine removal efficiency in the NF, DK, and HL membranes.

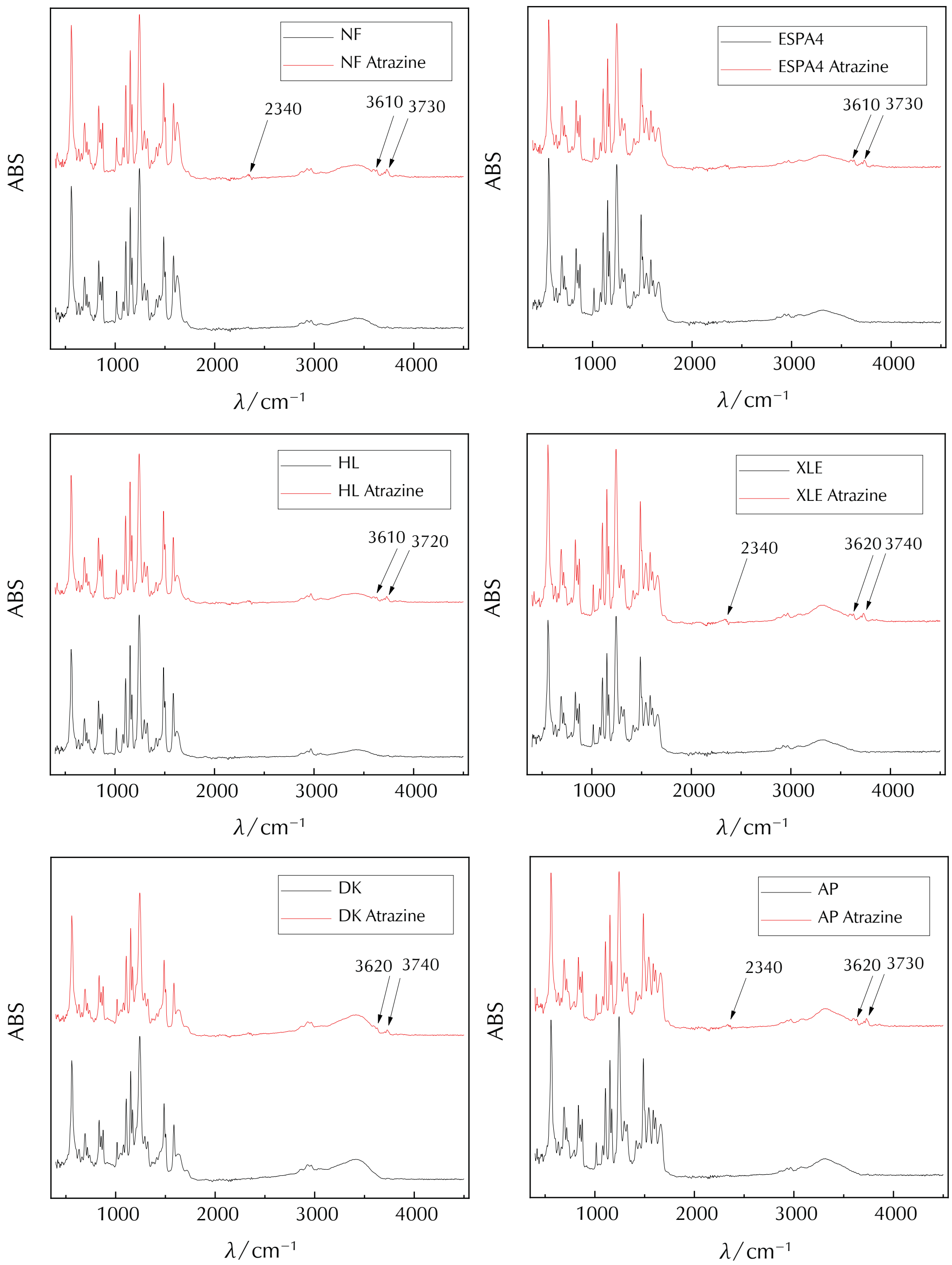


Fig. 1 – FTIR spectra of pristine membranes and membranes after atrazine treatment
Slika 1 – FTIR spektri čistih membrana i membrana nakon obrade atrazina

ACKNOWLEDGEMENTS

This research was funded by the NATO Science for Peace and Security Programme under grant ID. G6087 and the Croatian Science Foundation project DOK-NPOO-2023-10-8063.

List of abbreviations and symbols
Popis kratica i simbola

EU	– European Union – Europska unija
NF	– nanofiltration – nanofiltracija
RO	– reverse osmosis – reverzna osmoza
$MgSO_4$	– magnesium sulphate – magnezijev sulfat
NaCl	– sodium chloride – natrijev klorid
MWCO	– molecular weight cut-off – granična molekulska masa
$\log K_{O/W}$	– logarithm of octanol-water partition coefficient – logaritam koeficijenta raspodjele oktanol-voda
pK_a	– acid dissociation constant – konstanta disocijacije kiseline
M_w	– molecular weight – molekulska masa
μ	– dipole moment – dipolni moment
FTIR	– Fourier transformation infrared spectroscopy – infracrvena spektroskopija s Fourierovom transformacijom
R	– rejection factor – faktor zadržavanja
c_f	– concentration in feed – koncentracija u pojnoj smjesi
c_p	– concentration in permeate – koncentracija u pročišćenoj struji

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SAŽETAK

Procjena učinkovitosti nanofiltracije i reverzne osmoze za uklanjanje atrazina iz vode

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Povećanje poljoprivrednih aktivnosti dovodi do sve veće zabrinutosti oko otpuštanja pesticida u okoliš, zbog njihove mogućnosti putovanja kroz različite dijelove okoliša i akumulacije na neželjenim površinama i u vodenim tijelima. Zbog visoke toksičnosti pesticida prema živim bićima, kao i otpornosti na razgradnju, važno je istražiti učinkovite metode za njihovo uklanjanje iz vode. U ovom istraživanju, ispitano je uklanjanje pesticida atrazina iz binarne otopine uporabom komercijalno dostupnih nanofiltracijskih (NF) i reverzno osmotskih (RO) membrana s vrijednostima granične molekulske mase od 150 do 300 Da odnosno od 100 do 200 Da. Eksperiment je proveden na laboratorijskom RO/NF sustavu sa šest usporedno povezanih ćelija kroz razdoblje od 3 h. Da bi se ispitala učinkovitost uklanjanja, svi uzorci ulazne otopine i permeata analizirani su tekućinskom kromatografijom spregnutom s tandemskom masenom spektrometrijom. Rezultati su pokazali da je učinkovitost uklanjanja atrazina 16,0 – 84,9 % za NF, a 64,0 – 93,1 % za RO, što ukazuje na to da je glavni mehanizam uklanjanja efekt prosijavanja.

Ključne riječi

Pesticidi, atrazin, nanofiltracija, reverzna osmoza, efekt prosijavanja

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Izvorni znanstveni rad
Prispjelo 21. srpnja 2025.
Prihvaćeno 26. rujna 2025.