

EFFICIENCY ANALYSIS OF SOME ZEOLITE MEDIA IN THE TREATMENT OF TECHNOLOGICAL WASTEWATER IN AGRO-ZOOTECHNICAL UNITS

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ABSTRACT. The present study aimed to evaluate the efficacy of two filtration media—Rupea natural zeolite (ZNR) and Turbidex—in reducing the pollutant load of wastewater from agro-zootechnical units. Both media were tested under identical operational conditions to explore their potential for sustainable wastewater valorization with minimal environmental impact. Results showed that ZNR exhibited higher adsorption rates for nitrites, nitrates, and ammonium, whereas Turbidex achieved greater retention for COD, total suspended solids (TSS), and turbidity. Filtration through both zeolitic media effectively reduced microbial loads, as indicated by the elimination of coliform bacteria and enterococci, along with a significant decrease in heterotrophic plate counts at 22°C and 37°C. These findings demonstrate the potential of small-scale zeolite filtration systems as eco-friendly solutions for agro - zootechnical wastewater treatment.

Keywords: *adsorption, clinoptilolite, ZNR (natural zeolite from Rupea), Turbidex, wastewater*

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INTRODUCTION

Economic and urban development have increasingly intensified pressures on freshwater resources, resulting in a progressive decline in water availability and posing significant risks to human, economic, and environmental security [1–3]. The protection and sustainable management of natural freshwater reserves remain critical challenges, particularly in rural areas lacking access to potable water, where agricultural activities depend on reliable water supplies [2–4]. The Water Framework Directive (WFD) underscores the importance of maintaining water quality at both national and European scales, not only for environmental protection but also to support water-intensive sectors such as agriculture. Data from the WFD further demonstrate the substantial impact of agricultural practices on the deterioration of water quality, with uncontrolled wastewater discharge from small-scale agricultural units emerging as a major threat to surface water resources [4]. The increasing scarcity of freshwater resources underscores the need to explore alternative water sources, including the reuse of wastewater for irrigation [5]. Recent studies highlight that the integration of sustainable technologies and smart monitoring tools in agricultural practices constitutes a forward-looking approach to wastewater management and water resource protection. [6,7].

International research emphasizes the adoption of environmentally friendly materials capable of supporting innovative wastewater treatment technologies. In this context, ecological filter media have emerged as a viable alternative for managing wastewater from small agro-zootechnical units [8]. Zeolites have attracted significant attention due to their ability to remove both organic and inorganic pollutants from wastewater.

Their effectiveness is attributed to their crystalline and microporous structure, large surface area, uniform pore distribution, high stability, rapid adsorption - desorption kinetics and low energy consumption for regeneration [9-16].

Zeolites such as clinoptilolite, mordenite, chabazite, and erionite have demonstrated high efficiency in wastewater treatment [18]. Clinoptilolite is the most widely used zeolite for the removal of contaminants such as heavy metals, ammonia, dyes, phenols, and phenolic derivatives from aqueous solutions [18-21]. Recent research has also highlighted their role in reducing the ecological impact generated by the presence of nitrites and nitrates in wastewater [13,20]. In Romania, zeolites from Mârsid and Pâglișa have been successfully applied to decrease NH_4^+ , COD, and BOD_5 levels in wastewater [22].

Multiple studies have demonstrated the effective use of zeolites in targeted pollutant removal processes. For example, copper ions can be adsorbed using a clinoptilolite–chitosan composite, while NH_4^+ ions are

efficiently removed at concentrations of 10.4–12.3 mg/g from synthetic solutions at 20°C and pH 6.09 using natural zeolite from the Rupea region [23,24]. In addition, zeolites can also function as biofilters for the removal of pathogenic microorganisms.

The use of natural zeolites in water treatment is further supported by their low cost, environmental compatibility, high ion exchange capacity, cation selectivity, chemical and physical stability, as well as their renewability and reusability [24]. These properties make zeolites highly suitable for integration into sustainable wastewater management strategies, particularly in small-scale agro-zootechnical systems [25-28].

RESULTS AND DISCUSSION

Analysis of the physicochemical indicator parameters

The evolution of pH can be characterized by wide oscillations (6.31-8.10) in the set of raw and filtered wastewater samples. The distribution of recorded values reveals a clear tendency of alkalinization of the water upon filtration through the zeolitic material bed (Figure 1), a phenomenon attributable to the inherently elevated pH of the zeolite itself. The pH values recorded post-filtration of the wastewater are within the range outlined for agricultural irrigation water, which must not oscillate outside the permissible limits (6.5-8.5) allowed by current legislation [30].

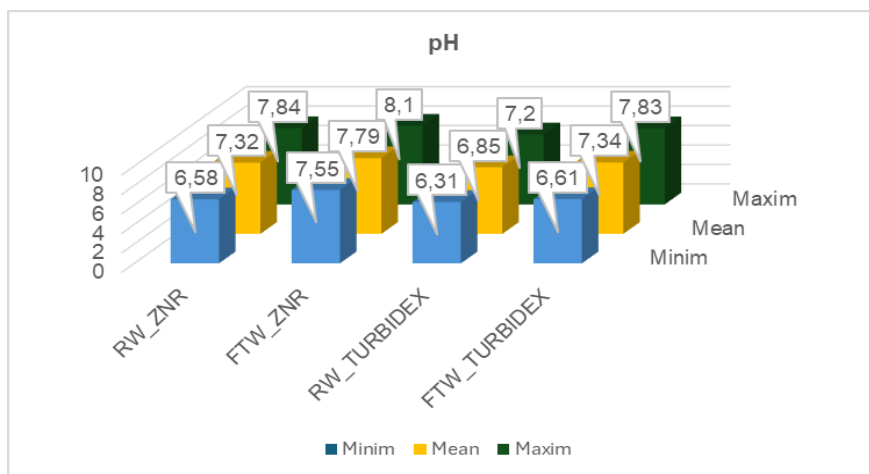


Figure 1. The evolution of the pH levels of wastewater [RW] and treated/ filtered water [FTW] on ZNR and Turbindex filtering media

The evolution of electrical conductivity (EC) was characterized by important oscillations, at the same operating temperature (25°C), for the wastewater that was filtered through Rupea zeolite (808-1320 $\mu\text{S}/\text{cm}$) and Turbidex (824-1117 $\mu\text{S}/\text{cm}$), respectively. A decreasing trend was observed after filtration, with mean values decreasing from 1277 $\mu\text{S}/\text{cm}$ to 1110 $\mu\text{S}/\text{cm}$ for Rupea zeolite and from 1025 $\mu\text{S}/\text{cm}$ to 933 $\mu\text{S}/\text{cm}$, for Turbidex.

This decreasing trend in conductivity can be correlated with the increase in alkalinity of the filtered water [21].

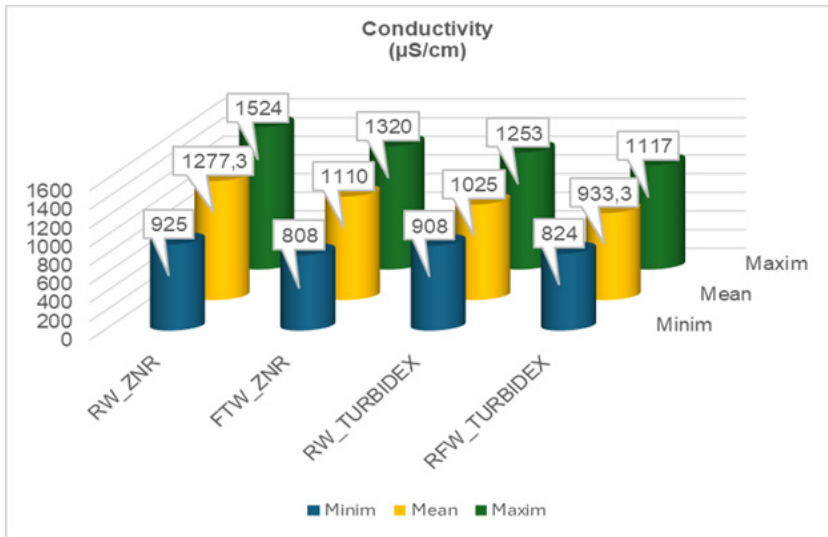


Figure. 2. The evolution of the electrical conductivity of wastewater [RW] and treated/filtered water [FTW] on ZNR and Turbidex filtering media

The use of the filter media (ZNR and Turbidex) allowed a significant reduction in the *turbidity* of agro-zootechnical wastewater. Thus, a decrease in the average turbidity values is observed from 18.44 to 5.45 NTU for ZNR and from 20.11 to 5.12 NTU for Turbidex, respectively. The quantification of these data indicated a good filtration efficiency of the two zeolite substrates, with a lower filtration rate for the Rupea zeolite (70.44%) compared to Turbidex (74.54%), but with very close average values (5.45 NTU and 5.12 NTU, respectively) (Figure 3).

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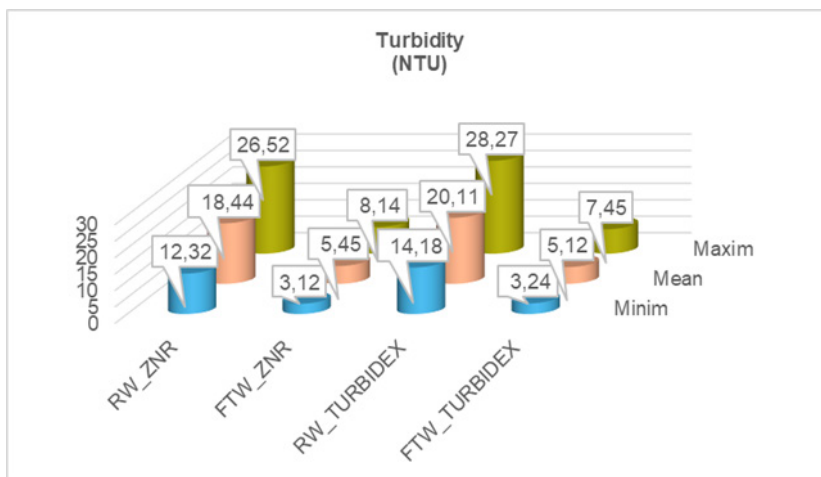


Figure. 3. The evolution of turbidity of wastewater [RW] and filtered/treated water [FTW] on ZNR and Turbidex filtering media

Turbidex demonstrated a higher average COD retention (35.35%) than Rupea zeolite (32.36%) (Figure 4). Retention efficiency declined with increasing influent concentrations, dropping to 25.8% for Rupea zeolite and 17.53% for Turbidex at concentrations above 150 mg/L, highlighting the need to optimize operational conditions according to both filter media properties and wastewater characteristics.

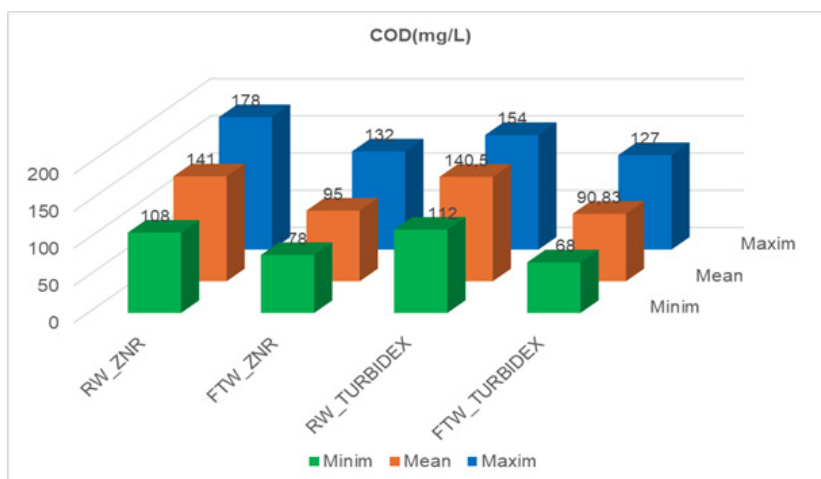


Figure 4. The evolution of COD of wastewater [RW] and filtered/treated water [FTW] on ZNR and Turbidex filtering media

As shown in the attached graph (Figure 5), the highest average percentage reduction in total suspended solids was achieved using Turbidex (74.54%) compared to Rupea zeolite (70.44%). The selected operational conditions, in relation to the type of filter media (Turbidex and Rupea zeolite), resulted in an effluent with TSS concentrations that comply with the standards established by NTPA 001/2002 [42].

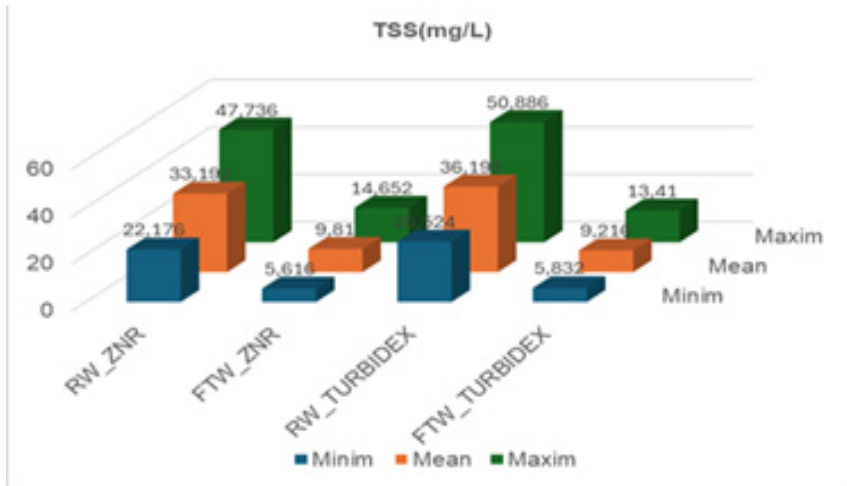


Figure 5. The evolution of total suspended solids of wastewater [RW] and filtered/treated water [FTW] on ZNR and Turbidex filtering media

The filtration of wastewater using Rupea zeolite and Turbidex resulted in high retention rates, with Rupea zeolite performing slightly better (88.54% vs. 86.85%). Despite this, under the operational conditions applied in this study, the average ammoniacal nitrogen concentration remained 3.74 mg/L for Rupea zeolite and 4.78 mg/L for Turbidex. This indicates that the adsorption of ammoniacal nitrogen was not sufficient to reduce its concentration to levels compliant with the effluent standards set by NTPA 001/2002, highlighting a limitation of the filtration process under the tested conditions [42].

When utilizing wastewater from agro-zootechnical units, particular attention is paid to the content of nitrites and nitrates. The discharge of insufficiently treated wastewater into outfalls generates the phenomenon of eutrophication, and in the case of distribution on land, it can lead to groundwater contamination.

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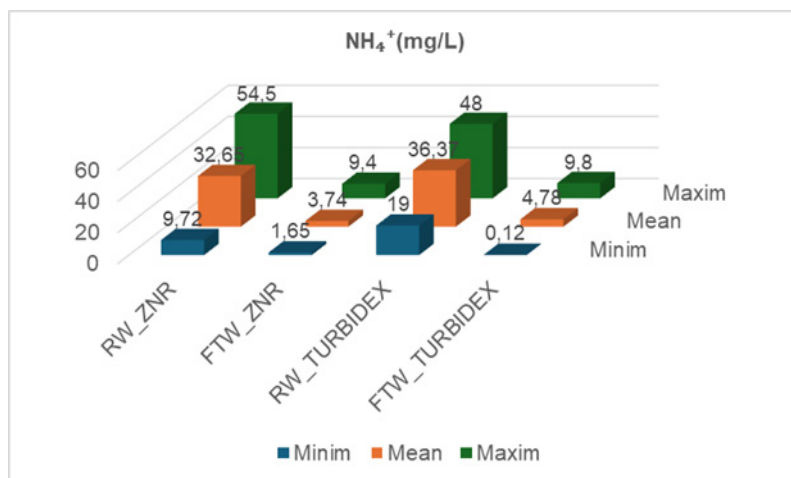


Figure. 6. The evolution of ammonium of raw water [RW] and filtered/treated water [FTW] on ZNR and Turbidex filtering media

The nitrites present in the raw wastewater exhibited levels slightly exceeding the permissible limits in the case of several samples. Following filtration through ZNR, the mean values decreased from 1.99 ± 0.33 mg/L to 1.33 ± 0.35 mg/L, and after filtration through Turbidex, they decreased from 2.31 ± 0.60 mg/L to 1.61 ± 0.42 mg/L (Figure 7). Monitoring of NO_2^-

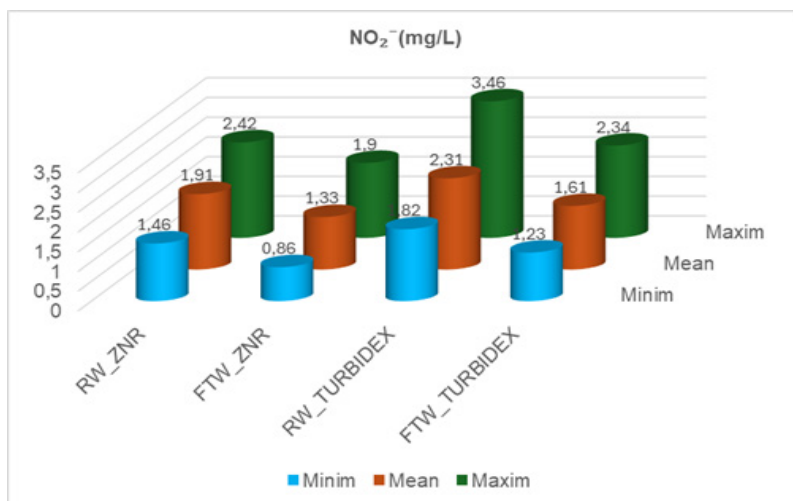


Figure. 7. The evolution of nitrites of wastewater [RW] and filtered/treated water [FTW] on ZNR and Turbidex filtering media

concentrations revealed a constant decrease in values post-filtration through both zeolite media, with these values falling within the permissible range for wastewater discharged into natural outfalls [46], except one sample whose values remained slightly elevated. The adsorption rate derived from these values outlined nitrite retention proportions from the wastewater of 30.36% for ZNR and 30.30% for Turbidex, respectively. Efficient treatment for nitrite removal can be based on denitrification, reverse osmosis filters or ion-exchange media, such as zeolitic materials [12, 18].

The nitrates detected in the raw wastewater exhibited concentrations slightly exceeding the permissible limits in most samples, with mean values reaching 59.42 ± 14.37 mg/L and 67.70 ± 17.93 mg/L, respectively. After filtration, the values decreased to 52.83 ± 10.16 mg/L and 62.05 ± 15.72 mg/L, respectively: the NO_3^- adsorption rate being 11.09% for ZNR and 8.34% for Turbidex, respectively (Figure 8). Overall, the evolution of this parameter showed slight decreases in values post-filtration for each analysed sample, with only 50% of them falling within the allowed range.

The obtained data is closely related to other research conducted in this field, especially the ones that investigated the use of clinoptilolite in the treatment of certain categories of wastewater [16-18].

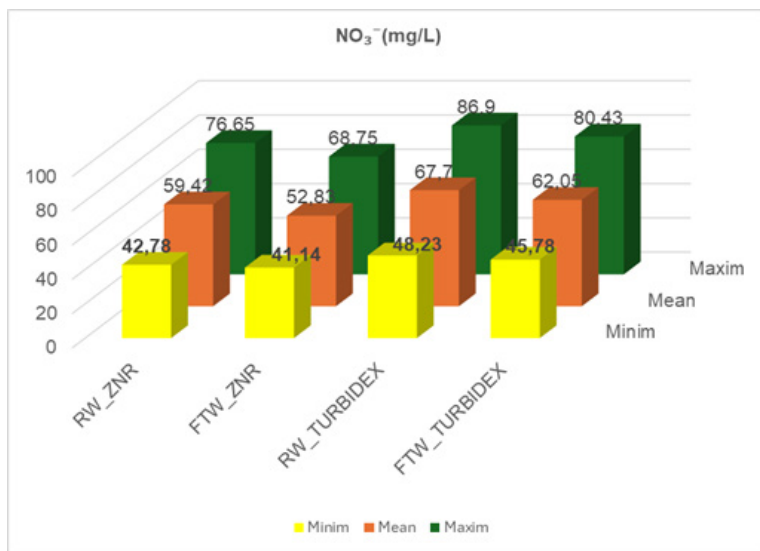


Figure. 8. The evolution of nitrates of wastewater [RW] and filtered/treated water [FTW] on ZNR and Turbidex filtering media

Analysis of microbiological parameters

This analysis included monitoring of indicative microbiological contaminants: *E. coli*, coliform bacteria, enterococci, the total colony count (CFU) at 22°C and the total colony count (CFU) at 37°C. Microbiological parameter testing revealed notable fluctuations in the obtained values, which are presented in (Table 1). The microbial load recorded in the case of influent for the ZNR substrate of the experimental station was characterised by 7 positive tests for coliform bacteria and enterococci, a level of 701.1 CFU/100 mL for the total colony count at 22°C parameter and 1132.7 CFU/100 mL for colonies at 37°C.

Table 1. Results from microbiological parameters testing of wastewater

Parameter	<i>Escherichia coli</i> (No/100mL)		B. Coliform (No/100mL)		Enterococci (No/100mL)		The total colony count at 22°C (CFU/mL)		The total colony count at 37°C (CFU/mL)	
MAC*	0		0		0		No abnormal changes		No abnormal changes	
Unit	RW	FTW	RW	FTW	RW	FTW	RW	FTW	RW	FTW
(A) Wastewater – ZNR substrate										
I	0	0	+	0	0	0	1025.0	323.0	1848.7	15.5
II	0	0	+	0	+	+	362.0	210.2	1264.6	12.0
III	0	0	+	+	+	0	865.3	163.0	1298.6	252.0
IV	0	0	+	0	+	0	820.0	23.4	711.7	20.0
V	0	0	-	-	0	0	736.2	25.0	1077.4	565.5
VI	0	0	0	0	-	-	398.0	12.0	595.4	171.6
Mean	0	0	+	0	+	0	701.1	126.1	1132.7	127.8
Maximum	0	0	+	+	+	+	1025.0	323.0	1848.7	565.5
Minimum	0	0	0	0	0	0	362.0	12.0	595.4	12
Standard deviation	-	-	-	-	-	-	266.1	127.3	453.1	216.5
RR [%]							82.01		88.71	
(B) Wastewater – Turbidex substrate										
VII	0	0	+	0	+	0	351.0	0	759.9	115.5
VIII	0	0	+	+	+	+	320.5	210.2	720.2	145.0
IX	0	0	+	0	+	0	315.2	163.0	731.5	123.0
X	0	0	+	+	+	+	220.0	23.4	578.4	115.3
XI	0	0	+	0	+	0	400.5	25.0	646.4	135.0
XII	0	0	0	0	0	0	180.7	12.0	358.9	110.3
Mean	0	0	+	0	+	0	297.9	72.3	632.6	124.7
Maximum	0	0	+	+	+	+	400.5	210.2	759.9	145.0
Minimum	0	0	0	0	0	0	180.7	0	358.9	110.3
Standard deviation	-	-	-	-	-	-	82.4	90.3	149.5	51.8
RR [%]							75.73		80.39	

RR- reduction rate, MAC*-Maximum admitted concentration [36]

The comparative analysis of the microbiological parameters of the effluent filtered by ZNR revealed that in 5 out of 7 tests, no coliform bacteria and enterococci were identified, and the total colony count at 22°C was reduced to 126.1 CFU/100 ml, respectively, to 127.8 CFU/100 ml at 37 °C.

Comparatively, raw wastewater from the Turbidex model system yielded positive results for both coliform bacteria and enterococci, which were recorded in 10 tests. The total colony count, following incubation at 22°C, was determined to be 297.9 the total colony count per 100 mL (CFU/100 mL) and respectively of 632.6 CFU/ 100 ml at 37°C.

Following filtration, neither coliform bacteria nor enterococci were detectable within 6 of the treated water samples. Moreover, total colony counts performed at 22°C and at 37°C, respectively, were significantly reduced (72.3 and 40.7 CFU/100 mL).

CONCLUSIONS

This study demonstrates that natural Rupea zeolite (ZNR) and the zeolitic substrate Turbidex are effective filter media for the treatment of wastewater from small agro-zootechnical holdings. Comparative results showed minor differences between the two materials, with ZNR exhibiting slightly higher adsorption for conductivity, ammonium, nitrites and nitrate, while Turbidex achieved superior retention of COD, turbidity, and total suspended solids. Both media significantly reduced microbial load, including complete removal of coliform bacteria and enterococci, and a marked decrease in colony counts at 22°C and 37°C.

These findings highlight the overall efficiency of zeolite-based filtration under pilot-scale conditions and emphasize the importance of optimizing operational parameters.

The integration of Rupea zeolite- and Turbidex-based filtration systems offers a sustainable approach for agro-technical wastewater management, supporting internal recycling and safe reuse of treated effluents for irrigation. Such systems contribute to environmental protection, nutrient recovery, and compliance with European and Romanian water quality standards, while aligning with international efforts to promote resource efficiency and sustainable water management.

EXPERIMENTAL SECTION

Wastewater samples

The study investigated the efficiency of eco-friendly filter materials, natural Rupea zeolite and Turbidex, for the sustainable management of wastewater originating from small agro-zootechnical units located in rural areas of the Someșul Mare river basin (Cluj and Bistrița-Năsăud counties). Research was conducted on three commercial micro-farms and nine traditional agricultural households, all discharging wastewater either into surface waters or onto adjacent land. Table 2 summarizes the coding and technological characteristics of the source units.

Table 2. The identification of wastewater [Ww] samples based on origin

Code water sample	Unit (Type)	Technological traits
I/ Ww	Micro farms	Young cattle fattening facility with septic tank
II/ Ww		Swine fattening facility without septic tank
III/ Ww		Dairy sheep farm, without septic tank
VI/ Ww	Farmstead	Mixed- swine and poultry with manure platform
V/ Ww		Mixed-lactating cows and swine with manure platform
VI/ Ww		Mixed-lactating cows and swine with manure platform
VII/ Ww	Farmstead	Mixed- swine and poultry without manure platform
VIII/ Ww		Mixed-lactating cows, with dairy facility and manure platform
IX/ Ww		Mixed- swine without manure platform
X/ Ww		Mixed- lactating cows with dairy facility and manure platform
XI/ Ww		Mixed- lactating cows, with dairy facility and manure platform
XII/ Ww		Mixed- swine and poultry without manure platform

The performance of the filter media was evaluated through key physicochemical parameters, including pH, electrical conductivity, turbidity, total suspended solids (TSS), chemical oxygen demand (COD), ammonium, nitrites, and nitrates. Additionally, the reduction of microbiological contaminants was closely monitored to assess the overall treatment effectiveness.

These indicators were specifically selected due to their critical role in determining the feasibility of reusing treated effluents in agricultural applications.

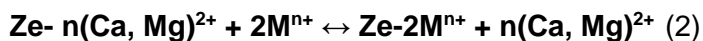
The filter media (ZNR and Turbidex)

The selection of zeolitic filter media for the treatment of agro-zootechnical wastewater was based on their inherent adsorption capacity, ease of regeneration, and potential for subsequent reuse as soil amendments. These considerations, supported by extensive literature, provided the basis

for investigating Rupea natural zeolite (ZNR) and the Turbidex substrate as adsorbent media in a pilot-scale wastewater treatment facility.

Rupea natural zeolite (ZNR) originates from a significant geological deposit in Romania and has well-established applications in water treatment, wastewater purification, environmental protection, and agriculture. ZNR primarily contains clinoptilolite, which exhibits a high ion-exchange capacity, making it suitable for the removal of a wide range of pollutants.

The adsorption mechanism involves the exchange of mobile cations (Na^+ , K^+ , Ca^{2+} , Mg^{2+}) within the zeolitic framework with cations (M^{n+}) present in the external solution, according to the following general relationship [29]:



The following two tables summarize the main compositional, mineralogical and physicochemical characteristics which are the basis of the ZNR characterisation [25, 31].

Table 3. Physicochemical characteristics of ZNR

Physical characteristics		Chemical composition [%]	
Softening point [°C]	1250	SiO_2	68.75-71.3
Melting point [°C]	1320	Fe_2O_3	1.90-2.1
Melting temperature [°C]	1400	Al_2O_3	11.35-13.1
Color	Grey-green	MgO	1.18-1.20
Smell	Odorless	CaO	2.86-5.2
Porosity (%)	32-44	Na_2O	0.82-1.30
Porous diameter (nm)	0.4-0.6	K_2O	3.17-3.40
Water hardness [Mohs scale]	3.5-4	Loss on ignition	8.75-8.86
pH [-]	8.75		
Density [Tons/m ³]	2.377		

Table 4. Mineralogical composition of ZNR

Evaluation type	Parameter / The principle of the test		UM	Values
Mineralogical composition	Clinoptilolite		%	87-90
	Plagioclase		%	2-5
	Anhydrite		%	2-3
	Cristobalite		%	4-5
Absorbent capacity	Pb	Absorption rate at different time intervals	%, 1a 2h	98,28
	Cd			99,51
	Cr			99,91
	Cu			99,46

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Evaluation type	Parameter / The principle of the test		UM	Values
Cation exchange capacity	Ca ²⁺	Determination of cationic adsorption or substitution and electrical charge differences	meq/100g	237.5
	Mg ²⁺			160.4
	K ⁺			38.4
	Na			39

Initially, the manufacturing company provided the study with three commercial series of ZNR, distinguished by size of granulation (0.5-1.5mm, 1.5-3 mm, and 3-5 mm). Following the preliminary literature reviews, the variant exhibiting the finest granulation (0.5-3 mm) was selected for inclusion in this study [9, 25, 32]. For the initial stage of the preparation of zeolite samples intended for laboratory investigations, a facile procedure, as delineated by Zeolites Group and corroborated by other pertinent research within this domain, was implemented [9,32]. This protocol involved the laving of the granulated zeolite with distilled water until the water remained clear, followed by desiccation at a temperature of 105°C, subsequent cooling, and finally, storage within a desiccator [17].

Turbidex is a naturally occurring zeolite extensively utilized in the United States of America due to its exceptionally high clinoptilolite content. It is identified by a characteristic chemical formula:



The expression of the adsorbent potential of this clinoptilolite medium, which is dictated by its distinct physical characteristics (ion exchange), is also influenced by the appropriate sizing and adaptation of the filter bed to the dimensions of the tank in which it is employed [46].

Table 5. The identification and composition of Turbidex zeolite substrate

	Characteristics
Chemical name	Clinoptilolite Zeolite/Potasium, Calcium, Sodium Aluminosilicate, Hydrated
Chemical formula	$K_2, Ca_2, Na_2)O \cdot Al_2O_3 \cdot 10SiO_2 \cdot 8H_2O$.
CAS Registry	12173-10-3
Natural zeolites granules	100%
Density	2.2.-2.4 Tons/m ³

Experimental plant and filtration of wastewater samples

The purification potential of zeolitic filter media was evaluated using an experimental water treatment station (Figure 6). The pilot plant featured a height of 750 mm and a zeolite filter volume of 40 L, operating at a filtration pressure of 3 bar and an influent flow rate of $1 \text{ m}^3 \cdot \text{h}^{-1}$. Filtration through both zeolite materials was performed under identical operational conditions, with at least two composite samples collected for analysis before and after each cycle. Each filtration cycle lasted approximately 2 hours and was followed by regeneration of the zeolitic medium.

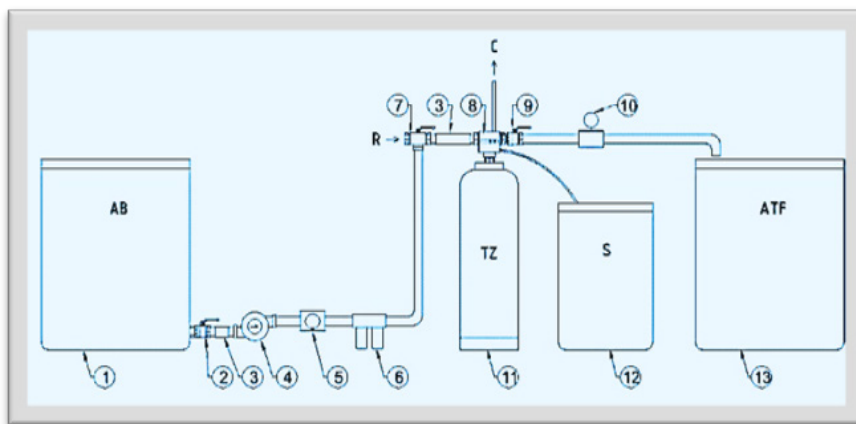


Figure. 9. The general blueprint for the experimental wastewater treatment plant: 1-raw wastewater recipient tank (AB); 2-faucet; 3-flexible connection; 4-pump; 5-electro pressure switch; 6-filter; 7-three-way valve Dn; 8-electro valve; 9-faucet; 10-water meter; 11-zeolite tank (TZ); 12-brine tank (S); 13-filtered water tank (WTF); R-water network; C-sewer discharge.

Methodology used for wastewater sample testing

The samples were organized into two experimental models, based on wastewater type and filter medium: Wastewater – ZNR (A) and Wastewater – Turbidex (B). Collected wastewater samples were transported to the laboratory and stored under refrigeration at 4°C until analysis.

Qualitative assessment was carried out by comparing the obtained data with the limit values established by the relevant national legislation in the field. The reliability of the chemical analyses was ensured through the use of high-purity reagents (Merck, România) and, certified experimental methods, alongside laboratory equipment validated for high-precision chemical measurements.

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Both influent and effluent were characterized to evaluate the treatment performance of Rupea Zeolite and Turbidex as filtration media.

The following table summarizes the analytical methods utilized for the monitored indicators.

Table 6. Tested parameters and the used methods

No.	Parameter [UM]	Method and equipment	NTPA- 001/2002
1.	pH [U. pH]	SR ISO 10523:2012; Water quality. Determination of pH, Romanian	6.5 – 8.5
2.	EC [mS/cm]	SR EN 27888:1997; Water quality. Determination of electrical conductivity	-
3.	Turbidity [NTU]	SR EN ISO 7027-1:2016. Water quality. Determination of turbidity. Partea 1:	-
4.	COD ⁻ [mg/L]	SR ISO 6060-96 Water quality Determination of the chemical oxygen demand	125.0
5.	TSS ⁻ [mg/L]	STAS 6953-81: Water Quality. Determination of total suspended solids	35.0 (60.0)
6.	NH ⁴⁺ [mg/L]	SR ISO 7150-1: 2001: Water Quality. Determination of ammonium content	2.0 (3.0)
7.	NO ²⁻ [mg/L]	SR EN 26777:2002/C91:2006; Water Quality. Determination of nitrite content. The Method by Molecular Absorption Spectrometry.	1.0 (2.0)
8.	NO ³⁻ [mg/L]	SR ISO 7890-3/2000; Water Quality: Determination of nitrate content: PART 3. Sulfosalicylic acid spectrometric method).	25 (37)
9.	<i>Escherichia coli</i> [CFU /100mL]	ISO 9308-1; Water quality. Enumeration of <i>Escherichia coli</i> and Coliform bacteria. Part 1: Membrane filtration method	0
10.	Total coliforms [CFU/100mL]	SR EN ISO 9308:1:2015/A:2017 Water quality. Enumeration of <i>Escherichia coli</i> and Coliform bacteria. Part 1: Membrane filtration method	<10
11.	Enterococci [CFU/100mL]	SR EN ISO 7899-2/2002. Water quality. Detection and enumeration of intestinal enterococci. Part 2: Membrane filtration method. (The determination was made after incubating the membrane, 48 h at 36°C, on Slanetz Bartley medium)	-
12.	The total colony count at 22°C [CFU/m]	SR EN ISO 6222:2004 Water quality. Enumeration of culture microorganisms. (Colony counting by seeding in agar-agar culture medium at a temperature of 22°C, incubated for 72 hours	-
13.	The total colony count at 37°C [CFU/m]	SR EN ISO 6222:2004: Water quality. Enumeration of culture microorganisms. (Colony counting by seeding in agar-agar culture medium at a temperature at 37°C incubated for 24 hours).	-

The adsorption capacity of the zeolitic substrates was evaluated by determining the retention rate of the analysed indicators, according to the following equation

$$R[\%] = \frac{C_i - C_f}{C_i} \times 100$$

where:

C_i represents the initial concentration (mg/L) of the analysed pollutant.

C_f represents the final concentration (mg/L) of the analysed pollutant.

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