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EVALUATION OF NATURAL ADSORBENTS IN THE REMOVAL OF AMMONIA AND OTHER NITROGEN COMPOUNDS IN WASTEWATER FROM THE VIACHA WASTEWATER TREATMENT PLANT

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ABSTRACT

This study evaluated the adsorption capacity of natural materials, namely limonite and orange peel, for the removal of ammonium and nitrogen compounds from wastewater at the Viacha Wastewater Treatment Plant (WWTP). Water physicochemical parameters were analyzed from August 2022 to November 2023, in a collaboration between IIDEPROQ and EMAPAV under the COSUDE CVG415 project. The results revealed high concentrations of ammoniacal nitrogen, with an average IQR of 119.5 ppm in the influent and 75.5 ppm in the effluent, alongside irregularities in the oxygen supply of the oxidation ditch. Orange peel exhibited limited adsorption, whereas limonite achieved a maximum removal of 45.5% under optimal conditions (pH 5.7, particle size 2.36 mm, and equilibrium time of 99.4 minutes). These findings support the implementation of a trickling biofilter as a tertiary treatment to enhance nitrification. Although natural adsorbents show potential for removing nitrogenous contaminants, their use at the Viacha WWTP is constrained by the high concentrations of these compounds. This study provides key data on non-conventional technologies for wastewater treatment in local contexts, emphasizing the importance of exploring innovative and sustainable solutions.

Keywords: Adsorption, sustainable solutions, ammonium removal, nitrification percolating filter, WWTP (Viacha).

1. INTRODUCTION

Water is an essential natural resource for life and socioeconomic development, especially in rural communities and growing cities, where access to basic services such as drinking water and sanitation is limited. In many regions of Bolivia, including rural and peri-urban areas, the lack of adequate wastewater treatment infrastructure has generated significant water pollution problems. This situation not only affects water quality but also poses a risk to public health and local ecosystems (INE, 2012).

Viacha, a municipality located in the province of Ingavi, department of La Paz, exemplifies this problem. With an estimated population of over 80,000, Viacha faces challenges related to wastewater management, particularly due to the discharge of domestic and industrial effluents into nearby water bodies, such as the Pallina River and Cohana Bay, which flows into Lake Titicaca. Recent studies have documented the enrichment of nutrients, especially nitrogen compounds, in these water bodies, leading to trophic changes, reduced biodiversity, and eutrophication events (Duwig et al., 2014; Archundia et al., 2016; Lazzaro et al., 2016).

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Figure 1. Wastewater inlet and outlet

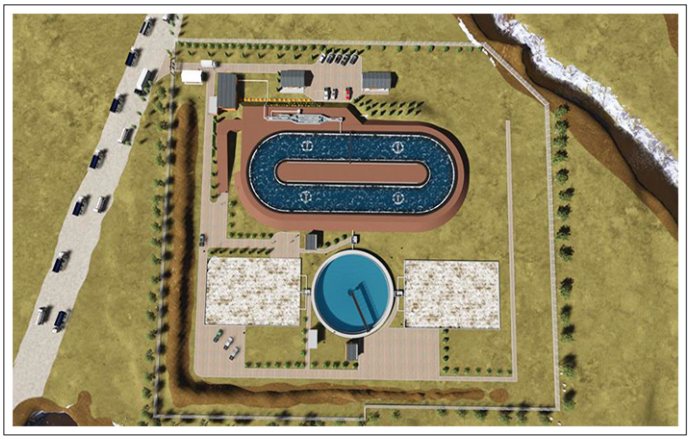


Source: Author's own work, photographs taken during water sampling

Nitrogen pollution, in the form of ammonium (NH_4^+), nitrites (NO_2^-), and nitrates (NO_3^-), is particularly concerning due to its environmental and human health impacts. Ammonium, for example, can transform into nitrites and nitrates, compounds that, in high concentrations, are associated with diseases such as methemoglobinemia (known as "blue baby syndrome") and potential cancer risks due to the formation of nitrosamines (Mohseni-Bandpi et al., 2013; Battas et al., 2019). Furthermore, the presence of these compounds in surface and groundwater, such as the Purapurani and Viacha aquifers, poses an additional challenge to the sustainable management of water resources in the region (MMAyA/VRHR, 2016).

In this context, the effluent discharge, as shown in Figure 1, exhibits a characteristic green color associated with eutrophication processes, indicating high concentrations of ammonium, nitrites, and nitrates discharged into the Pallina River. This exacerbates eutrophication problems in Cohana Bay and Lake Titicaca. In this scenario, the Viacha Wastewater Treatment Plant (WWTP), shown in Figure 2, plays a crucial role in mitigating water pollution. However, the plant faces technical limitations, such as the high organic load of the effluent, the presence of fats, and the inhibition of nitrifying bacteria due to the wide temperature variations characteristic of the Bolivian Altiplano. These factors hinder the efficient removal of nitrogen compounds.

Figure 2. Plan view of the WWTP (Viacha)



Source: Extracted from Consultoría y Construcción Gómez Dants (2015). Estudio TESA PTAR Viacha [Technical drawing No. 4]

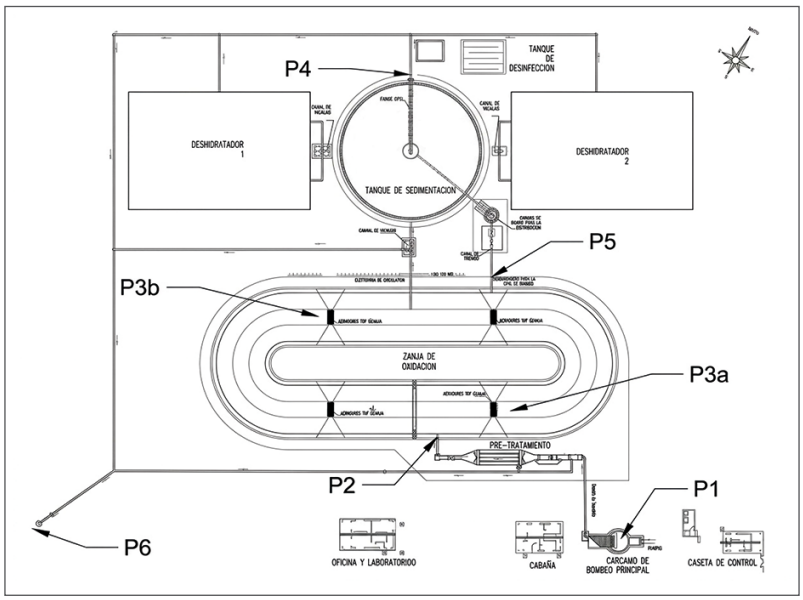
Given this problem, the main objective of this research is to evaluate the adsorption capacity of natural materials (limonite and orange peel) to remove ammonium and other nitrogen compounds from the wastewater of the Viacha Wastewater Treatment Plant. The results of this study aim to lay the groundwork for the development of innovative and sustainable technologies, thus contributing to improving the efficiency of wastewater treatment in the region, protecting local ecosystems, and safeguarding the health of surrounding communities.

2. MATERIALS AND METHODS

2.1. Wastewater monitoring at the Viacha WWTP

Wastewater monitoring is essential to ensure the quality of treated water and protect the environment. Specific monitoring points, shown in Figure 3, were established to collect accurate data on nitrogen compounds, pollutants, and other water quality indicators. Monitoring was conducted weekly from August 2022 to November 2023, ensuring continuous and detailed tracking.

Figure 3. Wastewater monitoring points



Source: Adapted from Manual de operación y mantenimiento: Planta de tratamiento de aguas residuales y emisarios (PTAR) - Viacha (p. 106), by ECOBLAD S.R.L., 2022.

The samples were collected following standardized procedures based on the Bolivian Standard NB/ISO 5667-10:2019 – Water Quality - Sampling of Wastewater, guaranteeing the consistency and reliability of the data.

2.2. Determination of physicochemical parameters

The physicochemical parameters of the wastewater were analyzed both in situ and in the laboratory; a summary is shown in Table 1.

Table 1. Parameters and methods used

Parameter	Method
Ammoniacal Nitrogen	SM-4500B SM-4500E HACH 8155 HI93733
Nitrate	HI93728
Nitrite	HI93708
Biochemical Oxygen Demand	SM 5210 D
Chemical Oxygen Demand	SM 5220 B
pH Temperature Turbidity Electrical Conductivity Oxidation-Reduction Potential Dissolved Oxygen	HI 9828

Source: List of parameters in APHA (2017). Analytical procedures from HACH (DR890) and Hanna (HI 9828) spectrophotometer manuals.

2.3. Methods for analysis

In the adsorption experiments and wastewater analysis at the Viacha Wastewater Treatment Plant (WWTP), both standardized methods (Standard Methods) and abbreviated methods (HACH, Hanna) were used. Wastewater analyses were performed at the Viacha WWTP using abbreviated methods, while adsorption analyses were carried out at IIDEPROQ using standardized methods, due to the availability of specialized materials and reagents.

2.3.1. Determination of Ammoniacal Nitrogen (NH_3-N)

2.3.1.1. Distillation Method

The SM-4500B method (distillation) followed by the SM-4500E method (titration) was used to determine the concentration of NH_3-N, the arrangement can be seen in Figure 4.

Figure 4. Working distillation equipment arrangement (IIDEPROQ)



Source: Author's own work, photograph taken at IIDEPROQ laboratories.

The process involved distilling a 100 mL sample of wastewater with borate buffer solution (pH 9) and boiling chips. The distillate was collected in boric acid and titrated with sulfuric acid (0.02 N H_2SO_4) to determine the concentration of NH_3-N using the formula:

$$N - NH_3 \left[\frac{mg}{L}\right] = \frac{(a - b) * 280}{V}$$

Where:

a= mL de H_2SO_4 0.02N used to titrate the sample
b= mL de H_2SO_4 0.02N used to titrate the sample
v= volume of the analyzed sample
280=14 x normality H_2SO_4 x 1000ugN

2.3.1.2. Rapid methods (HACH and Hanna)

Two spectrophotometric methods were used to determine ammonium. Using the HACH instrument, the 385 N, Ammonia, Salic program was used, preparing samples with salicylate and ammonium cyanurate reagents and measuring the absorbance after 15 minutes. Using the HANNA instrument, the Ammonium HR method was applied, adding reagents HI93733A-0 and HI93733B-0 and taking the measurement after 3 minutes and 30 seconds.

2.3.2. Determination of nitrates (NO_3^-)

The HANNA method for nitrates was used. The HI93728-0 reagent was added to the sample and the absorbance was measured after 4 minutes and 30 seconds.

2.3.3. Statistical analysis (Interquartile Range - IQR)

Interquartile Range (IQR) analysis was applied to assess the variability of the data for the analyzed parameters. This analysis allowed for the identification of outliers and predominant concentrations of nitrogenous pollutants.

2.4. Operational analysis of the Viacha Wastewater Treatment Plant

The performance of the activated sludge system was evaluated using a mass balance, considering the oxidation ditch and the settling tank as complete mixing reactors.

2.4.1. Adsorption study with biosorbents

2.4.1.1. Preparation of biosorbents

Two natural adsorbents were prepared: orange flavedo (Citrus sinensis), which was washed, dried at 60 °C, crushed and sieved (#100 mesh ASTM), and limonite, used in granule form and dried at 60 °C until a constant weight was reached.

2.4.1.2. Adsorption tests

Preliminary Tests: The adsorption of NH_3-N in synthetic solutions (10-100 mg/L) was evaluated with orange peel and limonite, varying pH, particle size, adsorbent mass and stirring time.

Experimental studies in batch reactors: Experiments were performed with limonite to evaluate the adsorption kinetics, varying the contact time (0.5, 1, 2, 4 and 8 hours).

2.5. Design of a percolating biofilter

A trickling biofilter was designed based on the Albertson and Okey model (1988, as cited in Water Environment Federation, 2010), using limonite as the packing material. The design considered zero-order and first-order kinetics, with an NH_3-N flux of 1.2 g/m²·dy and temperature corrections. (Water Environment Federation, 2010).

3. RESULTS AND DISCUSSION

3.1. Characterization of wastewater (Influent)

Wastewater from Viacha exhibits medium to high levels of contamination, indicating a municipal origin with a significant nutrient and solids load. The presence of emerging contaminants such as pharmaceuticals and surfactants was identified, which are not efficiently removed by conventional systems. Furthermore, the need for continuous monitoring of fats and oils, which can affect the efficiency of biological treatment, is highlighted; the results are shown in Table 2.

Table 2. Comparison of wastewater characterization

Parameter	High (Metcalf-Eddy)	High-Medium (Viacha)
BOD5 (mg/L)	400	246.9
COD (mg/L)	1000	377.8
ammoniacal N (mg NH3-N/L)	50	119.5
N nitrate mg (mg NO_3-N/L)	0	11.3
PT (mg P/L) (*)	15	14.25
Total Solids (TS)	1200	1219.2
Total Dissolved Solids (TDS)	850	802.3
Fixed Solids (mg/L)	525	530.7
Volatile Solids (mg/L)	325	549.7
Suspended solids (SS) (mg/L)	350	421.8
Total coliforms (MPN/100 mL) (*)	10^7-10^9	3.5 x 10^5

Reference: values from Metcalf & Eddy, (1998)

3.1.1. Evaluation of oxygen supply in the oxidation ditch

The oxygen supply provided by rotary aerators is insufficient to meet the demand for the degradation of organic and nitrogenous matter. The four aerators operating continuously would supply 1161.6 kg O_2/day, while the demand is 1231.8 kg O_2/day, resulting in a deficit of 70.2 kg O_2/day. Considering the recommended operating regime, the available oxygen is even lower, which negatively impacts the nitrification and ammonia removal processes.

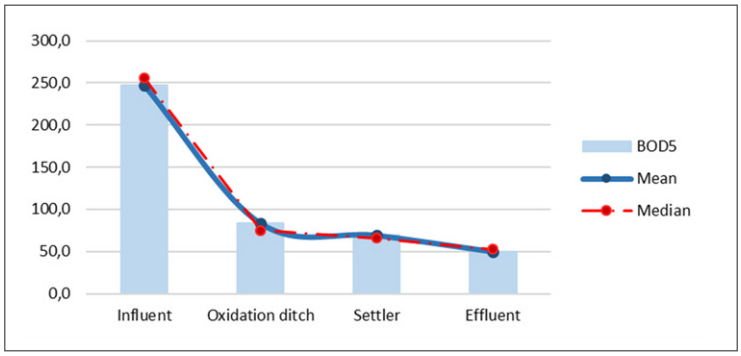
Improvement in the aeration system

To optimize oxygen transfer and improve treatment efficiency, the implementation of an aeration system with fine bubbles and mechanical agitators is recommended, which can increase oxygen dissolution efficiency to 6.6 kg O_2/h. Furthermore, considering the population growth of Viacha (113,453 inhabitants, INE 2024), the expansion of the wastewater treatment plant and the implementation of carousel-type oxidation ditches are suggested to guarantee future treatment capacity.

3.1.2. Catabolism of carbon and nitrogen compounds

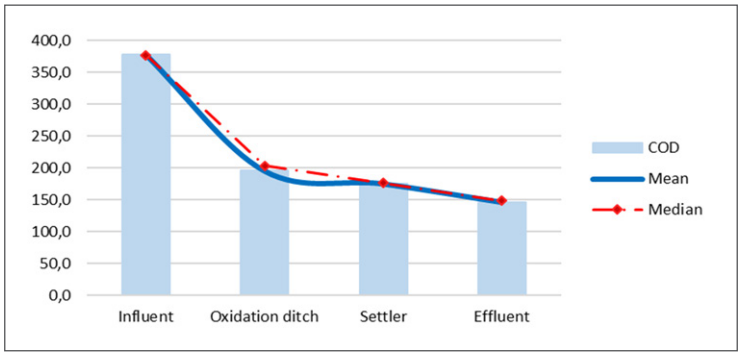
The degradation of pollutants in wastewater was assessed by measuring the reduction of BOD and COD, the variations of which are shown in Figures 5 and 6. However, insufficient oxygen in the process limits removal efficiency, suggesting the need to increase the hydraulic retention time. The lack of flow data prevented the calculation of system efficiencies, although trends in water quality were identified. The oxidation ditch showed the greatest reduction in organic matter due to the oxygen supply.

Figure 5. DBO5 Trend line



Source: From Ticona (2025, p. 91)

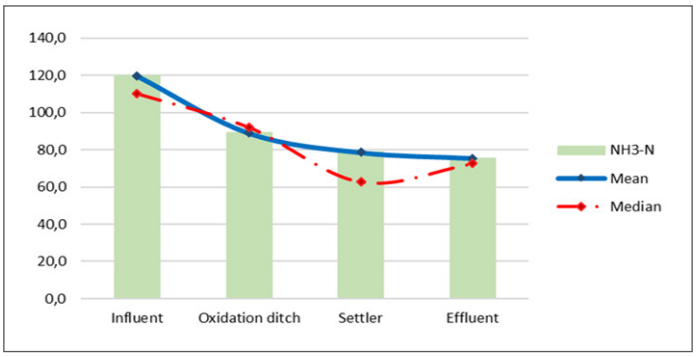
Figure 6. COD Trend line



Source: From Ticona (2025, p. 91)

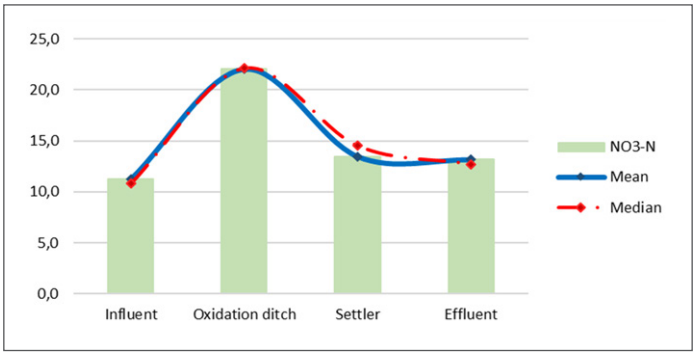
Nitrification was observed in two stages: conversion of NH_3 to NO_3^-, as shown in Figures 7 and 8, with low detection of NO_2^- due to its immediate conversion. Denitrification occurred in anoxic zones within the oxidation ditch. The activated sludge process exhibited limited nitrification due to the metabolic priority of carbonaceous compound degradation.

Figure 7. NH3-N Trend line



Source: From Ticona (2025, p. 91)

Figure 8. NO3-N Trend line



Source: From Ticona (2025, p. 91)

Reported optimal conditions for nitrification include temperature (~30°C), pH (7.2-8.5), and adequate oxygenation. However, the average temperature in the ditch was ~13°C, affecting the efficiency of the process. Hydraulic problems were identified in the settling tank, where the lateral inlet creates dead zones with solids accumulation, affecting contaminant removal and turning some areas into anaerobic reactors. Furthermore, inadequate control of the hydraulic level between the oxidation ditch and the sedimentation tank could allow the discharge of untreated water.

3.2. Preliminary tests with orange peel

The orange peel biosorbent showed an equilibrium time of 60 minutes, with rapid adsorption in the first 30 minutes due to the high surface area available. However, the adsorption capacity was limited, reaching a maximum removal of 7.9% for 0.15 mm particles (ASTM #100) and only 1.3% for 2.36 mm particles (ASTM #8).

These low values are attributed to the reduced surface area in larger particles and the material's permeability limitations, especially under acidic pH conditions (pH 5.5), which are incompatible with the neutral to basic pH of Viacha wastewater.

3.3. Experimental tests with limonite

The highest amount of ammonia adsorbed by limonite was reached at 240 minutes; however, from 90 minutes onwards, the capacity remained constant, reaching equilibrium. The experimental conditions are summarized in Table 3.

Table 3. Experimental kinetic conditions

Parameter	Value	Unit
pH	5.7	
Particle size	2.36	mm
Dose	4.1	g
Time	99.4	min
% Removal	45.4	%

Source: From Ticona (2025, p. 91)

The adsorption kinetics of limonite fit the pseudo-second-order model (Ho & McKay, 1999), with a correlation coefficient (R^2 = 0.989). The equilibrium adsorption capacity was 3.39 mg NH_3/g, confirming the material's effectiveness. The selected kinetic equation was:

$$q_t = \frac{t}{0.15 + \frac{t}{3.39}}$$

Donde:

q_t: Total amount of biosorbate adsorbed at any time (mg/g)
t: time (min)

Regarding the adsorption isotherm, the Langmuir model (Langmuir, 1918) showed a maximum adsorption capacity (q_max) of 49.75 mg NH_3/g, with moderate affinity (Langmuir constant b = 0.342 L/mg). The fit was optimal (R^2 = 0.9859), validating its use to describe the process. The selected Langmuir equation was:

$$q_e = \frac{17.01 * C_e}{1 + 0.34 * C_e}$$

Donde:

q_e: amount of adsorbate in the adsorbent at equilibrium (mg/g)
C_e: equilibrium concentration in the liquid phase (mg/L)

3.4. Design of a percolating filter

Calculations were performed for different filter media in the trickling biofilter, following the Albertson and Okey (1988, as cited in Water Environment Federation, 2010) kinetic model for nitrification. This model allows the determination of the surface area of the media required for zero-order nitrification, using a kinetic constant kn=1.2. The calculations were based on the characteristics of each material, considering its specific surface area and average density.

Given the significantly high concentration of NH_3-N in the Viacha WWTP effluent, a recalculation was performed for a scenario in which the treatment reduces the NH_3-N by half, reaching a value of 25 ppm. This value represents a more realistic intermediate condition for the biofilter design. The initial data used for this calculation were:

Flow rate (Q):	50 L/s
NTK-Ni:	32 ppm
NTK-Ne:	1.5 ppm
NH_3-Ni:	25 ppm
DBO:	25 ppm
sDBO:	5 ppm
Temperature:	15°C

The results are summarized in Table 4:

Table 4. Calculated values for percolating biofilter

	River rock	Crossflow PVC Plastic	Granular limonite	Units
SAA	62.3	138	1791	m^2/m^3
D	10.58	10.58	10.58	m
P	1574	7.11	0.55	m
WHL	0.57	0.57	0.57	L/m^2*s
TOL	0.08	0.17	2.24	kg/m_3*d

Where: SAA, Specific Surface; D, Filter Diameter; P, Depth of the filter medium; V, Total volume of the filter medium; WHL, Wet hydraulic loading; TOL, Total organic loading.

The results showed that, under these conditions, the biofilter dimensions can be optimized. For example, for limonite, a diameter of 10.58 m and a depth of 0.55 m were obtained. These dimensions allow for the implementation of two smaller biofilters, each 5.3 m in diameter and of the same depth, which simplifies their construction and operation.

This approach not only improves treatment efficiency, but also offers design flexibility, adapting to the specific needs of the Viacha WWTP and contributing to the effective reduction of nitrogen compounds in the effluent.

4. CONCLUSIONS AND RECOMMENDATIONS

- The physicochemical characterization of the water from the Viacha Wastewater Treatment Plant (WWTP), conducted from August 2022 to November 2023, shows a high load in the wastewater compared to international literature standards. The average ammonia nitrogen value is excessively high (119.5 ppm) compared to the high ammonia nitrogen range of 50 ppm to 75 ppm, indicating that the inflow should be controlled by identifying the source of this high quantity of nitrogenous nutrients.

- According to the analysis of data from report LAB-042-2023 EMAPAV, the oxygen supplied to the wastewater in the oxidation ditch is insufficient for the inflow rate, the concentration of contaminants, and the volume of water treated. The required amount is 1231.8 kg O2/day, but the aerator brushes only provide 1161.6 kg O2/day at maximum operating capacity. This deficiency prevents the nitrogen compounds from being oxidized.

- Water quality analysis of the effluent revealed average BOD and COD values of 49.6 mg O_2/L and 146 mg O_2/L, respectively, while historical minimums have been recorded at 8 mg O_2/L and 23 mg O_2/L. These results do not reflect the treatment of the entire incoming water flow, as a deficiency in the oxygen parameter required during operation was detected. This deficiency prevents achieving the reported values, which are expected to result in high efficiencies when all the water is treated. To confirm this situation, rigorous monitoring of both the water entering the reactor and the untreated water discharged via the bypass into the Pallina River is essential.

- Hydraulic and sanitary systems are inextricably linked in wastewater treatment plant design. If the hydraulics are not functioning properly, wastewater quality is compromised, as observed in hydraulic system observations involving the removal of nitrogen compounds from wastewater. Hydraulic head and the design of the sedimentation tank in the wastewater treatment plant (WWTP) negatively impact operation, increasing nitrogen compounds and potentially turning the sedimentation tank into an anaerobic reactor before the water even leaves the WWTP.

- The natural biosorbent orange peel exhibits very low adsorption capacity even under optimal conditions, hindering scalability despite its low cost. Its application in wastewater is more complex, as wastewater contains microorganisms that adhere to and degrade the material, increasing the water's organic load.

- The limonite biosorbent showed a good removal result, which had a maximum of 45.5%, whose optimal operating parameters are pH 5.7, particle size 2.36mm, equilibrium time 99.4 min and it was determined to be useful for performing the removal of ammonia nitrogen under the optimal adsorption conditions.

- The effectiveness of natural adsorbents is influenced by pH, as the best ammonia adsorption results are achieved under acidic conditions. However, the wastewater treated at the Viacha Wastewater Treatment Plant tends to be alkaline. Furthermore, the small particle size, when chosen for a filter medium, must ensure permeability and cross-flow of air and wastewater. These considerations complicate the implementation of non-conventional technologies.

- Implementing a trickling biofilter as a tertiary treatment process in wastewater is a viable alternative due to its compact design and the use of efficient materials such as limonite, which not only adsorbs nitrogen compounds

but also promotes biofilm formation thanks to its high specific surface area. With a flow rate of 50 L/s and an intermediate concentration of 25 mg/L of ammonia nitrogen (NH3-N), and a filter medium 10.58 m in diameter and 0.55 m deep, this unit is designed to progressively reduce the concentration of ammonia nitrogen and, after decreasing the carbonaceous organic load, enhance biological activity, achieving more efficient removal of nitrogenous pollutants.

- It is recommended to adjust the frequency of monitoring parameter analyses, prioritizing an operational approach. Parameters such as solids, which are inexpensive to measure, provide key information for the daily operation of the system, allowing for optimized control and performance.

- It is recommended to implement additional oxygenation equipment to improve the process at the Viacha Wastewater Treatment Plant, as the current mechanical aeration is superficial and insufficient. Furthermore, homogenization using mixers submerged in the oxidation ditch is inefficient, as evidenced by the low dissolved oxygen levels, which decrease drastically at depths greater than 0.5 m.

5. ACKNOWLEDGMENTS

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6. ABBREVIATIONS

BOD_5, Biochemical Oxygen Demand at 5 days, (mg O_2/L)
COD, Chemical Oxygen Demand, (mg O_2/L)
NH_4^+, Ammonium ion, key nitrogenous pollutant, (mg/L)
NO_2^-, Nitrite ion, intermediate in nitrification (mg/L)
NO_3^-, Nitrate ion, nitrification product (mg/L)
N-NH_3, Ammoniacal nitrogen, main contaminant analyzed, (mg/L)
O_2, Dissolved oxygen, essential for nitrification, (mg/L or kg/d)
pH, Potential of hydrogen
qe, Amount of adsorbate in the adsorbent at equilibrium (mg/g)
SAA, Specific surface area of the filter material, m^2/m^3

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