








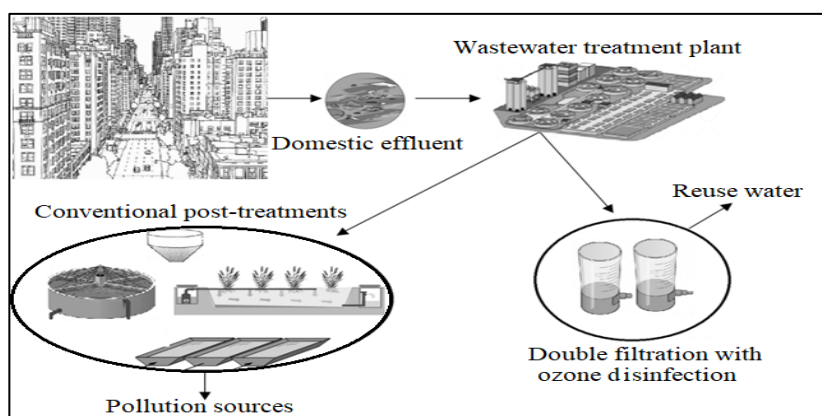
Full Paper | <http://dx.doi.org/10.17807/orbital.v13i4.1617>

Post-treatment of Anaerobic Reactor Effluent by Double Filtration with Gravel and Clinoptilolite and Ozone Disinfection

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Anaerobic reactors are an excellent alternative in the treatment of domestic effluents. However, this system presents the disadvantage of requiring post-treatment. Many technologies have been studied and rapid filtration systems have proven to be a viable alternative for the post-treatment of anaerobic reactor effluents. This study evaluates the efficacy of an anaerobic reactor effluent post-treatment system using double filtration (DF), which consists of an upward gravel filter and a downward filter of clinoptilolite, and disinfection by ozonation. The system was evaluated using physicochemical and microbiological parameters. The lowest ozone dose (0.4 mg L^{-1}) indicated possible oxidation of fractions of biodegradable organic matter with BOD removals above 95%. Higher ozone concentrations (5.0 and 7.0 mg L^{-1}) improved COD removal and total nitrogen removal. Regarding microbiological parameters, over 87% *E. coli* and 89% total coliforms could be removed only with the use of DF, while total inactivation was observed using doses above 5.0 mg L^{-1} ozone. The results showed that following the proposed treatment, the effluent treated with the DF/Ozonation combination meets the guidelines for reuse in toilets, irrigation of orchards, cereals, pastures, and other crops through runoff or by punctual irrigation system according to the NBR 13969/97. According to these results, DF using clinoptilolite followed by ozonation is a promising technology for the anaerobic reactor effluent post-treatment.

Graphical abstract



Keywords

Clinoptilolite
Double filtration
Post-treatment
Ozone

Article history

Received 14 April 2021
Revised 09 September 2021
Accepted 11 September 2021
Available online 30 September 2021

Handling Editor: Adilson Beatriz

1. Introduction

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The United Nations estimates that approximately 3.6 billion people around the world live in areas with potential water scarcity [1]. The irregular disposal of effluents in water bodies can compromise the use of water, since depending on the degree of contamination, treatment by conventional techniques aiming at public supply might be unfeasible [2]. This is because about 80% of the volumes of domestic and industrial effluents produced in the world are released into the environment without proper treatment [3].

Anaerobic reactors are technologies of sewage treatment plants consolidated in several countries [4,5]. The main advantages of such reactors are low sludge production, simplicity of operation, and reduced energy expenditure [6]. However, this technology is limited as it does not meet the effluent discharge standards adopted in several countries [7,8]. Therefore, the use of post-treatment systems becomes mandatory [6,9,10].

Several conventional techniques for post-treatment of effluents from anaerobic reactor such as polishing pond systems, biological filter, and activated sludge have been studied in recent decades [5,11–14].

Recently, the use of systems involving double filtration (DF) with an upward gravel filter and a downward sand filter in the post-treatment of effluent from anaerobic reactors has shown excellent results in the removal of several physicochemical parameters [9].

However, this system showed a limitation in the removal of ammoniacal nitrogen. As a result, the use of clinoptilolite to remove ammoniacal nitrogen from effluents was investigated and was found economically viable due to the low cost of large-scale production, high ammonia adsorption capacity, ease of operation, high biomass retention rates, as well as possible regeneration of the filter medium [15,16].

Advanced oxidative processes (POAs) and disinfection such as the use of ozone, hydrogen peroxide, and peracetic acid are efficient techniques that can improve the quality of the final effluent [17]. Ozone is a strong oxidizing agent being applied in wastewater treatment for the removal of drugs [18], algae [19], dyes [20] and elimination of bacteria, viruses, and protozoa [21,22].

Ozonation can be used in several stages of the effluent treatment. When used after coagulation or sedimentation, it aims to contribute to the oxidation of potentially toxic remaining substances, which will be more easily removed in the following steps [23,24]. The addition of ozone in the final stages of the process aims to convert part of the non-biodegradable organic matter into a biodegradable material [24,25]. In addition, one of the goals is to destroy microbial cells regardless of the stage in which ozonation is used [26].

This work employed physicochemical and microbiological analyses to evaluate the post-treatment of Upflow Anaerobic Sludge Blanket (UASB) reactor effluent by double filtration (DF) using gravel and clinoptilolite associated with ozone disinfection.

2. Results and Discussion

2.1 Experiments in Jar-Test

The ferric chloride concentration and coagulation pH used in the effluent treatment of the DF pilot unit were previously determined in bench tests using a Jar-test device. The main results are shown in Figure 1.

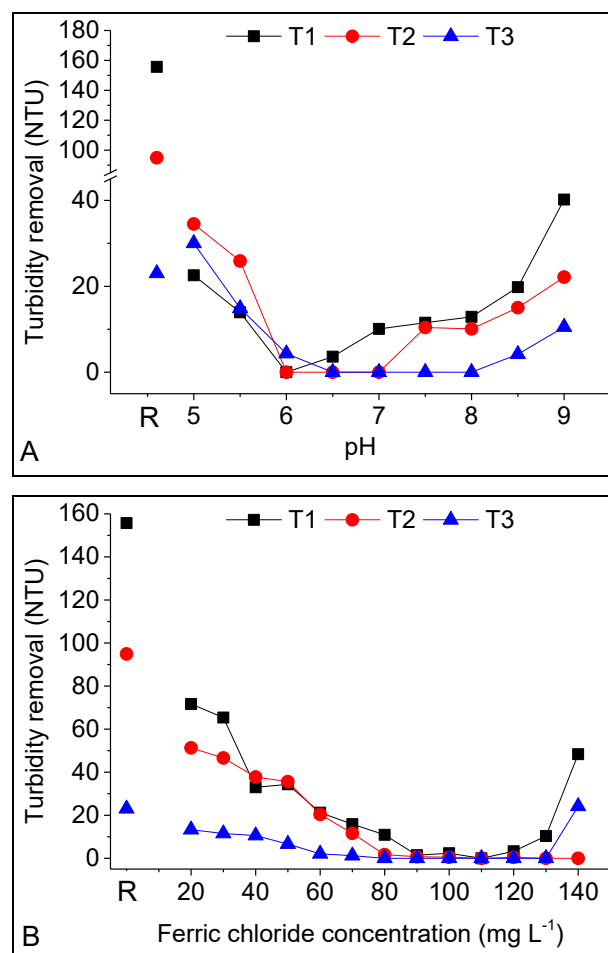


Fig. 1. Turbidity removal due to variations in pH and coagulant concentration. A: mixing gradient 300 s⁻¹; mixing time 15 s and ferric chloride concentration 80 mg L⁻¹; B mixing gradient 300 s⁻¹; mixing time of 15 s and effluent with natural pH 7.0 ± 0.2; R: corresponds to the initial turbidity condition of the anaerobic reactor effluent; T: corresponds to the evaluated treatment based on different turbidity conditions.

According to Figure 1-A, the best results obtained in the turbidity removal occurred at pH between 6.0 and 7.0. When the pH shifted to more acidic conditions, the turbidity removal efficiency was seen to decrease. At pH below 5.8, iron ions formed monomers of low molecular weight. In addition, excessive coverage of the surface of colloidal particles by negatively charged species occurred, resulting in the restabilization of suspended solids [27,28]. For pH values 6.0 and above, the restabilization was suppressed by improving coagulation and formation of sedimentable solids. This can be explained by a load destabilization mechanism that occurs on the colloidal surface, which is responsible for the formation of sufficient mass flakes to sediment [28]. The increase in pH above 7.0 favors the predominance of alkaline species, progressively increasing the restabilization of colloidal particles [27]. In addition, the increases in electronegativity at pH above 8.0 decreases the opportunities for contact by electrostatic repulsion [28], which decreases the efficiency of ferric chloride as a coagulant.

Considering the results obtained, we chose to use pH 7.0 for the post-treatment of anaerobic reactor effluent by DF and disinfection with ozone. Ozonation is more efficient in alkaline pH for which a reaction mechanism by the hydroxyl radical is characteristic of ozone [29,30]. However, increasing the pH to

basic conditions decreases the efficiency of the coagulant and increases operating costs due to the consumption of additional reagents for pH correction.

Regarding the coagulant concentration, the best results obtained in the turbidity removal (Figure 1-B) occurred in the range between 80 and 120 mg L⁻¹ ferric chloride. For concentrations above 130 mg L⁻¹, a reduction was observed in the turbidity removal efficiency, due to the progressive increase in the number of positive charges for each colloidal particle due to the increase in coagulant concentrations, which resulted in excessive adsorption and stabilization of colloids [27,28]. The variation range between 20 and 80 mg L⁻¹ ferric chloride corresponds to the range where the turbidity removal increases progressively. This behavior can be explained by the increased probability of adsorption between suspended solids and hydrolysis species [28,31]. The turbidity in anaerobic reactor effluents can vary considerably depending on operating and maintenance conditions, temperature, and raw effluent solids concentration, with values often varying from 25 to 150 NTU [7,32–34]. Thus, the 80 mg L⁻¹ concentration would be sufficient to meet several turbidity variations in anaerobic reactor effluents.

The complementary tests using a mixing gradient of 100 to 500 s⁻¹ and a 5.0 to 30.0 s rapid mixing time did not present significant differences. Since no significant variation was observed in the effluent quality improvement due to the variation of these parameters, we decided to operate the pilot system with the values already installed in the pilot unit (mixing gradient 300 s⁻¹ and rapid mixing 15 s), which made the construction of a new mixing system unnecessary. However, the adoption of a mixing system with a gradient of 100 s⁻¹ and a time of 5 s could be used without changing the efficiency of the system. Therefore, the conditions used for the other tests, namely 80 mg L⁻¹ ferric chloride concentration, effluent natural pH (~7.0), mixing gradient 300 s⁻¹, and rapid mixing 15 s were adopted in the next step.

2.2 Post-treatment tests by double filtration (DF) with ozone disinfection

The removal average for chemical oxygen demand (COD), biological oxygen demand (BOD), turbidity and total suspended solids (TSS) using double filtration with different ozone concentrations is shown in Figure 2.

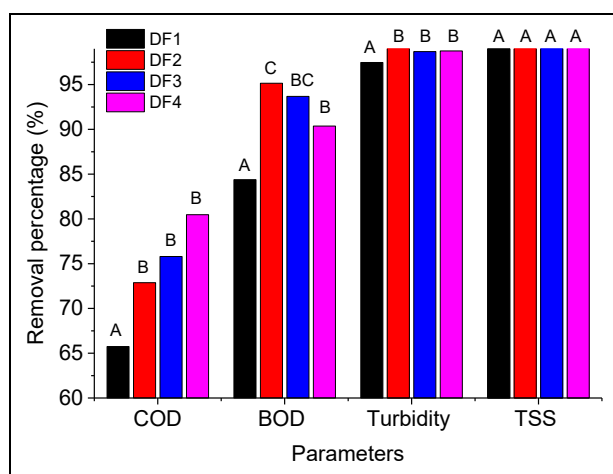


Fig. 2. Arithmetic mean of removal of COD, BOD, turbidity, and TSS parameters. DF1: Double Filtration; DF2: Double Filtration + 0.4 mgO₃ L⁻¹; DF3: Double Filtration + 5.0 mgO₃ L⁻¹; DF4: Double Filtration + 7.0 mgO₃ L⁻¹; Parameters with the same letter did not differ statistically.

The average COD concentration (Fig. 2) ranged from 150 to 51 mg L⁻¹ (66% removal) for DF1, from 148 to 40 mg L⁻¹ (79% removal) for DF2, from 150 to 36 mg L⁻¹ (76% removal) for DF3, and from 150 to 29 mg L⁻¹ (81% removal) for DF4. According to the analysis of variance, significant differences were observed in post-treatments by DF2; DF3 and DF4 when compared to DF1. However, among the treatments using ozone the variation of the means was not significant. Studies indicated that the COD of industrial wastewater can be halved in 5 min when using ozone [35]. In addition, ozonation of effluents treated by secondary systems for 5 min with 10 mg L⁻¹ ozone removed about 14% COD [36]. Secondary effluent treatments using 12 mg L⁻¹ ozone with 60 min of contact removed up to 52% COD [37]. These results suggest that the post-treatment using only DF was responsible for the greatest COD removal. When considering the ozone application, the increase from 0.4 to 7.0 mg L⁻¹ was observed to provide only 8% improvement in COD removal. The same trend was observed in domestic effluent post-treated by wetlands, and their effectiveness in removing COD decreased rapidly with the increase in ozone concentration [38]. According to Cavallini et al. [9], DF allows significant BOD removal. Thus, the COD seen in the post-treatment by DF with clinoptilolite may be less biodegradable, which contributes to the reduced effectiveness of this parameter by increasing ozone concentrations.

BOD average concentration ranged from 86 to 13 mg L⁻¹ (84% removal) for DF1, from 86 to 4 mg L⁻¹ (95% removal) for DF2, from 103 to 6 mg L⁻¹ (94% removal) for DF3, and from 103 to 10 mg L⁻¹ (90% removal) for DF4. According to the analysis of variance, significant differences were observed in the removal of this parameter in all different post-treatments. For lower ozone concentrations, greater efficiency in BOD removal was observed. This agrees with a similar study, where ozonation of domestic effluent post-treated by wetlands found improvement in BOD removal due to small concentrations of ozone, while larger amounts of ozone caused reduced efficiency [38]. A possible explanation for this behavior is that small concentrations of ozone can act in the breakdown of large organic molecules, transforming them into smaller and more easily biodegradable fractions [39]. Comparing the results of BOD removal using DF and peracetic acid presented by Cavallini et al. [9], in relation to DF and ozone, an improvement of about 15% was observed. In addition, these results are similar to those found in the post-treatment of anaerobic reactor effluent by triple filtration with activated carbon and calcium hypochlorite, which presented around 95% removal for the same parameter [5]. Much of the BOD was removed by the double filtration process with clinoptilolite, which is justified by its high porosity, ion exchange capacity, and adsorption of the material [15]. In addition, ozone favors the degradation of biodegradable organic matter as it is a powerful oxidizer, selectively reacting with organic substances either by direct reaction or indirectly via the hydroxyl radical [40].

Regarding turbidity, the average concentration was from 54 to 0.99 NTU (97% removal) for DF1, from 54 to 0.26 NTU (99% removal) for DF2, from 54 to 0.38 NTU (99% removal) for DF3, and from 54 to 0.36 NTU (99% removal) for DF4. Considering that the values obtained in all treatments were below 1.0 NTU, it is possible to state that the DF was efficient in removing suspended particles from the anaerobic reactor effluent. The high turbidity removal is consistent with the complete removal of total suspended solids (TSS). This can be confirmed when compared to the results of domestic effluent post-treatment by wetlands, which showed 60% in the

removal of turbidity using about 20 mg L⁻¹ ozone [38] and 58.9% for secondary domestic effluent treated with 17 mg L⁻¹ ozone for 120 min [41]. In addition, these results are better than those observed in the post-treatment of anaerobic reactor effluent using DF with ferrous sulfate as a coagulant and peracetic acid, which obtained the removal of approximately 90% turbidity [9].

The removal average for total nitrogen, ammoniacal nitrogen and total phosphorus using DF with different concentrations of ozone is shown in Figure 3.

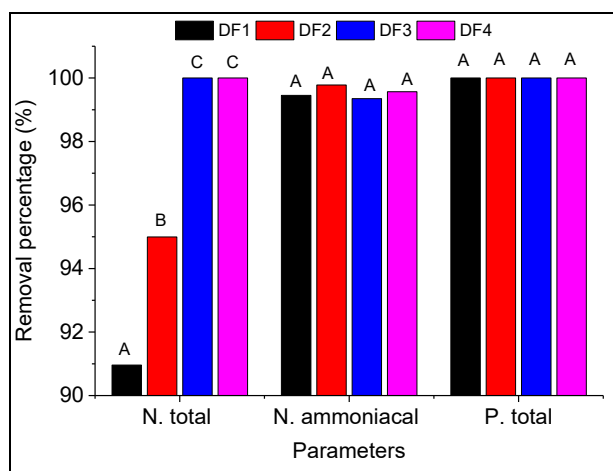


Fig. 3. Arithmetic mean of removal of total nitrogen, ammoniacal nitrogen and total phosphorus. DF1: Double Filtration; DF2: Double Filtration + 0.4 mgO₃ L⁻¹; DF3: Double Filtration + 5.0 mgO₃ L⁻¹; DF4: Double Filtration + 7.0 mgO₃ L⁻¹; Parameters with the same letter did not differ statistically.

Regarding total nitrogen (Fig. 3), the average concentration was from 73 to 6.5 mg L⁻¹ (91% removal) for DF1, from 73 to 3.6 mg L⁻¹ (95% removal) for DF2, from 73 to <0.5 mg L⁻¹ (>99% removal) for DF3, and from 73 to <0.5 mg L⁻¹ (>99% removal) for DF4. There was a significant difference in post-treatments by DF1 and DF2 in relation to DF3 and DF4. When considering ammoniacal nitrogen, the average concentration ranged from 46 to < 0.3 mg L⁻¹ (>99% removal) for DF1, DF2, DF3, and DF4.

Total nitrogen corresponds to various nitrogen species present in domestic effluent, such as organic nitrogen, nitrite, nitrate and ammoniacal nitrogen, while organic nitrogen includes various classes of substances such as proteins, peptides, nucleic acids, among others [42]. Considering that the concentrations of total nitrogen were slightly higher in treatments DF1 and DF2 when compared to treatments DF3 and DF4, it can be inferred that ozone in higher concentrations could break up larger mass molecules such as proteins and peptides. This trend agrees with a similar study, in which the domestic effluent obtained from a biological filter, air stripping, clarified and post-treated by ozonation, showed a reduction in the concentrations of substances with molecular masses greater than 1,000 Dalton as the ozone dose increased. However, the amount of compounds with a molecular weight of 253 Dalton has not changed significantly [39].

The high ammoniacal nitrogen removal can be explained by the adsorption and ion exchange capacity that clinoptilolite presents in relation to the ammonium ion, which significantly improves the removal of this parameter [43]. This statement can be confirmed when comparing the results obtained for removal of ammoniacal nitrogen using post-treatment

effluent from anaerobic reactor using DF, which obtained less than 20% removal [9], and triple filtration with activated carbon, which obtained 35% removal of the same parameter [5].

Total phosphorus removal was observed in all treatments evaluated, with no significant variation of this parameter. This removal is mainly explained by the initial stage where phosphate formation can occur in the hydrated oxide structures, with the formation of mixed cationic phosphates and ferric phosphate, which can be removed by sedimentation and filtration in the gravel initial stage [5]. This hypothesis is confirmed when analyzing the results of Verlicchi et al. [44], which used sand filtration without previous coagulation, obtaining phosphorus removal low efficiency. In addition, the removal of this parameter becomes more efficient using clinoptilolite, due to the ion exchange of the ammonium ion by Ca²⁺, which can be precipitated with residual phosphate [45].

The average removal for *E. coli* and total coliforms using double filtration with different concentrations of ozone is shown in Figure 4.

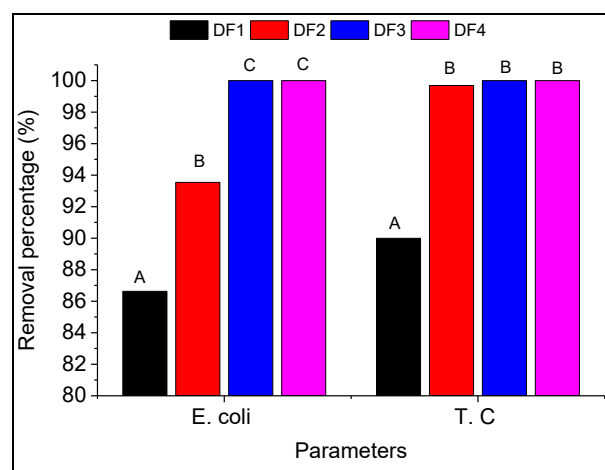


Fig. 4. Arithmetic mean of removal *E. coli* and Total Coliforms. DF1: Double Filtration; DF2: Double Filtration + 0.4 mgO₃ L⁻¹; DF3: Double Filtration + 5.0 mgO₃ L⁻¹; DF4: Double Filtration + 7.0 mgO₃ L⁻¹; Parameters with the same letter did not differ statistically.

Regarding *E. coli*, Figure 4 shows that the average concentration ranged from 5.7x10⁵ to 3.5x10⁴ CFU/100mL (93% removal) for DF1, from 1.3x10⁶ to 8.79x10⁴ CFU/100mL (93% removal) for DF2, reaching 100% of removal for DF3 and DF4. When considering total coliforms, the average concentration was from 1.1x10⁷ to 1.3x10⁶ CFU/100mL (89% removal) for DF1, from 5.6x10⁷ to 7.2x10⁴ CFU/100mL (99.7% removal) for DF2, and 100% of removal for DF3 and DF4. According to the analysis of variance, a significant difference in treatments DF3 and DF4 was observed in relation to DF1 and DF2.

According to these results, the use of double filtration with clinoptilolite was seen to be significantly effective in the removal of *E. coli* and total coliforms. Although the microorganisms present in the effluent are too small for gravity sedimentation to occur, such pathogens are often incorporated into larger particles, making it possible to remove a large portion through sedimentation tanks or granular media filtration processes [46]. In addition, the efficiency of disinfecting a sanitary effluent is directly affected by the presence of suspended materials as microorganisms, which can be located inside the particles, avoiding contact

with the disinfectant [9]. This explains the DF1 treatment high efficiency regarding microorganism removal. However, complete inactivation of these microorganisms was only achieved by using the ozone disinfection process. The presence of organic substances in suspension in domestic effluents decreases the ability to disinfect by ozonation, due to reactions that quickly consume oxidizing reagents [47]. Therefore, total removal of suspended solids was observed, which may have contributed to the inactivation of microorganisms when ozone disinfection was used. According to Paraskeva and Graham [48], ozone concentrations in the range of 2 to 6 mg L⁻¹ were sufficient to reduce the levels of total coliforms below 100 CFU/100 mL effluent treated by secondary systems. Ozonation of effluent treated by physicochemical and biological processes at a domestic wastewater treatment plant in Italy reported complete inactivation of fecal coliforms using concentrations ranging from 10 to 15 mg L⁻¹ ozone and a 30-min contact time [49]. Considering that complete inactivation of *E. coli* and total coliforms was obtained using 5 mg L⁻¹ ozone concentration with a 5-min contact time, it can be said that the proposed system was efficient in disinfecting the anaerobic reactor effluent.

Post-treatment with DF and clinoptilolite proved to be a promising alternative for the treatment of anaerobic reactor effluent. The results showed that by following the proposed treatment, the effluent treated with the DF/Ozonation combination met the guidelines for reuse in toilets, irrigation of orchards, cereals, pastures, and other crops through runoff

or by punctual irrigation system, according to the NBR 13969/97. In addition, all results obtained met the parameters established in the United States Environmental Protection Agency reuse manual [50] for urban reuse, restricted/unrestricted damming, creation and maintenance of wetlands, as well as industrial and agricultural reuse, provided that 1.0 mg L⁻¹ of residual chlorine was added to the final effluent to maintain the disinfection process.

3. Material and Methods

3.1 Double filtration unit (DF)

The effluent evaluated in this work came from the Gertrude sewage treatment plant, which is located in the municipality of Ponta Grossa - PR and was designed to serve a population of 27 thousand inhabitants, with a nominal flow of 30 L s⁻¹. The system in operation consists of a primary treatment for the removal of coarse material and a secondary system with two Upflow Anaerobic Sludge Blanket (UASB) in parallel, and post-treatment by biofilter.

The DF system included an upward gravel filter (UGF) arranged in four layers of 300 mm each with particle sizes in the following ranges: 19.1 to 38.1; 9.52 to 19.1; 4.8 to 9.52, and 2 to 4.8 mm. The second element was a downward clinoptilolite filter (DCF), disposed in 1200 mm monolayer and granulometry ranging from 0.42 to 2.36 mm. Figure 5 shows the diagram of the DF pilot unit.

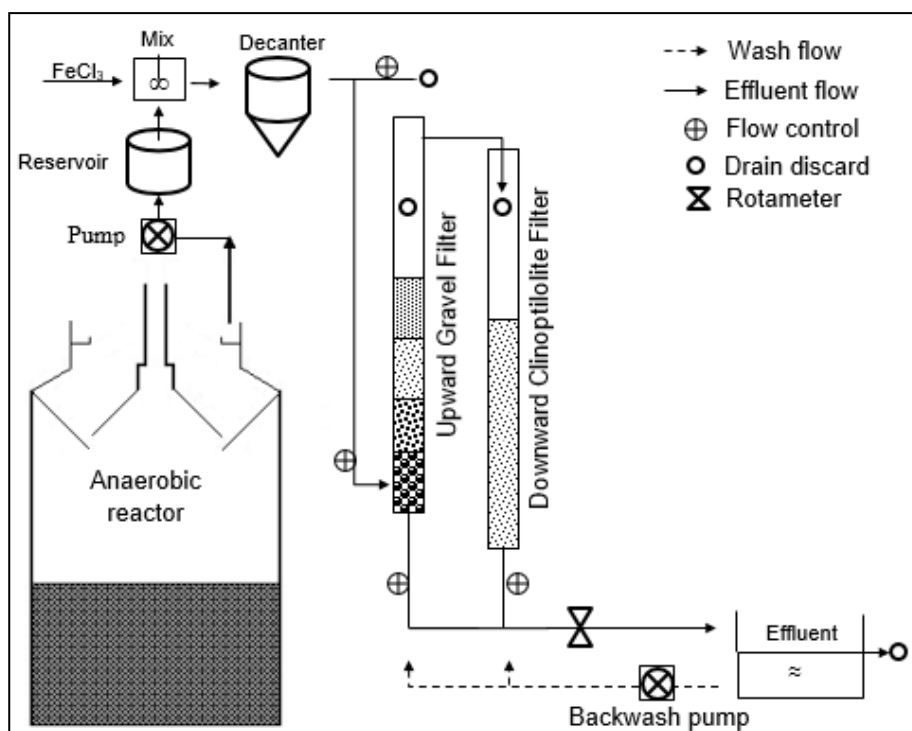


Fig. 5. Diagram of the double filtration pilot unit.

3.2 Experiments in Jar-Test

The pH, coagulant dosage, speed gradients and mixing time applied in the effluent treatment used in the double filtration pilot unit (DF) were previously determined in laboratory tests using jar test equipment. The mixing gradients 100, 300, and 500 s⁻¹ and times 5, 15 and 30 s were tested. The pH values evaluated ranged from 5.0 to 9.0 at 0.5 intervals for each test.

Ferric chloride concentrations ranged from 20 to 140 mg L⁻¹ at 10 mg L⁻¹ intervals. To prepare the stock solutions, a 38% ferric chloride concentrated solution was used. The pH was adjusted with sodium hydroxide and sulfuric acid solutions, both diluted to 1.0 mol L⁻¹.

3.3 Parameters analyzed

The removal efficiency was analyzed through physicochemical and microbiological parameters, according to the Standard Methods for the Examination of Water and Wastewater [42], presented in Table 1.

Table 1. Physicochemical and microbiological parameters assessed.

Parameters	Method
Turbidity (NTU)	Nephelometric 2130 B
Biochemical Oxygen Demand – BOD (mg L ⁻¹)	Oxymetric 5210 B
Chemical Oxygen Demand – COD (mg L ⁻¹)	Spectrophotometric (Hach 8000®) *
Total Phosphorus – TP (mg L ⁻¹)	Spectrophotometric 4500-PE
Ammoniacal Nitrogen – AN (mg L ⁻¹)	Spectrophotometric (Alphakit®) *
Total Nitrogen – TN (mg L ⁻¹)	Spectrophotometric (Spectroquant®) *
Total Suspended Solids – TSS (mg L ⁻¹)	Filtration/gravimetric 2540 D
Ozone – (mg L ⁻¹)	Spectrophotometric (Alphakit®) *
<i>E. coli</i> – (CFU/100 mL)	Membrane filter 9222 B
Total coliforms - TC (CFU/100 mL)	Membrane filter 9222 B

*kit

3.4 Post-treatment tests by double filtration (DF) with ozone disinfection

The anaerobic reactor effluent was pumped into a 150 L tank installed on top of the DF pilot unit. The natural pH of the effluent was used during the DF tests. The ozonation experiments were performed in a 1 L kitasato (reactor) using the effluent collected from the DF. A porous ceramic diffuser was placed at the bottom of the reactor to distribute the ozone. Compressed and dry atmospheric air was used for the ozone production by an ozone generator (Brazil Ozone®). The ozone concentration was measured using the Alfakit spectrophotometric equipment. To measure the ozone dosages, tests were performed using ultrapure water obtained by reverse osmosis and two flasks of ozone gas (off-gas). Ozonation time was adopted based on studies of ozonation of secondary effluents, in which no significant variations in the removal of organic matter after 5 min of contact [36] was observed. The effluent collected after the treatment by double filtration was submitted to disinfection by 0.4; 5.0 and 7.0 mg L⁻¹ ozone.

3.5 Statistical analysis

The normality of the data was calculated using the Shapiro Wilk test. The Tukey test was applied to data that showed significant normality, and the Kruskal-Wallis test for data that did not show significant normality. In all tests, a significance level of 0.05 was used.

4. Conclusions

Double filtration using clinoptilolite contributed significantly to the removal of the main physicochemical parameters such as turbidity, TSS, COD, BOD, nitrogen, and phosphorus from the anaerobic reactor effluent. The low ozone concentration (0.4 mg L⁻¹) indicated possible oxidation of fractions of biodegradable organic matter. We could observe that the highest ozone concentrations (5.0 and 7.0

mg L⁻¹) improved COD removal, but decreased BOD removal, indicating a possible increase in the mineralization of biodegradable fractions. The improvement in the removal of total nitrogen for treatments with a higher concentration of ozone, points to a possible breakdown of more complex molecules present in residual form in the treated effluent. In addition, the use of 5.0 mg L⁻¹ ozone was efficient in the complete inactivation of *E. coli* and total coliforms, resulting in a satisfactory disinfection of the final effluent. Through these results, double filtration using clinoptilolite followed by ozonation was seen to be a promising technology for the post-treatment of anaerobic reactor effluent. Both treatments, with 0.4 mg L⁻¹ and 5.0 mg L⁻¹ ozone, were shown to be promising in generating an effluent with potential for reuse in relation to BOD and COD removal. Therefore, the choice of the adequate ozonation process for the treatment depends on the quality of the effluent to be produced. For effluents with low COD and BOD concentrations, the use of 0.4 mg L⁻¹ ozone concentration is suggested, due to the high removal of biodegradable organic matter at this ozone concentration. For effluents with high COD and BOD concentrations, it is suggested that the treatment be carried out with a 5 mg L⁻¹ ozone concentration.

Acknowledgments

The authors are thankful to the Companhia de Saneamento do Paraná (SANEPAR) and Programa Paranaense de Pesquisas em Saneamento Ambiental (Fundação Araucaria/SANEPAR) - Finance Code 198/2019.

Author Contributions

Angela de Góes Lara Cardozo Costa: Conceptualization, Methodology, Investigation, Resources, Formal analysis, Writing - original draft, Writing - reviewing & editing. Cleber Pinto da Silva: Conceptualization, Methodology, Investigation, Resources, Formal analysis, Writing - original draft, Writing - reviewing & editing. Danilo Gabriel dos Santos Matos: Formal analysis & reviewing. Carlos Raphael Pedroso: Investigation and Resources. Carlos Magno Sousa Vidal: Supervision and Funding acquisition. Jeanette Beber de Souza: Supervision and Funding acquisition. Sandro Xavier de Campos: Supervision, Funding acquisition & reviewing.

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How to cite this article

Costa, A. G. L. C.; da Silva, C. P.; Matos, D. G. S.; Pedroso, C. R.; Vidal, C. M. S.; de Souza, J. B.; de Campos, S. X. *Orbital: Electron. J. Chem.* **2021**, *13*, 328. DOI: <http://dx.doi.org/10.17807/orbital.v13i4.1617>