

Development of a Methodology for Sizing Activated Sludge Reactor Volumes for Treating Wastewater from the Recycled Paper Industry

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ABSTRACT. Although the activated sludge system has been in use since the last decade, many industrial typologies still lack a precisely adjusted equation for sizing the optimal volume of these reactors. In response to the need for obtaining size parameters for activated sludge reactors used for treating effluents from the recycled paper industry, a new procedure, in batch mode, reactors were tested at 20°C under different hydraulic retention times (HRT) and 3 ranges of mixed liquor volatile suspended solids (MLVSS) concentrations. Kinetic equations for removing BOD₅ and COD were obtained, along with a model equation for calculating the volume of activated sludge reactors. Removals above 90% for BOD₅ and COD were achieved with 72 hours of aeration and MLVSS concentrations above 4 g L⁻¹. This new method with standardized equations specific to recycled paper industry wastewater can be considered validated and ready for use by users such as undergraduates and graduates, as well as wastewater treatment plant designers. Finally, it is advisable to use aerobic biological sludge from textile mills for inoculation of activated sludge systems in the paper industry.

Keywords: Pollutant removal; reaction volume; pulp and paper; removal kinetics.

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Introduction

The paper industry holds significant importance in the global landscape (Liang et al., 2021). This industrial activity generates large volumes of wastewater (WW), containing harmful organic and inorganic pollutants detrimental to the environment (Munir et al., 2022; Hämäläinen. 2022). According to Ashrafi et al. (2015), the production of one ton of paper generates 5 to 100 m³ of WW, which globally accounts for 42% of the three billion tons of industrial effluents produced on the planet (Toczyłowska-Maminska, 2017).

As a primary treatment, WW must undergo stages to separate non-biodegradable solids in flotation tanks (Munir et al., 2022). For secondary treatment, biological systems are recommended (Mendonça et al., 2022). Bacteria that thrive in aerobic treatment systems such as activated sludge exhibit enhanced biodegradation capabilities due to their wide pH tolerance, temperature adaptability, biochemical versatility, and environmental adaptability (Chandra and Singh, 2012; Kamali et al., 2015; Singh et al. 2021). Consequently, aerobic systems like aerated lagoons and activated sludge have been successfully used worldwide for treating wastewater from the paper and pulp industries.

In the last decades, both in its conventional and modified forms, the activated sludge process has played a crucial role in treating effluents from paper and pulp mills, meeting the effluent limits of secondary treatment (Buyukkamaci and Koken, 2010; Mendonça et al., 2022). In activated sludge bioreactors, aerobic heterotrophic communities efficiently remove organic substances, nutrients, as well as toxic compounds, and pathogens from the produced wastewater (Wells et al., 2011). In this context, it has been demonstrated that applying a consortium of different bacteria may exhibit greater efficiency in reducing BOD₅ and COD in effluents from the paper and pulp industry.

With the advancement of recycling in Brazil, the increase in paper and cardboard recycling plants highlights a commitment to resource recovery. However, this growth is also linked to the rise in wastewater production and environmental pollution. In this context, developing methodologies and reactors specifically designed for treating wastewater from recycled paper and cardboard factories is essential.

Although the activated sludge system is widely adopted globally for treating wastewater from conventional paper and pulp industries, there is a lack of references in the literature to specific techniques, methods, or equations for sizing this powerful system for the recycled paper and cardboard industry. This study aimed to fill this gap by exploring and developing specific approaches to adapt the activated sludge system to this growing industry.

Considering the need to establish a reliable guideline for sizing treatment plants for wastewater from the recycled paper industry, this study tested activated sludge reactors in batches to remove pollutants and nutrients from this type of wastewater. The study outlined parameters for sizing and proposed an equation to determine the volume of reactors.

Material and methods

Wastewater characteristic

The wastewater from the paper and cardboard recycling industry used in the present experiment was collected after a Krofta flotation unit, operated with the addition of coagulant and flocculant. Preceding the flotation unit there was an aerated equalization system and a parabolic static screen. The average characteristics of the tested wastewater in the proposed activated sludge system are described in Table 1. Analytical procedures were performed following American Public Health Association [APHA] (2017).

Table 1. Parameters analyzed in the wastewater from the recycled paper and cardboard industry after passing through a flotation unit.

Parameters	Values ¹
pH	7.5 _(0.5)
BOD ₅ (mg L ⁻¹)	1100.0 _(10.0)
COD (mg L ⁻¹)	2400.0 _(64.0)
COD/BOD ₅	2.18
O&F ² (mg L ⁻¹)	30.0 _(0.5)
Surfactants (mg L ⁻¹)	8.0 _(0.02)
N-NTK ³ (mg L ⁻¹)	11.0 _(1.0)
TSP ⁴ (mg L ⁻¹)	4.0 _(0.0)

¹The values in parentheses represent the standard deviation observed between three replications; ²O&F - Oil and Fats; ³NTK - Total Kjeldahl Nitrogen;

⁴TSP - Total soluble phosphorus.

The values observed for the different parameters are in accordance with the typical characteristics of wastewater effluents from paper and cellulose industries presented by Patel et al. (2021).

Inoculum and metagenomic analysis

Each reactor was inoculated with 250 mL of sludge with an age (θ_c) of approximately 12 days. The inoculum volume corresponded to 1/6 of the reactor (Mendonça et al. 2018). The main bacterial species in the inoculum were *Zooglea ramigera*, *Sphaerotilus natans*, *Microthrix parvicella*, *Thiothrix* sp, *Beggiatoa* sp, and *Pseudomonas* sp, detected through metagenomic analysis (Laudadio et al., 2019). This inoculum containing these bacterial strains was extracted from a textile wastewater treatment plant and subsequently adapted to the conditions imposed in the present project.

Reactors and experimental conditions

Bench-scale tests were conducted in three 1.5 L reactors (R1, R2, and R3) with diffused air aeration ($DO \leq 3.0$ mg L⁻¹) and reaction times of 12, 24, 32, 48, 60, and 72 hours to express the removals of BOD₅ and COD. The reactor model used was a cylindrical biological fermenter adapted with a diffused air aeration system at its lower part. The system was equipped with measurement sensors, including pH control systems, dissolved oxygen, temperature, feeding, agitation, and gas monitoring.

The sedimentation time for each test was 30 minutes. Three ranges of mixed liquor volatile suspended solids (MLVSS) concentrations were tested: 3,000-3,500 mg L⁻¹ for R1, 4,000-4,500 mg L⁻¹ for R2, and 5,000-5,500 mg L⁻¹ for R3. After each batch in its consecutive cycle time, the sludge was separated from the liquid by sedimentation in the reactor for 40 minutes. Subsequently, the supernatant was poured into falcon tubes and frozen at -50°C for further analysis of pollutant removal using a spectrophotometer (Hach 3900, Italy).

To maintain the BOD:N:P ratio at 100:5:1, 5 mg of nitrogen (N) and 1 mg of phosphorus (P) were added. Urea (40 mg L⁻¹) was used as the nitrogen source, and phosphoric acid (7.8 mg L⁻¹) as the phosphorus source. The pH was

kept between 6.5 and 7.5, and the temperature at $20^{\circ}\text{C} (\pm 1^{\circ}\text{C})$. The three batch tests were repeated 15 times, and samples were analyzed in triplicates. Analytical procedures were performed in accordance with APHA (2017).

For the analysis of organic matter removal kinetics, the parameters BOD_5 and COD were used. After determining the optimal reaction time associated with the best concentration of mixed liquor volatile suspended solids (MLVSS), the removal was analyzed for oils and fats (O&F) and surfactants, in addition to the nutrients, total nitrogen (N-TKN) and total soluble phosphorus (TSP).

Figure 1 presents the complete experimental layout, from wastewater collection in the field, inoculum collection, routing to the laboratory, and key parts of the methodology, to obtaining the results as a flowchart.

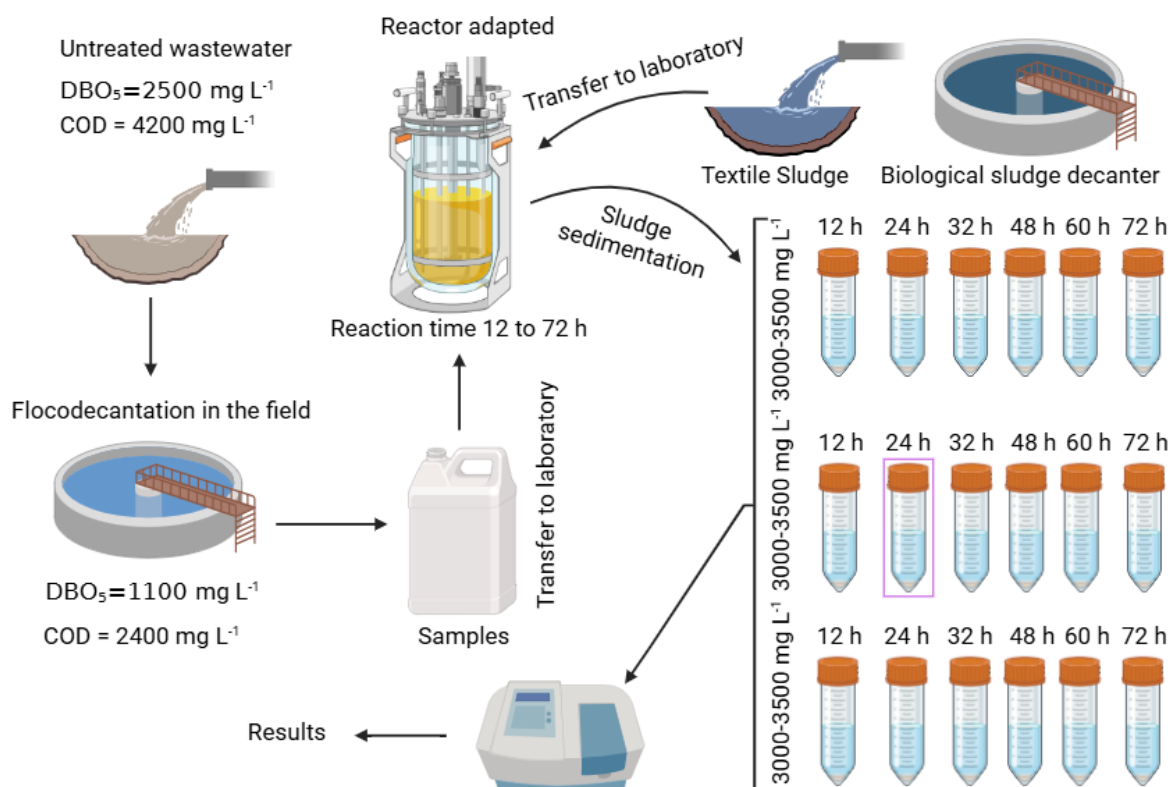


Figure 1 Schematic summary in the form of a flowchart of the experiment for validating the methodology.

Statistical analysis

Significant differences between treatment means were checked by the Tukey test at a 95% confidence level. Before the parametric tests, the normality of the data was confirmed by the Shapiro-Wilk test using PAST software (Hammer et al. 2021).

Deriving the equation

Organic load (F):

The organic load (available food) in the reactor is:

$$F = Q \times S_0 \quad (1)$$

Where:

Q = influent flow rate or volume ($\text{m}^3 \text{ day}^{-1}$),

S_0 = influent BOD_5 concentration (mg L^{-1}).

The total organic load (F) is the amount of BOD_5 entering the reactor per day, i.e., the wastewater flow rate multiplied by the BOD_5 concentration.

Microorganisms (M):

The microorganism's concentration is represented by the active biomass in the aeration tank, which is the product of the reactor volume (V) and the concentration of volatile suspended solids ($\text{VSS} = \text{MLVSS}$), representing the active biomass (X).

$$M = V \times X \quad (2)$$

Where:

V = aeration tank volume (m³),

X = volatile suspended solids concentration in the aeration tank (mg L⁻¹).

Assembling the F/M equation

Now, we can divide the organic load (F) by the biomass amount (M) to get the F/M ratio:

$$F/M = (Q \times S_0)/(V \times X) \quad (3)$$

Considering that part of the food will be degraded, we can replace S₀ with (S₀-S_f), that is, the initial BOD₅ concentration minus the initial BOD₅ concentration. Next, the volume was also isolated in the equation, and this was used as a root to develop the calibrated equation.

$$V = \frac{Q \times [S_0 - S_f]}{1,000 \times F/M \times MLVSS} = \frac{Q \times [BOD_{in} - BOD_{out}]}{1,000 \times F/M \times MLVSS} \quad (4)$$

Note: SF values were obtained after batches and described in Figure 2.

Results and discussion

BOD₅ Removal and Equation for Reaction Volume Sizing

The BOD₅ removals were above 90% for the 72-hour HRT under the conditions of applying MLVSS concentrations of 4,000-4,500 and 5,000-5,500 mg L⁻¹, making this the ideal recommendation for the sizing of activated sludge systems (Figure 2). For the 48-hour HRT and the same MLVSS concentrations, the BOD₅ removals ranged between 85 and 87%, which can also be adopted when discharge standards in watercourses are less stringent. However, for the 24-hour HRT, the BOD₅ removals were between 50 and 73%, and thus, not recommended.

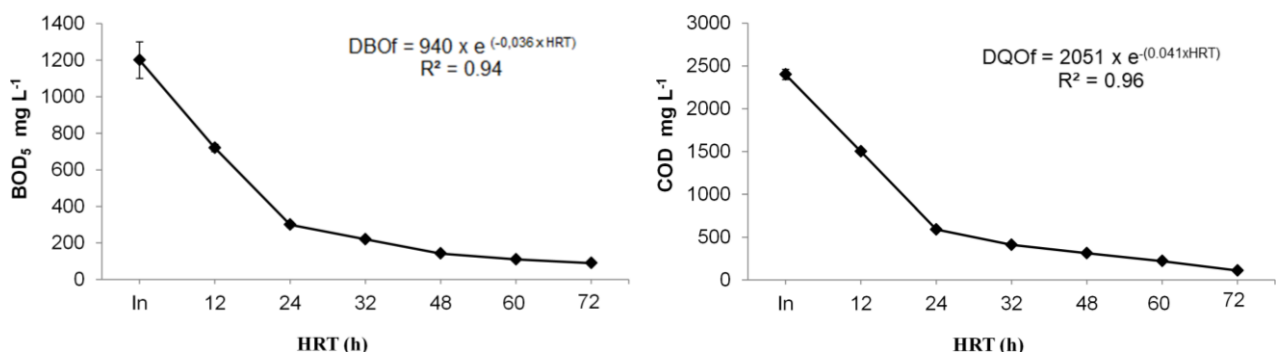


Figure 2. BOD₅ (a) and COD (b) removal kinetics for different reaction time (HRT) and MLVSS between 4,000-4,500 mg L⁻¹: These graphs represent the intermediate scenario between the tests. It is considered the ideal situation.

Figure 1a shows that the decay of BOD₅ was more intense up to 24 hours, reaching up to 73% removal. However, even with the significant MLVSS concentration, the 90% efficiency was only achieved after 72 hours of HRT. There was no statistical difference for the 72-hour HRT when applying MLVSS concentrations of 4,000-4,500 and 5,000-5,500 mg L⁻¹.

The equation determining the decay kinetics obtained in Figure 1a was used in the proposed equation 5 for reactor sizing.

$$V = \frac{Q \times [BOD_{in} - BOD_{out}]}{1,000 \times F/M \times MLVSS} = \frac{Q \times [BOD_{in} - 940 \times e^{(-0.036 \times HRT)}]}{1,000 \times F/M \times MLVSS} \quad (5)$$

Where:

V = optimal volume of activated sludge reactor; Q = flow rate (m³ d⁻¹); BOD_{in} = initial BOD₅ concentration (mg L⁻¹); HRT = hydraulic retention time (hour); F/M = food-to-microorganism ratio, MLVSS (g L⁻¹).

The term in the equation: $940 \times e^{(-0.036 \times HRT)} = BOD_{out}$, was obtained from the kinetic curves of the bench tests and corresponds to the final BOD₅ obtained by the treatment system as a function of the HRT (Figure 1a).

Equation 5 can be used under the following conditions:

BOD_{in} (influent to the reactors) $\leq 1,200 \text{ mg BOD}_5 \text{ L}^{-1}$;

MLVSS range of 4,000 - 5,500 mg L^{-1} (equivalent to 4 to 5.5 g L^{-1});

F/M (Food to Microorganism ratio) range of 0.075 - 0.10;

HRT (Hydraulic Retention Time) between 48 and 72 hours for 87 - 92% BOD_5 removal.

Sludge age (θ_c) 18 to 30 days.

Under these conditions, Equation 1 can be applied for sizing activated sludge reactors. For Example:

$$V_r = \frac{100 \times (1,000 - 940 \times e^{(-0.036 \times 72)})}{1,000 \times 0.08 \times 4} = 290 \text{ m}^3$$

To determine the hydraulic retention time (HRT) in the previous example, the quotient of the activated sludge reactor volume obtained by the inlet flow was calculated, as demonstrated in equation 6.

$$HRT = \frac{290 \text{ m}^3}{100 \text{ m}^3/\text{d}} = 2,9 \text{ d} \quad (6)$$

Confirming the kinetics data outlined in Figure 1, 48 to 72 hours for 87 to 93% BOD_5 removal. Therefore, the potential practical applicability of the adjusted equation is confirmed.

COD Removal

COD removals above 95% were achieved only for the 72-hour HRT with MLVSS concentrations of 4,000-4,500 and 5,000-5,500 mg L^{-1} , which is the recommended range for full-scale unit sizing (Table 2). During the same period (72 h) and at concentrations of 3,000-3,500 mg L^{-1} , COD removals of 88% were recorded. For COD removal, a similar pattern to BOD_5 decay was observed up to 24 hours of HRT (Figures 1a and 1b). However, after 24 hours, 75% of the applied COD was already removed in the reactor operated with MLVSS concentrations of 4,000-4,500 mg L^{-1} . Under these conditions, the reactor showed a COD removal of 91% from 60 hours onwards. Similar to BOD_5 , there was no statistical difference for the 72-hour HRT when testing MLVSS concentrations of 4,000-4,500 and 5,000-5,500 mg L^{-1} .

In a complementary and summarized manner, Table 2 compiles the data for each treatment, highlighting the input and output values for each treatment. It is worth noting that the input values were consistently the same since the same wastewater was used in all tests as a standardization measure.

Table 2. Average BOD_5 and COD values for different hydraulic retention times (HRT) in activated sludge reactors with different mixed liquor volatile suspended solids (MLVSS) concentrations.

Parameters HRT (h)	MLVSS (mg L^{-1})		
	3,000-3,500	4,000-4,500	5,000-5,500
	BOD_5 (mg L^{-1})		
0	1,100 _(10.0)	1,100 _(10.0)	1,100 _(10.0)
24	550 _(20.0) ^a	300 _(5.0) ^b	292 _(1.1) ^b
48	360 _(14.0) ^a	142 _(3.0) ^b	134 _(2.4) ^b
72	229 _(6.2) ^a	90 _(0.5) ^b	80 _(0.3) ^b
	COD (mg L^{-1})		
0	2,400 _(64.0)	2,400 _(64.0)	2,400 _(64.0)
24	950 _(12.0) ^a	590 _(26.0) ^b	500 _(7.0) ^b
48	605 _(9.0) ^a	312 _(10.0) ^b	273 _(1.1) ^b
72	290 _(0.6) ^a	110 _(2.3) ^b	100 _(1.9) ^b

The values in parentheses represent the standard deviation observed between three replications; Values followed by the same letter on the same line did not show a significant difference using the Tukey test at a 5% confidence level.

COD and BOD_5 removal compared to other studies

As there are no specific studies on the treatment of wastewater from the recycled paper and cardboard industry for activated sludge systems, Table 3 has been prepared to compare the use of the activated sludge system and its variants or complementary techniques in treating various types of wastewater from paper industries.

The research conducted by Tsang et al. (2006) on the activated sludge system for the treatment of pulp industry wastewater showed a substantial removal of COD, which exceeded the findings of the present study.

Although the organic load addressed in their investigation was lower than that considered here, this comparison suggests that the activated sludge system can be successfully employed in treating wastewater generated by the recycled paper and cardboard industry, both in Brazil and in the global context.

Table 3. The removal of COD and BOD₅ in the present study compared with activated sludge reactors and their variants in different types of wastewaters from the paper industry.

Paper industry typology	COD removal	BOD ₅ removal	Technique/system used	Reference
Pulping industry wastewater	*NR	98.1 %	Conventional operation: two aeration columns and a sedimentation in continuous flow	Tsang et al. (2006)
	92%	99%	Continuous stirred batch reactor pre-treated with NaBH ₄ as a reducing agent	Ghoreishi and Haghighi (2007)
Pulp and paper mill wastewater	60-70%	95%	Activated sludge treatment process with wastewater samples fractionated by microfiltration and ultrafiltration into different size fractions	Leiviskä et al. (2008)
	74.8% - 92.1%	NR	activated sludge process using a sequence batch reactor + advanced oxidation processes (Fenton method) at a bench scale	Abedinzadeh et al. (2018)
Pulp mill wastewater	76	≈ 81%	**Three bacteria Combination in aerated system	Tiku et al. (2010)
Paper mill wastewater	91.5%	NR	Activated sludge with the addition of digestate powder and microalgae	Talapatra and Ghosh (2022)

Ghoreishi and Haghighi (2007) tested an anaerobic system like activated sludge in a continuous stirred batch reactor pre-treated with NaBH₄ as a reducing agent. In this case, the authors treated wastewater from the pulp mill using a pre-treatment to improve the biodegradability conditions of the effluent, achieving high removal above 90% for COD and BOD₅. Some wastewater from the paper industry requires pre-treatment to optimize the efficiency of the biological stage. In the present research, non-biodegradable compounds were efficiently removed by the chemically assisted primary treatment in a Krofta flotation unit. In other words, only the flotation unit as the primary treatment for the studied wastewater was sufficient to eliminate suspended solids and non-biodegradable colloids derived from lignin, cellulose, etc.

Similar to previous authors, Leiviskä et al. (2008) worked with wastewater from the pulp and paper mill, collecting samples before and after the activated sludge treatment process. Additionally, the samples were fractionated into different sizes by microfiltration and ultrafiltration. The results showed that the reduction in BOD₅ was around 95%, and for COD it was 60–70%. According to these authors, the filtration process helps to increase efficiency in the wastewater treatment process as it was found to reduce these chemical parameters even for the largest pore size.

Bengtsson et al. (2008) evaluated the production of polyhydroxyalkanoates in wastewater from activated sludge treatment from a paper mill, carried out on a laboratory scale. The process consisted of three steps: acidogenic fermentation to convert wastewater organic matter into volatile fatty acids, an activated sludge system operating under feast/famine conditions to enrich polyhydroxyalkanoate-producing organisms, and finally PHA accumulation in batch experiments. The authors found that after 250 days of operation, 95% removal of soluble COD was possible. For the authors, the enrichment process enabled high COD removal efficiency along with the production of a value-added byproduct such as polyhydroxyalkanoates. Abedinzadeh et al. (2018) assessed the removal of COD and color from wastewater from the pulp and paper industry by applying the activated sludge process in a sequential batch reactor combined with advanced oxidation processes (AOPs) on a bench scale. In the stage with activated sludge (pre-treatment), with an initial COD condition of 1,000 mg L⁻¹, and a cycle time of 24h, 74.8% COD removal was achieved. Then, using Fenton oxidation as post-treatment (dosages of 3 mM Fe²⁺ and 6 mM H₂O₂ at pH 3.0 for a reaction time of 30 min), a 92.1% COD reduction was obtained for combined treatment. Therefore, the study showed that using AOPs as post-treatment can be very useful, both operationally and economically.

Assalin et al. (2009) investigated the removal of COD, TOC, total phenols, and color from paper mill effluent using combined processes of activated sludge and advanced oxidation process by ozonation. The effluent was obtained after the first alkaline extraction stage (E1), with an initial characteristic of 1,500 to 2,500 mg L⁻¹ of COD. The treatment with activated sludge, ozone, and pH 10 allowed the removal of more than 80% of the original COD. The authors concluded that the combined treatment increased the degradation

rate for all parameters studied, with lower ozone consumption than the single ozonation treatment, leading to cost reduction.

In the treatment of pulp mill wastewater with a mix of three bacteria, Tiku et al. (2010) achieved removals above 75% for COD and above 80% for BOD₅. One of the bacterial genera used by the authors, *Pseudomonas* sp., was also detected in the sludge used as inoculum in the present experiment, indicating that this genus has good adaptability for growth in wastewater from the recycled paper and cardboard industry.

Jagaba et al. (2022a) and Jagaba et al. (2022b) used activated sludge systems with the addition of natural biosorbents derived from rice. These studies reported excellent treatment efficiencies for the types of wastewaters produced from a paper packaging biorefinery and pulp and paper industry (with combined sewage).

Most cited authors employed some strategy to increase the concentrations of N and P in wastewater, which was also necessary in the present study, as all wastewater from the paper industry does not meet the minimum BOD:N:P ratio for aerobic treatment (100:5:1).

After comparison with the cited literature, only the activated sludge system, operating with the inoculum used in the present research and adjusting the BOD:N:P ratio, would be enough to treat the wastewater without the need for the addition of other chemicals, other microorganisms such as fungi or microalgae, or biosorbents, although there is still room for more advanced studies from this perspective. The biological treatment alone was able to efficiently treat the residue, as its biodegradability after passing through the primary system improved, resulting in a COD/BOD₅ ratio of 2.18. This indicates that, for this type of wastewater, COD/BOD₅ ratios should be at least 3.0 for the adoption of the biological system.

Removal of other parameters

The other four operational parameters evaluated in this study are listed in Table 4.

Table 4. Average removal values of parameters analysed in the effluent after different hydraulic retention times (HRT) in activated sludge reactors with different mixed liquor volatile suspended solids (MLVSS) concentrations.

Parameters HRT (h)	MLVSS (mg L ⁻¹)		
	3,000-3,500	4,000-4,500	5,000-5,500
	O&F ⁺ (mg L ⁻¹)		
0	30 _(0.5)	30 _(0.5)	30 _(0.5)
24	20 _(0.3) ^a	15 _(0.1) ^a	10 _(0.3) ^b
48	12 _(0.1) ^a	9 _(0.02) ^a	4 _(0.01) ^b
72	10 _(0.0) ^a	0 _(0.0) ^b	0 _(0.0) ^b
	Surfactants (mg L ⁻¹)		
0	8 _(0.02)	8 _(0.02)	8 _(0.02)
24	2 _(0.0) ^a	1.1 _(0.03) ^a	0.5 _(0.0) ^b
48	0.5 _(0.0) ^a	0.6 _(0.0) ^a	0.0 _(0.0) ^b
72	0.0 _(0.0) ^a	0.0 _(0.0) ^a	0.0 _(0.0) ^a
	N-NTK (mg L ⁻¹)		
0	50 _(3.0)	50 _(3.0)	50 _(3.0)
24	12 _(0.2) ^a	10 _(0.0) ^a	8 _(0.01) ^a
48	8 _(1.0) ^a	3 _(0.0) ^b	1 _(0.0) ^b
72	3 _(0.02) ^a	0 _(0.0) ^a	0 _(0.0) ^a
	TSP ⁺⁺ (mg L ⁻¹)		
0	20 _(0.2)	20 _(0.2)	20 _(0.2)
24	18 _(0.5) ^a	15 _(0.02) ^a	13 ₍₁₎ ^a
48	15 _(0.1) ^a	10 _(0.0) ^b	8 _(0.0) ^b
72	10 _(0.1) ^a	4 _(0.0) ^b	3 _(0.0) ^b

⁺O&F – Oils and fats; ⁺⁺TSP – Total soluble phosphorus; The values in parentheses represent the standard deviation observed between three replications; Values followed by the same letter on the same line did not show a significant difference using the Tukey test at a 5% confidence level.

For 72-hour HRT and MLVSS concentrations above 4,000 mg L⁻¹, the values of TKN (Total Kjeldahl Nitrogen), O&F, and surfactants were below the method detection limit, and phosphorus (P) was removed with an efficiency of 80-85%, the latter being considered an efficient removal level.

Conclusion

BOD₅ and COD were satisfactorily removed with an HRT between 48 and 72 hours and an MLVSS concentration of 4,000 mg L⁻¹. On the other hand, TKN, O&F, and surfactant concentrations were completely removed with the 72-hour HRT and MLVSS above 4,000 mg L⁻¹. Therefore, the parameters for sizing activated

sludge systems should be in the range of MLVSS concentrations of 4,000–5,000 mg L⁻¹ and F/M between 0.075 and 0.1. The equation obtained from the BOD₅ decay curves can be applied to reactor sizing. The present equation developed and adjusted specifically for this type of wastewater, when applied with the correct factors listed above, has significant potential for designing reaction volumes in batch and extended aeration-activated sludge process.

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