Determination of kinetic constants on thermophilic aerobic wastewater treatment of Kraft bleaching cellulose effluent

Determinação de constantes cinéticas do processo de tratamento aeróbio termofílico de efluente de celulose branqueada Kraft

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ABSTRACT
Recalcitrant compounds found in pulp and paper mill effluents, which are usually generated at high temperatures, have shown the potential for thermophilic treatment. Using bleached kraft pulp mill effluents, this study aims to evaluate the acclimation conditions of mesophilic aerobic sludge to thermophilic conditions and determine the kinetic parameters. Two feeding strategies were used for this purpose: in the first approach, the first reactor R1 was fed with bleached kraft pulp effluent, while in the second strategy the reactor R2 was fed with easily biodegradable synthetic effluent and then the gradual substitution for bleached effluent was performed after sludge acclimation to a thermophilic environment. Both reactors were operated in batch at 55ºC. The second strategy has a slight advantage in COD (chemical oxygen demand) degradation and a considerable higher biomass production. The kinetic parameters ($K_s$, $\mu_{max}$, $Y$, $K_d$) for both approaches were obtained using the Monod model. Those values indicated that the approach used for reactor R2 provided higher reaction speed.

Keywords: kinetic parameters, kraft bleaching cellulose effluent, thermophilic aerobic wastewater treatment.
RESUMO
Efluentes de fábricas de papel e celulose, constituídos de compostos recalcitrantes com alta carga orgânica, são normalmente gerados em altas temperaturas e tem potencial para tratamento termofílico. O presente trabalho teve como objetivo a determinação dos parâmetros cinéticos do processo de tratamento de efluente de celulose branqueada Kraft, com prévia adaptação de lodo aeróbio de mesofílico a termofílico. Para tal propósito foram utilizadas duas estratégias de alimentação: na primeira estratégia, o primeiro reator (R1) foi alimentado com efluente de branqueamento de polpa Kraft; enquanto na segunda estratégia, o segundo reator (R2) foi alimentado primeiramente com efluente sintético facilmente biodegradável para, então, ser feita a troca gradual por efluente de branqueamento, após a adaptação do lodo. Ambos os reatores foram operados em batelada a 55°C. A segunda estratégia ofereceu uma ligeira vantagem na degradação do COD (demanda química de oxigênio) e uma maior produção de biomassa. Os parâmetros cinéticos \((K_s, \mu_{max}, Y, K_d)\) foram determinados usando o modelo de Monod. Estes valores indicam que a metodologia aplicada no reator R2 providenciou uma maior velocidade de reação.

Palavras-chave: efluente de celulose branqueada kraft, parâmetros cinéticos, tratamento aeróbio termofílico de efluentes.

1 INTRODUCTION

Paper and cellulose industries have significant economic importance in many countries in the world. However, those industries produce a great quantity of pollutants. In Brazil, where cellulose and paper industries have exponential growth, 60 m³ of effluents are emitted for every ton of produced cellulose (da Silva and Xavier, 2021). For that reason, effluents generate during cellulose production must be treated to their impact to the environment. Different techniques were proposed in literature to reduce the complexity, duration, costs and environmental impacts of the treatment of wastewater produced by cellulose industries (Mascharenas et al. 2021; da Silva and Xavier, 2021; De Almeida et al., 2020).

Industrial effluent treatments based on high-temperature thermophilic aerobic biological wastewater processes offer potential benefits such as accelerated biodegradation, low net biomass production, decreased need for cooling (Tripathi and Allen, 1999), increased solubility of poorly soluble substrates and rapid inactivation of pathogens (Abeynayaka and Visvanathan, 2011). The results of many researches, the high energy requirements for cell maintenance and the high coefficients of microbial decay in thermophilic processes indicate that the amount of excess sludge in these cases may be much smaller for the thermophilic treatment in relation to the mesophilic one (Jahren et al., 2002).
However, it is important to understand the design and operating parameters of the thermophilic aerobic process to gain its advantages. The physical, chemical and biological characteristics of the thermophilic aerobic process are so different from the conventional process that the conventional operational process knowledge base is unusable (Lapara and Alleman, 1999). Besides, literature indicates that the thermophilic aerobic process can be used to treat efficiently many types of wastewaters at high temperatures.

Pulp and paper industries are demanding activated sludge reactors operating in the thermophilic temperature zone, due to factors such as the increment of process wastewater, limited availability of fresh water for cooling, and reduced accessibility to cooling systems (Barr et al., 1996). Besides, industries can harness the thermal energy of the effluents in their production processes (Vaccari, et al., 2003). As a result, the number of researches about the technical feasibility of thermophilic biological treatment has increased in the last years.

Thus, the objective of this study was the acclimation of mesophilic aerobic sludge to a thermophilic temperature of 55°C, using kraft pulp bleach plant wastewater and determination the kinetic constants \((K_S, \mu_{max}, Y, K_d)\) of this process using the Monod model. Two feeding strategies were applied: In the first strategy, the reactor was fed with bleached kraft pulp effluent, while in the second approach a reactor was fed with easily biodegradable synthetic effluent and gradually substituted for bleached effluent.

2 MATERIALS AND METHODS

2.1 AEROBIC SLUDGE ACCLIMATION

For the mesophilic aerobic sludge acclimation, two strategies were applied:

**Strategy I:** This test used a first reactor (R1), with a capacity of 1 L, fed with the wastewater from a kraft pulp mill bleach plant, prepared as described below. Nitrogen and phosphorus were also added in an amount sufficient to maintain the ratio COD:N:P in 300:5:1. In addition to these nutrients, 1.0 ml L\(^{-1}\) of a micronutrients solution (Table 1) was used.

**Strategy II:** In this test, the second reactor (R2) was fed with a synthetic substrate containing sucrose, phosphorus, nitrogen, starch, and 0.42 g L\(^{-1}\) of meat extract. Thus, a total COD close to the value obtained with the bleaching substrate in the first strategy (2640 mg L\(^{-1}\)) was obtained. After the sludge acclimation period, a gradual substitution
for the industrial pulp bleach effluent was performed. The industrial wastewater was prepared as in the strategy I.

From the start-up, the temperature of both reactors was set at 55 °C ± 1 °C. Both reactors were filled with 0.4 L of aerobic sludge and 0.6 L of substrate and operated in batches, with an equivalent hydraulic retention (HRT) time of 5 days. At every 24 hours it was left to sediment for 10 minutes and a sample of 0.2 L of the supernatant was removed, adding the same amount of a new substrate and adjusting the pH to 7.0. The sample taken was used for analysis. The oxygen supply at an average flow of 1.4 L min\(^{-1}\) produced a dissolved oxygen concentration of approximately 2.6 mg L\(^{-1}\). All physical and chemical analysis were done using Standard Methods for the Examination of Water and Wastewater (1998), three times a week. Microbiological tests were performed with the samples collected once a month.

The wastewater was prepared in the laboratory mixing the acid and alkaline extraction discharge from the bleach plant, as indicated in Table 2. Thus, the experiments described in this work simulate the treatment of the segregated stream of a bleach plant. The same proportion found in their discharge, 60% acid extraction and 40% alkaline extraction, was used in this work. The wastewater and the sludge were supplied by Ripasa S.A. Limeira-SP, Brazil, a large integrated pulp and paper mill that processes eucalyptus.

### Table 1: Micronutrients and buffer solution.

| Compound                                    | Concentration (g L\(^{-1}\)) |
|---------------------------------------------|------------------------------|
| **Micronutrient solution**                  |                              |
| Ferric chloride (FeCl\(_3\).6H\(_2\)O)      | 0.25                         |
| Calcium chloride (CaCl\(_2\))              | 27.5                         |
| Magnesium sulfate (MgSO\(_4\).7H\(_2\)O)   | 22.5                         |
| **Phosphate buffer solution**               |                              |
| Potassium dihydrogen phosphate (KH\(_2\)PO\(_4\)) | 8.5                           |
| Dipotassium hydrogen phosphate (K\(_2\)HPO\(_4\)) | 21.75                     |
| Sodium phosphate dibasic heptahydrate (Na\(_2\)HPO\(_4\).7H\(_2\)O) | 33.4                        |
| Ammonium chloride (NH\(_4\)Cl)             | 1.7                          |

Source: Authors.

### Table 2: Main characteristics of the bleach plant wastewater.

| Parameter                          | Concentration (mg L\(^{-1}\)) |
|------------------------------------|-------------------------------|
| Chemical Oxgen Demand (COD)        | 2640                          |
| Biochemical Oxgen Demand (BOD)     | 1090                          |
| Nitrogen                           | 5.4                           |
| Phosphorus                         | 0.90                          |

Source: Authors.
2.2 TESTS TO DETERMINE THE KINETIC CONSTANTS

To determine the kinetic constants, tests were performed for the two adapted reactors (R1 and R2). For these tests, 500 mL of the mixed liquor supernatant was removed from the reactors after sedimentation, leaving only the acclimated aerobic sludge and then the reactors were filled with a new bleaching substrate to complete the volume of 1 L, which is the starting point of the tests \( t = 0 \). During the test, samples were taken at different time intervals for 96 hours (4 days) to determine COD (Chemical Oxygen Demand) and VSS (Volatile Suspended Solid), obtaining the experimental points that were used to fit the kinetic constants of the thermophilic aerobic system.

A smoothed curve was obtained from those experimental points using a nonlinear regression (Nelles, 2001). With this smoothed curve as input, the algorithm estimated the kinetic parameters using a mathematical model. This model considered a batch reactor, in which a mass balance was performed for both bacterial growth (biomass) and for substrate consumption, using the Monod model for the specific growth rate \( \mu \), equations (1) and (2):

\[
\frac{dX}{dt} = \mu_{\text{max}} \frac{S}{K_S + S} X - K_d X
\]

(1)

\[
\frac{dS}{dt} = \frac{\mu_{\text{max}}}{Y} \frac{S}{K_S + S} X
\]

(2)

where \( X \) is the biomass concentration in the reactor, measured as VSS (mg\text{VSS L}^{-1}); \( \mu_{\text{max}} \) is the maximum specific growth rate (d\text{\textsuperscript{-1}}); \( t \) is time (d); \( K_d \) is the endogenous respiration coefficient (d\text{\textsuperscript{-1}}); \( S \) is the limiting substrate concentration (mg\text{COD L}^{-1}); \( K_S \) is the saturation concentration (mg\text{COD L}^{-1}); \( Y \) is the cell yield coefficient (mg\text{VSS mg\text{COD}^{-1}}).

The model developed was fitted to the smoothed experimental data obtained for both reactors (R1 and R2), while the estimation algorithm uses the least squares method to find the kinetic parameters. The growth phase was used for estimating \( \mu_{\text{max}}, K_S \) and \( Y \), using equation (1) without the term corresponding to the biomass decay, simultaneously.
with equation (2). The term $K_d$ was estimated by simultaneously integrating equations (1) and (2), using the parameter values previously determined. The analysis software was made in MATLAB.

3 RESULTS AND DISCUSSION

3.1 AEROBIC SLUDGE ACCLIMATION

After 95 days of operation, the acclimation of the mesophilic aerobic sludge to thermophilic was achieved for both strategies. After this period, the reactors were operated for an additional 94-day period.

Figure 1 shows the pH values during the acclimation and operation periods for both reactors. The pH at the reactor R1 has increased considerably in the first 10 days of acclimation, reaching values close to 9.0. In the remaining period of the adjustment the pH remained oscillating between 8.0 and 8.5. For the reactor R2, the behavior of this parameter was different in both acclimation stages. In the first stage, the reactor was fed only with the synthetic substrate and the pH ranged from 6.5 to 8.2. Once the gradual change with the bleaching substrate began, the second stage, the pH gradually increased, reaching approximately 9.0. This effect probably happened due to the characteristics of the wastewater.

![Figure 1: Values of pH over time for both acclimation strategies.](image)

Source: Authors.
Both reactors presented similar behavior after complete substitution, with the pH oscillating between 8 and 9. Other authors (Tripathi and Allen, 1999) also indicate a higher pH inside the reactor than in the feed wastewater. Since their experiments were performed with the final wastewater from the bleached kraft pulp mill and this experiment was performed with the bleach plant effluent, it seems that the high pH at the operation is a characteristic of the thermophilic treatment of pulp mill wastewaters. It should be mentioned that the wastewater from this work differs substantially from the wastewater used by Tripathi and Allen (1999).

Figure 2 shows the COD values determined during the acclimation period of the aerobic sludge and during the remaining operating period of both reactors. As can be seen in the first 65 days of operation, both reactors shown similar tendencies, differing in the greater COD removal achieved by the strategy II (in reactor R2), when compared with the strategy I (in the reactor R1). When, on day 65, the gradual substitution of the synthetic substrate by the bleaching effluent began in the reactor R2, the COD effluent tends to increase in its concentration value, reaching the same level as the value seen in the reactor R1 at the end of the acclimation period. In the second operating period, the COD concentrations for the reactor R1 remained practically constant, reaching a 60% removal rate, while a 64% removal rate was obtained for the reactor R2. Visual inspection of the results indicates that the efficiency in the reactor R2 was gradually increasing, while the efficiency in the reactor R1 was constant. However, the authors do not have sufficient data to estimate the final efficiency attainable with the reactor R2. Similar results were reported by Tripathi and Allen (1999) and Jahren et al. (2002), operating at 55°C; and by Suvilampi et al. (2005) operating at the same temperature with a synthetic effluent prepared from samples collected at a paper and pulp mill. However, Vogelaar et al. (2002) treated wastewater from a recycling paper plant at 55°C, obtaining only 48% COD removal.
Figure 2: Effluent COD values for reactors R1 and R2.

![COD concentrations over time](image)

Source: Authors.

Figure 3 shows the values of TSS (Total Suspended Solids) and VSS (Volatile Suspended Solids) determined during the acclimation and operating period. A similar behavior was observed for both parameters. Figure 3b shows the biomass (VSS). A considerable loss of biomass was observed initially in the effluent for both strategies, probably due to the thermal shock. After the first ten days of acclimation, both reactors presented approximately the same amount of biomass. However, after this initial period of adjustment, the amount of biomass remained practically constant at R1 up to the end of the experiment, while for R2, after the initial period, the biomass increased up to the beginning of the gradual bleach substrate substitution. From this point on, the biomass decreased considerably, a process possibly caused by the toxic compounds found in the bleaching substrate, combined with a reduced availability of readily biodegradable matter. For the final operating period (steady state), both reactors showed similar behavior: apparently both achieved stability, with higher biomass concentration at R2, which was also observed by Tripathi and Allend, (1999). It is noticeable, however, that from day 98 on, the biomass in reactor R2 shows an overall tendency to increase (1340 mg VSS d⁻¹) while at reactor R1 the biomass tends to decrease (500 mg VSS d⁻¹). The sludge from the reactor R1 presented better sedimentation properties than the sludge from the reactor R2, as observed by empirical inspection of the effluents. Furthermore, for both reactors the
effluent exhibited high turbidity, also observed by other researchers (Jenkins et al., 1993; Vogelaar et al., 2002; Suvilampi et al., 2003).

Figure 3: Values of TSS and VSS (biomass) determined over time during acclimation of the aerobic sludge and operation for both strategies studied.

Figure 4 shows the microorganisms of the biomass after the sludge acclimation for a period of 3 months. Figure 4a, which corresponds to R1, shows that there is a smaller number of filamentous microorganisms and a larger number of inert material in comparison with R2 (Figure 4b). This may explain the greater sedimentation readiness of the first strategy when compared with the second one. Besides, a reduction in bacterial
growth was observed in both reactors at this operating temperature, an aspect related to its high requirement maintenance, as mentioned elsewhere (Jenkins et al., 1993; Vogelaar et al., 2002).

Figure 4: Biomass formed during the adaptation of aerobic sludge in R1 (a) and R2 (b).

3.2 KINETIC CONSTANTS

Table 3 shows the values of the kinetic parameters determined according to the procedure described above. As can be seen in Table 3, the values of the kinetic parameters determined for $\mu_{\text{max}}$ and $Y$ are similar for both reactors, differing only in the saturation constant ($K_S$): this value for R1 (the reactor whose sludge was adapted with a bleaching substrate) is eight times greater than R2 (the reactor whose sludge was acclimated with a synthetic substrate). Hence, considering that the other parameters are equal for both reactors, the substrate consumption in reactor R2 is always faster than in reactor R1. For both reactors, low values of specific growth rate were determined, probably on account of the high operating temperature that causes greater maintenance needs and consequently the reduction of bacterial growth (Jenkins et al., 1993). The coefficient values of the cellular yield were lower than those determined by Jackson (1993) for the effluent of the box board manufacturing operating at 53 ºC and by Couillard et al. (1989) for industrial effluent operating at 52 ºC. A lower biodegradability appeared in this study which is an expected characteristic of the bleach plant effluent used in this experiment.

The estimation for the endogenous respiration coefficient, also seen in Table 3, were similar for both reactors, and lower than those values reported by Jackson (1993) and Couillard et al. (1989). However, the mentioned authors dealt with wastewaters that
are more biodegradable than the raw bleach plant effluent from a pulp mill (Jackson, 1993; Couillard et al., 1989).

Table 3: Results of the kinetic parameters obtained by adjusting the model of both reactors and industrial waste in the literature

| Kinetic parameters | R1 5°C | R2 55°C | Jackson Industrial waste (53°C) | Couillard Industrial waste (52°C) |
|--------------------|--------|---------|---------------------------------|----------------------------------|
| \( \mu_{\text{max}} \) (d\(^{-1}\)) | 0.5  | 0.4  | 3.4  | 6.0  |
| \( K_S \) (mgCOD L\(^{-1}\)) | 22  | 2  | 2  | 0.6  |
| \( Y \) (mgVSS mgCOD\(^{-1}\)) | 0.1  | 0.7  | 0.6  | 0.3  |
| \( (\text{error})^2 \) (*) | 3.43  | 0.63  | 0.5  | 0.3  |
| \( K_d \) (d\(^{-1}\)) | 21  | 2.35  | 3  | 0.3  |
| \( (\text{error})^2 \) (**) | 2.21  | 3.35  | 2  | 0.3  |

* for the estimate of \( \mu_{\text{max}}, K_S \) and \( Y \), ** for the estimation of \( K_d \).

Source: Authors.

Figures 5 and 6 show the graphs of the COD and VSS concentration as a function of time for reactors R1 and R2, with the smoothed experimental data and mathematical model used to estimate \( \mu_{\text{max}}, K_S \) and \( Y \). For both reactors, a good fit of the smoothed experimental data to the model can be observed, especially for the biomass (VSS) concentration, as verified by the small (error)\(^2\) presented in Table 3.
Figure 5: Adjustment of the model to experimental data to determine the kinetic parameters ($\mu_{\text{max}}$, $K_S$, and $Y$) for R1 (a, b).

Source: Authors.
Figure 6: Adjustment of the model to experimental data to determine the kinetic parameters parameters ($\mu_{\text{max}}$, $K_S$ and $Y$) for R2 (a, b).

Figures 7 and 8 illustrate the graphs of the COD and VSS concentration as a function of time for reactors R1 and R2 with the smoothed experimental data and mathematical model used to estimate $K_d$. For both reactors a reasonable fit of the smoothed experimental data to the model is observed, as can be verified by the $(error)^2$ in Table 3.
Figure 7: Adjustment of the model to experimental data to determine the endogenous respiration coefficient ($K_d$) for R1 (a, b).

Source: Authors.
Figure 8: Adjustment of the model to experimental data to determine the endogenous respiration coefficient ($K_d$) for R2 (a, b).

4 CONCLUSIONS

This work found that, for this type of substrate (bleach plant wastewater from a kraft pulp mill), the best acclimation strategy of mesophilic aerobic sludge to thermophilic conditions is to first perform the acclimation with the synthetic substrate and then gradually conduct the substitution with the bleaching wastewater. Thus, it is obtaining the formation of a larger amount of biomass, despite the sludge sedimentation difficulty at the beginning of the operation, which has improved over time. The amount of biomass in the acclimated reactor with strategy II (R2) after 189 days of operation was kept around 1340 mg L$^{-1}$, a value that is within the suitable range for the activated sludge process. For the reactor R1, acclimated according to strategy I, the concentration remained at around 500 mg L$^{-1}$, an inadequate amount for aerobic treatment by activated sludge. The kinetic values inferred by the algorithm reinforce the difference in how the biomass acclimation was performed in the thermophilic process, thus indicating the importance of a good start-up procedure in order reach high efficiencies in the wastewater.
treatment plant. The value of cellular yield coefficient ($Y$) of reactor R2, which is two times the value found for R1, is explained by its better acclimation to the thermophilic environment.
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