



Moving bed biofilm reactor for treatment of Kraft pulp effluent with high organic load rate

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ABSTRACT

The pulp industry uses more than 40 m³ of water per ton of pulp produced, generating high effluent flows. In general, it presents high concentrations of organic matter, color and ecotoxicity. The most widely used effluent treatment systems in the pulp industry are biological, including moving bed biofilm systems that are efficient in removing biodegradable organic matter. This work evaluated the removal of organic matter, total phenolic compounds, color and lignin derivatives in the treatment of Kraft cellulose effluent using the moving bed biofilm reactor (MBBR), and also evaluated the support media biofilm development by solid analysis and scanning electron microscopy. The parameters evaluated during treatment were: BOD₅, COD, color, total phenolic compounds, lignin derivatives, and solids, with tests performed on organic loads from 0.7 and 8.9 kg_{COD} m⁻³ d⁻¹. Organic matter removal remained stable, being over 80% to BOD₅ and over 42% to COD. The color and the total phenolic compounds were removed up to approximately 7 and 28%, respectively. Over 19% removal of derivatives of lignin compounds was observed in both organic load rates. In the MBBR, biofilm was confirmed and enabled this biological system to treat the cellulose effluent in a stable way.

Keywords: organic matter, recalcitrant compounds, SEM.

Reator de leito móvel para tratamento de efluente de celulose Kraft em carga orgânica volumétrica elevada

RESUMO

A indústria de celulose utiliza mais de 40 m³ de água por tonelada de celulose produzida, gerando grandes volumes de efluentes. Em geral, possui altas concentrações de matéria orgânica, cor e ecotoxicidade. Os sistemas de tratamento de efluentes mais amplamente utilizados na indústria de celulose são os biológicos, incluindo sistemas de reator de leito móvel que são eficientes na remoção de matéria orgânica biodegradável. O presente trabalho teve como objetivo avaliar a remoção de matéria orgânica, compostos fenólicos totais, cor e derivados de lignina no tratamento de efluente de celulose Kraft através do reator de leito móvel (MBBR), bem como avaliar o desenvolvimento de biofilme de meios de suporte por análise de sólidos e microscopia eletrônica de varredura. Os parâmetros avaliados durante o tratamento foram: DBO₅, DQO, cor, compostos fenólicos totais, derivados de lignina e sólidos, com testes



realizados em cargas orgânicas de 0,7 e 8,9 kg_{DQO} m⁻³ d⁻¹. A remoção de matéria orgânica permaneceu estável, sendo superior a 80% para DBO₅ e acima de 42% para DQO. A cor e os compostos fenólicos totais foram removidos acima de 7 e 28%, respectivamente. Foi observado remoção de compostos derivados de lignina acima de 19% nas cargas avaliadas. O biofilme formado no MBBR foi observado e confere estabilidade ao sistema biológico no tratamento do efluente de celulose Kraft.

Palavras-chave: compostos recalcitrantes, matéria orgânica, MEV.

1. INTRODUCTION

The Kraft pulping process is the most widely used pulp production in the world. There is high water consumption in the pulp industry, as more than 40 m³ of water is consumed per ton of pulp produced, which in turn generates large volumes of effluent. In general, this effluent is rich in organic matter, suspended solids, resin acids, lignin, color and ecotoxicity (Bachmann, 2009; Bakraoui *et al.*, 2020; Furley *et al.*, 2015; 2018; Hossain and Ismail, 2015; Toczyłowska-Mamińska, 2017).

Most industrial effluent treatment systems are biological, and these are also the most used in the pulp and paper industry (Cabrera, 2017). In these industrial sectors, activated sludge, biological filters, aerated lagoons, moving bed biofilm reactors (MBBR), etc. are most often used. (Hubbe *et al.*, 2016).

The MBBR system consists in keeping part of the mixed liquor in suspension in the liquid mass, and part adhered to the support media. It uses hydraulic retention times of 3 to 48 hours. The support media provide adhesion surface for microbial biomass and present high mobility (Leyva-Díaz *et al.*, 2017). The support media applied in this type of biological treatment are generally made of high-density polyethylene or polypropylene. They are inert and have a high specific surface area (>1000 m² m⁻³) (Leyva-Díaz *et al.*, 2017).

Among them, there is AQUAPOROUSGEL[®] (APG) from Nisshinbo Chemical Inc. It presents specific surface area greater than 3000 m² m⁻³, specific mass of 30 drykg/wetm³, multiporous, and its material is based on polyethylene-glycol (Sakuma, 2004). According to the manufacturer, the filling ratio for an efficient treatment is 10% of the reactor volume, a value lower than other support media, which are in the range of 25 to 70% (Haandel and Lubbe, 2012; Sakuma, 2004).

This work evaluated the performance of a moving bed biofilm reactor with spongy support media with regard to organic matter, total phenolic compounds, color, and derivatives lignin compounds removal, and also evaluated biofilm development in the support media during treatment.

2. MATERIALS AND METHODS

The effluent used for the treatment with MBBR (Moving Bed Biofilm Reactor) was obtained from an industry of unbleached Kraft pulp in Curitiba region, Brazil. The effluent was collected twice at different times before biological treatment, transported and stored at 4°C protected from light in 30 L vessels. The treatment occurred in a continuous flow reactor, in lab scale, with 1 L of volume, filled with 10% of spongy support AQUAPOROUSGEL[®] (APG), 8 cm³ per unit, by Nisshinbo Inc. The MBBR reactor operation was at two different organic load rates (OLR) over a total of 48 days of operation.

The operation of the reactor occurred at two different organic loading rates (OLR): 0.7 kg_{COD} m⁻³ d⁻¹ and 8.9 kg_{COD} m⁻³ d⁻¹ (Phases I and II, respectively). The hydraulic retention times (HRT) were 13.3 and 2.8 h to achieve those two loads worked. Those organic load rates were

based on Peitz and Xavier (2017) and Vanzetto *et al.* (2014), who applied a load of $9.0 \text{ kg}_{\text{COD}} \text{ m}^{-3} \text{ d}^{-1}$ and HRT between 2 and 3 h with real Kraft pulp effluent and AMB media support.

The treatment lasted 48 days, 30 days in Phase I, as an adaptation step for growing biomass by itself without inoculation, and 18 days in Phase II until steady state. The steady state was reached when the system got as far as organic matter removal variation (COD and BOD_5) of up to 10%.

Both Influent and Effluent were characterized regarding the analytical parameters: Biochemical Oxygen Demand – BOD_5 ; Chemical Oxygen Demand – COD; Total Phenolic Compounds – TPC ($\text{UV}_{215\text{nm}}$); color ($\text{Vis}_{440\text{nm}}$), aromatic compounds ($\text{UV}_{254\text{nm}}$), lignin derived compounds ($\text{UV}_{280\text{nm}}$) and liginosulfonic acid ($\text{UV}_{346\text{nm}}$), in samples filtered with nitrocellulose membrane of $0.45 \mu\text{m}$ porosity. All of them were monitored during continuous biological treatment (APHA *et al.*, 2012; Çeçen, 2003; Morales *et al.*, 2015).

Adhered solids (AS) were considered as the biofilm formed in the spongy support during biological treatment. The biofilm adhered to the spongy carrier was quantified by suitability of the APHA *et al.* (2012), in which the APG samples were subjected to a 100 mL volume ultrasonic bath in deionized water for one (1) hour, being sequentially conducted according to the same method. Suspended solids (SS) were considered as the biomass present in the mixed liquor and were measured according to APHA *et al.* (2012). Both determinations were performed in triplicate during the steady state of each applied organic load rate (Phase I and II). The results were expressed by total and volatile adhered solids (TAS and VAS) and total and volatile suspended solids (TSS and VSS), as mg L^{-1} (APHA *et al.*, 2012).

Scanning electron microscopy (SEM) analyses were performed at the Multiuser Materials Characterization Center of the Federal Technological University of Paraná (UTFPR). For the analyses, samples of the spongy support medium were collected at the end of each applied organic load rate (Phase I and Phase II), dried in a lyophilizer and stored in a desiccator for further analysis of SEM.

3. RESULTS AND DISCUSSIONS

3.1. Characterization of the effluent

In Table 1 is shown the characterization of two samples collected from the same industry of Kraft mill effluent treated over 48 days by moving bed biofilm reactor (MBBR).

Table 1. Characterization of samples treated during biological treatment and operation conditions.

Parameter	OLR ^e / ($\text{kg}_{\text{COD}} \text{ m}^{-3} \text{ d}^{-1}$)	
	0.7	8.9
COD ^a (mg L^{-1})	371.6 ± 57.8	1025.0 ± 138.2
BOD_5^{b} (mg L^{-1})	125.6 ± 23.5	220.4 ± 41.2
BOD_5/COD	0.34	0.21
TPC ^c (mg L^{-1})	73.1 ± 49.1	385.8 ± 70.7
Color ($\text{Vis}_{440\text{nm}}$) (1 cm x1 cm)	0.222 ± 0.092	0.402 ± 0.194
Aromatic compounds ($\text{UV}_{254\text{nm}}$) (1 cm x1 cm)	3.191 ± 0.937	6.285 ± 0.914
Lignin derived compounds ($\text{UV}_{280\text{nm}}$) (1 cm x1 cm)	2.786 ± 0.627	6.352 ± 0.943
Liginosulfonic acid compounds ($\text{UV}_{346\text{nm}}$) (1 cm x1 cm)	0.764 ± 0.378	1.381 ± 0.631
Operation (days)	30	18
HRT ^d (h)	13.3	2.8

Note: ^aChemical Oxygen Demand. ^bBiological Oxygen Demand. ^cTotal Phenolic Compounds. ^dHydraulic Retention Time. ^eOrganic Load Rate. During Phase I, n=10, and during Phase II, n=7.

Table 1 showed that the two different samples of effluent from the same industry present organic matter, color, phenolic compounds, and derivatives of lignin compounds. During Phase I, the sample treated had the highest BOD₅/COD ratio (0.34), which was favorable to the biological treatment (Hubbe *et al.*, 2016). The effluent treated during OLR 0.7 kg_{COD} m⁻³ d⁻¹ had total phenolic compounds, 73.1 mg L⁻¹, with low concentration compared to literature (Hossain and Ismail, 2015; Morales *et al.*, 2015).

In the next Phase, with effluent from another collection from the same industry, the BOD₅/COD ratio decreased to 0.21; however, it is still favorable to the treatment. According to Hubbe *et al.* (2016), pulp and paper effluent has BOD₅/COD between 0.05 – 0.50, and the closer to 1.0, the better the biological treatment. This value of 0.21 for BOD₅/COD ratio indicated higher recalcitrance in the sample, and there is a greater presence of lignin derivatives compounds, aromatic and phenolic compounds, all difficult to biodegrade and with high color, as proposed by Maria *et al.* (2014).

In general, the characteristics of the samples in this work agree with those found by different authors (Hubbe *et al.*, 2016).

3.2. Organic matter, color, total phenolic, and lignin derivatives compounds

Figure 1 shows the removals calculated during treatment of the Kraft pulp mill effluent in the MBBR. Regarding organic matter, the system had an average efficiency of 42.6% of COD removal at the lowest load, remaining stable, and 41.8% removal of this parameter in the OLR of 8.9 kg_{COD} m⁻³ d⁻¹. Compared with the MBBR system treating Kraft pulp effluent with phytosterols, in HRT of 3h, COD removal was 40% (Peitz and Xavier, 2017).

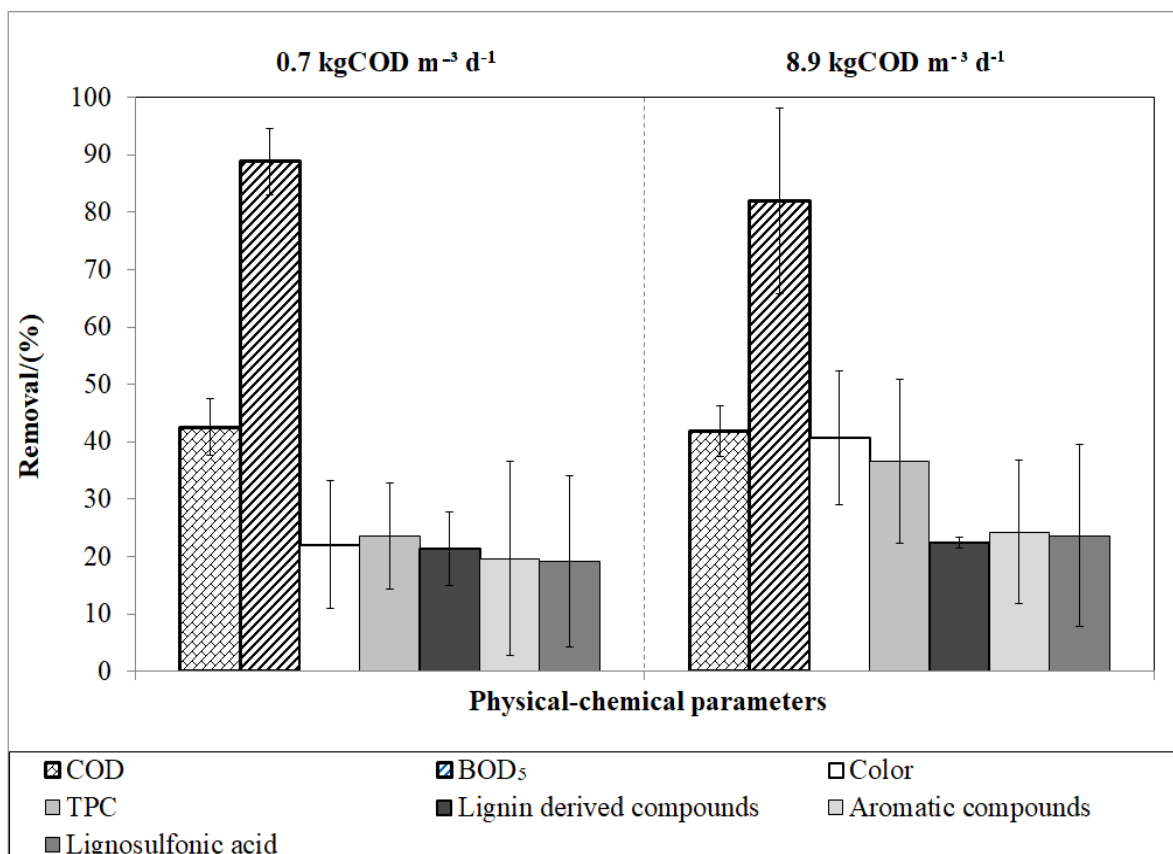


Figure 1. Removals of physical-chemical parameters obtained in the MBBR treating Kraft pulp effluent.

Regarding BOD₅ removal, even with the difference between the BOD₅/COD ratio in these

phases, (Table 1, 0.34 \rightarrow 0.21), a decrease of only 7.7% in the efficiency average removal was found. In the load of 0.7 kg_{COD} m⁻³ d⁻¹, 88.8% of BOD₅ removal was obtained and in the next phase it was 81.9%, showing how stable the MBBR is with high organic loads and shock loads, as was observed by other authors with different media support (Huang *et al.*, 2015; Leyva-Díaz *et al.*, 2017; Qiqi *et al.*, 2012). The treatment of Kraft pulp effluent by MBBR with APG presented stable removal of organic matter even with low value hydraulic retention time (2.8 h).

Regarding color, removal of this parameter was observed in both organic loads, between 22.1% and 40.7% on average. These results were better than those of Peitz and Xavier (2017), who obtained 3% color removal at a HRT of 3 h. This was unexpected in a biological treatment, in which an increase in the effluent color was observed in reactors similar to the moving bed biofilm reactor (Cabrera, 2017; Kamali and Khodaparast, 2015; Wahyudiono *et al.*, 2008).

This behavior can be explained by the fact that the spongy support has adsorbed the effluent color during treatment by MBBR, i.e., the lignin compounds present in the effluent of the Kraft pulp industry, which are mainly responsible for the color, were adsorbed on the sponge material of the APG, which was initially white and in the early stages of the reactor operation became brown. This color changed from brown to black at around 45 days of operation. It is also possible that the MBBR-treated samples were predominantly built of low molecular weight compounds that were metabolized due to their size (Cabrera, 2017).

Regarding the removal of total phenolic compounds (TPC), also shown in Figure 1, the efficiency of TPC removal was about 21% in Phase I and 36.6% in the next load. Peitz and Xavier (2017) obtained 36.4% of TPC removal in OLR 9.0 kg_{COD} m⁻³ d⁻¹, in a similar condition with real Kraft pulp effluent. These removal levels could be explained because of the action of certain bacterial communities. There are some effective species in breaking down recalcitrant compounds like lignin derivatives and phenolic compounds, reducing the color and phenolic compounds of pulp mill effluent this way, even if the removal of color and phenolic compounds in aerobic treatments is not common, as observed by others authors (Cabrera, 2017; Hubbe *et al.*, 2016; Muñoz *et al.*, 2019; Singh *et al.*, 2019).

During the treatment, the removal of the aromatic (UV_{254nm}), ligninic (UV_{280nm}) and lignosulfonic (UV_{346nm}) compounds was observed in all organic loading. Removals were greater than 19% in all parameters in both organic loads applied. The highest removal level was during OLR 8.9 kg_{COD} m⁻³ d⁻¹, 22.4% – 24.3% for lignin derivatives compounds.

Regarding UV_{254nm}/UV_{280nm} ratio, the reduction of the values occurred during the biological treatment of 1.18 to 1.14, during ORL 0.7 kg_{COD} m⁻³ d⁻¹ and 1.04 to 1.01 in Phase II. These values indicated higher amounts of aromatic compounds (UV_{245nm}) in all samples compared to the lignin compounds (UV_{280nm}), which is in agreement with other studies with cellulose effluent (Çeçen, 2003; Morales *et al.*, 2015; Peitz and Xavier, 2017; Villamar *et al.*, 2009; Machado, 2017).

3.3. Biomass adhered and suspended

The result of the quantification of biomass adhered to the support media (AS) and suspended in MBBR (SS) measured during steady state is presented in Figure 2. During the treatment, the system was not inoculated with sludge.

During the aerobic treatment, the biomass grew, mainly attached to APG support media. Volatile Adhered Solids (VAS) of 770.4 mg L⁻¹ were observed in the load 0.7 kg_{COD} m⁻³ d⁻¹ (Figure 2A). Volatile Suspended biomass (VSS) reached a concentration of 233.3 mg L⁻¹ at load 0.7 kg_{COD} m⁻³ d⁻¹, lower than expected (1500–5000 mg L⁻¹ for conventional activated sludge), and 3.3 times lower than VAS. This preference for adhered solids in support media was observed by other authors (Minegatti *et al.*, 2012; 2014; Qiqi *et al.*, 2012; Vanzetto *et al.*, 2014). In the next phase, OLR 8.9 kg_{COD} m⁻³ d⁻¹, both concentrations grew up to 3419.2 and 1432.5 mg L⁻¹, respectively (Sperling, 2014; Toczyłowska-Mamińska, 2017).

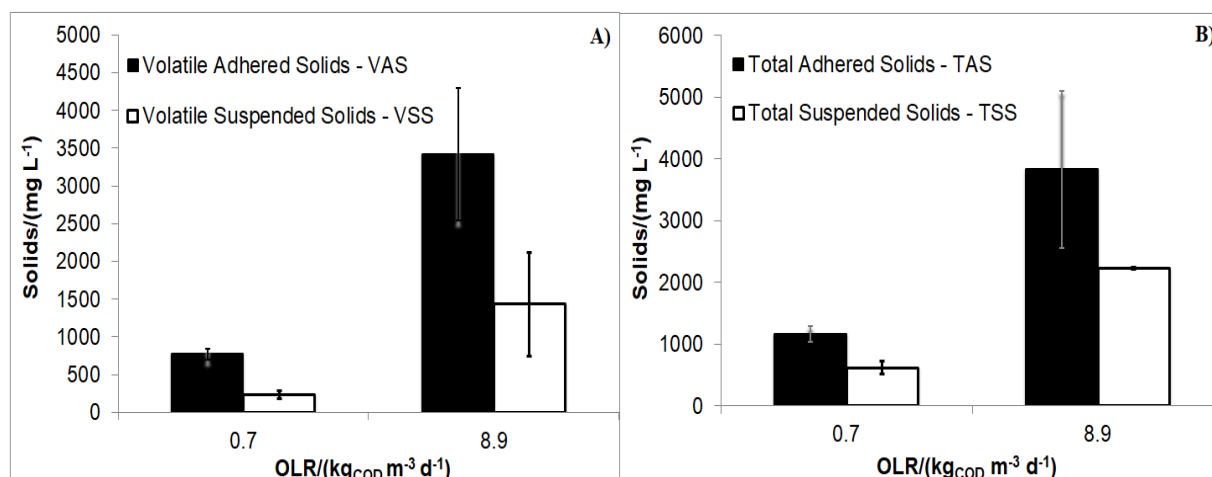


Figure 2. Adhered and suspended solids in the reactor.

Regarding Total Solids, TS, Figure 2B, it arrived at 3831 mg L⁻¹ in the support media (TAS), in agreement with the literature, 3000 – 4000 mg L⁻¹ (Barwal and Chaudhary, 2014). Regarding Total Suspended Solids, the concentration was 2227.5 mg L⁻¹. Table 2 shows the ratios for the different loads applied during treatment by the APG spongy support MBBR reactor.

Table 2. Biomass ratio.

OLR/ (kg _{COD} m ⁻³ d ⁻¹)	VAS ^a /TAS ^b	VSS ^c /TSS ^d
0.7	0.66	0.38
8.9	0.89	0.64

^a: Volatile Adhered Solids (mg L⁻¹). ^b: Total Adhered Solids (mg L⁻¹). ^c: Volatile Suspended Solids (mg L⁻¹) ^d: Total Suspended Solids (mg L⁻¹).

For OLR 0.7 kg_{COD} m⁻³ d⁻¹, the VSS/TSS ratio was 0.38, characterized as a stabilized sludge, with load increase in Phase II, this ratio up to 0.64, as a partially stabilized sludge with high organic matter content, since the biomass was not removed from the system during the treatment (Sperling, 2014). Compared with the VAS/TAS ratio, the sludge is characterized as a partially stabilized sludge, mainly due to the increase of organic load in the system.

These results are in accordance with SEM images, Figure 3, in which were verified biofilm growth in all phases.

In Figure 3A-B, the APG support media has a high surface area with pores of different diameters for biofilm growth. During aerobic treatment, biomass grew mainly linked to support media such as biofilm. In Figure 3B-C, Phase I, with 13.3h HRT, biofilm growth did not fully cover the micropores, allowing biofilm development. At OLR 8.9 kg_{COD} m⁻³ d⁻¹, Figure 3E-F, growth continued and the increase was observed by VAS analysis and formation of extracellular polymeric material seen by SEM.

With the increase in organic load there was a greater development of biofilm (Figure 3E-F), which cooperated in the removal of the parameters analyzed in this biological treatment.

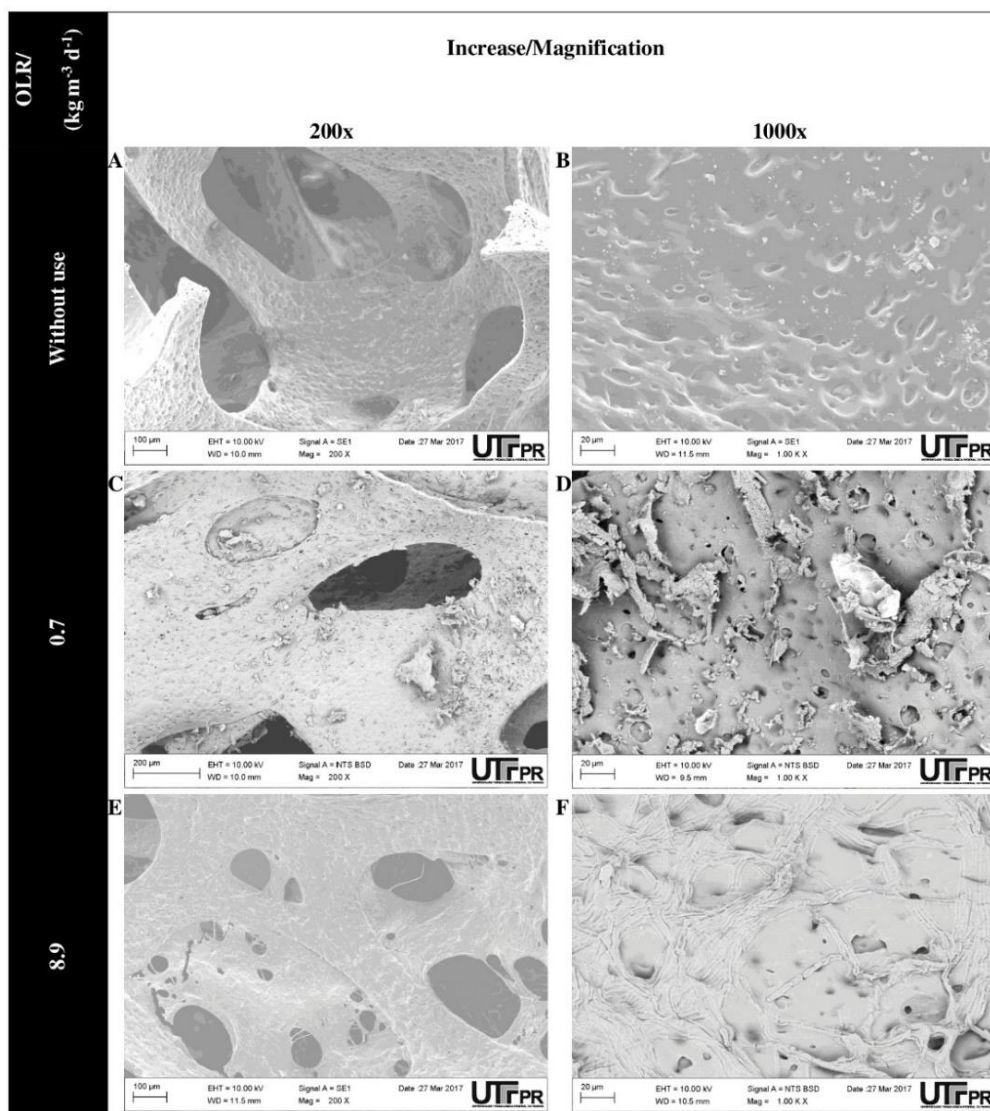


Figure 3. Scanning electron microscopy of APG in each organic load rate applied.

4. CONCLUSIONS

The removal efficiency of organic matter remained stable in relation to COD, remaining above 40%, not presenting large discrepancies between the applied loads, constituting MBBR with APG as a stable system. Considering the removal of BOD₅, a drop of 7.7% being justified by the low biodegradability of treated Kraft pulp effluent, showing the stability of the system in load shocks.

The values achieved for color removal were close to 40% on average for the highest load, which was not expected from a biological treatment. The removal efficiency for total phenolic compounds (TPC) was not negatively affected by the 15-times increase in load, which could be considered another favorable point for the stability of the MBBR with spongy support APG. Regarding biomass, its affinity with the support medium APG was verified because its concentration was 3.3 and 2.4 times higher than the biomass suspended in the reactor in each organic load rate applied. The VSS/TSS ratio for suspended solids was a partially stabilized sludge.

To sum up, it can be affirmed that the MBBR system based on support medium APG on OLR 8.9 kg_{COD} m⁻³ d⁻¹ has the potential to treat Kraft cellulose.

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