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Evaluation of effluent post-treatment by slow filtration and adsorption with activated carbon produced from spent coffee grounds in surfactant removal in sewage treatment

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Marcelo Pinheiro Ribeiro*; Alexandre Botari

Departamento de Tecnologia. Universidade Estadual de Maringá (UEM), Avenida Ângelo Moreira da Fonseca, n° 1800, CEP: 87506-370, Umuarama, PR, Brazil. E-mail: abotari@hotmail.com
*Corresponding author. E-mail: marcelopr@sanepar.com.br

ABSTRACT

Environmental pollution is a worldwide concern, especially when caused by sewage dumping into water bodies. Many substances are present in industrial or domestic wastewater, causing contamination in superficial water collection. Surfactants stand out for being widely used both industrially and domestically. The use of detergents and many types of surfactants was increased during the Covid-19 pandemic period, a fact observed in the levels in the effluent sample analysis from a Sewage Treatment Plant (STP) - Vila City around 21 and 39 mg L-1 in this period. This work evaluated the surfactant concentrations in the primary and secondary treatment units of the Vila City STP, in the city of Paranavaí-PR.-Brazil. In addition, the use of a post-treatment by slow sand filtration and adsorption by activated carbon produced from spent coffee grounds in the complementary removal of surfactants was proposed. A mixed bed with sand and activated carbon columns was made on a pilot scale, and filtration/adsorption runs were performed simulating slow filtration with rates of approximately 15 m3 m-2 day-1. The parameters used for the efficiency removal evaluation in a pilot plant run were: turbidity (NTU) and surfactant concentrations. The removal of surfactant concentrations was about 9% and 7% in the Upflow Anaerobic Sludge Bed reactors (UASB-RALF) and in the secondary treatment, respectively, at the STP – Vila City units. In the post-treatment proposed by filtration/adsorption, bed columns on a pilot scale plant obtained a reduction of approximately 94% in terms of turbidity (NTU) and 95% in terms of surfactant removal.

Keywords: activated carbono, coffee poder, effluent, surfactants.

Avaliação do pós-tratamento de efluentes por filtração lenta e adsorção com carvão ativado produzido a partir da borra de café na remoção de surfactantes em estações de tratamento de esgoto

RESUMO

Poluição ambiental é uma preocupação mundial, especialmente causada por despejos de esgotos nos coleção hídrica. Diversas substâncias compõem os despejos e resíduos industriais e esgotos domésticos. Os surfactantes destacam-se por serem amplamente usados tanto industrialmente como domesticamente. O consumo de detergentes e diversos tipos de tensoativos foi incrementado durante o período de pandemia da Covid-19, fato observado nos

teores de surfactantes no efluente das Estações de Tratamento de Esgoto (ETE) - Vila City entre 21 e 39 mg L-1 neste período de tempo. Este trabalho avaliou as concentrações de surfactantes em diferentes etapas da ETE da Vila City, na cidade de Paranavaí, PR.- Brasil. Além disso, propôs-se a utilização de um pós-tratamento por filtração/adsorção por carvão ativado produzido a partir da borra de café na remoção complementar dos surfactantes. Foram confeccionadas colunas de adsorção com meio filtrante de areia e carvão em escala piloto e realizados ensaios de filtração/adsorção simulando filtração lenta com taxas de aproximadamente 15 m3 m-2 dia-1. Os parâmetros utilizados na avaliação da eficiência nos ensaios em escala piloto foram: turbidez e surfactantes. Obteve-se como resultado, as remoções das concentrações de surfactantes de cerca de 9% e 7% no reator anaeróbio de leito fluidizado (RALF) e no tratamento secundário na ETE, respectivamente. No pós-tratamento proposto em ensaios de filtração/adsorção em escala piloto obteve-se uma redução de aproximadamente 94% de turbidez (uT) e de 95% de surfactantes, respectivamente.

Palavras-chave: carvão ativado de borra de café, efluente, surfactante.

1. INTRODUCTION

Environmental pollution is a worldwide concern, mainly caused by the discharge of sewage into water bodies. To measure the level of pollution and contamination, environmental legislation uses some quality indicators in samples collected, such as oils and greases, surfactants, organic matter, or phosphorus (Metcalf and Eddy, 2014). Among these parameters, surfactants stand out for being widely used in industries, and daily in households as a detergent; in this way, surfactants are among the main pollutants found in sewers (Gül, 2020).

The significant use of detergents in Brazil and the lack of domestic sewage treatment in urban regions have caused undesirable environmental effects (Gül, 2020). The presence of these can cause the inhibition or paralysis of the natural or artificial purification due to the formation of foams, disturbances in the oxygen conduction through the membranes of the aquatic organisms, and the eutrophication of surface waters due to the presence of phosphates present in the surfactants (Costa *et al.*, 2007).

Studies show that the average concentration of surfactants in the sanitary sewer is 4.55 mgL-1 (Amigo, 1998). However, there are no maximum values for surfactants in federal legislation that may be present in effluents to be released into superficial water collection. Considering the presence of surfactants in industrial and domestic sewers, which often exceed the limit concentrations for dumping in river waters, it is necessary to implement techniques and processes to reduce the concentration of these compounds, such as, for example, filtration and adsorption.

It is known that the soluble coffee processing industries generate considerable volumes of residues from the coffee extraction stage, the spent coffee dreg sludge. It is estimated that 48% of the amount of raw material used is converted into spent coffee dreg. In 2005, 6 million tons of coffee powder was generated worldwide (Tokimoto *et al.*, 2005). The coffee residue is generally collected as organic waste and disposed of in landfills, where it decomposes, generating methane, thus contributing to climate change. Such residue is formed by high organic content, such as carbohydrates, proteins, fibers, caffeine, polyphenols, tannins, and pectins, compounds that can be treated or valued in different ways to reduce the environmental impact. The major component of coffee residue is carbon, which can be used in adsorption processes such as activated carbon (Jutakridsada *et al.*, 2016).

Considering the need to control the levels of surfactants and having spent coffee dreg as a by-product and adsorbent potential, the present study aims to verify the efficiency of surfactant removal at the Vila City Sewage Treatment Plant (STP) in the city of Paranavaí - Paraná - Brazil

and to analyze the efficiency of the pilot-scale plant filtration/adsorption process as a post-treatment using activated carbon produced from spent coffee dreg and slow sand filtration to remove surfactants and turbidity, respectively.

2. MATERIAL AND METHODS

The surfactant removal experiments were carried out using a sustainable method, at the Vila City STP in Paranavaí City-Paraná-Brazil, located at Avenida Mariano Morangueira. The STP Vila City has an average flow of 130.5 m3 h-1 and a maximum effluent flow of 240 m3 h-1. At this station, 40% of sanitary sewage is received from the municipality of Paranavaí. All the experiments carried out in the present study with activated carbon derived from spent coffee dreg were developed at the Sewers Analysis Regional Laboratory of the Companhia Paranaense de Saneamento (SANEPAR), in the city of Paranavaí

2.1. Surfactants Determination Methods

For surfactants, concentration measurements used the methylene blue method for anionic surfactants, according to the 5540-C method (APHA *et al.*, 2012). In this method, the samples' anionic surfactants react with the methylene blue dye to form a blue complex, which is extracted with chloroform. Therefore, the samples were analyzed immediately after their collection; 10 collections were carried out from May 2019 to February 2020. The collection points are located at the arriving sewage in the treatment plant, after the Upflow Anaerobic Sludge Batch Reactor (UASB-RALF), and after secondary treatment – final effluent.

The samples were diluted by adding 2.5 mL of the effluent, making up to 100 mL with deionized water. These were added in a 500 mL separating funnel, where 1 M sodium hydroxide solution was added by droplets until the solution was alkaline, with basicity identified by phenolphthalein (pink color). Then, the 1 M sulfuric acid solution was added by droplets until the solution became colorless. Another 10 mL of chloroform and 25 ml of methylene blue reagent were added (previously prepared by diluting 100 mg of methylene blue in 100 mL of water, 30 ml of which were pipetted in a 1000 mL volumetric flask).

The separating funnel was stirred for 30 seconds, and this procedure was performed with three successive extractions with 10 mL aliquots of chloroform each. Moreover, 50 mL of the washing solution was added to the organic phase, prepared from 6.8 mL of concentrated sulfuric acid, 50 g of monobasic sodium phosphate monohydrate, added to 1000 mL with deionized water, the extraction being repeated twice.

After that, the organic phase was collected in a 100 mL flask and the volume was completed with chloroform, the blue color of the organic phase being determined spectrophotometrically at 652 nm wavelength. The reading given in mg L-1 was multiplied by 40, considering the initial dilution of 2.5 per 100.

2.2. Turbidity Measurements

The turbidity of the samples was determined using the Turbidimeter Model AP2000 WT (Policontrol). After the instrument was properly calibrated, the cuvette was completed with the homogenized sample up to the mark. The turbidity values of the sample in (NTU) are shown on the equipment display.

2.3. Efficiency removal in Sewage Treatment Plant Vila City – Paranavaí -PR

The variations in the surfactant concentrations samples were evaluated by Standard Deviation (*SD*) and the Coefficient of Variation (*CV*). The standard deviation (Equation 1) is a measure that expresses the degree of dispersion of a data set, that is, the standard deviation indicates how uniform a data set is.

$$SD = \sqrt{\frac{\sum(x_i - \bar{x})^2}{n-1}} \quad (1)$$

Where: *SD* is the Standard Deviation; x_i is the value at position *i* in the data set; n is the number of samples and $\underline{x}$ is the arithmetic average.

The coefficient of variation (Equation 2) provides the variation of the data concerning the average. The lower its value, the more homogeneous the data will be. The coefficient of variation is considered low (pointing to a very homogeneous data set) when it is less than or equal to 25%.

$$CV = \frac{SD}{\underline{x}} 100 \quad (2)$$

The removal efficiency (*E%*) of surfactants by the wastewater treatment process (STP – Vila City) was calculated according to Equation 3.

$$E\ (\%) = \frac{=C_{in} - C_{out}}{C_{in}} . 100 \quad (3)$$

Where C_{in} is the surfactants concentration in the sewage inlet unit reactor from STP Vila City and C_{out} is the surfactants concentration in the sewage outlet unit reactor from STP Vila City.

2.4. Precursor material preparation for the activated carbon production

A total of 50 g of spent coffee dreg previously dried in a kiln were weighed on an analytical balance (Shimadzu). With the aid of qualitative filter paper (Whatman 90 mm), funnel, kitassato, and a vacuum pump, the sample was washed with deionized water to remove soluble compounds. This procedure was carried out until the water resulting from the washing was clear. After washing, the sample was placed in a kiln at a temperature of 120ºC for 2 hours for drying. After that time, the samples were cooled in a desiccator and stored in closed containers (Ahsan *et al.*, 2018).

2.5. Activation Process

The chemical activation process was adapted from Figueiredo *et al.* (2017). For this, 50 g of washed and dried spent coffee dreg were weighed in a porcelain capsule, and with a beaker, 50 mL of a solution of the activating agent (H_3PO_4 - phosphoric acid 85%) was added, ensuring a ratio of 1:1 (in mass) (spent coffee dreg /activating agent solution).

The powder (spent coffee dreg) was homogenized with a spatula, so that the activator would impregnate the entire mass of spent coffee dreg, and was left in dynamic contact for 12 hours in a desiccator, protected from light and contaminants at a controlled temperature of approximately 20ºC.

After that time, the samples were filtered with the aid of a vacuum pump, kitassato, and qualitative filter paper (Whatman 90 mm) to remove the excess activating solution, and transferred again to a porcelain capsule for carbonization in the muffle furnace. The increase of the temperature was progressive in these steps: 350ºC, 450ºC, 550ºC, 650ºC, 750ºC and 850ºC, for 1 hour in each temperature range. Subsequently, the sample was cooled to environmental temperature. Further, was stored in a desiccator, then the activated carbon was macerated with a porcelain mortar and pestle passed through a 3 mm sieve and then stored in closed containers and protected from light.

2.6. Fixed bed filtration and adsorption essays (Pilot Plant Scale Filtration)

To carry out the study of filtration and adsorption in a fixed bed, a filter was assembled filled with the carbon produced and activated with phosphoric acid. The filter, shown in Figure 1, was constructed with a PVC pipe, which has a diameter of 100 mm and a height of 1.5 m. A perforated PVC plate was inserted to support the layer and the porous media bed filter. The porous media layer was constituted by two layers of sand with 10 cm each with their respective sizes starting from the bottom layer: 1.18 mm (medium-sized sand) and 0.3 mm (smaller-sized sand), a layer of 10 cm of activated carbon produced by spent coffee dreg (powder) and two sand layers of 10 cm each were repeated into the top of filter media, the same as described in the bottom ones.

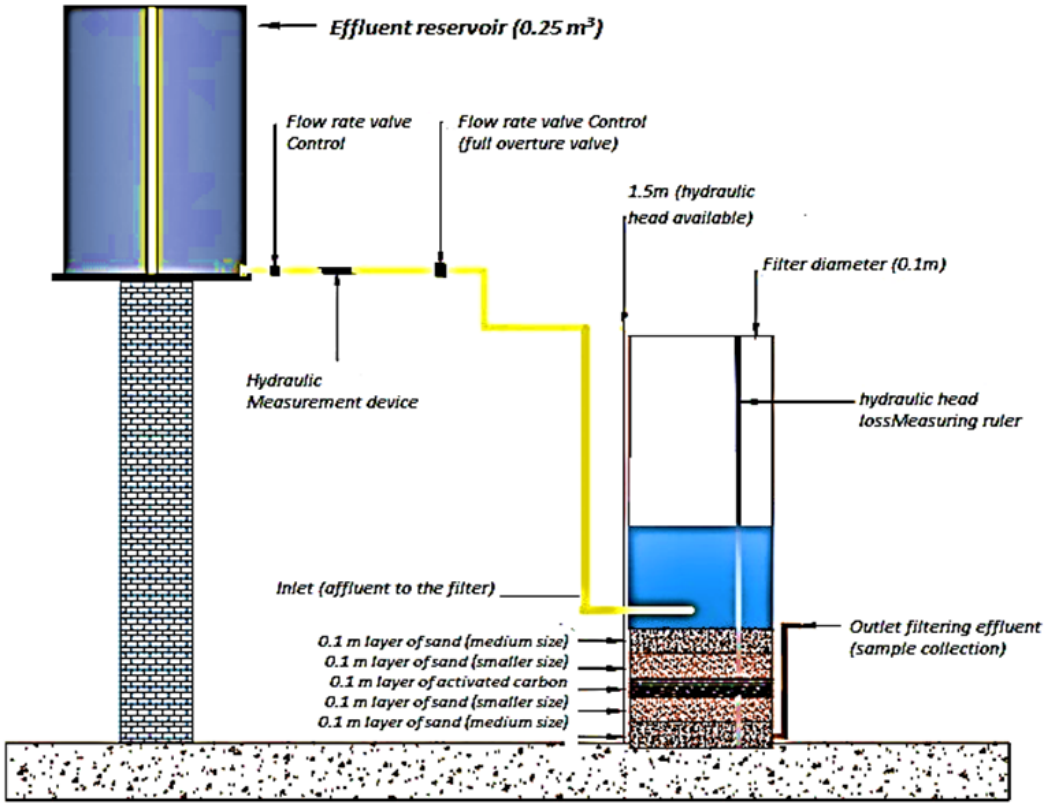

Figure 1. Schematic diagram of the installation on a pilot scale of slow filtration/adsorption using activated carbon produced from spent coffee dreg.

Figure 1 shows the Pilot Plant Scale Filtration apparatus was placed in a reservoir with a capacity of 500 L (of which it occupied 250 L), located above the filters, therefore allowing the hydraulic flow by gravity. The solution from the reservoir to the filter was conducted by pipeline and the flow was controlled by hydraulic valves. In addition, at the bottom of the filter, a device was also installed that allows positive pressure in the internal porous media bed filtration/absorption medium. The outlet pipeline also allowed the sample collection of filtered effluent.

The samples were collected hourly for 9 hours of experimental run. Four experimental filtration and adsorption runs were conducted. The sample measurements were surfactant concentrations or contaminant concentrations and turbidity. The samples were measured until removal efficiency reached values below 20%. During the running of the experiment, the hydraulic head loss measure was carried out in the Filtration Pilot Plant.

The slow filtration experimental runs were performed with a rate of 15 m3 m-2 day-1 and natural pH of the solution (temperature around 25ºC). Before being used, the sands were washed with deionized water before beginning the tests for slow filtration.

3. RESULTS AND DISCUSSION

3.1. Evaluation of the efficiency of removal of surfactants at Vila City STP

The descriptive statistics of the data referring to surfactant analysis at the beginning of the season, after the UASB-RALF and after the secondary treatment, is shown in Table 1.

Table 1. Descriptive statistics of surfactant data in samples at Vila City STP.

Parameters	Effluent	UASB-RALF	Secondary post-treatment
Number of samples	10	10	10
Average (mg/L)	23.15	21.43	21.83
Standard deviation (mg/L)	9.72	10.78	9.90
Coefficient of variation (%)	42	50	45
Minimum (mg/L)	6.94	1.97	3.67
Maximum (mg/L)	39.12	34.32	37.04

For the effluent, the surfactants varied between 6.94 and 39.12 mg L-1, after the UASB-RALF they varied between 1.97 and 34.32 mg L-1 and after the secondary treatment, they varied between 3.67 and 37.04 mg L-1. The average value observed for the effluent is very close to values reported in the literature, of 14.40 mg L-1 (Carosia *et al.*, 2014) and 15.00 mg L-1 (Braz *et al.*, 2021).

It can be observed that the surfactant concentrations found in the UASB-RALF and after the secondary treatment indicate removals of about 9% and 7%, respectively, in relation to the effluent. Between UASB-RALF and sewage after secondary treatment, the average values considered show an increase of 2% in the concentration of surfactants. The observed values are much lower than those reported in the literature, about 45% of removal of the concentration of surfactants in the UASB-RALF (Braz *et al.*, 2021; Carosia *et al.*, 2014). Such values justify the need to adjust the effluent to the maximum values of surfactants designated by the legislation, by means of complementary methods, such as, for example, post-treatment by activated carbon filtration.

3.2. Study of post-treatment by filtration / adsorption in a fixed bed of synthetic effluent

3.2.1. Surfactants Removal Results – Synthetic effluent

The removal of surfactants by fixed bed filtration with carbon adsorbent produced from coffee dreg and activated with phosphoric acid is shown in Figure 2 (a), which also contains the bars that express the errors attributed to the equipment and glassware used in the filtration. In this first analysis, to test the filter capacity, an aqueous solution with detergent diluted in deionized water was used. The concentration of surfactants (commercial detergent and deionized water) in the initial solution was 31.60 mg L-1. In the first hour of the filtration run, the concentration of the filtrate was 4.20 mg L-1, with removal efficiency of around 87%. Over the filtration / adsorption time the filtrate concentration gradually increased, and after 9 hours of testing the concentration was 27.20 mg L-1, with 14% removal efficiency. Such values indicate that at the beginning, with the "clean" coal and the clean filter medium, the adsorption / filtration process occurs more effectively, and over time, as the surfactant molecules occupy the active sites of the carbon, this efficiency gradually decreases, due to the decrease in the availability of sites for adsorption to occur.

In this case, the evaluated filtration period does not allow the calculation of the breaking point (Figure 2 (b)), exhaustion point or adsorptive capacity, since the breaking point is considered to be 5% of the initial solute concentration ($C_{post\ filter}/C_{initial} = 0.05$); that is, it would be for the concentration of 1.58 mg L-1 of surfactants. However, the first measurement, made

in 1 hour of filtration, already has a concentration higher than this. The exhaustion point, which occurs when the mass transfer zone (ZTM) reaches the bottom of the bed and the concentration of the solute at the exit of the column reaches around 95% of the initial concentration ($C_{post\ filter}/C_{initial}$= 0.95), that is, close to 30 mg L-1, it was also not reached in this study, since the concentration in 9 hours of filtration was 27.20 mg L-1(Kleinübing and Silva, 2008). This was due to the end of the filtration / adsorption row due to the loss of maximum hydraulic pressure, see Figure 3.

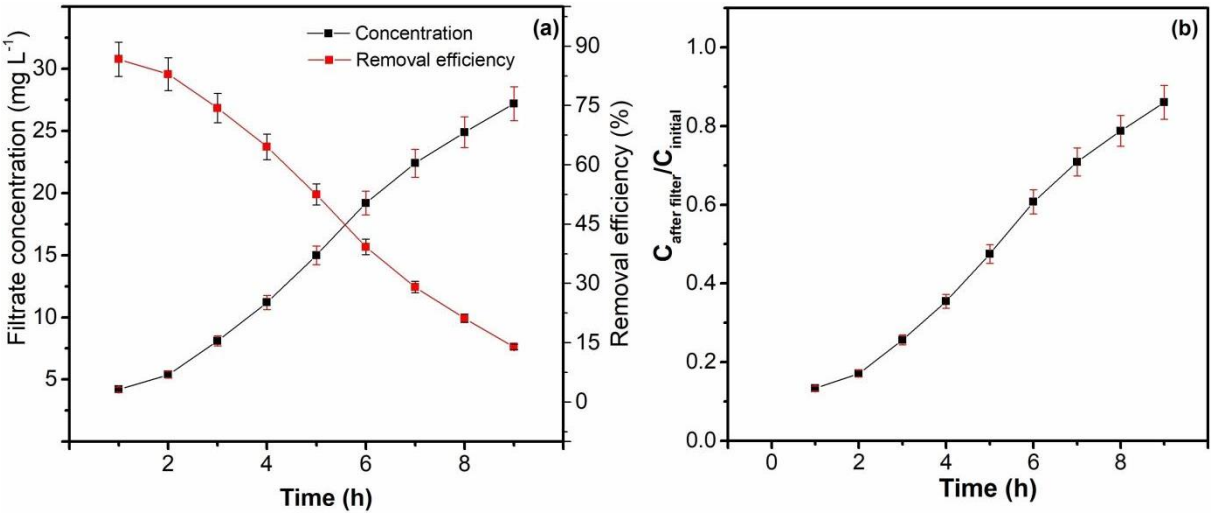

Figure 2. Evaluation of surfactant removal (a) and surfactant removal rupture curve (b), of the samples of the synthetic effluent by filtration in a fixed bed with carbon produced from coffee dreg and activated with phosphoric acid – example run.

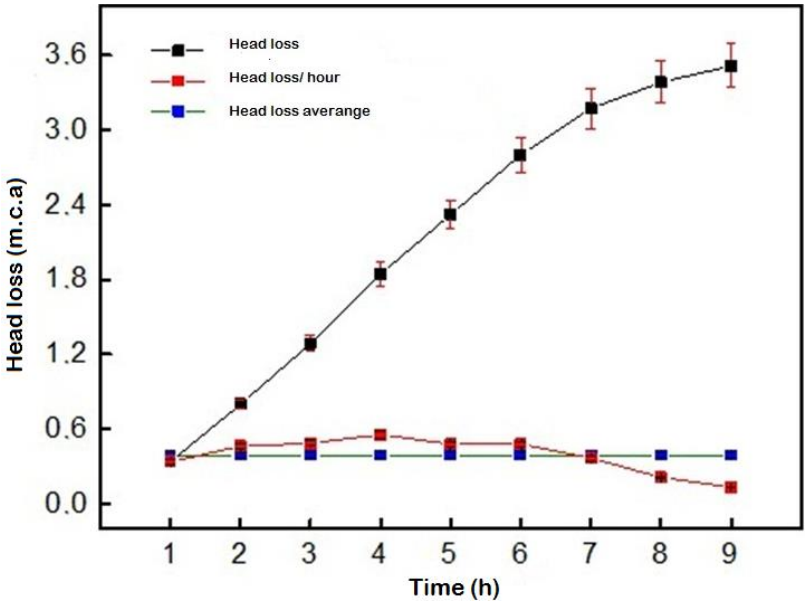

Figure 3. Example of head loss in the filtration/adsorption essay using activated carbon produced from spent coffee dreg in the pilot plant presented in Figure 1 and data presented in Figure 2.

The retention of surfactant solution above the filter layer was evaluated in order to verify the behavior of the filtration rate, as shown in Figure 3. It can be seen that the volume of the surfactant solution column above the filter layer increases approximately linearly until about 7 hours of filter operation and that the volume retained per hour remains close to the average value retained (ratio between the volume total retained and the number of hours of filtration or

career), thus indicating that during this period the filtration rate is constant (15 m3 m-2 day-1). After 7 hours of filtration, the height of the retained solution column possibly puts pressure on the solution in the filter medium, slightly increasing the filtration rate (decreasing the retained volume per hour).

3.2.2. Turbidity Removal Results – Synthetic effluent

Evaluation of turbidity of post-filtration samples along with the concentration of surfactants was also determined. The initial turbidity of the surfactant solution used in filtration was 0.32 (NTU). After filtration, the turbidity of the samples varied between 0.88 (NTU) (after 1 hour of filtration) and 0.55 (NTU) (after 9 hours of filtration), indicating that part of the carbon particles was washed away, even after washing the carbon prior to filter assembly. However, these values remain below 1.00 (NTU), which corresponds to the maximum allowed even by Consolidation Ordinance No. 5 Annex XX modified by Resolution No. 888 of the Potability Standard (Brasil, 2021).

3.3. Study of post-treatment by filtration / adsorption in a fixed bed to remove surfactants present in real effluent

3.3.1. Surfactants Removal Results – Real effluent

The removal of surfactants from the effluent by fixed bed filtration with carbon adsorbent produced from coffee dreg and activated is shown in Figure 4 (a). The concentration of surfactants in the initial solution was 1.36 mg L-1, and after just 1 minute of filtration, it dropped to 0.14 mg L-1, with a removal efficiency of about 90%. After 3 minutes, the concentrations of surfactants present in the samples were so low that it did not allow their quantification (limit of quantification of the technique used is 10 mg L-1).

Considering that the concentration of remaining surfactants in the effluent of Vila City STP was around 21.00 mg L-1 after the secondary treatment, it is observed that the results obtained by the filtration/adsorption tests on carbon produced from the coffee dreg and activated with phosphoric acid proved to be efficient in removing surfactants from this effluent, with lower final values than those detectable by the limit of quantification of the technique used.

The breaking point can be seen on the curve (Figure 4 (b)) and corresponds to 0.07 mg L-1 of the surfactant; however, this value is below the limit of quantification of the technique. Thus, it was not possible to determine the exact moment at which the breaking point is reached.

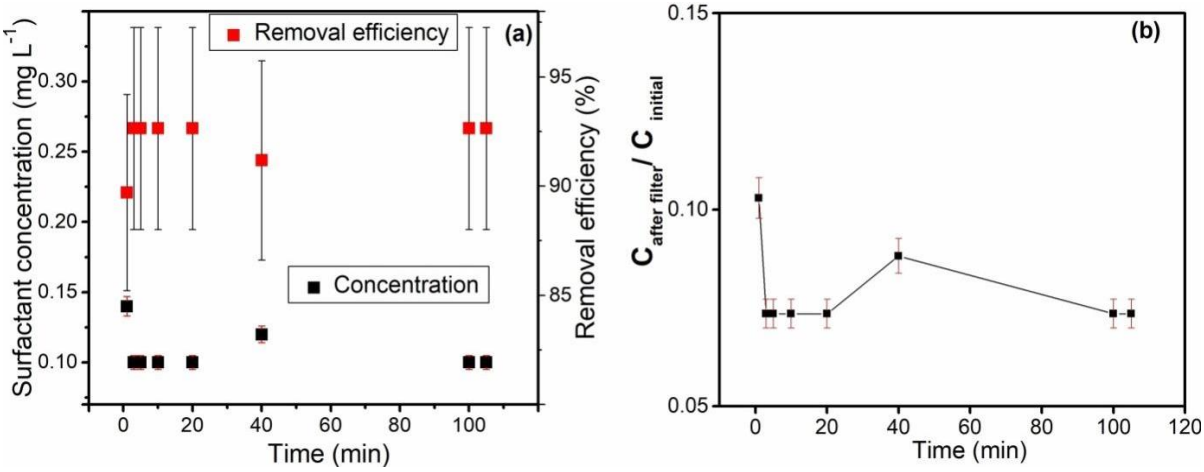

Figure 4. Example of evaluation of the surfactant removal (a) and rupture curve in the surfactant removal (b), of the samples of the real effluent by filtration in a fixed bed with phosphoric acid activated coffee dreg.

3.3.2. Turbidity Removal Results – Real effluent

The turbidity values of the effluent as a function of time during the filtration process were also determined. The initial turbidity of the solution was 7.8 (NTU) and a decrease in turbidity was observed as a function of the filtration time. This decrease occurred up to 40 min, reaching the minimum value of 0.5 (NTU). After 40 minutes of filtration, these values started to increase, indicating that in the beginning, with the "clean" carbon, the adsorption process occurs more effectively, and over time, as the particles occupy the active sites of the carbon, this efficiency may gradually decrease by decreasing the availability of sites for adsorption to occur, beside the fact of particle retention in the porous medium (sand-bed layers), evidently. All turbidity values presented in this study, below 8.0 (NTU), indicate that concerning this parameter the effluent is suitable for release into bodies of water, since according to CONAMA Resolution 430 (CONAMA, 2011), the discharge of effluents into Class 2 water should not exceed turbidity of 100.0 (NTU).

4. CONCLUSION

The results showed that in the conventional process used by the sewage treatment plant the removal of surfactants in the effluent was around 9% after UASB-RALF and 7% after secondary treatment. Thus, there is a need for a post-treatment of this specific parameter of the effluent before it is discharged into the receiving water body.

The efficiency of the post-treatment by filtration/adsorption using activated carbon produced from coffee dreg for the removal of surfactant evaluated in a pilot installation proposed in this work was: for 1 hour of filtration/adsorption, the removal efficiency was approximately 90%; for 9 hours of filtration/adsorption run, it was around 14%.

In this case, the filtration rate was constant in the order of 15 m3 m-2 day-1 during the 7 hours of the filtration/adsorption run. The removal of the analytical parameters with their respective efficiency averages was also carried out, obtaining the removal of approximately 94% of turbidity and 95% of surfactants, respectively.

The results provide a sustainable option for the post-treatment of effluent from ETE's contaminated with surfactants. The activated carbon produced from the coffee dreg in association with the sand filtration used in the pilot filtration/adsorption station proved to be efficient. It should be noted that the removal of effluent surfactants is even more necessary due to the pandemic caused by Covid-19, in which there was a significant increase in the use of hygiene and cleaning products containing surfactants.

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