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Biosurfactant and Bacterial Cellulose Applied to Textile Effluent Treatment

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The textile industry is one of the largest in the world, being of a great economical impact in many countries. The high water consumption and large effluents production with high concentrations of chemical products and residual dyes are amongst the industry's biggest environmental impacts. The application of biosurfactants (BS) in many processes in this industry is already established. However, its exploration in the effluent treatment stages can still be improved. Natural surfactants have diverse structure, are biodegradable, biocompatible, selective, and have good stability. They can be greatly explored in filtering processes. Bacterial cellulose (BC), a high purity biopolymer, is formed by a three-dimensional (3D) nanofibrillar network, with high water absorption capacity and high tensile strength. The present study aimed to evaluate, individually and in combination, the use of BS and BC both produced in media supplemented with industrial waste, aiming for its application in the treatment of textile effluents. The tests were carried out with synthetic effluents with navy blue dye at concentrations between 7.5 – 30.0 mg/L. BS was added to the effluent in the critical micelle concentration (CMC) of 1x CMC, 3 x CMC and 10 x CMC, to observe the agglutinating action. The BC pellicle with approximate thickness of 3 mm was evaluated as a filtering element. The color removal index of the treated effluent was determined through spectrometry as the parameter of effectiveness. The results showed an efficiency in color removal of up to 45 % solely with BC as a filtering agent and of 65 % when treated with BS before filtration with BC. The results demonstrate the potential of reducing the color parameter with the BS/BC association for a low-cost and sustainable filtration system to be applied in textile effluent treatment.

1. Introduction

Wastewater from the textile industry processes contains dyes and other components that impair the photosynthetic activity of aquatic organisms, in addition to having a toxic effect after decomposition. This can contaminate the soil, sediments, and surface water, therefore affecting the environment and human health (Al-Tohamy et al., 2022).

About half of global synthetic dyes are classified as non-biodegradable and carcinogenic (Rashid et al., 2020). The aromatic structure of dyes is responsible for such, reinforcing the need to develop methodologies for their removal from effluents (Nandhin et al.,2019). During the textile's dyeing process, 30 to 50 % of the dye is not fixed in the fabric (Rashid et al., 2020).

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Therefore, textile effluents are a topic of interest for environmentalists, researchers, and government agencies as they impact United Nations Sustainable Development Goals (SDGs) (Yaseen and Scholz, 2019).
There are studies associating ultrafiltration with synthetic and natural surfactants, demonstrating efficiency in the removal of dyes from wastewaters (Purkait et al., 2004; Samal et al., 2017; Verma et al., 2020). These techniques utilize surfactants to increase the hydrodynamic size of smaller solutes due to their amphipathic properties. Biosurfactants show a great new perspective for their application in bio discoloration treatment processes of textile effluents (Nor et al., 2021). BS have a great biodegradability and solubility, low toxicity, tolerance to extreme conditions, and ecological acceptability becoming a substitute for synthetic surfactants, which can be toxic to the environment (Shakeri et al., 2020). Rhamnolipids, sophorolipids, glycolipids and lipopeptides are some of the types of BS studied for the treatment of textile effluents (Santos et al., 2023). The anionic lipopeptide *Rufisan* produced by *Candida lipolytica* (UCP 0988), for example, shows a good action for removing hydrophobic contaminants and heavy metals (Rufino et al., 2011, 2013).
Bacterial cellulose (BC) also stands out as a versatile biotechnological material, being an alternative to the exploitation of vegetable cellulose. Researchers have demonstrated its use in the textile industry (Galdino et al., 2019), electronic field (Souza et al, 2021), pharmaceutical and cosmetics industry (Amorim et. al, 2019), among others. Bacteria of the genera *Komagataeibacter* (previously known as *Gluconacetobacter*), *Agrobacterium* and *Sarcina* are able to produce BC (Gomes et al., 2013). BC has a high purity, and due to its 3D nanofibrillar network, it shows high water holding capacity (with approximately 98 % water in its structure) (Galdino et al., 2019) and high tensile strength, in addition to being biodegradable and biocompatible (Medeiros et al., 2021). Galdino et al. (2020) obtained promising results regarding the efficiency, durability, and resistance of the bacterium's *Komagataeibacter hansenii* BC membrane for the treatment of oily water generated during industrial activities. Alves et al. (2020) demonstrated the efficiency of BC for dye removal for up to ten cycles.
In this sense, the present study aimed to produce the biosurfactant of *C. lipolytica* and bacterial cellulose membranes of *K. hansenii*, both produced in low-cost media with industrial wastes, for a combined use in the treatment of textile effluents.

2. Methods

2.1 Biosurfactant (BS) production

The biosurfactant was obtained via fermentation of *Candida lipolytica* (UCP 0988) (Rufino *et al.*, 2011). The BS was produced in a mineral medium containing 6 % of soybean oil from refinery waste and 1 % glutamic acid. The fermentation time was 72 hours, under orbital stirring at 150 rpm. Afterwards, the fermented product was filtered through Whatman No. 1 paper and centrifuged at 2000 x g for 20 minutes. The cell-free filtrate was subjected to extraction with chloroform (1:1 v/v) in a separatory funnel at a temperature of 25°C. The aqueous phase was lyophilized.

2.2 Bacterial cellulose (BC) production

The bacterium *Komagataeibacter hansenii* (UCP1619) was used for the membrane's production. A modified Hestrin Schramm (HS) (1954) medium was used with the following composition (w/v): 1.5 % glucose, 2.5% corn steep liquor, 0.27 % dibasic sodium phosphate, 0.15 % citric acid, adjusted to pH 5 (Costa et al., 2017). After 8 days of cultivation, membranes with a thickness of 3mm were obtained. They were cleaned by immersion in a 4 % sodium hydroxide solution for 2h to eliminate the retained bacterial cells . Afterwards, the membranes were washed with deionized water until neutral pH was reached.

2.3 Agglutination and filtration tests

The synthetic effluents (SE) were obtained from dilutions of the Coratex® navy-blue dye, RGB 43 46 67, for concentrations of 30.0 mg/L, 15.0 mg/L and 7.5 mg/L, named SE1, SE2 and SE3, respectively.
The agglutination of the dye consisted of adding the BS at concentrations of 1X CMC, 3X CMC, 10x CMC with 0.3, 0.9 and 3.0 % (w/v) of the SE, respectively. The solutions were placed on an orbital shaker (150 Hz, 28 °C, 2 h).
The used filtration system was adapted from Galdino et al. (2020), a negative pressure of 1.5 atm was achieved with a double-stage vacuum pump (Prisma Tech 131). The separation processes were carried until total filtration was achieved with the different dye concentrations.

2.4 Analytical method

The obtained effluents were analyzed through absorbance, at the maximum absorption wavelength (λmax) of the dye (590 nm) using a UV-vis spectrophotometer (Spectrophotometer, single beam, Uv-M51 Bel). Efficiency was analyzed by comparing the initial and post-process absorbance.

3. Results and discussion

3.1 Biosurfactant agglutination

The biosurfactant produced by *C. lipolytica* is a polymer formed by carbohydrates, proteins, and lipids, of anionic character (Rufino et al., 2011). In this sense, the BS's responsible for allowing the hydrophobic moiety to bind with the dyes, forming micelles containing dyes and water (Nor et al., 2021). A bioremediation study done by the same authors demonstrated that a lipopeptide BS from *Bacillus cereus* resulted in a discoloration of 47 % in a textile effluent of 30% (v/v).
The linear regression of the navy-blue dye showed excellent linearity, with a correlation coefficient of 0.9995. With the obtained equation (Eq. 1), it was possible to acquire the value of the concentrations of the post-treatment dye solutions, using the absorbance analyzed in the spectrophotometer.

$$y = 0.0223x - 0.0035 \quad (1)$$

Table 1 shows that the addition of the BS increased the absorbance of the effluent samples. The BS with 10 x CMC indicated a greater interaction with the dye, therefore, a better agglutination. The obtained results demonstrated a greater agglutination with the BS at low dye concentrations. It is worth mentioning that the concentration of the added surfactant to the effluent behaves in a non-proportional manner to the dye concentrations (Rashid *et al*., 2020).

Table 1: Percentage increase in absorbance of synthetic effluent after addition of biosurfactant at different concentrations, 1x critical micelle concentration (CMC), 3x critical micelle concentration (3 x CMC) and 10x critical micelle concentration (10 x CMC). Data is expressed as mean ± Standard Deviation for triplicate determinations.

Sample name	Synthetic effluent concentration (mg/L)	1 x CMC	3 x CMC	10 x CMC
SE1	30.0 ± 1.0	3.45 %	9.98 %	28.60 %
SE2	15.0 ± 1.0	5.63 %	11.62 %	25.36 %
SE3	7.5 ± 1.0	11.35 %	25.53 %	50.71 %

3.2 Filtration with bacterial cellulose

The BC membranes have a porosity of around 65 %, as demonstrated by Galdino et al., 2020. The results for the filtration using BC, can be seen in Figure 1, highlighting the greatest result with sample SE3. The dye reduction is shown in Table 2. The use of BC showed a good color removal efficiency without the use of additives in its polymeric matrix.The dye reduction indices were found between 41 and 45 % for all samples. Alves et al. (2020) demonstrated the use of BC from *Komagataeibacter xylinus* in the filtration of a blue-colored raw textile effluent with reduction results of 67.8 and 100 %. With that being said, the present study was carried out with synthetic effluent, and that other elements present in raw effluents such as the presence of solids in suspension can positively influence the dye removal in filtration processes.

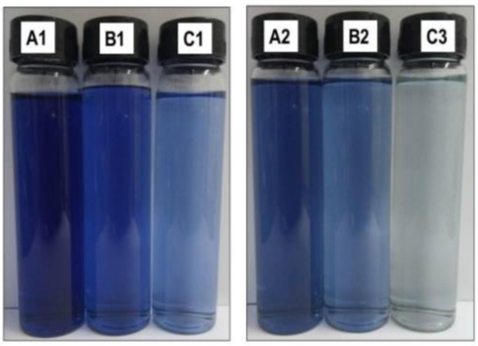

Figure 1: Synthetic effluent at initial concentrations of SE1 (A1), SE2 (B1) and SE3 (C1) and after the filtering process (A2, B2 and C2).

Table 2: Dye concentrations on the synthetic effluent before and after the filtering process. Data is expressed as mean ± Standard Deviation for triplicate determinations.

Sample name	Synthetic effluent post-filtration (mg/L)	Dye reduction (%)
SE1	16.4 ± 0.4	43.3
SE2	8.6 ± 0.4	41.6
SE3	4.2 ± 0.4	45.5

The BC fibers swell in contact with water due to the hydrophilic nature of this biopolymer, therefore BC are able to interact with water-soluble dyes (Costa *et al.*, 2019). Based on this, it is possible to infer that BC naturally absorbs the dye during the filtration process even without the addition of other agents to the feed effluent, and this can be seen in Figure 2.

Figure 2: Bacterial cellulose membranes after filtration of synthetic effluent at concentrations of SE1 (A), SE2 (B) and SE3 (C).

3.3 Biosurfactant and bacterial cellulose association

As the BS has the ability to trap the dye within micelles, this results in an increase of the size of the particles that pass through the membrane, resulting in a better dye retention. Aryanti et al. (2021) demonstrated an ultrafiltration process with the association of a synthetic polyethersulfone (PES) membrane with a saponin BS at a concentration of 2 x CMC and obtained great results for remazol and naphthol dyes
The 10 x CMC concentration demonstrated the best results, being the selected sample for the association tests.. Table 3 contains the reduction values. A greatest reduction of dye in the effluent is observed when compared with the filtration using only the BC membrane. The best result was with the sample that had the effluent at a concentration of 7.5 mg/L, of 65.9 %.

Table 3: Dye concentration in the synthetic effluent with biosurfactant (10 x CMC) before and after the filtering process. Data is expressed as mean ± Standard Deviation for triplicate determinations.

Sample name	Synthetic dye concentration post-filtration (mg/L)	Dye reduction (%)
SE1	14.1 ± 0.4	51,2
SE2	7.8 ± 0.4	50.4
SE3	3.4 ± 0.4	65.9

The interaction of the BS with the dye was visually observed by the change in the hue of the color of the effluent (as seen in Figures 1 and 3). After the filtration with BC, the samples SE2 and SE3 became more translucent when compared with the process without the use of BS. Overall, sample SE3 demonstrated the best results, the addition of BS to the filtration process with BC increased the removal efficiency by 44.83%

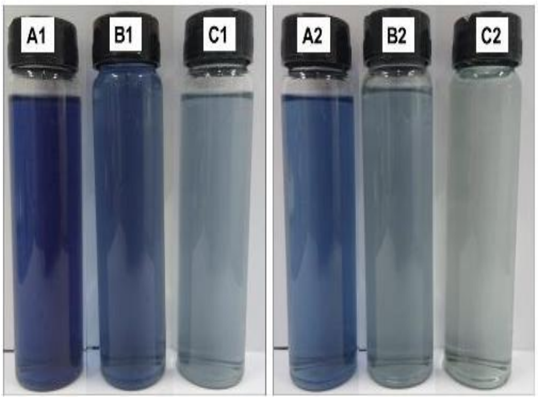

Figure 3: Synthetic effluent with biosurfactant (BS) at initial concentrations 30 mg/L (A1), 15 mg/L (B1) and 7.5 mg/L (C1) and after the filtering process (A2, B2 and C2).

4 Conclusion

The development of sustainable technologies for the treatment of colored effluents is important to preserve the planet's water resources. BS in interaction with dyes decreases its solubility. The BC membrane is responsible for the retention of the dye in its fibers and nanopores. The association of the BS with the BC enhanced the efficiency of the filtering process. This filtering association used for the treatment of effluents is very promising, however, further studies are still needed in order to improve the process and to better evaluate the incorporation of other materials into the BC's matrix that allow a better particle retention and dye adsorption prevention.

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