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Evaluation of operational parameters from a microfiltration system for indigo blue dye recovery from textile dye effluent

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ABSTRACT

This study aims to evaluate the use and operational parameters for microfiltration system (MF) for recovery of insoluble indigo blue present in the dye bath water of the cotton yarn, in order to reuse the pigment and water washing. The results showed that MF process is a promising technology for the recovery of insoluble indigo blue presents in a synthetic wastewater. The longitudinal module, when compared with the submerged module, presented a better performance showing removal efficiency of color and chemical oxygen demand (COD) of 99 and 94%, respectively. The dye recovery operation was performed at low pressure, specifically 0.3 bar, value determined by analysis of critical flux. The process was carried out under a laminar flow with Reynolds values of 1,900 and 2,850 and a turbulent flow with Reynolds values of 3,800 and 4,700 that achieved a recovery rate of 89.7% under minimal fouling conditions.

Keywords: Indigo blue; Microfiltration; Recovery; Reuse

1. Introduction

In the textile industry, during the fiber dyeing process, the water is regarded as a vital raw material, whether for use in the dyeing process itself or for auxiliary processes such as washing, bleaching, heating, and drying of the fiber [1]. Among these stages, the dyeing process is of primary environmental concern. The effluent from the dyeing process contains a large amount of dyes and other auxiliaries, and frequently does not meet the legislation or the reuse standards, even after undergoing conventional treatment processes commonly used, such as coagulation–flocculation and activated sludge. Conventional effluent treatment processes do not remove the remaining color resulting in an aesthetic concern, given that, generally, dye concentration higher than 0.005 mg/L in a water surface can usually be detected with the naked eye [2]. They can also cause serious environmental

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problems such as reduction of light penetration, change in quality of the receptor and toxicity to food chain organisms and aquatic life, in general.

Indigo blue ($C_{16}H_{10}N_2O_2$) is a synthetic organic dye widely used in textile industries, specifically in the jeans industry,[3] and due to its complex chemical structure it is considered as a persistent substance [4,5]. Indigo blue is insoluble in aqueous solutions and for the dyeing process it needs to be reduced to its soluble form (leuco form), which has chemical affinity with the cellulose fiber. This reduction is performed in alkaline solutions (pH ranging from 11 to 14) using a strong reducing agent such as sodium dithionite [6]. Fig. 1 shows the oxidation/reduction mechanism of indigo blue.

Indigo blue penetrates and adheres to the cotton fiber when immersed in the dyeing bath and when the fiber is exposed to air, the indigo is oxidized to its insoluble form and retained between the fibers of the fabric [7,8]. While 80% of the indigo is absorbed into the fibers of the cotton yarn, 5–20% is discarded during the washing step. The typical concentration of indigo blue in the washing bath in the dyeing process varies from 0.01 to 0.1 g/L [9–11].

The treatment of a textile effluent containing indigo blue dye usually consists of physicochemical, biological, and advanced oxidation processes or a combination thereof [12–14]. The physicochemical processes are effective for color removal, but use more energy and chemicals than biological processes [6]. However, due to the low biodegradability of the indigo dye and the presence of the chemicals used in the dyeing process, the conventional biological treatment does not always generate an effluent that meets the discharge or reuse standards [15]. Other typical problems of biological and physicochemical processes are the generation of intermediate products which are sometimes more toxic than the dye itself, and the large volume of sludge generated. The sludge has a significant amount of dye adsorbed during the treatment process of this effluent, generated in the industry. Conventional systems require large areas; have high cost; and bring little or no return (Pay-back). Therefore, from the environmental and economic point of view, it is important that the dye is recovered prior to disposal, simplifying and reducing the cost of treatment, allowing compliance with reuse standards, as well as ensuring financial return to the industry through the recovery of the dye as well as the reuse of water.

Given this context, the membrane separation processes (MSP) are a promising technology for the treatment of the indigo blue dye effluent, as it enables simultaneously the recovery of the dye, and the reduction of the color and organic load biochemical oxygen demand (BOD) and chemical oxygen demand (COD) of the effluent. This is only possible because indigo blue molecule is stable, insoluble in aqueous media and the microfiltration process (MF) may be used to recover this pigment and return it to the dyeing process. This technology is also suitable for the removal of the colloidal dyes of the exhausted dye bath and subsequent washings, such as bath and washes solutions of indigo blue dyeing, in which the indigo is found mainly in its oxidized form. The use of MF allows significant dye recovery and the production of a permeate which can still present other typical chemical products of the process, but can be used as water for less restrictive uses [16]. Owing to problems regarding fouling on the membrane surface due to pigment retention, the use of MF systems that operate in the hydrodynamics of the flow to minimize this effect is recommended [17]. MF can also be used as a pretreatment to nanofiltration (NF) or reverse osmosis (RO) yielding a permeate with excellent quality, adequate to be reused in situations that demand higher quality water, such as in cooling towers or boilers [18,19].

Thus, the aim of this study is to evaluate the use and operational conditions of a MF for the recovery of the indigo blue dye in their insoluble fraction present in a synthetic wastewater, in order to reuse the pigment and the water of the cotton yarn dye bath.

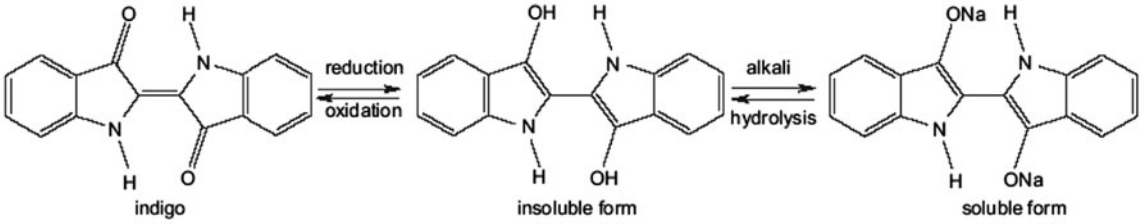

Fig. 1. Oxidation/reduction mechanism of indigo blue.

2. Methodology

2.1. Effluent

In this study, a synthetic effluent of a solution of 1 g/L of indigo blue dye prepared with deionized water was used. The indigo blue dye (Dye B Indigo Gran) was supplied by *Quimanil Corantes e Auxiliares/ São Paulo* and was used with no pretreatment so that the dye is present in its insoluble form, similar to that in the dyeing washing effluent. In order to standardize and analyze the dye concentration, a calibration curve was constructed correlating the color used in the UV–vis spectrophotometer (trade mark) with the concentration of COD [20].

2.2. Experimental apparatus

This study evaluated two configurations for the MF system. The first used longitudinal modules in a crossflow system and the second, submerged modules in a submerged system. The permeation system in the longitudinal modules uses the tangential feed flow. The tangential flow aims at reducing both the polarized layer and the accumulation of material on the membrane surface. Fig. 2 shows a schematic of the permeation system for the longitudinal module. The crossflow MF consists of a feed reservoir, a circulating feed pump, a microfiltration membrane module, pressure gauge, flow-meter (feed and permeate), and two tanks, one for the collection of the permeate and the other for the collection of the retentate, purchased from *PAM Membranas Seletivas Ltda*.

In the submerged system, unlike the crossflow system, the microfiltration module is located inside the feed tank. To minimize the effects of the concentration polarization and accumulation of material on the membrane surface during the permeation process, aeration was used at the base of the module. Aeration at the base of the module and parallel to the surface of the fibers acts as shear stress of the deposited material. Fig. 3 shows a schematic of the permeation system for the submerged system.

The submerged membrane MF has a 6 L membrane tank and five processing lines: system feed line, containing the effluent to be treated; compressed air line for aeration of the membrane module, when appropriate; microfiltrate effluent line; vacuum line; permeate line for backwash.

2.3. Longitudinal module vs. submerged module

The longitudinal and submerged membrane modules consisted of hollow fiber membranes made of poly (ether-imide) and selective external layer with 0.5 μm average pore size. The membranes were supplied by *PAM Membranas Seletivas LTDA*. The longitudinal module presents a conformation, similar to a shell and tube heat exchanger, and a membrane with an effective filtration area of 0.027 m^2 and packing density of 300 m^2/m^3. The module feeding was conducted inside the housing and the permeate was collected from within the hollow fibers. The submerged module consisted of a membrane with an effective filtration area of 0.046 m^2, and packing density of 523 m^2/m^3. The submerged module is closed at one end while the other is open to allow the drainage of the permeate, which takes place inside the fibers. The performance of the submerged module was assessed with and without aeration between the fibers. To compare the performance of the two

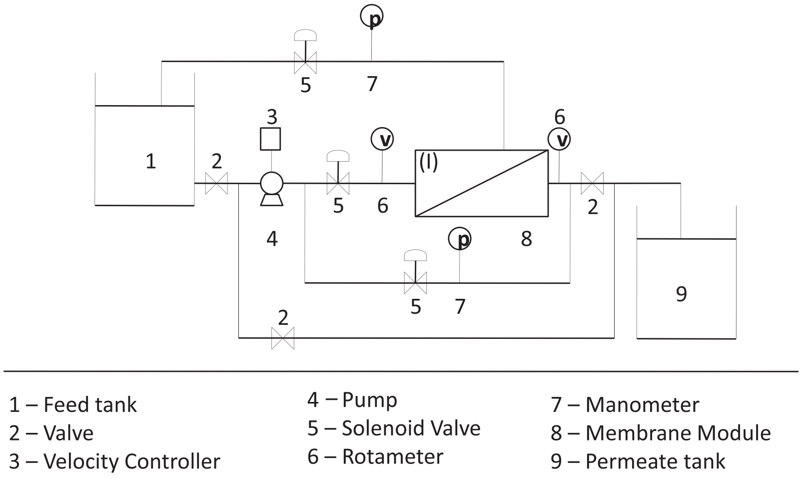

Fig. 2. Crossflow MF system.

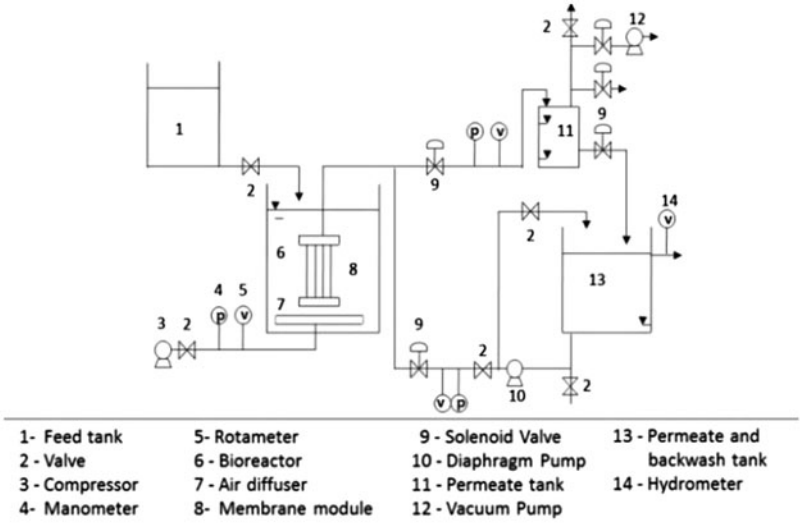

Fig. 3. Submerged MF system.

configuration membrane modules, the hydraulic permeability was measured with water and synthetic effluent applying both positive and vacuum pressure in the same range of values (0.3, 0.4, 0.5 and 0.6 bar). The feed flow in longitudinal module was 144 L/h and aeration flow in aerated submerse module was 0.5 Nm^3/h. The rejection of the dye by the membrane was evaluated by monitoring the concentration in the feeding and the permeated both in terms of COD.

2.4. Operation under high pressure vs. low pressure

Longitudinal membrane modules typically operate at higher pressures than submerged modules. However, under these operating conditions of applied higher pressure more fouling may occur, especially in the case of effluents with high concentration of suspended solids and colloidal material. Taking this into consideration, the performance of the longitudinal module was assessed under low and high pressure. Performance was assessed through the monitoring of hydraulic permeability with synthetic effluent compared to hydraulic permeability of the membrane with water by applying pressure in the range of 0.3, 0.4, 0.5 and 0.6 bar (low pressure) and 0.75, 1.0, 1.25 and 1.5 (high pressure) and of the rejection of the dye by the membrane in terms of COD.

2.5. Evaluation of the flow regime (Re)

The evaluate hydrodynamic conditions was conducted only for the pressurized module. the variation in the flow regime (Re) was 1,900, 2,850, 3,800, and 4,700. The hydrodynamics effect was evaluated from the response of the hydraulic permeability of the membrane with the synthetic effluent, in relation to hydraulic permeability of the membrane with microfiltrated water, rejection capacity of the membrane in terms of COD and color. At the end of each test, the module was subjected to a physical cleaning process (water recirculation and backwash) and a chemical cleaning (the membrane was immersed in 500 mg/L of sodium percarbonate for 20 min in an ultrasonic bath).

2.6. Critical flux

Given an optimum flow rate, the operating pressure was evaluated by determining the critical flux in pre-established conditions. The critical flux for microfiltration is the one below which a *decline* of *flux* with *time* does not *occur*; and *above* which *fouling* is *observed*, and whose value depends on the hydrodynamics of the process [21]. To obtain the critical flux for this system and for the longitudinal modules, the pressure was fixed and the permeate flux was monitored in 15 min intervals. After each increment of the pressure, the permeate flux was monitored. To determine the critical flux the pressure values assessed were 0.20, 0.25, 0.35, 0.30, 0.40, 0.45, and 0.50 bar.

2.7. Recovery rate

The previous tests were conducted with permeate and concentrate returned to the feed tank in order to maintain constant the concentration in the feed tank. To assess the real situation, only the concentrate was returned to the feed tank in order to determine the permeate flux profile with increasing concentration in the feed tank. In this same test, the dye recovery rate was assessed to evaluate the possibility of returning the dye concentrate to the dyeing process. During permeation, the pressure applied was kept at 0.3 bar (value below the critical pressure, which was obtained in the critical flux tests, and Reynolds of 2,805). Both the permeate and concentrated feed were analyzed throughout the concentration test regarding the concentration of COD and color in accordance with Standard Methods for the Examination of Water and Wastewater [20].

3. Results

3.1. Longitudinal vs. submerged module

To evaluate the performance of longitudinal and submerged modules (both manufactured with the same membrane type) the hydraulic permeability and the relation hydraulic permeability of effluent and hydraulic permeability of water, known as K_e/K_w was assessed. The performance of the submerged module was assessed with and without aeration between the fibers. Aeration between the fibers aims to minimize membrane fouling due to shear stress.

The hydraulic permeability was 142, 37, and 43 for longitudinal and submerged modules without and with aeration, respectively and relation K_e/K_w was 0.54, 0.62, and 0.88 for longitudinal and submerged modules without and with aeration, respectively. It can be observed that the submerged module was less likely to fouling than the longitudinal module. This result can be visualized from the values obtained for K_e/K_w, that is, the higher the value of this parameter, the lower the permeability loss with the MF using the effluent. The lower fouling effect on the submerged module with aeration is due to aeration at its base, minimizing significantly the deposition of the material on the membrane. In this module configuration, aeration acts as a shearing force minimizing the thickness of the polarization layer and fouling formation. The result shows that the shear stress effect caused by the bubbles proved more efficient than the flow shear stress within the longitudinal module housing. In the submerged module, the permeability reduction to the effluent compared to its hydraulic permeability was only of 12%. The submerged module presented less fouling, whereas the longitudinal module presented greater permeability. This behavior is due to the small size of the pressurized module. In this configuration and size, may also occur areas of recirculation of the liquid inside of module resulting in a minimization of the effective area of the membrane permeation process, i.e. one cannot observe a fully developed flow inside the module. This behavior has been observed previously by NETA FRANCE [22] for longitudinal modules.

Regarding the removal/recovery of Indigo Blue, was observed that the longitudinal and submerged modules without and with aeration showed removal efficiency of 94, 82, and 99%, respectively in terms of COD. The concentration of COD in the permeate of longitudinal and submerged modules without and with aeration was 76, 232, and 8 mg/L, respectively. The data shows that the highest rejection values or highest recovery of Indigo blue were obtained for the submerged modules with aeration and the longitudinal modules. Probably, this result is due to the fact that in both settings turbulence is promoted close to the membrane surface reducing the thickness of the polarized layer and consequently, minimizing the passage of the dye from the feed to the permeate and the deformation of the indigo blue particle. In the submerged module the shear effect close to the surface of the membrane is caused by bubbles, in the longitudinal module it occurs with the feed flow tangential to the membrane surface, which reduces the difference in concentration between the feed side and the permeate side, thereby reducing the flow of the dye across the membrane. In addition to this, the aeration in the submerged module may have contributed to the oxidation of the indigo blue fraction that was probably solubilized in the effluent, increasing the rejection by this module, since MF membranes are not able to retain the indigo blue in its soluble form.

To highlight the results obtained in the previous test, the same behavior was observed for the permeated color analyses of the longitudinal module and submerged module with and without aeration, which were 23, 61, and 29 uH, respectively. Considering the quality standards for water reuse in the textile industry in terms of COD and color, in wet processing, values are 178–218 mg/L for COD and 20–30 uH for color [1]. Based on this, it can be said that both the crossflow module and the submerged module with aeration can be used for microfiltration of effluent. Due to greater permeability obtained in the longitudinal module and the satisfactory removal of indigo in this type of module, it was selected to continue the operational tests in this study. Due to the higher permeability values obtained in pressurized modules it was selected to continue the sequence of operational tests in this study. It is noteworthy that a satisfactory removal of indigo in a submerged module is required to use aeration which results in a higher operational cost, which is not justified as acceptable efficiency is achieved with pressurized module. Furthermore, in smaller capacity plants, pressure systems is typically the most economically viable solution. Crossflow systems are typically skid mounted, easy to install and do not require heavy construction. Submerged systems for smaller capacities are perceived as being more expensive and as having higher installation costs. Due to more fouling observed in longitudinal modules, optimum operational conditions were investigated to minimize this effect. The operating parameters for minimizing fouling were investigated in terms of the flow regime and the critical flux.

3.2. Operating under low and high pressure conditions

Most longitudinal membrane modules are operated at high pressure and high recirculation rate and are characterized by providing higher permeate flux when compared to submerged modules. However, in some cases the operation at high pressure, mainly in the case of effluent with high concentration of suspended materials, can result in increased fouling, demanding increased energy expenditure, higher cleaning frequency, and the reduction of the membrane useful life. In these cases, it is best to operate the systems at low pressures and high flow rates to minimize the polarized layer near the surface of the membrane, thus increasing the permeate flux values. Fig. 4 shows the hydraulic permeability values of the longitudinal module under low and high pressure operated with microfiltered water and synthetic effluent.

The reduction in the effluent permeability compared to the water permeability was lower in modules operated at low pressure, showing decay values around 42%. In the modules operated at high pressure this decay of permeability was 75%. The higher value of decay of permeability is in agreement with data from literature, which emphasize that with the increase in the operating pressure of the system there is an increase in the convective flow toward the surface of the membrane, increasing the concentration of particulate matter on the membrane surface. The increase in pressure also contributes to more compression of the cake formed on the surface of the membrane, thereby increasing the resistance to transport. Fig. 5 shows the values of COD removal using longitudinal module at high and low pressure.

Most COD removal occurred in the module operated under high pressure when compared to the module operated under low pressure. This result can be associated with the contribution of the cake formed on the surface of the membrane. The increase in operating pressure resulted in compression of the cake deposited on the surface of the membrane. This cake acts as a dynamic membrane by increasing the membrane rejection to the constituents present in the feed. Taking into consideration the quality standards for reuse in the textile industry in terms of COD, already previously presented here, the permeate obtained, both under low- and high-pressure operation, meets reuse patterns. The best operating condition can be determined based on higher productivity which, in this case, was achieved under low pressure operation.

3.3. Evaluation of the effect of the flow regime

Another way to control fouling in longitudinal modules is to operate them at high flow rates, turbulent flow close the membrane surface, and low operating pressures. The increase in the flow rate will lead to an increase in the shear stress near the surface of the membrane removing fouling material and increasing the permeated flux. According to BACCHIN [23], reduced

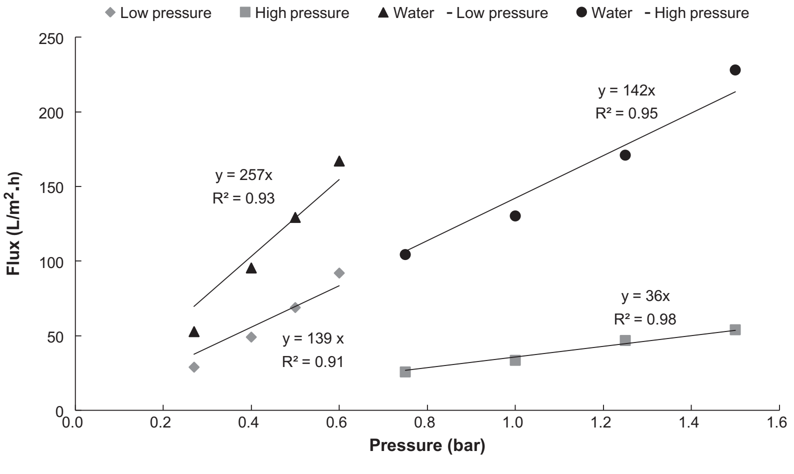

Fig. 4. Hydraulic permeability to the effluent of the longitudinal module under low and high pressure (Re = 2,850).

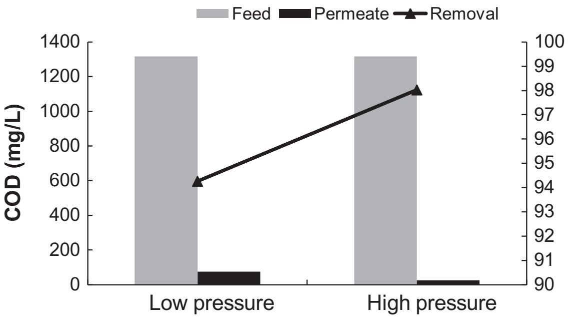

Fig. 5. COD removal using a longitudinal microfiltration module under low and high pressure (Re = 2,850).

pressures and increased shear forces combined are enough to minimize deposition of particles on the surface of membrane. Fig. 6 shows the results obtained for operation under different flow rates, which are equivalent to a flow regime (Reynolds number) of 1,900, 2,850, 3,800, and 4,700, ranging from the laminar to the turbulent regime. The variation of the flow regime was related to the reduction in the permeability of the membrane with the effluent in relation to hydraulic permeability of the membrane (K_e/K_w).

With the increase of the flow regime from 1,900 to 2,850, there was a reduction in the permeability loss of the membrane with effluent in relation to hydraulic permeability from 51 to 46%. However, with the increase in the flow regime from the laminar to the turbulent regime, from 3,800 to 4,700, there is an increase in permeability loss of the membrane in relation to the effluent from 59 to 61%. The result obtained is not corroborated by data from literature in which increasing feed flow favors increased permeability of the membrane for effluent, due to the action of shear forces. The difference between the behavior of theoretical and experimental data may be explained from the flux paradox [17]. In some cases, these deviations occur due to the flow regime and the thickness of the flow channel, or systems operated under high shear conditions and narrower flow channels may exhibit discrepancy between the theoretical and experimentally obtained. In this study, we have used the longitudinal modules with 0.2 m length, which is not enough for the full development of the flow within it. The flow of liquid inside these modules may cause recirculation zones fluid, thereby generating a poor distribution of the liquid and thus minimizing the effective area of the membranes used for the separation of the pigment. This behavior explains the decrease in the K_e/K_w parameter obtained experimentally with increasing flow regime. Fig. 7 shows COD removal using the longitudinal module under different flow regimes.

The increase in flow regime leads to an increase in COD removal in the permeate flux which is directly associated with the reduction of the cake layer and the concentration polarization effect on the membrane surface reducing the passage of the pigment particles to the permeate side or diffusive effects of mass transfer, if there are solubilized pigment particles. The reduction of the concentration polarization layer on the membrane surface reduces the flux of the dye across the membrane by diffusive effect between the pores, whose driving force is the difference in solute concentration (indigo blue) on the surface of the membrane and the side of the permeate. Fig. 8 shows color removal using longitudinal module under different conditions of flow regime.

The effect of the flux rate on color removal by membrane processes operated under different conditions of flow regime is similar to that obtained for COD removal, i.e. the higher the flow regime, the greater the color removal. The color removal obtained was significantly superior to that found by [1] that removed only 30% and used a membrane with 0.45 μm average pore diameter at an operating pressure of 0.48 bar. The membrane rejection in all conditions of flow regime analyzed, meets the reuse standards of the textile industry regarding the removal

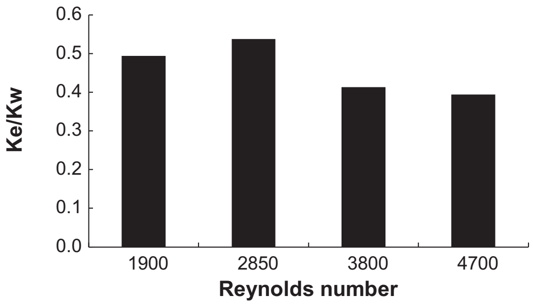

Fig. 6. Effect of the flow regime in the reduction of the permeability of the membrane to the effluent.

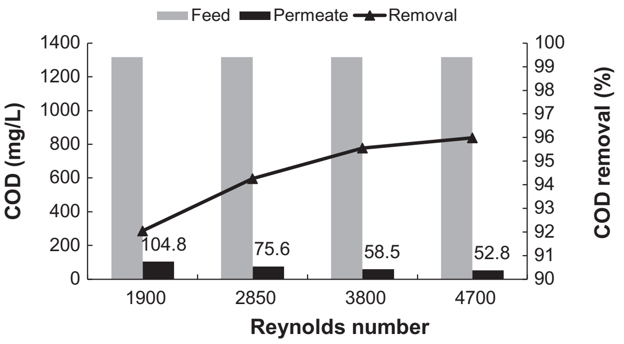

Fig. 7. COD removal using longitudinal module under different flow regimes.

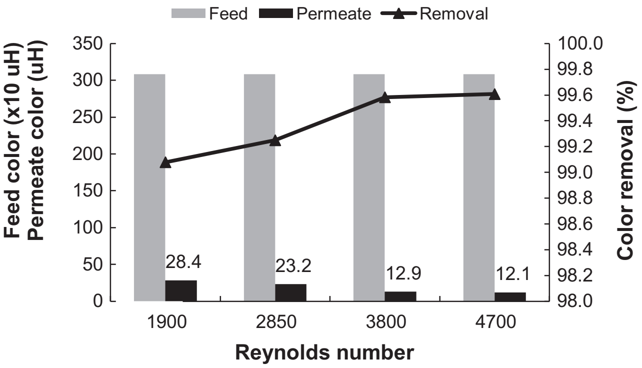

Fig. 8. Color removal using longitudinal module under different conditions of flow regime.

of COD and color. The selection of the best operating conditions will be associated with the conditions that result in greater productivity of the permeate system, that is, a lower loss of membrane permeability in relation to the operation with the effluent compared to water permeability (K_e/K_w).

3.4. Critical flux

Critical flux tests were conducted to determine the optimal operating pressure in order to prevent fouling. The values of critical flux and critical pressure depend on both the concentration of the feed and the hydrodynamics flow in the longitudinal module.

Fig. 9 shows the relationship between the permeate flux and the system operating pressure to determine critical flux. The flow regime in which the critical flux measurements were carried out was at Re 2,850 to avoid the effect the flux paradox, as mentioned earlier.

The increase in the difference of pressure from 0.3 to 0.4 bar led to a change in the behavior of the permeate flux. The reduction in the permeate flux at constant pressure in a given time interval is an indication that the critical flux value for this operating system was obtained. Therefore, for a sustainable operation, i.e. at constant permeate flux values and without increasing the operating pressure, the MF system will need to be operated with permeate flux lower than 27 L/m^2 h.

3.5. Recovery rate

With the optimal operating conditions established, that is, operation at low pressure, specifically 0.3 bar, determined from critical flow tests and flux regime at Re 2,850, the recovery rate was determined. Fig. 10 shows the relationship between recovery rate, membrane permeability to effluent, in addition to the COD and color analysis of the permeate.

The data obtained for the dye concentration test indicate that during the 560 min of the test there was no significant change in values regarding membrane rejection capability in terms of COD and color. The procedure carried out under optimum operational conditions and shows that the membrane permeability reduction to effluent containing the dye was only 36% suggesting that to the permeation of the effluent in question, a recovery rate of 89.7% can be applied. UZAL [1] used 0.45 μm microfiltration membranes and obtained a recovery rate of 90%, similar to that found with permeability reduction of 38%. Based on the

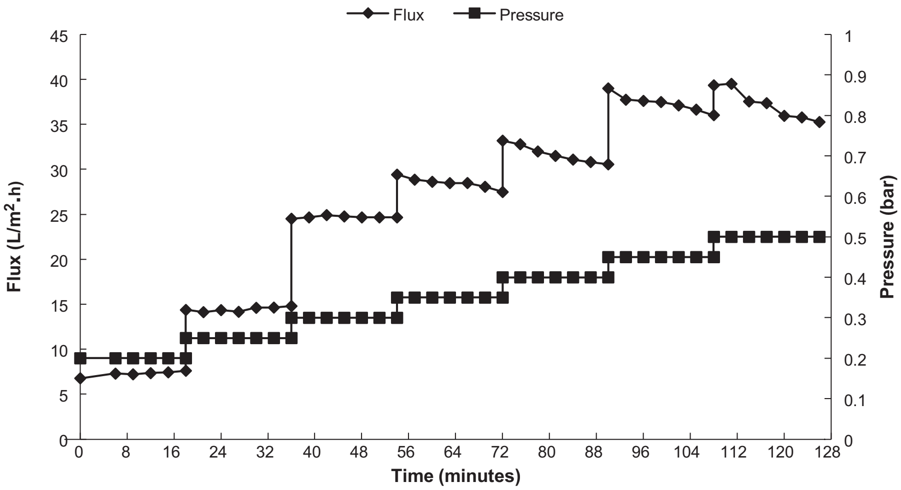

Fig. 9. Relationship between the permeate flux and the operational pressure to obtain the critical flux.

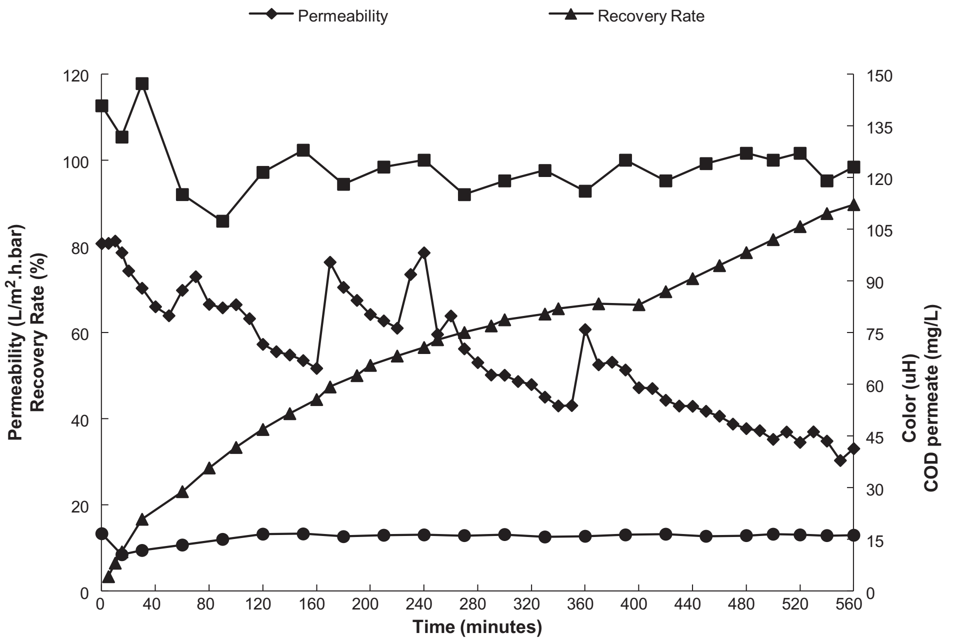

Fig. 10. Relationship between recovery rate, membrane permeability to the effluent, color and COD of permeate.

results obtained for the recovery of indigo blue dye in its insoluble form, the microfiltration separation process is feasible in the recovery of this constituent.

4. Conclusion

In view of the results obtained with experimental tests presented in this study, the operational conditions obtained for microfiltration process presents itself as a promising alternative in the recovery of indigo blue dye in its insoluble form discarded in bath dyeing processes. The results of the quality of the permeate obtained, leads toward the use of this process as water for reuse or as pre-treatment technique to generate better quality water to be used, for example, in boilers or cooling towers. However, the adequacy of permeate water for reuse and dye concentration for later reuse in dyeing tanks is related only to the insoluble fraction of the dye. To validate this data, it will be necessary to carry out tests with real effluent from dyeing tanks to confirm the results obtained at this stage of the study. The longitudinal module presented greater permeability in the processing of synthetic effluent with indigo blue dye concentration of 1 g/L and showed removal efficiency in terms of color and COD of 99 and 94%, respectively. The operation under low pressure conditions, specifically 0.3 bar, value obtained from the analysis of critical flux, and a flux regime of 2,850, shows a recovery rate of 89.7% under minimum fouling conditions.

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