



Case Report

Treatment of water from the textile industry contaminated with indigo dye: A hybrid approach combining bioremediation and nanofiltration for sustainable reuse

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ABSTRACT

In recent decades, the extensive use of synthetic dyes in dye-based industries, particularly the textile sector, has led to environmental contamination, raising concerns about water quality and human health. This study aimed to develop a sustainable approach for treating wastewater contaminated with indigo dye through a hybrid approach combining bioremediation and nanofiltration for reuse. In the first stage, two bioaugmentation strategies were compared: using native microorganisms in the wastewater (R-1) and activated sludge from a municipal wastewater treatment plant (R-2). Both strategies effectively reduced chemical oxygen demand (COD) to 200 mg/L and eliminated over 90% of the color. Statistical analysis showed no significant difference in COD removal efficiencies between R-1 and R-2. Correlation analysis revealed a strong association (>99%) between COD, suspended solids (SS) concentration, and color in the reactors, providing insights into sustainable monitoring for wastewater treatment, with potential cost reductions and environmental benefits. In the second stage, nanofiltration (NF) with organic membranes was employed to obtain high-quality permeate for reuse in denim washing. Permeability measurements for R-1 demonstrated similar membrane behavior (NF 270 and Alfa Laval NF) at various transmembrane pressures (TMPs). Both membranes achieved color rejection rates exceeding 96% and COD removal efficiency over 80% for all TMPs. NF tests with R-2 effluent revealed higher permeate flux of 23.71 L·h⁻¹·m⁻² at a TMP of 5 bar, with Alfa Laval NF membrane exhibiting superior COD and color rejection compared to NF 270 membrane. The techno-economic analysis showed the hybrid approach of biological treatment/NF to have a mean unit cost of 0.97 USD/m³, indicating economic feasibility and competitiveness against commercial purification processes for sustainable water reuse.

1. Introduction

The global fashion and clothing market were valued at USD 993.6 billion in 2021, with an anticipated annual growth rate of 4.0% from 2022 to 2030 [1]. This growth will result in increased water demand for raw material processing, estimated to be equivalent to 93 billion cubic meters of water annually, representing 20% of the world's wastewater production [2]. Shockingly, approximately 1.84 billion tons of textile wastewater are discharged each year without proper treatment [3].

In recent decades, dyes have surged due to the rapid expansion of dye-based industries and the growing demand for textiles and clothing.

Consequently, substantial amounts of synthetic dyes, extremely resistant to biodegradation, have accumulated in the environment. These dyes are employed in the textile industry and find applications in food, cosmetics, paper, rubber, and other sectors [4].

The quantity of dyes released into wastewater after the dyeing process depends entirely on the type of dyes used. Simple dyes result in a loss of only 2%, while reactive, indigoid, or azo dyes can lead to a loss of up to 50% of the dyes [5]. Consequently, water quality parameters such as biological oxygen demand, chemical oxygen demand, total suspended solids, and optical characteristics significantly impact the growth of aquatic flora and fauna [6]. Even at low concentrations, the presence of

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dyes in effluents is highly visible, and the oxidation products of these dyes are often carcinogenic. Moreover, these dyes exert various toxic effects on living organisms, including humans, with neurotoxic, cytotoxic, genotoxic, mutagenic, hypersensitive, and mitochondrial toxicant properties being observed in several dyes. Numerous countries have implemented stringent environmental regulations governing the textile industry to address this concern [4,7].

Indigo, widely used in the textile industry for applications like jeans dyeing, is prominent among the most commonly employed dyes. Cotton fabrics dyed with indigo are commonly referred to as denim. The global indigo market experiences an annual demand increase of 5%, consuming more than 4.5 billion meters of water [8].

In the present context of the circular economy, the effective management of textile effluents is crucial for enabling wastewater reuse, as the textile sector is widely acknowledged as a major polluter, particularly concerning water pollution. The World Bank has observed that the textile industry is responsible for approximately 20% of all water pollution globally [9].

The wastewater treatment approach requires a synergistic method involving a blend of conventional processes (biological, physicochemical) and cutting-edge techniques, such as oxidation processes (ozone, Fenton, among others) [10], adsorption [11], or membrane technologies [12].

Most traditional methods for treating wastewater of textile origin can remove more than 80% of the dye, with some exceeding 90%. However, these methods come with certain limitations. Electrocatalytic systems, for example, are effective but often associated with high operating costs. Membrane systems can face fouling issues, particularly in the presence of high dye concentrations [13].

Advanced oxidation systems, which may involve adding chemical compounds like hydrogen peroxide in Fenton reactions, pose chemical hazards due to the corrosive nature of these compounds. This limits their application on an industrial scale.

Additionally, in adsorption systems, contaminants are transferred from the liquid phase to the solid phase. This necessitates the remediation of the contaminated adsorbent material, leading to increased environmental management costs [14].

Several authors have reported on the combination of biological reactors and nanofiltration for the treatment of textile wastewater [15, 16]. Specifically for denim wastewater, Sahinkaya [17] also documented the application of this combined process for water reuse. In their study, the biological step followed the inoculation of the reactor with activated sludge from a wastewater treatment plant. In contrast to these authors and other studies focusing on the biological treatment of textile wastewater [18–20], the present study also evaluates the feasibility of working with native microorganisms.

This study investigates the treatment of denim-washing wastewater containing indigo through a two-stage process. The first stage involves bioremediation, comparing the results obtained using native microorganisms with those achieved by adding activated sludge from a wastewater treatment plant to the reactor. The second stage comprises nanofiltration (NF) utilizing organic membranes to obtain high-quality permeate suitable for reuse. Additionally, the study includes a technical-economic analysis to assess the unit costs of treatment, employing multiple scenarios with Monte Carlo algorithms. To the best of our knowledge, there are no papers in the bibliography reporting results of bioremediation for this type of water using native microorganisms alone, i.e., only the microorganisms present in the wastewater itself, with no addition of either seed sludge or previously acclimated sludge.

2. Materials and methods

2.1. Textile wastewater

Textile wastewater was obtained from jeans washing machine with a

capacity of 25 L. The washing bath consisted of a detergent (ASUTOL FTT), a wetting agent (PRODER MAC), and two softeners (Asumin H-RP micro and Asumin WK). This composition ensured the presence of fibers, indigo from the jeans, and auxiliary products in the wastewater. The wastewater sample was provided by a textile industry located in the Valencian Community, Spain.

2.2. Treatment of textile wastewater

The textile industry wastewater containing indigo dye underwent a two-stage treatment: bioremediation in a batch reactor and nanofiltration (NF), as depicted in Fig. 1, to obtain treated water suitable for industrial reuse.

2.2.1. Bioremediation stage

The bioremediation process was conducted aerobically using bioaugmentation and biostimulation. For bioaugmentation, two strategies were employed. The first strategy involved using the native microorganisms in the washing wastewater without adding inoculum (R-1). The second strategy utilized activated sludge obtained from a municipal wastewater treatment plant (WWTP) as the inoculum (R-2). Two batch reactors, each with a reaction volume of 4 L, were set up for these two strategies. Aeration was provided by an air pump (EHEIM, Germany) at an airflow rate of 200 L/h, and mechanical agitation at 150 rpm was employed to maintain the homogeneity of the mixed liquor. The experiments were conducted at an operating temperature of 24 °C for 30 days. Biostimulation was done by adding 0.5 g/L of EC fertilizer NPK 5-5-5 (Productsflower) due to the lack of nutrients in the wastewater samples.

2.2.2. Nanofiltration stage

The biologically treated wastewater underwent settling (120 min) and centrifugation (9600 rpm, 15 minutes). A SIGMA-6-16KS centrifuge (Fisher, Germany) was used for this purpose. This treatment was a preliminary stage before nanofiltration was performed to minimize membrane fouling.

Two operation modes were employed for nanofiltration: dead-end and cross-flow. The membranes used in the experiments were NF270 (FilmTec™, USA) and Alfa Laval NF (Alfa Laval, Sweden).

2.2.2.1. Dead-end nanofiltration tests. The dead-end nanofiltration tests were conducted using a stirred cell (HP 4750) provided by Sterlitech (USA), with a membrane-active surface area of 14.66 cm². Feed pressure (5, 7, and 9 bar) was controlled using nitrogen. A feed sample of 200 mL was introduced into the cell, which was stirred at 500 rpm to minimize membrane fouling. Fig. 2 illustrates the setup of the dead-end nanofiltration laboratory plant.

The water permeability of the membranes was determined after compaction through experiments with osmotic water at 5, 7, and 9 bars. Permeate flux values required to calculate the permeability were obtained using Eq. (1)

$$J_p = \frac{V}{A_m t}, \quad (1)$$

where:

J_p : Permeate flux (L·m²·h⁻¹)

V: Permeate volume (L),

A: Membrane active surface (m²),

t: Time taken to collect the permeate sample volume (h).

The permeability value for each membrane was obtained from the linear regression slope of the permeate flux versus the feed pressure. A pristine membrane was tested for each feed pressure, and the water membrane permeability was measured before each test. The rejection of

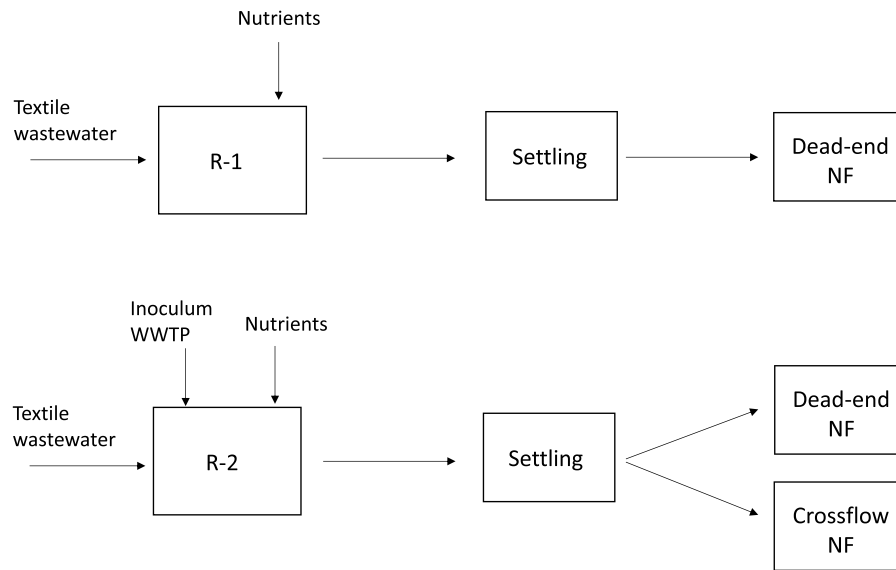


Fig. 1. Block diagram of textile wastewater treatment. In the upper part, the bioremediation process with native microorganisms from washing wastewater (R-1), and in the lower part, the bioremediation process with the addition of municipal wastewater treatment plant (WWTP) as inoculum (R-2).

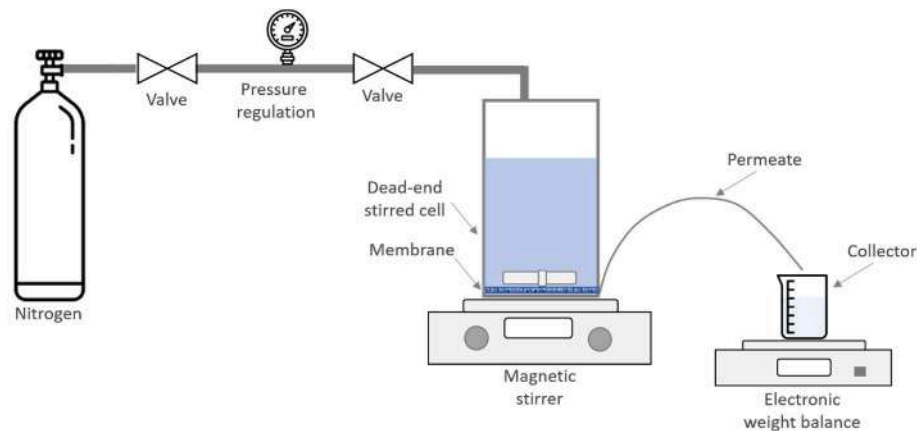


Fig. 2. Schematic diagram of the dead-end laboratory plant.

chemical oxygen demand (COD) and color was calculated using Eq. (2)

$$R (\%) = \frac{C_f - C_p}{C_f} \times 100, \quad (2)$$

where C_f and C_p are the COD concentration (mg/L) or color value in feed and permeate streams, respectively.

2.2.2.2. Cross-flow nanofiltration tests. The cross-flow experiments utilized pristine membrane samples of the same types as those used in the dead-end nanofiltration tests. Fig. 3 depicts the setup of the cross-flow laboratory plant. The plant was equipped with a flat module containing two flat-sheet membranes, each with an active surface area of 90 cm². A transmembrane pressure of 5 bar, determined from the dead-end tests, was applied. Three experiments were conducted at constant volume (recycling both permeate and retentate to the feed tank to operate at constant concentration) and constant temperature (20 °C), with varying cross-flow velocities of 0.89 m/s, 1.33 m/s, and 1.77 m/s [21]. The effect of cross-flow velocity on permeate flux and membrane rejection was investigated.

2.3. Analysis and instrumentation

pH and conductivity measurements were performed using a pH-Meter GLP 21+ and EC-Meter GLP 31+ (Crison, Spain). Chemical oxygen demand (COD) was determined using kits (Merck, Germany) following the experimental protocol provided by the manufacturer. Color measurements were carried out using a spectrophotometer (Hewlett Packard HP-84-53, Waldbronn, Germany) by determining the absorbances at 436 nm, 525 nm, and 620 nm and calculating the color according to ISO 7887. Suspended solids in the reactors were measured following the Standard Methods [22]. Statistical analysis of the bioremediation monitoring variables involved conducting a linear correlation analysis of parameter pairs using a symmetric Pearson Product-Moment correlation matrix. One-way analysis of variance (ANOVA) was performed to determine significant differences between reactors.

The membrane pieces used in the nanofiltration tests were characterized using Field Emission Scanning Electron Microscopy (FESEM) with an Auriga Compact instrument from Zeiss (Orsay Physics Kleindiek Oxford Instruments, UK). Imaging parameters included a 1.5 kV accelerating voltage, a 5.5 mm working distance, and a secondary electron (SE) detector to obtain surface information for membrane fouling evaluation.

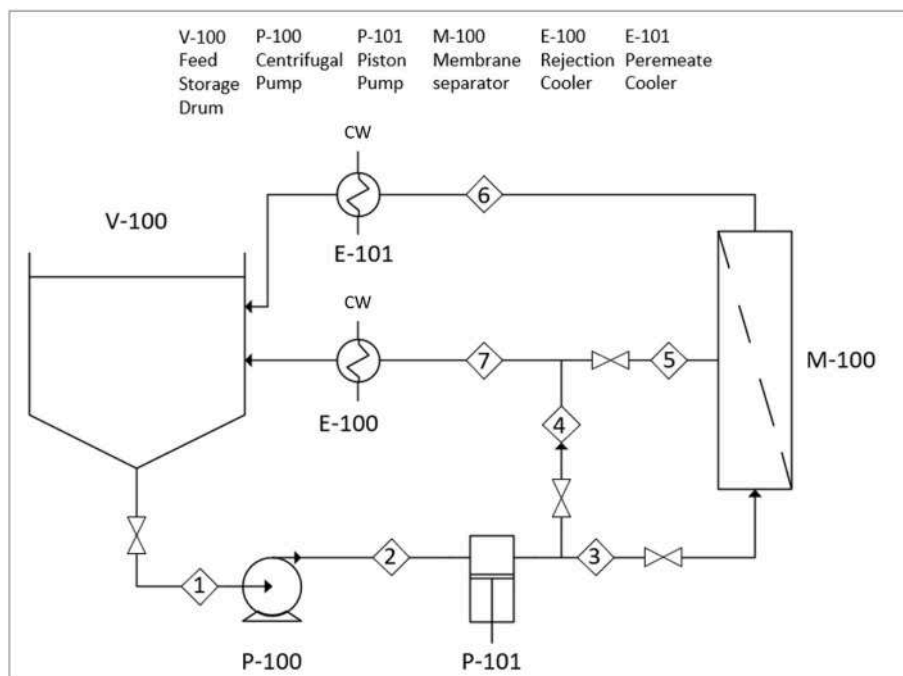


Fig. 3. Schematic diagram of the cross-flow laboratory plant. CW is the cooling water service.

2.4. Techno-Economic study

A Monte Carlo algorithm was employed to calculate the value distribution of unit costs for the combination of bioremediation and nanofiltration. The discount rate, assumed to follow a normal distribution, ranged from 9.27% to 12% based on the Weighted Average Cost of Capital (WACC) for the chemical sector from 2003 to 2023. Considering the found unit costs, the present value (C_{PV}) was calculated using Eq. (3).

$$C_{PV} = \frac{C_L}{(1+i)^t}, \quad (3)$$

where: C_L is the cost for the technology (USD), i is the discount rate with normal distribution, and t (years) corresponds to the number of years elapsed between the year of the found cost and the current year.

3. Results and discussion

Indigo carmine was considered a contaminant in this study because the effluent to be treated comes from the textile industry, specifically from the jeans washing and finishing stages. The matrix resulting from the washing process contains a mixture of soaps, surfactants, softeners, cotton fibers, and indigo carmine dye. The latter has been categorized as a toxic dye due to its harmful effects on human health upon skin and eye contact. Furthermore, from a chemical perspective, the structure of indigo carmine comprises four benzene rings and two negatively charged sulfonates, rendering it non-biodegradable, highly toxic, and carcinogenic to both humans and aquatic life [23].

The pollution effects of indigo carmine that are not fixed in the fabric are also associated with increased turbidity, which reduces the photosynthesis process, posing a threat to aquatic ecology. Therefore, it is of vital importance to remove indigo-colored materials from wastewater to mitigate environmental impacts and comply with environmental regulatory guidelines [24].

3.1. Wastewater characterization

The wastewater characterization of the raw effluent was conducted, focusing on pH, conductivity, chemical oxygen demand (COD),

suspended solids (SS), and color. The mean values obtained for these parameters are presented in Table 1.

The results reveal that the wastewater exhibits alkaline properties, with a pH value of 9.21. Furthermore, the COD concentration in the wastewater is significantly higher when compared to municipal wastewater. The effluent exhibited a distinct blue color, with the highest absorbance observed at 620 nm, aligning with the expected indigo spectrum [25].

The alkaline nature of the wastewater indicates a potential need for pH adjustment during the treatment process to optimize the removal of pollutants. The high COD concentration highlights the complex and organic nature of the wastewater, indicating the presence of various organic compounds that require effective treatment strategies. The intense blue color of the wastewater, particularly at 620 nm, confirms the presence of indigo compounds commonly used in denim dyeing processes [26,27]. The absorption peaks at different wavelengths further suggest the presence of other chromophores contributing to the overall color of the wastewater [28].

3.2. Bioremediation previous to nanofiltration

The textile wastewater's biological treatment was conducted before the nanofiltration process. Fig. 4 illustrates the variation of pH and COD with the operating time for both reactors. The pH remained relatively constant throughout the experiment at around 9 in both reactors, with a slight decrease at the beginning (reaching 8.7 in R-1 and 8.3 in R-2). The analysis of pH behavior during the treatment revealed an initial decrease

Table 1
Textile wastewater characterization.

Parameter	Effluent
pH	9.21
Conductivity ($\text{mS} \cdot \text{cm}^{-1}$)	3.220
COD (ppm)	3042
Suspended solids (g/L)	0.066
Color 436 nm	1.050
Color 525 nm	1.234
Color 620 nm	1.825

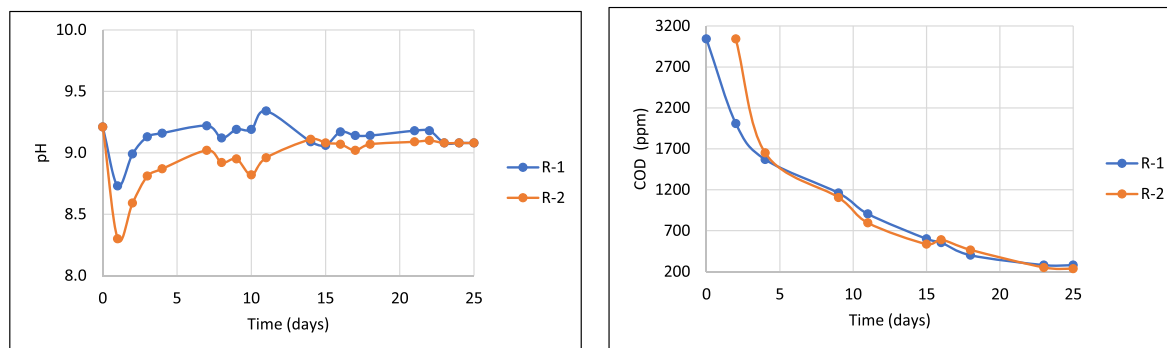


Fig. 4. Variation of pH and COD in the treated wastewater from both reactors.

in the process. This decrease can be attributed to an inoculum dilution factor added to the bioremediation stage, as reported by Trivedi et al. [29] in studies of azoic methyl orange dye bioremediation using activated sludge from domestic wastewater. These studies demonstrated a similar pH trend, starting from a pH value of 7.5 in the initial dye solution. During the initial days, they observed a decrease until reaching a stable pH near 5.8 [29]. This suggests that the pH change is associated with the microorganism's adaptation phase to the nutrient medium rather than physical-chemical reactions [30].

Regarding COD, a continuous reduction in COD concentration was observed in the effluents from both reactors. The COD degradation rate decreased over time, reaching a final value of approximately 200 mg/L. The values measured for both reactors were practically identical from the 4th day on. As depicted in Fig. 5, statistical analysis indicated no significant differences between the two reactors' mean COD (ppm) values. The F-ratio (0.224) and the p-value (0.6421) suggest no statistically significant difference between the COD removal efficiencies of the two reactors.

Fig. 6 demonstrates the variation in reactors' suspended solids (SS) concentration. As anticipated, SS levels increased sharply during the initial days due to rapid organic matter assimilation. It should be noted that SS in the reactor serves as an indirect measure of microbial concentration. In the first ten days, different SS concentrations were observed in both reactors due to the contribution of microorganisms from the inoculum in R-2. After this period, SS concentrations became very similar in both reactors, increasing until the 15th day. Subsequently, SS concentration remained relatively constant at around 1.8 g/L, mainly due to the scarcity of organic substrate.

Table 2 summarizes the removal efficiencies of COD and color in both reactors at the end of the experiments. Both reactors achieved high

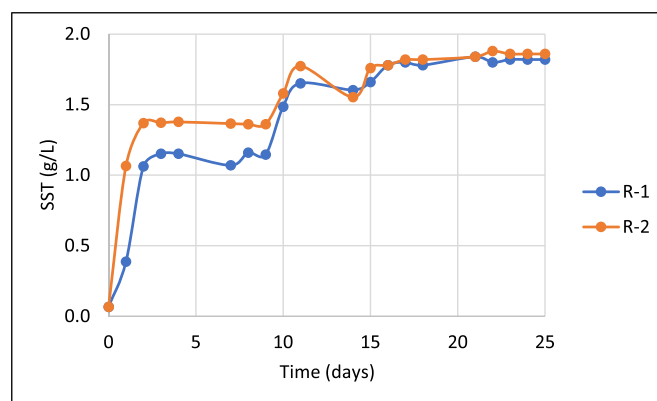


Fig. 6. Variation of SS concentration in both reactors.

Table 2

Performances of both reactors at the end of the experiments.

Parameter	R-1 removal efficiency (%)	R-2 removal efficiency (%)
COD (ppm)	90.8	92.3
Color		
436 nm	81.0	85.9
525 nm	91.3	93.2
620 nm	94.1	95.4

COD removal efficiencies, surpassing 90%. Color removal, measured in terms of absorbances at 525 nm and 620 nm, also exceeded 90%. At 436 nm, the color removal percentages were 81.0% for R-1 and 85.9% for R-2. Thus, the final absorbance values were very low (between 0.1 and 0.2) independently of the wavelength. That means that color was removed at a higher extent for the three wavelengths. In this way, the highest removal percentages corresponded with the wavelength at which the highest initial absorbance was measured.

In a study by Ref. [17], dilution of denim wastewater was required for microbial acclimation. After 56 days of acclimation, wastewater was fed to the reactor without dilution, resulting in COD removal efficiencies ranging from 84% to 91%. The removal percentage for color was reported as 75%.

A correlation analysis was performed to assess the relationship between the measured variables in the treated wastewater (COD and color) and SS in the reactors. An asymmetric Pearson product-moment correlation matrix was utilized for this purpose [31]. The Pearson matrix, depicted in Fig. 7, reveals a correlation higher than 99% between COD and color, which aligns with expectations considering that indigo is an organic molecule.

Similarly, a correlation was observed between SS concentration in the reactor and color and COD concentrations. As SS concentration increased, there was a decrease in COD and color in the effluent, as

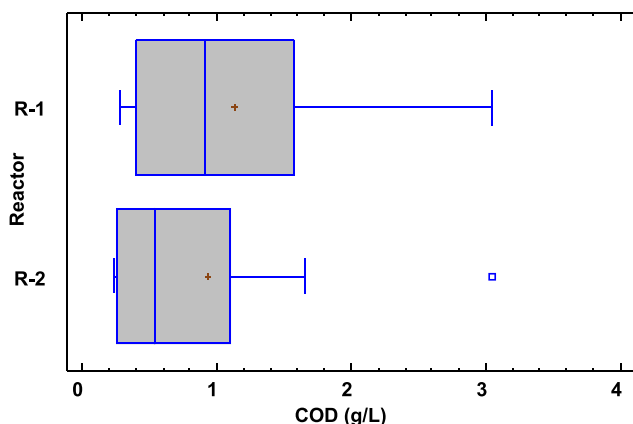


Fig. 5. Tukey box/box- and -whisker plot for the treated wastewater from both reactors.

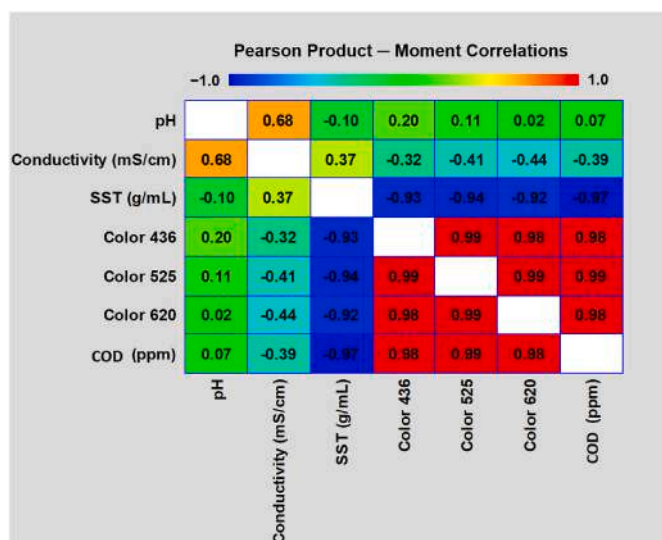


Fig. 7. Pearson product-moment correlation matrix for the process.

anticipated, due to microbial growth resulting from organic matter degradation. Conversely, pH and conductivity did not correlate significantly with other process variables. Table 3 shows the p-value matrix for each Pearson value estimated, indicating that the found correlation between COD and color, or SS and color are statistically significant (p-value < 0.05).

In conclusion, the biological treatment of the textile wastewater demonstrated effective COD and color removal. The reactors achieved high removal efficiencies, exceeding 90% for both parameters. The correlation analysis further confirmed the relationship between the reactors' COD, color, and SS concentration. These findings contribute to our understanding of the performance and dynamics of the treatment process, highlighting the importance of biological treatment as a crucial step before nanofiltration for the successful remediation of textile wastewater.

3.2.1. Sustainable monitoring

The correlations found between COD, color, and SS concentration in the reactors have significant implications for enhancing the sustainability of wastewater treatment processes and promoting water reuse. This is particularly critical in terms of cost reduction and minimizing the environmental impact associated with traditional COD determination methods.

Color, being a rapid and cost-effective variable to measure, offers a distinct advantage over COD and SS, which typically necessitate sample treatment and the use of toxic chemicals for analysis. By establishing a robust linear correlation between color and these parameters, we can effectively utilize color measurements as a viable proxy for monitoring and managing the treatment process.

Utilizing color as a surrogate parameter for COD and SS facilitates a streamlined monitoring approach, thereby reducing the need for extensive and resource-intensive laboratory analyses. This reduction in

the number of tests not only saves valuable time but also significantly reduces the consumption of chemicals, energy, and laboratory resources. Consequently, this leads to a substantial reduction in the overall operational costs of the treatment process, contributing significantly to its economic sustainability.

Furthermore, employing color as a monitoring parameter aligns seamlessly with the principles of green chemistry and sustainability. Green chemistry places emphasis on designing and implementing chemical processes that are environmentally friendly, resource-efficient, and economically viable [32]. By minimizing our dependence on COD and SS measurements, which entail the use of hazardous chemicals and result in waste generation, we can promote a more sustainable and environmentally conscious approach to process monitoring. A similar approach was proposed by Ref. [33], which involved the development of a prototype for an in-situ seawater COD monitoring sensor based on UV-Vis absorption spectroscopy, aimed at reducing secondary pollution associated with monitoring.

One crucial environmental consideration, especially pertinent to COD determination, is the reliance on oxidation reactions involving reagents categorized as toxic. These reagents, including $K_2Cr_2O_7$, Ag_2SO_4 , $FeSO_4 \cdot 7H_2O$, $Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$ [34] contribute to the generation of hazardous chemical waste during research phases and analytical processes. The utilization of correlation techniques plays a pivotal role in reducing the generation of hazardous chemical waste in industrial-level analytical laboratories.

3.3. Dead-End nanofiltration tests

3.3.1. Membrane permeability

Fig. 8 shows the water permeability of the NF270 and Alfa Laval membranes, showing minimal differences between them. Both membranes exhibited a permeability of approximately $10 \text{ L} \cdot \text{h}^{-1} \cdot \text{m}^{-2} \cdot \text{bar}^{-1}$.

3.3.2. NF tests using the pretreated effluent from R-1

Fig. 9 presents the behavior of the NF membranes in the dead-end

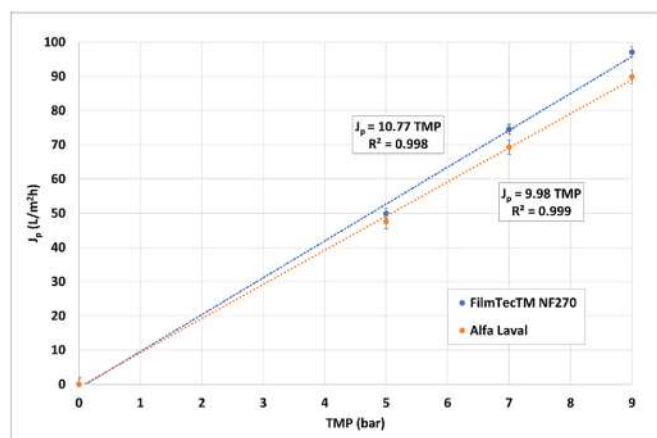


Fig. 8. Membrane permeability for NF270 and Alfa Laval membranes.

Table 3
p-Value correlation matrix for the process.

p-value	pH	Conductivity (mS/cm)	SST (g/mL)	Color 436	Color 525	Color 620	COD (ppm)
pH	–	4.39×10^{-2}	7.98×10^{-1}	6.06×10^{-1}	7.78×10^{-1}	9.59×10^{-1}	8.58×10^{-1}
Conductivity (mS/cm)	4.39×10^{-2}	–	3.27×10^{-1}	4.01×10^{-1}	2.73×10^{-1}	2.36×10^{-1}	2.99×10^{-1}
SST (g/mL)	7.98×10^{-1}	3.27×10^{-1}	–	2.79×10^{-4}	1.64×10^{-4}	4.41×10^{-4}	1.50×10^{-5}
Color 436	6.06×10^{-1}	4.01×10^{-1}	2.79×10^{-4}	–	3.26×10^{-7}	3.65×10^{-6}	3.65×10^{-6}
Color 525	7.78×10^{-1}	2.73×10^{-1}	1.64×10^{-4}	3.26×10^{-7}	–	3.26×10^{-7}	3.26×10^{-7}
Color 620	9.59×10^{-1}	2.36×10^{-1}	4.41×10^{-4}	3.65×10^{-6}	3.26×10^{-7}	–	3.65×10^{-6}
COD (ppm)	8.58×10^{-1}	2.99×10^{-1}	1.50×10^{-5}	3.65×10^{-6}	3.26×10^{-7}	3.65×10^{-6}	–

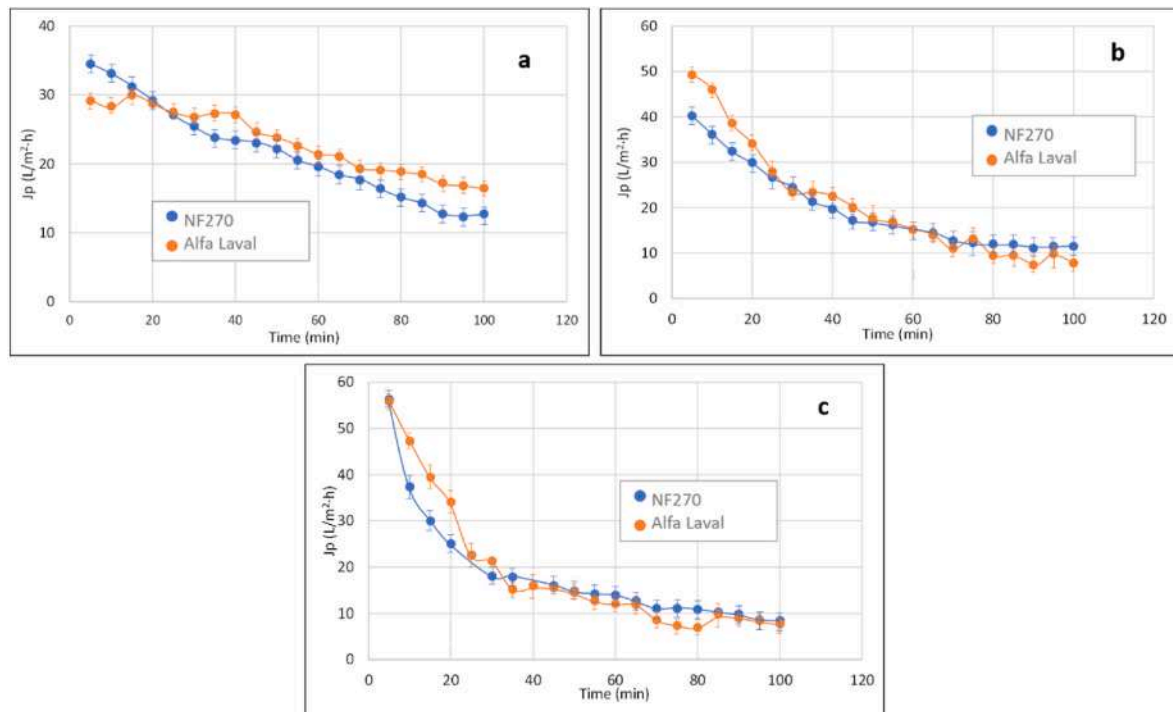


Fig. 9. Permeate Flux Values at Different Operating Conditions for R-1 Effluent as Feed (a. TMP = 5 bar, b. TMP = 7 bar, c. TMP = 9 bar).

tests at different transmembrane pressures (TMPs) of 5, 7, and 9 bar. As expected, the permeate flux decreased over time due to a combination of pore blocking and pore shrinking, consistent with the findings of [35].

Both membranes displayed similar behavior at all three TMPs, with final permeate fluxes of approximately $10 \text{ L}\cdot\text{h}^{-1}\cdot\text{m}^{-2}$ for 7 and 9 bar. The lowest TMP (5 bar) achieved the highest final flux values, reaching 17.4 and $13.8 \text{ L}\cdot\text{h}^{-1}\cdot\text{m}^{-2}$ for Alfa Laval and NF270, respectively. These results indicate that fouling increased with the TMP.

Fig. 10 illustrates the influence of membrane type and TMP on color and COD rejection.

Color rejection percentages exceeded 96% for both membranes, with no significant differences observed between them. The COD removal efficiency was consistently higher than 80% at all TMPs, with increasing TMP correlating to enhanced COD removal efficiency. These results align with previous reports on textile wastewater nanofiltration [36]. While both membranes showed similar color rejection, the Alfa Laval NF membrane demonstrated higher COD rejection than NF270. Cabrera-González et al. [37] also reported higher retention values for NF270. However, this study found the opposite trend for textile wastewater

treatment, possibly due to the effluent's higher pH (9.21).

3.3.3. NF tests using the pretreated effluent from R-2

Fig. 11 shows the permeate fluxes obtained in the experiments with both membranes at the established TMPs.

Similar to the R-1 effluent, the membranes behaved comparably for the R-2 effluent, especially at 5 and 7 bar TMPs. For TMP = 9 bar, there was a sharper flux decay for both membranes due to the higher fouling (fouling increase with TMP). The initial flux values were very different for both membranes, being the NF270 membrane the one with higher flux. This is in agreement with the membrane permeability values showed in Fig. 8, in which it is observed that the flux difference between membranes increases with the TMP. The final flux decreased with increasing TMP, attributed to a more compact cake formation. The highest final flux was measured at 5 bar (21.89 and $23.71 \text{ L}\cdot\text{h}^{-1}\cdot\text{m}^{-2}$ for NF270 and Alfa Laval NF, respectively), with a flux decay remaining below 45%. Sahinkaya et al. [17] reported a flux decay of 45% for a TMP of 5 bar. Notably, at 9 bar TMP, NF270 displayed higher initial permeate fluxes compared to Alfa Laval NF, but the final fluxes were similar.

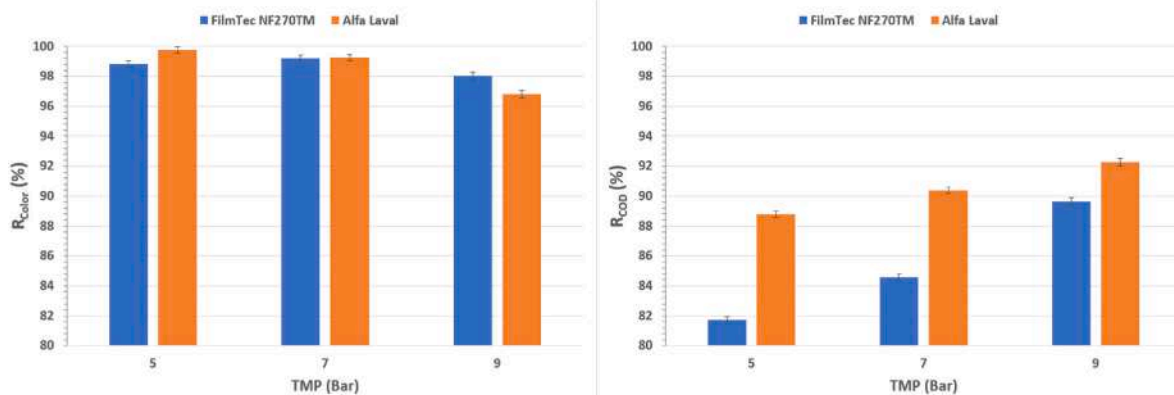


Fig. 10. Membrane Rejection to Color and COD at different TMPs for R-1. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

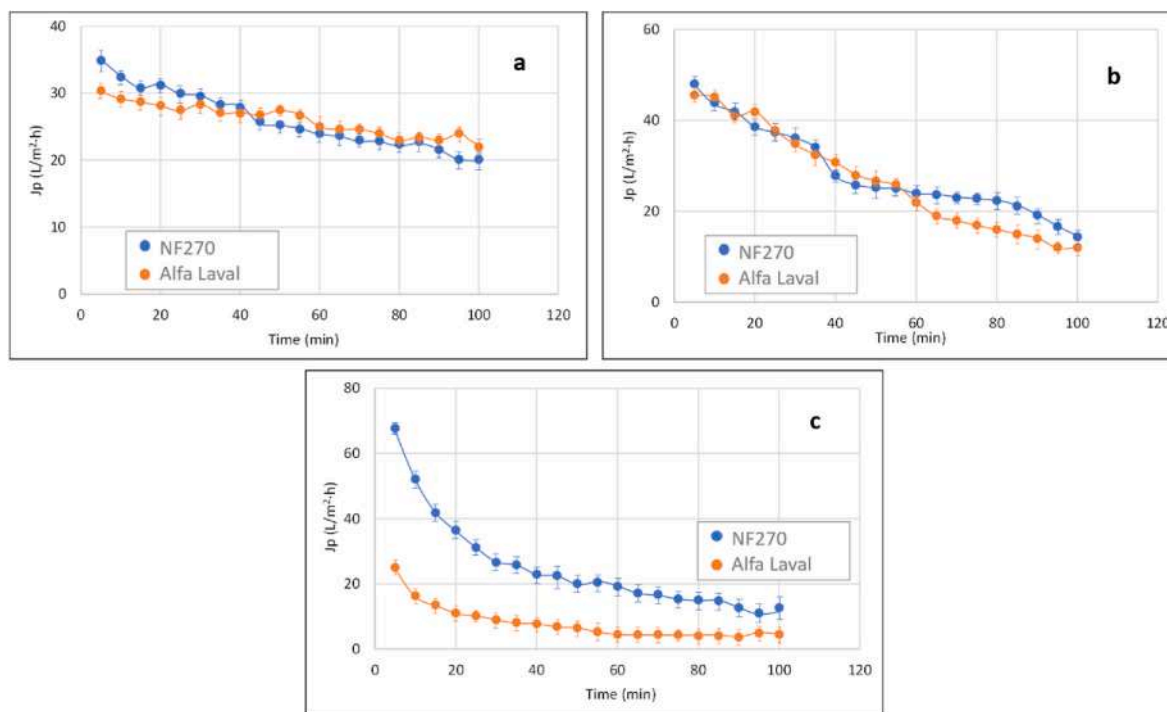


Fig. 11. Permeate flux values at different operating conditions for R-2 effluent as feed (a. TMP = 5 bar, b. TMP = 7 bar, c. TMP = 9 bar).

COD and color rejections were consistently higher than 80% and 90%, respectively, with no significant differences between the membranes (Fig. 12). Unlike for R-1 effluent, TMP did not significantly influence the process. It could be due to a higher solute accumulation on membrane surface than for R-1 effluent. The fouling layer leads to reduce the influence of TMP on COD rejection. The higher fouling for R-2 effluent can be also checked in Figs. 9 and 11 since the flux decay is higher in the case of R-2 effluent. These results align with [17] findings, where COD rejection was around 65%, and color removal was nearly complete (98%).

Overall, the NF tests demonstrated the effectiveness of both membranes in achieving high COD and color rejection, offering the potential for sustainable wastewater treatment and reuse in textile industries.

3.4. Cross-flow nanofiltration tests

Cross-flow NF tests were conducted using the effluent from R-2 at a TMP of 5 bar, as this effluent demonstrated higher permeate flux for both membranes using this effluent as a feed stream. Initially, membrane permeability was calculated, resulting in measured values of 7.85 and

4.3 L·h⁻¹·m⁻²·bar⁻¹ for NF270 and Alfa Laval NF, respectively. It is important to note that these experiments were performed with pristine membrane pieces, which could account for the differences compared to the membrane pieces used in dead-end experiments.

Fig. 13 illustrates the final permeate fluxes obtained for each membrane at various cross-flow velocities. No variation in permeate flux with cross-flow velocity was observed for both membranes. However, the permeate flux of the NF270 membrane was significantly higher than Alfa Laval NF, likely due to its lower permeability.

Once the initial flux decay occurred, no additional fouling was observed during the experiments. This fact indicates that the previous biological treatment effectively eliminated the organic compounds that could have hindered mass transfer through the membrane due to their electrical properties [38].

Regarding rejection, the Alfa Laval NF membrane showed higher COD and color rejection. COD of the permeate was 14.5 mg/L, and absorbances at 436, 525, y 620 nm were 0.003, 0.002, and 0.002, respectively. However, rejection of NF 270 membranes was lower, with a COD value in the permeate of 36.4 mg/L and absorbances values of 0.013, 0.010, and 0.011 for 436, 525, and 620 nm, respectively. It can be

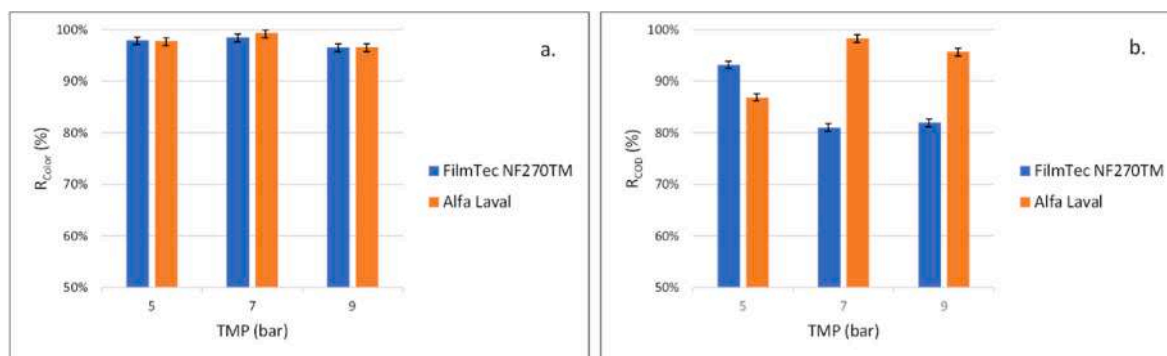


Fig. 12. Membrane Rejection to Color and COD at different TMPs for R-2. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

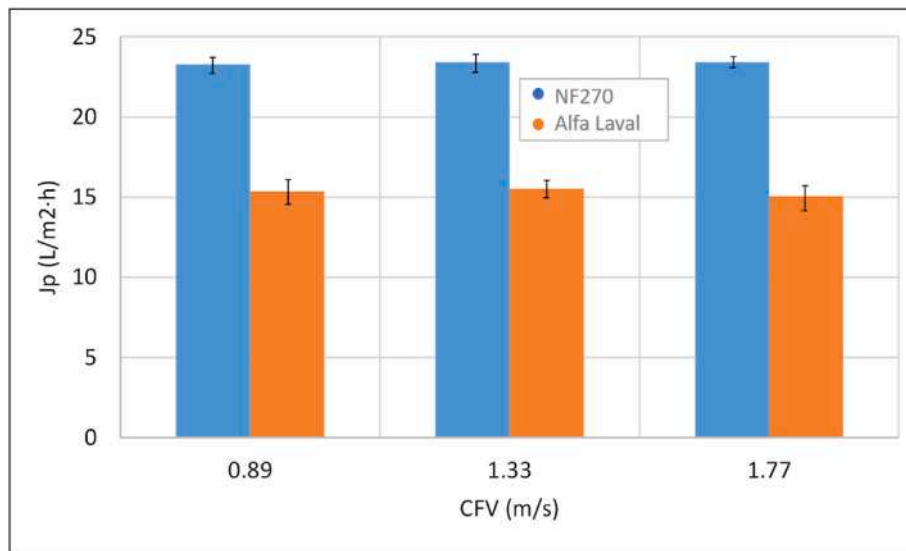


Fig. 13. Influence of CFV on Permeate flux for R-2 between NF270 and Alfa Laval membranes.

observed that the membrane with the lowest permeability yielded the highest COD and color rejection.

Regarding rejection, the Alfa Laval NF membrane exhibited higher COD and color rejection. The COD of the permeate was 14.5 mg/L, and the absorbances at 436, 525, and 620 nm were 0.003, 0.002, and 0.002, respectively. In contrast, the rejection of NF270 membranes was lower, resulting in a COD value in the permeate of 36.4 mg/L and absorbance

values of 0.013, 0.010, and 0.011 for 436, 525, and 620 nm, respectively. Notably, the membrane with the lowest permeability achieved the highest COD and color rejection. These results suggest that pores of Alfa Laval NF membrane have a lower size than those of NF 270 membrane.

Fig. 14 displays the FESEM images of the membrane surface after the NF tests once the membranes were rinsed. NF270 membrane showed

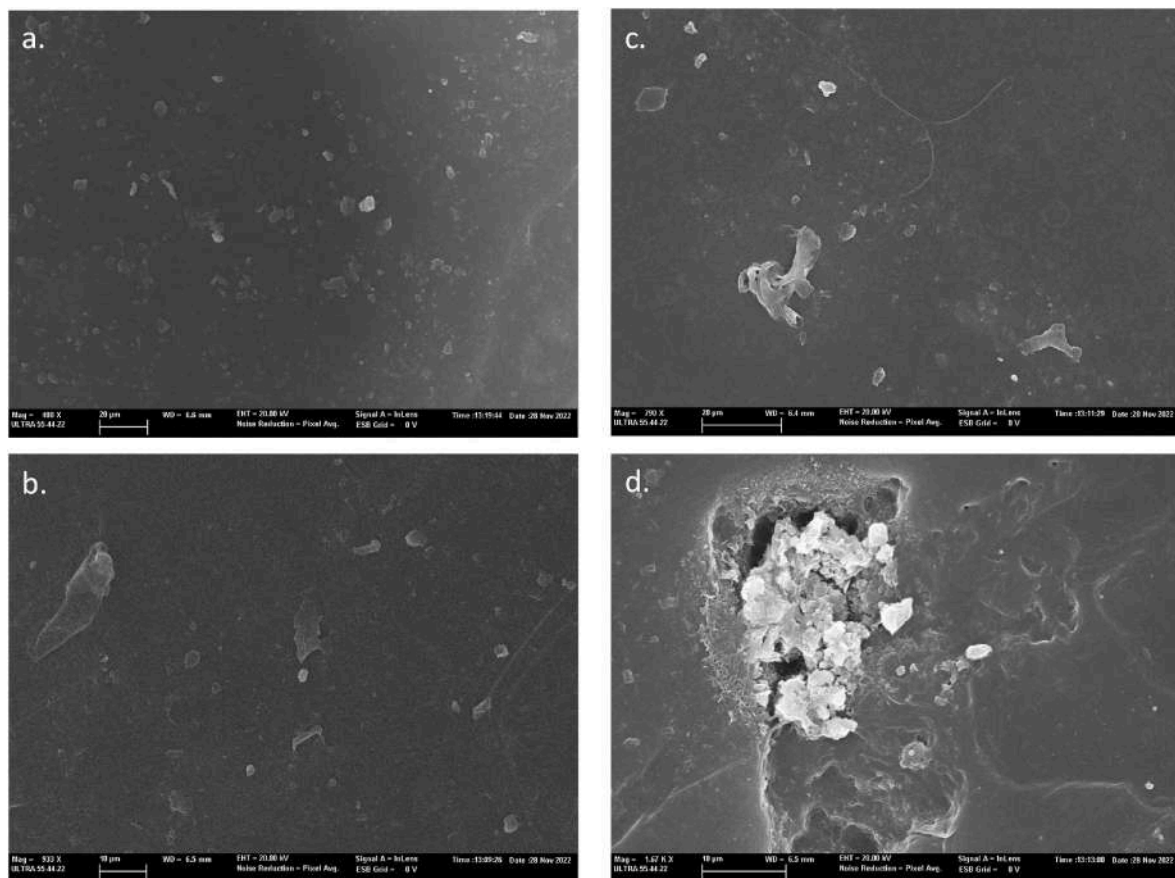


Fig. 14. FESEM membranes surface after the rinsing process. a. NF270 surface 20µm, b. NF270 surface 10µm, c. Alfa Laval surface 20 µm, d. Alfa Laval surface 10 µm.

94% permeability recovery, while the Alfa Laval NF membrane demonstrated 90% recovery of its initial permeability. The remaining fouling is visible in the FESEM images. As expected, the Alfa Laval NF membrane, with the lowest permeability, exhibited more fouling, as shown in Fig. 14c and d, where precipitated solids with an asymmetric morphology can be observed.

3.5. Techno-Economic study

In addition to the examined combination of biological treatment and NF, various technologies have been developed for treating textile wastewater. Table 4 summarizes different alternatives along with their respective unit costs, adjusted to 2023 values using a discount rate of 9.6% based on industry estimates [39].

These techniques used for the treatment of textile wastewater can yield varying performance in contaminant removal and effluent recovery, depending on operational factors. Nanofiltration, for instance, offers low operational costs (\$0.24 USD/m³) but has a low efficiency on its own in the recovery of treated effluent (36%) [45]. Furthermore, it produces a concentrate that requires an additional stage for contaminant reduction and faces fouling issues, leading to membrane pore blockage and a reduction in permeate flow or an increase in transmembrane pressure (TMP) depending on the operating mode [46].

Advanced oxidation techniques such as electrocoagulation, while achieving a 99% recovery of treated effluent due to contaminant reduction, come with a high cost (\$3.00 USD/m³) due to energy consumption of 13.2 kWh [47], 3.8 times higher than conventional wastewater treatment [48]. This cost factor can limit its industrial application, and it also generates a significant amount of solids [14]. Photochemical treatments of dyes (H₂O₂/O₃/UV) offer a 90% effluent recovery with a cost of \$58.80 USD/m³ associated with reagents and operational costs in ozone production [49]. However, disadvantages of scaling this approach include the risk of hydrogen peroxide (H₂O₂) corrosion and the introduction of complexity into the treated water, posing risks to humans, ecological systems, and the environment [50].

On the other hand, hybrid treatment involving ozone and biological processes comes at a cost of \$94.70 USD/m³, allowing for a 94% recovery of treated effluent. Which is the highest unit cost due to the high-energy requirements for ozone generation (2.54 kW/m³) [51]. Although this process can handle wash baths rich in salt and high dye concentrations, it may face challenges with ozone use, as ozone can be depleted in the presence of organic compounds such as humic acids (commonly found in natural water), reducing disinfection efficiency as less oxidant will be available for microorganisms [52].

Treatment methods that combine membrane filtration equipment in their operation have become attractive options for textile, food, paper, printing, and vehicle production industries [53]. However, the use of this technique is limited by fouling of the membrane layer, reduced flow, and membrane regeneration [54]. Effluent recovery through a membrane bioreactor reached 73% with a unit cost of \$0.24 USD/m³. In contrast, for the activated sludge + nanofiltration treatment technique,

Table 4
Unit costs of different alternatives for textile wastewater treatment.

Technology	Unit cost USD/m ³	Year	Equivalent to 2023 USD/m ³ unit cost	Percentage of dye reduction	Reference
Nanofiltration	0.24	2021	0.29	36%	[40]
Electrocoagulation	3.00	2020	3.91	99%	[41]
H ₂ O ₂ /O ₃ /UV	58.80	2010	186.19	90%	[42]
O ₃ /Biological treatment	94.70	2003	557.66	94%	[43]
Membrane bioreactor	0.21	2022	0.23	73%	[44]
Activated sludge + nanofiltration	0.29	2005	1.45	94%	[43]

the operational cost was \$0.21 USD/m³ for a 94% recovery of treated effluent. This combination offers a competitive alternative, harnessing the advantages of both techniques, achieving the removal of various types of dyes, low decolorization times, and dye degradation through different metabolic pathways [14]. In conclusion, the proposed hybrid treatment is deemed feasible as the nanofiltration membrane is fed with effluent from biological treatment with low organic matter concentration.

A Monte Carlo simulation with 20,000 iterations was conducted to assess different scenarios of unit cost distribution for the proposed treatment, as depicted in Fig. 15.

The statistical analysis revealed a mean unit cost of 0.97 USD/m³ for the hybrid technology (biological treatment + NF). Scenarios with less than 5% probability indicate a minimal unit cost of 0.39 USD/m³ and a maximal cost of 1.51 USD/m³. Hence, the proposed treatment emerges as highly competitive among other technologies, providing a promising solution for reusing textile wastewater.

The results obtained from the study, showcasing the successful combination of biological treatment and nanofiltration in reducing COD levels to 200 mg/L and eliminating over 90% of color in textile indigo dye wastewater, present a significant advancement in water reuse for the textile industry. This hybrid technology aligns well with the principles of Cleaner Production and the Blue Economy strategy [9].

Traditionally, the textile industry has heavily relied on high-quality freshwater for its production processes, leading to extensive water consumption and the discharge of untreated wastewater into sewage systems. Due to their high costs, the introduction of advanced wastewater treatment technologies has been limited, particularly for small and medium-sized textile companies [55]. However, the new findings demonstrate that combining biological treatment and nanofiltration offers a cost-effective alternative for treating textile effluents.

With a mean unit cost of 0.97 USD/m³, the hybrid technology (biological treatment + NF) proves economically feasible and competitive compared to existing commercial purification processes and alternative solutions. Notably, this cost is considerably lower than the mean of 1.85 USD/m³ reported by Vajnhandl [55] for a different effluent recycling system based on physical-chemical pre-treatment, ultrafiltration, and ozonation. As such, hybrid technology offers a more cost-efficient approach to achieve substantial pollutant removal and water quality improvement.

Additionally, the hybrid technology's impressive COD reduction and color elimination enhance the potential for water reuse in the textile industry. Reusing treated wastewater conserves freshwater resources and minimizes the environmental impact of discharging effluents into natural water bodies. This approach aligns with the broader goals of sustainable water management and circular economy principles [56].

4. Conclusions

This study successfully demonstrated a sustainable approach for treating wastewater contaminated with indigo dye through a hybrid approach combining bioremediation and nanofiltration for reuse.

The bioremediation stage using native microorganisms and activated sludge from a wastewater treatment plant (WWTP) effectively reduced chemical oxygen demand (COD) to 200 mg/L. It eliminated over 90% of the color in denim washing wastewater contaminated with indigo dye. This successful COD and color removal highlights the potential of both bioaugmentation strategies for sustainable wastewater treatment.

The statistical analysis revealed no significant difference in COD removal efficiencies between the two bioaugmentation strategies (using native microorganisms and activated sludge). This finding suggests that both strategies can be equally considered for the bioremediation of indigo dye-contaminated wastewater, offering flexibility and adaptability to different treatment scenarios.

The strong correlation (>99%) between COD and color in the Pearson matrix indicates the organic nature of the indigo dye, validating

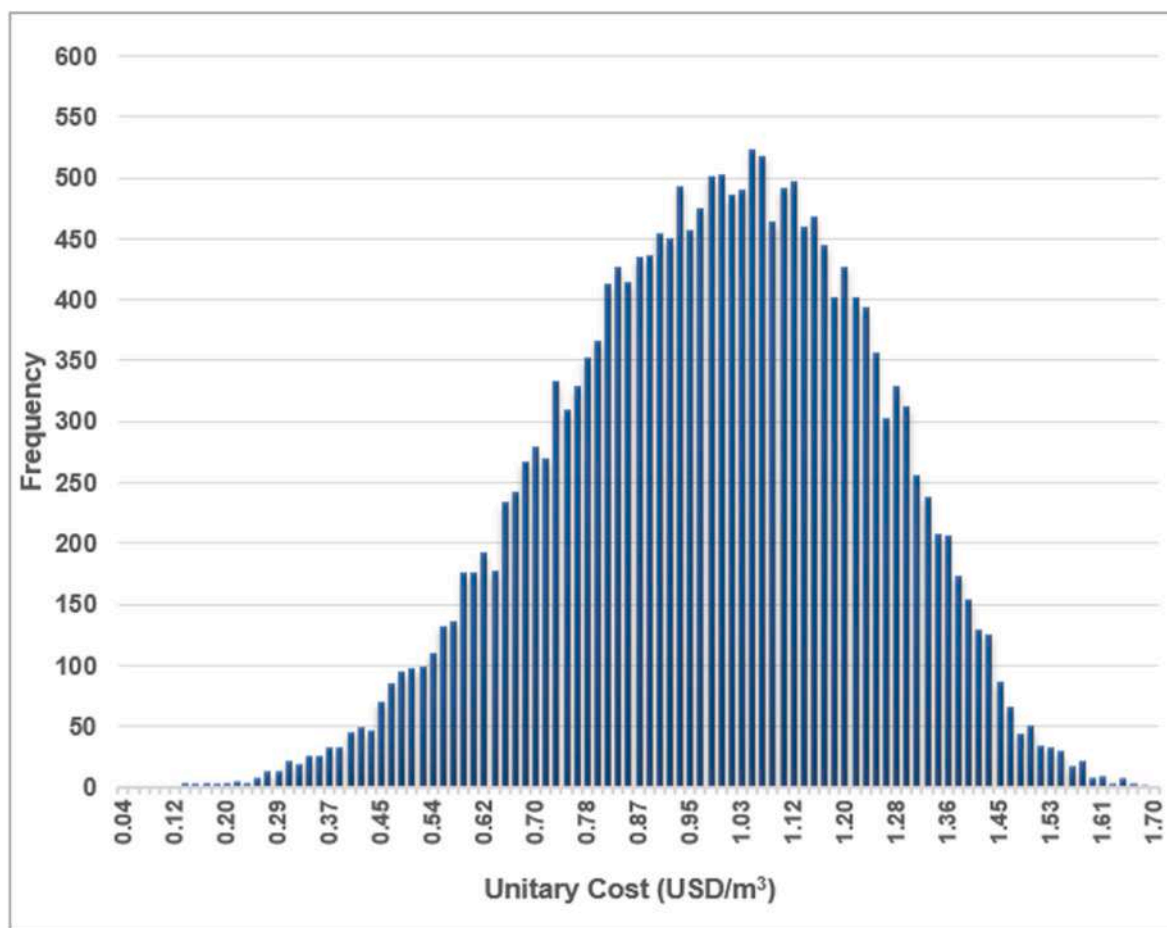


Fig. 15. Results of the Monte Carlo simulation for the unitary cost distribution in USD/m³.

color as a reliable and cost-effective proxy for COD measurement during the treatment process. Furthermore, the correlations between suspended solids (SS) concentration and color and COD concentrations provide valuable insights into sustainable wastewater treatment and water reuse, with potential cost reductions and environmental benefits.

The nanofiltration (NF) stage demonstrated high color rejection rates (>96%) and COD removal efficiency (>80%) for both NF 270 and Alfa Laval NF membranes. Although fouling increased with transmembrane pressure (TMP), the overall performance of the membranes remained satisfactory, suggesting their suitability for sustainable wastewater treatment. The highest flux of 21.89 and 23.71 L h⁻¹ m⁻² for NF270 and Alfa Laval NF, respectively, was obtained at 5 bar.

In cross-flow nanofiltration tests using the effluent from the reactor employing activated sludge (R-2), the Alfa Laval NF membrane exhibited higher COD and color rejection compared to NF 270 membrane. This finding highlights the potential superiority of Alfa Laval NF membrane for achieving higher quality permeate suitable for reuse in denim washing processes.

The FESEM images confirmed the feasibility of membrane recovery, with NF 270 and Alfa Laval NF membranes recovering 94% and 90% of their initial permeability after rinsing. No irreversible fouling of the membranes was observed.

The techno-economic analysis underscored the economic feasibility and competitiveness of the hybrid approach combining biological treatment and NF for wastewater treatment. With a mean unit cost of 0.97 USD/m³, the hybrid technology is a promising and cost-effective alternative to existing commercial purification processes and other solutions.

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Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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