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Application of membrane processes in closing of water cycle in a textile dye-house☆

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ABSTRACT

In this study, investigations were made to close water cycle in dye-houses. At the first stage, optimum operation conditions of a membrane nanofiltration were determined. Experiments were carried out for DL membranes (Osmonics, USA). The effect of temperature, pressure and pH was investigated. Investigations were performed for different types of real wastewater produced in a laboratory-scale Pyrotec S dyeing apparatus (Roaches) for various types of reactive dyes. An over 90% dye hold-up was obtained for all dye baths.
At the final stage of the experiments, dyeing processes were carried out with the use of a filtrate from nanofiltration as water applied for dyeing, rinsing and washing after dyeing. In cooperation with the Technical University of Lodz, we have managed to apply biological degradation of the concentrate, carried out in two different reactor systems.

1. Introduction

Nanofiltration is a known and efficient method for the separation of pollutants from water which allows us to close the circulation of water used in dye-houses [1–5]. However, after the process of nanofiltration, a concentrated mixture of dyes and auxiliary substances (salts, acids, alkalies, organic compounds) remains, which causes a serious problem. The problem must be solved to enable a successful implementation of this technology in industry. It follows from our earlier works that concentrate coming from the nanofiltration processes and reverse osmosis can be utilized with biological methods [6].

Biological methods are generally considered as environmentally friendly. Furthermore, they can lead to complete mineralization of organic pollutants at relatively low costs [7,8]. It is known that conventional aerobic processes used in municipal wastewater treatment plants are insufficient in dye degradation, especially azo dyes, which represent the largest colorant's class and are the most commonly used in the textile industry [9]. However, the azo bonds can be biodegraded in the anaerobic conditions in the presence of the external electron donors. The total biodegradation of azo dyes generally requires a combination of two stages, anaerobic reduction of azo bond and aerobic degradation of formed aromatic amines [10].

A strong salinity of the wastewaters is one of the serious problems of the textile industry. The salt is applied to increase the affinity of dye molecule to cellulose fiber in dyeing with reactive dyestuffs and direct dyestuffs. Therefore, the research on developing a closed cycle of brine arouses a growing interest [11].

The goal of this study was to develop a complex method of closing the circulation of water and brines in dye-houses in combination with the biological utilization of concentrate by biological methods [12,13]. A subject of this study was wastewater, which was obtained after dyeing of knitted fabric with reactive dyes. After the filtration processes, the wastewater in the form of filtrate was turned back for the dyeing processes of different knitted fabrics.

2. Experimental methods

2.1. The optimization of the nanofiltration process

In the first stage of this research, the optimum working parameters of a DL nanofltration membrane (TF polymer, reaction size 96 $MgSO_4$, 25 °C, pH range 2–11, typical flux/psi 31/100) were determined. The experiments were carried out for the model wastewater coming from dyeing with reactive dye C.I. Reactive Red 120.

Apart from the hydrolyzed unbound dye, the dye bath contained considerable quantities of salt (NaCl) and soda (Na_2CO_3) which are usually applied in the process of dyeing with reactive dyestuffs. In the solution there were also small quantities of Dekol SN — an anionic surface-active agent used for washing of knitted fabrics after dyeing, and acetic acid (CH_3COOH) which was used to neutralize pH of the rinsing bath (Table 1).

A parametric assessment of the process was made on the basis of filtration rate, DFZ and specific conductance of a filtrate and concentrate.

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Table 1
Stages and parameters of the dyeing process.

• dyeing (dyeing baths contained 3% dye, 50 g/dm^3 NaCl, and 15 g/dm^3 Na_2CO_3), dyeing temperature 80 °C;
• 1st rinsing after dyeing 70 °C;
• 2nd rinsing after dyeing 60 °C;
• neutralization with CH_3COOH to pH 7 − 8
• washing: 98 °C, Dekol SN — anionic surfactant 2 g/dm^3;
• 1st rinsing after washing 70 °C;
• 2nd rinsing after washing 40 °C.

The DFZ parameter was calculated in accordance with DIN — 38404/1 standard:

$$DFZ = \frac{1000 \cdot E(\lambda)}{d} [1 / \mathrm{m}]$$

where: $E(\lambda)$ — absorbance for a given wavelength and d — thickness of the absorption cell [mm].

2.2. Biodegradation of concentrate

Experiments with the post-filtration concentrate biodegradation were conducted in two systems — single SBR and the sequential anaerobic–aerobic two-sludge system.

The key element of the SBR set-up was a bioreactor with a total and working volume of 1 and 0.8 L, respectively, equipped with a propeller stirrer (IKA, type Eurostar Digital, mixing speed 100 rpm). The experiments were conducted at ambient temperatures of 20 to 25 °C. The second system was a two-sludge system consisting of two reactors and a transition tank. One (working volume 0.4 L) was operating under anaerobic conditions at the constant temperature (37 ± 1 ºC). The second one (working volume 0.8 L) was operating under aerobic conditions at the ambient temperature (20–25 °C). The peristaltic pumps (Vertex, type PPO1) and digital timers (Metron, type PCg.O3) enabled filling and drawing of bioreactors and the transition tank. The SBR bioreactor and aerobic bioreactor were aerated by an air pump, through a rotameter. Gas distributors were placed on the bottom of the reactors (aeration rate — 0.8 vvm). The SBR bioreactor and anaerobic reactor were mixed by the propeller stirrer (IKA, type Eurostar Digital, mixing speed 100 rpm).

Both bioreactor systems were working in 24 h cycles: sedimentation 60 min, drawing 20 min (10 L min^{-1}), filling 20 min (10 L min^{-1}), reaction 22 h 20 min (single SBR: 19 h 20 min mixing, 3 h aeration; aerobic reactor — only aeration; anaerobic reactor — only mixing).

The single SBR reactor was inoculated with 320 mL of the surplus activated sludge SAS and 80 mL of the fermented sludge (FS). The aerobic reactor was inoculated with 400 mL of the surplus activated sludge (SAS), while the anaerobic one was inoculated with 200 mL of the fermented sludge (FS).

During the first week of operation synthetic wastewater was treated in both systems in order to adapt microorganisms to the new conditions.

As textile wastewater does not contain all substances which are necessary for biomass growth, the concentrated solution of a synthetic domestic wastewater was used in the bioreactor experiments. The solution had the following composition (g L^{-1}): casein peptone (1.56), dry broth (1.05), NH_4Cl (0.20), NaCl (0.07), $CaCl_2 \bullet 6H_20$ (0.075 g), $MgSO_4 \bullet 7H_2O$ (0.02), KH_2PO_4 (0.20), K_2HPO_4 (0.50).

Synthetic wastewater solution was diluted ten times and supplemented with 1 mL of the acetic acid per liter. Thereafter, an adaptation of sludge to concentrates started. Every two or three days 10% more concentrate was added until 90% of the concentrate in the feed was achieved. Additionally, it contained also 10% of the synthetic wastewater and different amounts of the acetic acid. Both systems were fed with the identical wastewater mixture 200 mL in volume. The progress of acclimatization was controlled by taking samples of the influents and effluents.

2.3. Verification of the applicability of purified wastewater

In the final stage of this work a series of tests were done to establish the possibility of closing the water and brine cycle in dye-house. In this part of the research the real wastewater coming from one Polish textile factory was used for filtration. In order to have result repeatability and to eliminate variation, the wastewater was taken from one batch directly from the dyeing machine. To model the wastewater considerable amount of salt, soda and anionic surfactants were added.

The nanofiltration process was run at a constant volume rate (ca. 2 dm^3/min), pressure 1.5 MPa, at the temperature 31–40 °C, with a DL membrane (TF polymer, reaction size 96 $MgSO_4$, 25 °C, pH range 2–11, typical flux/psi 31/100). The reverse osmosis process was run at the pressure 2.2 MPa, and temperature 31–40 °C, with a SG membrane (Desalination Systems, USA).

In the second stage of the test, filtrates from nanofiltration and/or reverse osmosis were applied as water used for dyeing process. Tests were performed in a laboratory dyeing machine, *Pyrotec S,* Roaches (UK) for knitted fabrics with the following composition: cotton 100%, polyester 100%, and viscose/Lycra mix (95%/5%) (Table 2).

Dyed samples were analyzed for their functional quality. Relative dyeing intensity (color strength) of samples dyed in purified process wastewater in comparison to dyeing intensity obtained in dyeing in standard water was measured with a Spectraflash 500 spectrophotometer (Datacolor Int.).

3. Results and discussion

3.1. Optimization of nanofiltration

Running costs of membrane filtration depend mainly on working parameters. Volume rate, the efficiency of filtration and durability of membranes depend on a number of parameters such as temperature, pressure, filtrate pH and its chemical composition. Some chemicals cause a considerable decrease in the filtration rate, and some may even permanently damage the membrane. One of the first tasks before implementing the closed water circulation using membrane techniques is the optimization of filtration process considering specificity of a given factory, and careful selection of wastewater streams, in order to separate the chemicals which cause permanent membrane damage.

The rate of both nanofiltration and reverse osmosis increases with pressure growth, however, this is not the only factor which we should take into account in the optimization of filtration in order to close the process water circulation in the textile industry. Textile industry is quite specific in respect to requirements for water applied in processing. Poor water quality can make it impossible to obtain the desired effect of dyeing or other textile finishing processes. Therefore, to use the filtrate as water, it must be wholly colorless and free from suspended matter and dissolved organic compounds, which is not always possible. We observed that with pressure increase the filtrate quality deteriorated. Hence, some optimum pressure value should not be exceeded.

An important parameter is the degree of concentration. With an increase of concentrate concentration the content of salt both in the

Table 2
List of applied dyes used in this work for knitted fabrics.

Knitted fabric	Cotton 100%	Polyester 100%	Viscose/Lycra (95%/5%)
Dyes applied	Doractiv Crimson LXE Doractiv Navy Blue LXE	Lumacron Rot EFB 2% Lumacron Navy Blue SEGS 1%.	Holux Supra Blue BL (C.I. Direct Blue 106)