

Toward Resource Recovery from Textile Wastewater: Dye Extraction, Water and Base/Acid Regeneration Using a Hybrid NF-BMED Process

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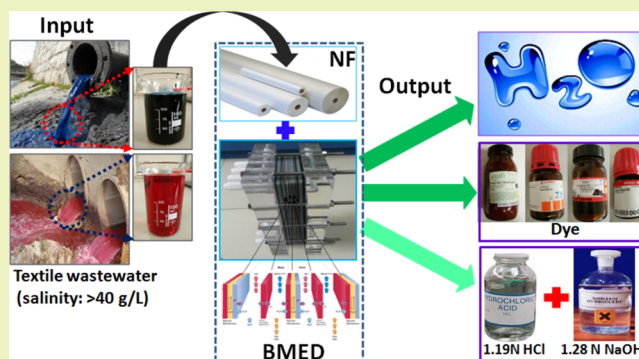
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S Supporting Information

ABSTRACT: In this work, textile wastewater is explored for resource recovery in a hybrid loose nanofiltration (NF)-bipolar membrane electro dialysis (BMED) process for fractionation of dyes and salt, in view of dye purification and water and salt reuse. A loose nanofiltration membrane, i.e., Sepro NF 6 (Ultura), found to have a low salt rejection (0.27% in 120 g·L⁻¹ NaCl solution) and high rejection for direct dyes and reactive dyes (≥99.93%), was used for fractionation of dye/salt mixtures through diafiltration. In diafiltration, the addition of pure water with a volume factor of 5.0 can effectively remove the NaCl salt by using Sepro NF 6 with an invariable dye concentration, in view of the recovery of high purity dyes. The overall salt rejections in diafiltration for the dye/salt mixtures with 40, 50 and 60 g·L⁻¹ NaCl are 2.2%, 1.8% and 1.1%, respectively, enabling a further treatment by BMED. Subsequently, application of BMED for reuse of salt-containing NF permeate demonstrates that desalinated water with ~100 ppm of NaCl can be obtained, and base/acid can be produced from the salts without any membrane fouling by dyes. Therefore, the hybrid loose NF-BMED process allows for resource (i.e., dye, salt and pure water) extraction from textile wastewater, which closes the salt and water cycle, in view of process intensification.

KEYWORDS: Hybrid loose NF-BMED process system, Dye/salt mixture, Resource recovery, Zero liquid discharge



INTRODUCTION

Having contributed to human civilization for several millennia, the textile industry is today considered one of the major industries of modern society. Because of the exponential population growth in the 21st century, textile products are in large demand. Currently, China is the largest textile producer and exporter, providing 40% of world textile and clothing exports with a total value of US \$65.4 billion in 2009, and potentially stimulating its economic growth scenario.¹

Textile wastewater is generated during the dye production and textile dyeing process and typically has a high salinity (>6.0% NaCl or >5.0% Na₂SO₄).^{2–5} A typical 200–350 m³ of pure water is required for producing one ton of dyed textile products, which in turn leads to the generation of a huge amount of textile wastewater.^{6,7} According to the China Environment

Statistical Yearbook in 2012, around 2.37 × 10¹⁰ tons of textile dyeing wastewater was discharged, posing a severe threat to the aqueous environment in China.⁸ This kind of wastewater contains high concentrations of dyes in the wastewater, with a detrimental toxicity for the environment and for humans through, e.g., azo functional groups. Furthermore, upon exposure to light, the chromophore structure of the dyes absorbs the light. This phenomenon hinders the photosynthetic activity of aquatic organisms.^{6,9,10} Therefore, these severely polluted streams should be appropriately treated before disposal.

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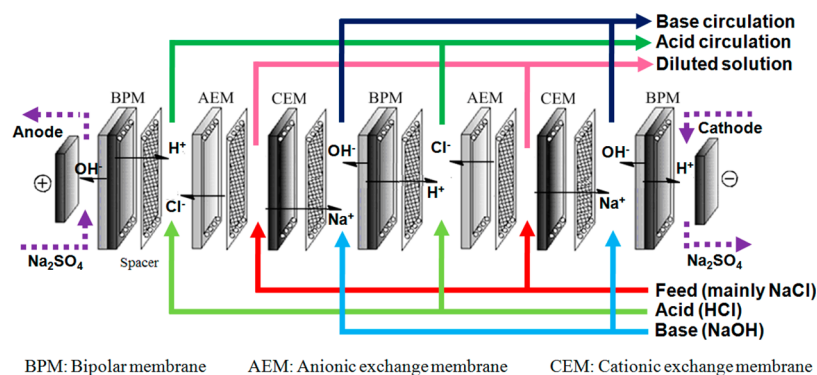


Figure 1. Configuration of BMED stack for base/acid production and water recovery.

In the current concept of sustainability for wastewater treatment, wastewater is to be considered a resource rather than a waste, in view of energy production, recycling of plant fertilizing nutrient and other resources as well as water reclamation.^{11–14} The reuse of dyes from textile wastewater not only remarkably reduces the expenditure of dyes, but also tremendously lowers the toxicity to the aqueous environment.^{9,15–18} However, conventional approaches (i.e., adsorption, coagulation, biological treatment and advanced oxidation processes) mainly focus on dye removal or destruction, reducing the possibility for dye recovery and salt reuse from textile wastewater.^{19–22} Therefore, state-of-the-art treatment technologies focusing on recovery of valuable resources from textile wastewater are on the research agenda.

Currently, bipolar membrane electrodialysis (BMED) offers an environmental friendly and sustainable platform in industrial application by combining the conventional electrodialysis process and water dissociation in a bipolar membrane. It allows for clean production of base/acid,²³ combinatorial synthesis of ionic liquids,²⁴ power generation,²⁵ as well as CO₂ extraction.^{26,27} Specifically, the conversion of salt into base and acid by BMED from wastewater containing a high salinity and organic matter gives a new avenue to raw material recycling and zero liquid discharge.^{28–30} Applying BMED, Shen et al. successfully fractionated glyphosate and NaCl from glyphosate neutralization liquor (1.28% glyphosate and 16.8% NaCl). This serves a double target: glyphosate recovery and a closed loop for the recycling of NaOH and HCl from NaCl conversion in glyphosate production.³¹ Wu et al. proved the feasibility of BMED for salt recovery from 1,3-propanediol fermentation broth.³² However, the fouling of ion exchange membrane limits the separation efficiency of BMED. Macromolecules carrying positive or negative electrical charges (i.e., polyelectrolytes, humic acids, surfactants and dyes) tend to be preferentially deposited on the surface or on the pore wall of ion exchange membranes, dramatically increasing the membrane resistance under a direct current.^{33–37} Additionally, BMED may not be sufficient as a stand-alone technology.²⁸ The integration of BMED with other techniques, such as a fluidized pellet reactor,³⁸ microbial fuel cell,³⁹ diffusion dialysis⁴⁰ and nanofiltration (NF),^{41,42} may be explored, in view of a synergic effect, to realize the extraction of valuable materials from (textile) wastewater.

NF technology can be an alternative pretreatment to BMED for textile wastewater, due to the high dye removal efficiency. However, available commercial dense NF membranes with a high salt retention would have a high osmotic pressure difference, which substantially decrease the permeate flux.⁴³

Consequently, some of the valuable dyes in the textile wastewater cannot be easily purified, due to the high amount of salt as impurities. Furthermore, the high salt retention for these dense NF membranes only yields a small amount of salt in the NF permeate, reducing the possibility of salt reuse. Therefore, NF membranes that allow a fractionation of dyes and salts from textile wastewater are of interest for simultaneous dye recovery and salt reuse. Previous work has demonstrated that loose NF membranes have a good fractionation performance for the direct dye/salt mixtures, showing over 99.6% rejection for direct dyes and ~97.5% salt permeation when 40.0 g·L⁻¹ NaCl is present.⁴ The high concentration of salt and trace amounts of dye in NF permeate potentially facilitates salt reuse by BMED.

In this study, resource recovery from a dye (direct and reactive dye)/NaCl system by a hybrid loose NF-BMED process is proposed, in view of sustainability. Fractionation of NaCl and diverse dyes (direct red 80 and reactive blue 2) in the simulated textile wastewater with high salinity by using loose NF in diafiltration was explored, based on the reasonable mathematic simulation. Subsequently, BMED was applied to investigate the desalination of the salt-containing permeate from the loose NF membrane and acid/base production from the salt by fractionation. An environmental assessment for the integrated NF-BMED process in the treatment of a dye/salt mixture system was carried out as well.

THEORETICAL BACKGROUND

Diafiltration by NF Membrane for Fractionation of Dye/Salt Mixture. In industrial application, diafiltration is a useful method to achieve high purification rates for macro/microsolute systems with an economically acceptable productivity.^{44,45} Diafiltration by applying an NF membrane extends the range of separation to the molecular or ionic level.^{46,47} This fractionation generally yields a high target product rejection and nearly complete byproduct permeation.

To quantify the concentration of solutes (salt and dye) in the permeate and feed solution, a derivation for simulation for diafiltration in a constant volume mode was carried out following the procedure proposed by Wang et al.⁴⁴ The concentration of salt and dye in the feed (C_f) and permeate (C_p) is expressed by

$$C_f = \exp[-\eta \cdot (1 - R_T)] \cdot C_{f,0} \quad (1)$$

$$C_p = \exp[-\eta \cdot (1 - R_T)] \cdot C_{f,0} \cdot (1 - R_T) \quad (2)$$

where $C_{f,0}$ is the initial concentration of solute in the feed solution, η is the number of diavolumes, defined as the ratio of the total collected filtrate volume (V_w) to the constant feed volume (V_0) during the diafiltration process, and R_T is the observed rejection for solutes in the diafiltration procedure.

Bipolar Membrane Electrodialysis (BMED) Process.

BMED combines a conventional electro dialysis and water dissociation by a bipolar membrane in which a water layer is split into proton (H^+) and hydroxide ions (OH^-). A schematic diagram of BMED is shown in Figure 1. When a direct current is established, the cations (i.e., K^+ and Na^+) and anions (i.e., SO_4^{2-} , NO_3^- and Cl^-) in the feed are transported through cation- and anion-exchange membranes, combining with OH^- and H^+ split by a bipolar membrane, to form base and acid, respectively.

Bases and acids generated by BMED can be potentially recycled as raw materials for dye synthesis. Furthermore, the reclamation of water from the salt-containing NF permeate by BMED is essential to realize zero emission of wastewater. The diluate from BMED can be reused for the diafiltration of dye/salt mixtures by NF. To minimize the amount of salt in this diluate, BMED in a constant-voltage mode (20 V) is adopted in this case.⁴⁸

The energy consumption (E) is calculated by extrapolating the results for the production of 1 kg of NaOH based on eq 3.⁴⁹

$$E = \int \frac{U \cdot I \cdot dt}{(C_t - C_0) \cdot V \cdot M} \quad (3)$$

where U is the voltage drop across the BMED stack; I is the applied current; M is the molecular weight of target solute (NaOH); C_t and C_0 are the concentrations of target solute at times t and 0, respectively; V is the circulated volume of solution.

The desalination efficiency (δ) of the feed solution was calculated according to the following equation:

$$\delta(\%) = \frac{C_{NaCl,0} - C_{NaCl,t}}{C_{NaCl,0}} \times 100 \quad (4)$$

where $C_{NaCl,t}$ and $C_{NaCl,0}$ are the concentrations of NaCl at times t and 0, respectively.

METHODS AND MATERIALS

Chemicals. Direct red 80 (DR 80) and reactive blue 2 (RB 2) supplied by Sigma-Aldrich (Belgium) were used as model dyes. NaCl with analytical grade was provided by Sigma-Aldrich (Belgium). All chemicals were used without any further purification. Figure 2 shows the molecular structure of tested dyes. Deionized water was used throughout all the experiments.

Loose NF Membrane Filtration. Membrane Samples and NF Setup. A thin film composite NF membrane, Sepro NF 6, which was kindly provided by Ultura (USA), was used in this study. The membrane properties are shown in Table 1.

For each experiment, a NF membrane coupon with a total effective area of 22.9 cm² was loaded in the permeation cell of a laboratory scale cross-flow nanofiltration unit described elsewhere.⁴ The pressurized cross-flow filtration tests were conducted with a feed of deionized water containing salts and dyes in recirculation mode at 6 bar and 25.0 ± 1 °C. All membrane samples loaded in the module were precompact with deionized water under 10 bar for 2 h to ensure a stable performance.

Subsequently, around 350 mL of DR 80/NaCl (dye, 1 and 2 g·L⁻¹; NaCl, 40 and 60 g·L⁻¹) and RB 2/NaCl (dye, 1 and 2 g·L⁻¹; NaCl, 40 and 50 g·L⁻¹) mixed solutions were applied as the model solutions for diafiltration. Pure water was continuously added to the feed to maintain

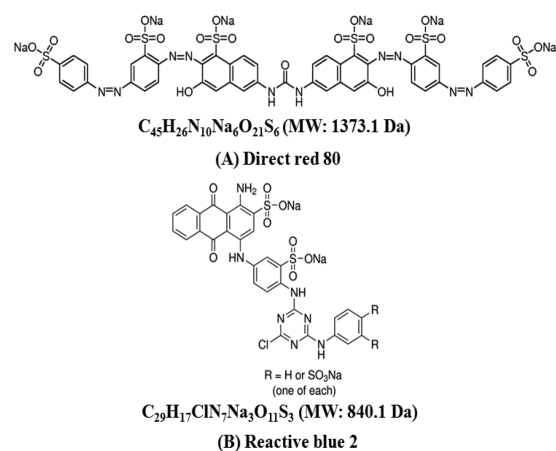


Figure 2. Molecular structures of the dyes used in the experiment.

Table 1. Properties of Loose NF Membranes Tested in This Study⁴

membrane	Sepro NF 6
composition top layer	piperazine-based polyamide
permeability (L·m ⁻² ·h ⁻¹ ·bar ⁻¹) ^a	13.7
max. temperature (°C)	50
process pH limitations	3.0–10.0
ζ-potential at pH 7.2 (mV)	−45.58
molecular weight cutoff (Da)	847
NaCl rejection (%) ^b	10

^aPure water flux provided by manufacturer. ^b2000 ppm of NaCl solution at 10.6 bar and 25 °C.

the system at the constant volume. Feed and permeate solutions were sampled at a fixed volume factor of added pure water to assess the variation of dye and NaCl concentration in the feed and permeate during diafiltration.

Membrane Flux and Solute Rejection. The flux (J_w) was calculated based on the time to collect a fixed volume of water at 6 bar, which is expressed as

$$J_w = \frac{V}{A \cdot t} \quad (5)$$

where V is the water volume collected during the time t , and A is the effective membrane area for water permeation.

The observed rejection (R_T) of salts and dyes can be calculated by the following equation:

$$R_T(\%) = \frac{C_f - C_p}{C_f} \times 100 \quad (6)$$

where C_f and C_p are the solute concentrations in the feed and permeate, respectively.

BMED for Base/Acid Recovery and Pure Water Production.

The lab-scale setup for BMED consisted of a cathode and an anode, made of titanium coated with ruthenium, and a membrane stack. Five pieces of anion exchange membranes (AMV, Asahi Glass, Japan), five pieces of cation exchange membranes (CMV, Asahi Glass, Japan) and six pieces of bipolar membranes (FT-BM, Fuma-Tech, Germany) with dimensions 27 cm × 11 cm (effective membrane area of 21 cm × 9 cm) were arranged between the anode and cathode chambers, which were isolated by spacer gaskets with 0.75 mm thickness. Distilled water was added in the acid and base compartment for producing acid and base. In the study, different salt solutions (i.e., 20, 40 and 75 g·L⁻¹ NaCl) with a trace amount of reactive blue 2 (~1.0 ppm) solutions were applied as the feed. A 3.0 wt % Na₂SO₄ solution was employed as the electrolyte rinsing solution for both electrode compartments. The voltage and current across the BMED stack were directly recorded by a

Table 2. Properties of the Membranes Used for BMED Stack

membrane type	thickness (μm)	IEC ($\text{meq}\cdot\text{g}^{-1}$)	area resistance ($\Omega\cdot\text{cm}^2$)	voltage drop (V)	selectivity (%)
AMV	130		2.5		96
CMV	130		3		96
FT-BM	200		<3	1.2–2.2	98

regulated CV/CC power supply (WYL 1703 \times 2, Hangzhou Siling Electrical Instrument Co. Ltd., China). Once the conductivity of feed with different salt concentrations was below $0.5 \text{ mS}\cdot\text{cm}^{-1}$, the BMED operation was interrupted as 1 cycle.

Furthermore, fouling property of the ion exchange membranes by dyes in the BMED stack was investigated in different cycles with NaCl solutions (20 and $40 \text{ g}\cdot\text{L}^{-1}$) to which 1.0 ppm reactive blue 2 was added. The BMED operation was performed in the 20 and $40 \text{ g}\cdot\text{L}^{-1}$ NaCl solution for 3 cycles.

The characteristics of the ion exchange membranes and bipolar membrane in this study are shown in Table 2.

Analytical Methods. The concentration of direct dye and reactive dye was determined by a Shimadzu UV-1601 double beam spectrophotometer (Japan). For single salt solutions, the concentration of NaCl was measured by a conductivity meter (Thermo Scientific Orion Star A212 Benchtop Conductivity Meter, Belgium). The concentration of NaCl in the dye/NaCl mixture solution in the diafiltration process was determined by ion chromatography (Dionex ICS-2000 System, USA). The corresponding concentrations of NaOH and HCl produced by the BMED process were determined by titration with a $0.01 \text{ mol}\cdot\text{L}^{-1}$ HCl and NaOH standard solutions, respectively.

RESULTS AND DISCUSSION

Diafiltration of Dye/Salt Solutions. To optimize the diafiltration process for dye/salt mixtures, the variation in the dye and salt concentration in the feed and permeate is shown in Figure 3.

As shown in Figure 3, the concentrations of DR 80 and RB 2 in the feed fluctuate slightly, with the addition of pure water for diafiltration. With multiple negatively charged sulfonate groups for both dyes, the high retention of dyes (>99.92%) was obtained by this loose NF membrane with negative surface during the diafiltration through the combination of Donnan repulsion and size exclusion.^{4,50} Sepro NF 6 shows a slightly lower rejection for RB 2, compared to DR 80 with a larger molecular size, which is consistent with the results in Figure S1. In the NF permeate, no color was observed, indicating the potential viability of reusing salt-containing permeate by BMED without causing any contamination to the ion exchange membranes.

Furthermore, the amount of salt in the feed solution decreased with the addition of pure water during diafiltration. The NF permeate had a slightly lower salt concentration than the feed solution, resulting from the low salt rejection for Sepro NF 6. The simulated overall salt rejections of Sepro NF 6 for diafiltration of dye/salt mixtures with 40, 50 and $60 \text{ g}\cdot\text{L}^{-1}$ NaCl are 2.2%, 1.8% and 1.1%, respectively, which are similar to the salt rejections in the pure NaCl solutions (see Figure S2). This low salt rejection is advantageous for the fractionation of salt/dye mixture, requiring low water consumption. Around 99.0% desalination efficiency for the dye/salt mixtures was obtained by Sepro NF 6 after the addition of pure water with the number of diavolume of 5.0.

Furthermore, Figure 3 indicates that the measured salt concentration in the feed and permeate fits well with the calculated data using eqs 1 and 2. As the diafiltration proceeds, the measured salt concentration has a slight deviation from the calculated data, when the salt in the feed decreases to a low

concentration. This is caused by the increase in rejection of the Sepro NF membrane at low NaCl concentrations, because the Donnan effect is strengthened with a decreasing NaCl concentration. This is consistent with the results in Figure S2.

BMED for Base/Acid and Pure Water Production. To close the material loop and achieve zero liquid discharge, BMED was applied for the reuse of salt from the fractionation of dye/salt mixtures. The variation of the operational parameters in the BMED stack is shown in Figure 4.

Figure 4A shows the variation of conductivity in the feed solutions. It was observed that the conductivity of the feed decreased under the current field, due to the continuous transportation of Na^+ and Cl^- ions through the ion exchange membranes. In this case, the conductivity of the feed was below $0.5 \text{ mS}\cdot\text{cm}^{-1}$, indicating an adequate desalination of BMED stack at constant voltage mode.

Figure 4B shows the evolution of current across the BMED stack for different salt solutions. As indicated in Figure 4B, the current across the membrane stack shows a reverse “U” shape when it is run at the mode of constant voltage. Initially, the membrane stack had a high electrical resistance, because the deionized water was used as the medium in the base and acid compartment, resulting in a low current across the membrane stack. Under the established electrical field, Na^+ and Cl^- ions transported from the bulk feed solution to the base and acid compartment through the ion exchange membranes, and hence the electrical resistance for the stack started to decrease, resulting in an abrupt current increase across the membrane stack. Similar with BMED stack run at constant current density mode, a quasi-steady stage for BMED operation at constant voltage mode was obtained.⁵⁰ During the steady operation, the increase in acid/base concentration in the BMED stack was observed to compensate the increase in the electrical resistance of the feed compartment, keeping the electrical resistance of the BMED stack constant. In the ultimate stage, the full exhaustion of salt in the feed resulted in a high resistance of the membrane stack (see Figure 4A) with a sharp decrease of the current.

Figure 4C shows the base yield of the BMED stack for different salt solutions. It can be seen that the concentration of NaOH increases linearly during the stable operation stage. The Na^+ and Cl^- ions in the feed were continuously transported to the base and acid compartment under an electrical field. In the final stage, the mass transfer of Na^+ and Cl^- ions diminished, due to the exhaustion of NaCl in the feed, as shown in Figure 4A. The final concentrations of NaOH in 20, 40 and $75 \text{ g}\cdot\text{L}^{-1}$ NaCl solution were 0.40, 0.77 and $1.28 \text{ mol}\cdot\text{L}^{-1}$, respectively. During the steady operation stage, the NaOH yield for different salt solutions had the same linear evolution, suggesting that most of the energy is effectively applied for the transportation of salt in this phase. Because of the stronger proton leakage, those corresponding salt solutions yielded acid solutions with lower concentrations of 0.36, 0.72 and $1.19 \text{ mol}\cdot\text{L}^{-1}$, respectively, compared to NaOH production (see Figure 4D).³⁸

The Cl^- content in the feed, Cl^- removal efficiency, and the purity of NaOH produced by BMED are shown in Table 3.

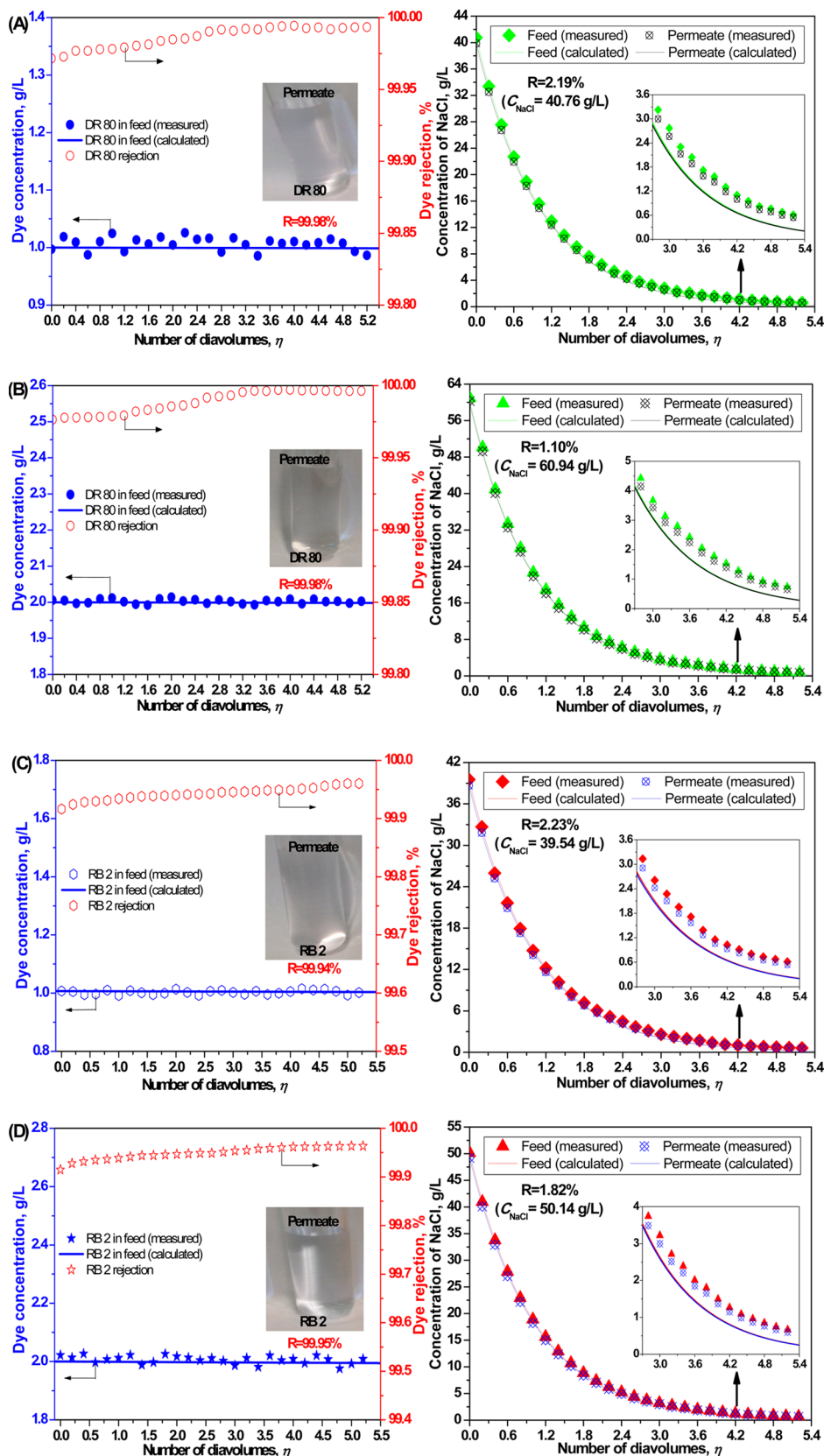


Figure 3. Diafiltration for different DR 80/NaCl and RB 2/NaCl solutions by the loose NF membrane. (A) $1 \text{ g}\cdot\text{L}^{-1}$ DR 80 and $40 \text{ g}\cdot\text{L}^{-1}$ NaCl; (B) $2 \text{ g}\cdot\text{L}^{-1}$ DR 80 and $60 \text{ g}\cdot\text{L}^{-1}$ NaCl; (C) $1 \text{ g}\cdot\text{L}^{-1}$ RB 2 and $40 \text{ g}\cdot\text{L}^{-1}$ NaCl; (D) $2 \text{ g}\cdot\text{L}^{-1}$ RB 2 and $50 \text{ g}\cdot\text{L}^{-1}$ NaCl.

It can be seen that the BMED stack run in the mode of constant voltage obtained over 99.3% desalination efficiency, only allowing ~ 100 ppm of Cl^- present in the feed. In the base

compartment, the Cl^- concentration increased due to the stronger salt diffusion with the increase of NaCl concentration in the feed.⁵¹ NaOH produced by BMED had a purity of

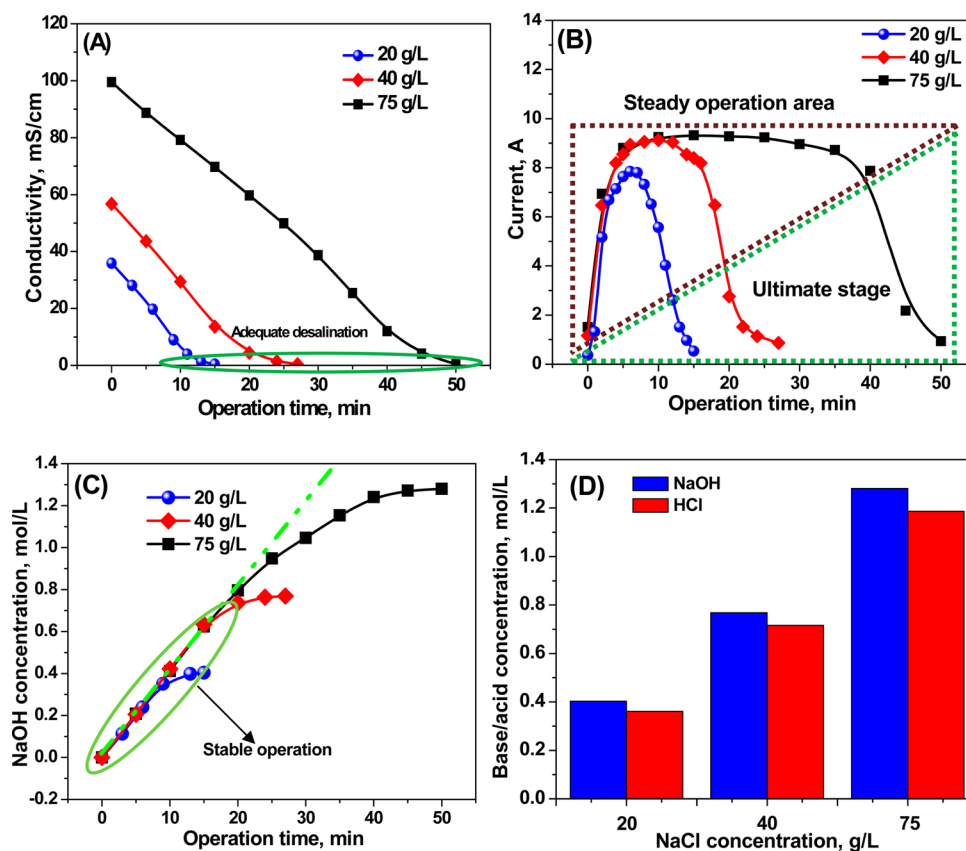


Figure 4. (A) Conductivity variation of feed; (B) evolution of current across BMED stack; (C) base yield of BMED stack; (D) final base/acid concentration at BMED stack.

Table 3. Overview of BMED Performance for Base Production

NaCl concentration ($\text{g}\cdot\text{L}^{-1}$)	Cl^- content in the feed (ppm)/ Cl^- removal (%)	Cl^- content in NaOH (ppm)	purity of NaOH (%)
20	101.2/99.3	192.9	98.0
40	96.7/99.6	467.5	97.5
75	112.7/99.8	837.6	97.3

$\sim 97.5\%$, which is probably not suitable for commercial purposes. However, it is acceptable for the reuse as raw material for dye synthesis.

Figure 5 shows the energy consumption in the BMED stack for base production in NaCl solution with different concentrations. The energy consumption increased from 2.86 to 4.06 $\text{kWh}\cdot\text{kg}^{-1}$ as the salt concentration in the feed rose from 20 to

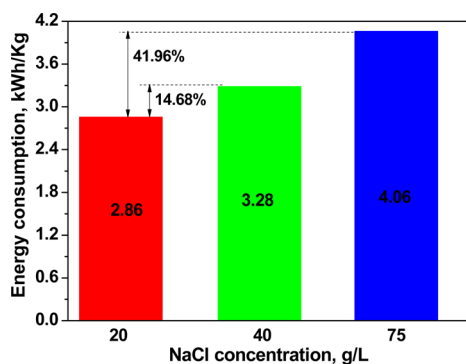


Figure 5. Energy consumption in BMED stack for different salt solutions.

75 $\text{g}\cdot\text{L}^{-1}$. The increment in the energy consumption was 14.68% and 41.96% in the 40 and 75 $\text{g}\cdot\text{L}^{-1}$ NaCl solution, compared to the 20 $\text{g}\cdot\text{L}^{-1}$ NaCl solution. The increasing energy consumption of the BMED stack in the different solutions originates from the intensification of coion leakage and diffusion through the ion exchange membrane.^{28,52,53}

Fouling of BMED Stack. Membrane fouling is an important issue for the BMED performance, which can shorten the life span of membrane and increase the operation cost. In this case, BMED operation in different cycles is carried out, which is shown in Figure 6.

It is observed in Figure 6 that BMED has a similar performance (i.e., evolution in the stack, feed conductivity and base yield, as well as Cl^- content in the feed) for salt solutions in different cycles. The stable operation of BMED for the desalination of salt-containing NF permeates indicates that the trace amount of dye (~ 1.0 ppm) in the NF permeate caused no contamination to the ion exchange membranes. During the operation for NaCl solutions in different cycles, the BMED stack maintained its high desalination efficiency ($\sim 99\%$) and strong capacity for base/acid production. Hence, Sepro NF 6 is an excellent pretreatment for textile wastewater to avoid the membrane fouling in a post-treatment strategy, i.e., BMED process.

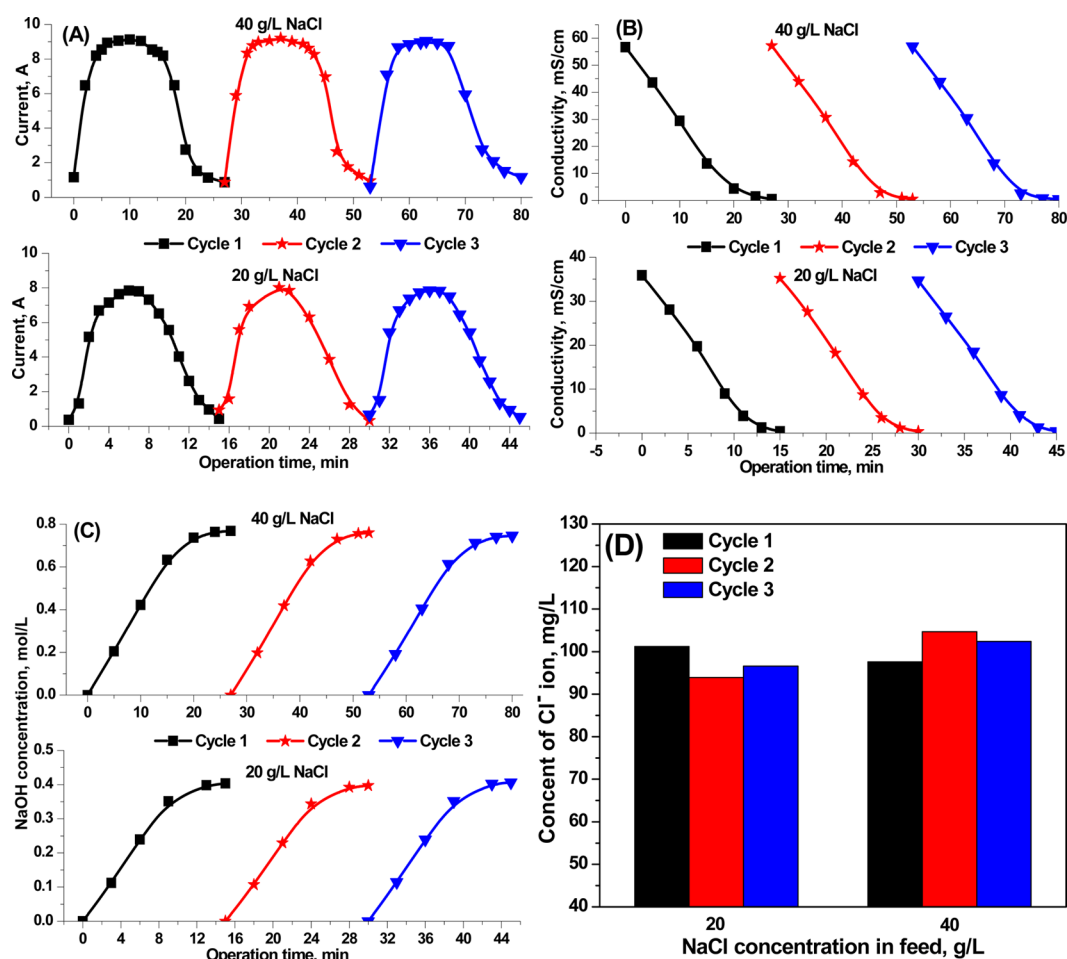


Figure 6. BMED operation in different cycles. (A) Evolution of current across BMED stack; (B) conductivity variation of feed; (C) base yield of BMED stack; (D) Cl^- content in the feed.

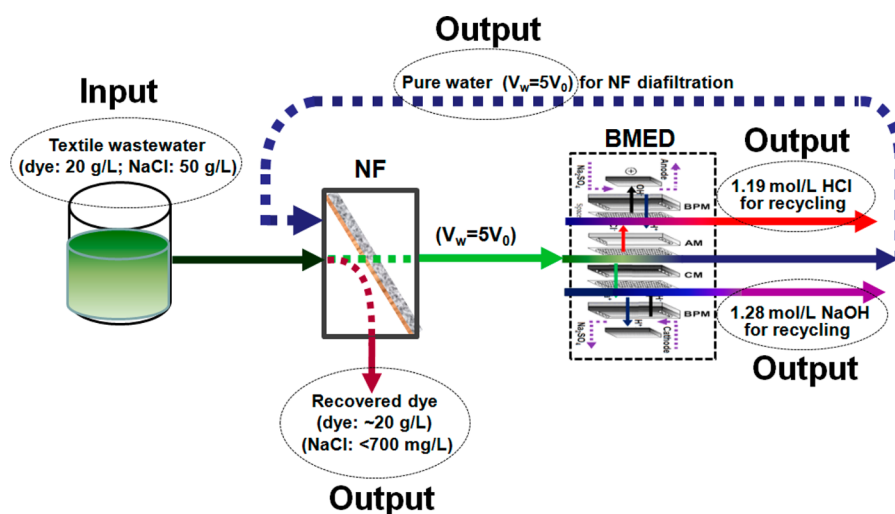


Figure 7. Scheme of resource recovery from textile wastewater by loose NF-BMED hybrid process.

Industrial Flowsheet Using Hybrid Membrane Technology for Resource Recovery from Dye/Salt Mixtures. The results demonstrate that the use of a loose NF membrane can be technically feasible for the fractionation of dye/salt mixtures. On the other hand, it can prevent membrane fouling by dyes during the post-treatment (e.g., BMED) of the salt-containing NF permeate, due to the complete rejection of direct

dyes and reactive dyes by the loose NF membrane (Sepro NF 6). The integration of the loose NF-BMED process may lead to resource recovery and zero liquid discharge for dye/salt mixture streams (especially in the dye synthesis process). The overall process is shown in Figure 7.

In this case, the textile wastewater from the dye synthesis procedure is taken into consideration, because it contains a high

amount of salts and dyes. The dye is to be purified to commercial standards.^{54,55} It is assumed that the textile wastewater from dye synthesis process contains 20 g·L⁻¹ dye and 50 g·L⁻¹ NaCl, and is applied as the feed solution. The application of loose NF membrane for diafiltration can yield dyes with >96.6% purity, after the addition of pure water with a volume factor of 5.0, which is prior to the commercial dyes (DR 80 with 25% purity and RB 2 with 60% purity from Sigma-Aldrich). Furthermore, NaOH with a concentration of 1.28 mol·L⁻¹ and HCl with a concentration of 1.18 mol·L⁻¹ from salt conversion by BMED is recycled for dye synthesis. The diluate from BMED, with a low salt content (~100 ppm), can be reused for diafiltration of dye/salt mixtures by loose NF membrane to close the water loop.

CONCLUSIONS

This study investigates the applicability of a hybrid loose NF-BMED process system for sustainable dye extraction, water and salt reuse from textile wastewater. The loose NF membrane (Sepro NF 6, Ultura) has an excellent diafiltration performance for the fractionation of (direct and reactive) dye/salt mixtures, allowing a free passage of salt ($R \leq 2.2\%$) and high retention of dyes (>99.93%). The addition of pure water with a volume factor of 5.0 can yield ~99.0% salt desalination efficiency for dye/salt mixtures. The supplement of BMED can realize the production of pure water, acid and base from the salt-containing NF permeate without membrane fouling. Especially, at the sacrifice of energy consumption with 4.06 kWh·kg⁻¹, bases and acids with a high concentration (1.28 and 1.19 mol·L⁻¹, respectively) can be yielded from a 75 g·L⁻¹ NaCl solution, as well as the diluate with ~100 ppm of NaCl. Therefore, this hybrid process can technically recover the dyes with >96.6% purity and close the salt and water loop from textile wastewater, showing a high potential in industrial application.

ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acssuschemeng.5b00234.

Filtration performance of loose NF membrane in the single-component solutions (PDF).

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Author Contributions

The paper was written through contributions of all authors. All authors have given approval to the final version of the paper.

Notes

The authors declare no competing financial interest.

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