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Study on Recycling Alkali from the Wastewater of Textile Mercerization Process by Nanofiltration

Yun Zhang\textsuperscript{a}\textdagger, Shuai Shao\textsuperscript{a,b}, Weicheng Yu\textsuperscript{a}, Fenglin Yang\textsuperscript{a}, Xiaochen Xu\textsuperscript{a}

\textsuperscript{a}Key Laboratory of Industrial Ecology and Environmental Engineering (MOE), School of Environmental Science and Technology, Dalian University of Technology, Linggong Road 2, Dalian 116024, P. R. China
\textsuperscript{b}School of Innovation Experiment, Dalian University of Technology, Linggong Road 2, Dalian 116024, P. R. China

Abstract

This study has researched the treatment ability of nanofiltration technology in printing and dyeing mercerization process alkali wastewater and investigated the effect of different pressures and alkalinities on membrane flux. The effect of different operating conditions on COD removal rate and alkali penetration rate has been also investigated in this study. The experimental results show that nanofiltration technology can remove the COD in printing and dyeing mercerization process wastewater effectively and the removal rate is above 80\%. We conclude that the membrane flux tends to increase as the operating pressure increases, and decrease as the concentration of alkali in feed liquor and COD increases. The infiltration liquor through membrane can be satisfactorily reused by the method of concentration, and the final recovery efficiency of alkali by nanofiltration membrane treatment is above 90\%.

Keywords: nanofiltration technology; nanofiltration membrane; printing and dyeing mercerization process; Wastewater;

1. Introduction

The wet process technology with freshwater as a medium, has been widely employed in textile printing and

\textdagger\ Corresponding author. Tel.: +86-411-8470-6152; fax: +86-411-84706152.
E-mail address: zhangyun@dlut.edu.cn.
dyeing industry in China. The process is divided into 3 sections: pretreatment, dye and after treatment, including multiple processes such as desizing, rinsing, mercerizing, dyeing, sizing and etc. Almost every process needs huge quantity of freshwater and steam in printing and dyeing industry and discharges desizing, mercerizing and dyeing wastewater, which is the representative of high pollution, alkalinity and chroma, resulting in high load and difficulty in the end treatment.

In order to improve fiber tension, increase fiber surface gloss, decrease fiber contractility and enhance the affinity of dye, the sodium hydroxide solution is used to treat the textile in mercerization process. The wastewater of textile mercerization is a high alkali waste liquor and always is mixed with other wastewater in production processes to be treated in the end treatment system. Because of its high alkalinity, large amount of acid is consumed to neutralize the alkali when the wastewater is treated; otherwise, it will cause the difficulty in the subsequent bio-treatment process and have a negative effect on the equipment. Therefore, it is necessary to purify and recover the alkali from the waste alkali liquor of the mercerization, reducing the quantity of alkali into end processing system.

Nanofiltration (NF) is a kind of membrane technology which is rapidly growing and widely recognized as superior water and wastewater treatment in recent years, and has the separation characteristics of sieve effect and Donnan charge effect [1]. Generally, nanofiltration can retain the divalent and higher-valence ions; whereas the monovalent ions such as sodion and chloridion can pass through an NF membrane. Thus, NF technology is widely used in de-salting of the process stream [2, 3]. In this study, nanofiltration membrane separation technology is used to purify and recover alkali from the waste alkali liquor of the mercerization; the influence factors in operation process of membrane separation are investigated to offer the technical support to wastewater treatment using nanofiltration technology.

2. Materials and Methods

2.1 Experimental materials

The compositions of mercerizing wastewater of printing and dyeing are uncertain, therefore, wastewater with different concentrations should be studied when NF membrane technology is used to treat printing and dyeing wastewater. In this research, the wastewater was collected from the mixture of multi-effect mercerizing rinsing waste alkali liquor and normal mercerizing waste alkali liquor of BY Printing and Dyeing Co., Ltd., Haicheng city, Liaoning province, China, which contained 10.1%wt of alkalinity, 185NTU of turbidity, 1320mg/L of COD, and 342mg/L of SS. Three experimental samples with alkalinities of 20.16g/L, 30.24g/L and 71.2g/L respectively were obtained by diluting the original water sample. The index of COD and turbidity are shown in table 1.

<table>
<thead>
<tr>
<th>No. of liquor sample</th>
<th>Alkalinity (g/L)</th>
<th>COD (mg/L)</th>
<th>Turbidity NTU</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>20.16</td>
<td>330</td>
<td>4.6</td>
</tr>
<tr>
<td>2</td>
<td>30.24</td>
<td>376</td>
<td>6.8</td>
</tr>
<tr>
<td>3</td>
<td>71.2</td>
<td>1075.7</td>
<td>13</td>
</tr>
</tbody>
</table>

Table 1 The water quality of various influent

Special alkali-resisting nanofiltration membrane, SelRO®MPS-34-pH Stable Membrane 4040 A2Z from Koch Industries (USA.) was used in this study.
2.2 Technological process

Original liquor enters into the coagulation reaction tank, where it is heated to the specified temperature, coagulum is added, stir is applied, keep it still, and then deliver the liquor to raw water tank by a pump. The liquor in raw water tank enters into a bag filter and safety filter, and then into nanofiltration membrane. The flowrate and press are controlled by adjusting the opening on the high concentration side of the NF membrane, and the flowrate of high concentration water is controlled by adjusting the opening of the sampling valve on the high concentration side, thus controlling the recovery efficiency. In the experiment, quantity of reducing agent (sodium hydrogen sulfite) was directly added into the tank according to calculation.

2.3 Experiment method

The sample liquor from coagulation was pumped into another tank; clean water was added; then the alkalinites of the sample water after dilution were measured. In this study, the diluted samples with alkalinites of 20.16g/L, 30.24 g/L and 71.2 g/L, respectively were used to examine membrane flux and quality of infiltration fluid under different temperatures and pressures. Under the optimal operation conditions, condensing experiments of waste alkali liquor with typical concentration were carried out, and the recovery efficiency and quality of recovered water were measured.

The relevant parameter calculation methods from nanofiltration membrane are shown in table 2.

Table 2. The relevant parameter calculation methods from nanofiltration membrane

<table>
<thead>
<tr>
<th>Relevant parameter</th>
<th>Units</th>
<th>calculation methods</th>
</tr>
</thead>
<tbody>
<tr>
<td>Transmembrane Differential Pressure TMP</td>
<td>Pa</td>
<td>( \text{TMP}=P_f-P_c )</td>
</tr>
<tr>
<td>Membrane penetrate flux ( J )</td>
<td>m(^3)/m(^2)·h</td>
<td>( J=V_p/A_t )</td>
</tr>
<tr>
<td>Membrane recovery efficiency ( \eta )</td>
<td>%</td>
<td>( \eta=V_p/V_f \times 100% )</td>
</tr>
<tr>
<td>Alkalinity recovery efficiency ( \zeta )</td>
<td>%</td>
<td>( \zeta=C_pV_p/C_fV_f \times 100% )</td>
</tr>
<tr>
<td>Feed liquor condensing multiple ( \omega )</td>
<td>times</td>
<td>( \omega=V_t/V_0 )</td>
</tr>
</tbody>
</table>

\( P_f \): feed liquor pressure; \( P_c \): concentrate liquor pressure; \( V_p \): penetrate liquor volume(m\(^3\)); \( A_t \): membrane area(m\(^2\)); \( T \): running time(h); \( V_f \): feed flow liquor volume(m\(^3\)); \( C_p \): alkalinity of recovery liquor (g/L); \( C_f \): alkalinity of feed flow liquor (g/L); \( V_f \): recovery liquor volume(L); \( V_f \): feed flow liquor volume (L); \( V_t \): final condensing liquor volume(m\(^3\)); \( V_0 \): original liquor volume(m\(^3\)).

Based on the feed liquor potassium hypermanganate oxidation experiment, the quantity of reducing agent (sodium hydrogen sulfite) was 126 mg/L.

3. Result and Discussion

3.1 Treatment of dye liquor with the membrane

3.1.1 The effect of pressure liquor and alkalinity on the membrane flux

Membrane flux was analyzed based on the feed pressure from 2.0MPa, 2.5MPa and 3.0MPa under the 35°C. The results are shown in figure 1.
Fig. 1. The changes of flux with feed pressure among influents with different alkalinitities

The membrane flux are 58 L/h·m², 46 L/h·m² and 40 L/h·m², respectively under the different conditions shown in the figure. We conclude that the membrane flux tend to decrease as the alkalinitities in feed liquor increase. The high concentration of COD in original liquor also increases the pollution of membrane.

3.1.2 The effect of feed liquor quality on concentration of COD and removal efficiency of infiltration fluid

The concentration of COD and removal efficiency of infiltration fluid were analyzed based on the feed pressure of 2.0MPa, 2.5MPa and 3.0MPa under 35°C. The results are shown in figure 2.

Fig. 2. Comparison of COD removal rate among different samples

The removal efficiency of COD is 81%-88% by nanofiltration membrane treatment. As the concentration of COD increases, the removal efficiency increases first, then the changes tend to decrease. The removal efficiency of COD reaches around 87% and 81% by nanofiltration membrane when concentration of COD is between 300-400mg/L and 1000mg/L in original liquor, respectively.
3.1.3 The effect of feed liquor alkalinity on the alkalinity of nanofiltration membrane infiltration fluid and alkali permeability.

The alkalinity of infiltration fluid and alkali penetration efficiency was analyzed based on the feed pressure from 2.0MPa, 2.5MPa and 3.0MPa under the 35°C. The results are shown in figure 3.

![Fig. 3. Comparison of alkali transmittance among different samples](image)

The figure indicates that alkali penetration efficiency reaches around 93%, 93% and 98% when alkalinitities of feed liquor are 20.16g/L, 30.24g/L and 71.6g/L respectively, and reaches maximum of 98.31% when the feed pressure was 2.5Mpa at 35°C. As the alkalinity of feed liquor increases, the alkali penetration efficiency tends to increase. This trend also is caused by the increase of the ratio of the alkalinity to the volume of the penetrating fluid per unit time. On the other hand, the increase of alkalinity and the solute concentration of membrane boundary layer arouses the increase of diffusion rate, resulting in the increase of alkali penetration efficiency.

3.2 Membranes condensing multiple experiments

Condensing multiple experiments are used to demonstrate alkali removal efficiency of membrane in practical application. Under the specified temperature and 3.0MPa of feeding pressure, wastewater flows into original liquor tank, infiltration fluid flows into produced liquor tank, and condensing multiple is 20 times; record the starting time and running flux data, measure membrane flux when the ratios of volume of infiltration fluid to original are 50%, 70% and 95%, respectively. The quality of infiltration fluid, infiltration fluid in final produced water tank and final effluent are also measured in the experiment. The liquor sample is NO.1 in table 1, at 35°C. The water quality of the final effluent in the condensing experiment are shown in Table 3. The change curve of flux and treating time with condensing factors are shown in figure 4.

| Table 3 The water quality of the final effluent in the condensing experiment |
|-------------------------------|-----------------|-----------------|-----------------|
| liquor sample                 | alkalinity (g/L)| COD (mg/L)      | Volume (L)      |
| Original liquor               | 20.16           | 330             | 270             |
| Final penetration liquor      | 19.52           | 73.96           | 256.5           |
| Final condensing liquor       | 24              | 1143            | 13.5            |
Table 3 indicates that the COD concentration of final infiltration fluid and remaining infiltration fluid are 73.96mg/L and 1143mg/L, respectively, meaning that the membrane has a pretty high interception efficiency of COD (77.59%). The alkali recycling efficiency reaches 91.98% based on the calculation method in table 2. The recycling liquor quality completely meets the requirements for the reuse in printing and dyeing or other processes. The above results are of great significance in practical engineering. In practical application, the data in condensing experiment can be used as a reference for the discharge volume and rate of condensing fluid. When the concentration of alkali and COD in original fluid increase, the discharge volume and rate can be increased accordingly, so that the stable running of membrane equipment can be ensured.

4. Conclusion

1. When the alkali concentrations in feed liquor are 20.16g/L, 30.24g/L and 71.2g/L, the alkali permeability can reach 93-98%; the flux of membrane reaches 58 L/h·m², 46 L/h·m² and 40 L/h·m² in optimum condition, respectively; the removal rate of COD in feed liquor reaches around 81%-88%.

2. The recovery efficiency of alkali reaches 91% and COD removal efficiency was 77% respectively when the alkalinity of feed liquor is 20.16g/L in condensing experiments.

Acknowledgment

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References